Contents

1. General

Commercially available reagents were used without additional purification. E. Merck Kieselgel 60 was used for column chromatography.

Thin-layer chromatography (TLC) was performed on silica gel 60 F254 aluminum sheets (MERCK). Visualization was performed using UV light (254 or 312 nm) or staining with $KMnO_4$.

NMR spectra were recorded on a Bruker Avance III 800 (with a 5-mm CPTXI cryoprobe) and Bruker Fourier 300. Chemical shifts were reported relative to residue peaks of DMSO-d_6 (2.51 ppm: for ^1H and 39.5 ppm: for ^{13}C).

Melting points were measured on a SMP 30 apparatus without correction.

High-resolution mass spectra (HRMS) spectra were recorded on AB Sciex TripleTOF® 5600+ System using electrospray ionization (ESI). The measurements were done in a positive ion mode (interface capillary voltage – 5500 V); mass range from m/z 50 to m/z 3000; external or internal calibration was done with ESI Tuning Mix, Agilent. A syringe injection was used for solutions in acetonitrile, methanol, or water (flow rate 20 μl/min). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

For substances **9-11** HRMS spectra were recorded on mass spectrometer Orbitrap LTQ XL with an ESI ion source; needle voltage 3.5 kV/5 kV for negative/positive ions; capillary temperature 350 °C; gas flows — Sheath: 50, Aux: 10; mass range: 100-2000. Samples were introduced via an Agilent 1100 chromatograph without a column. The eluent was water/acetonitrile (1/1 v/v) with 0.1% formic acid additive, flow rate 0.35 mL/min, injection volume 1-10 μL.

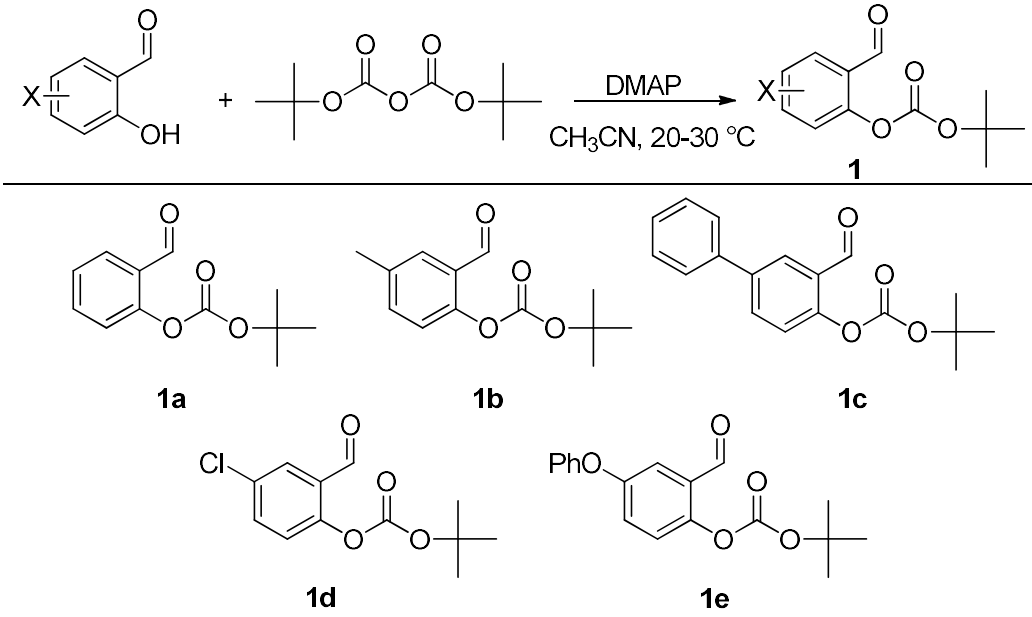
IUPAC compound names were generated using ChemDraw Pro 12.0 Software.

Photoinduced processes were performed on Evoluchem™ PhotoRedOx box. 365 nm (LG, HCK1012-01-006, 25 mW/cm^2) light-emitting diode (LED) lamp from Evoluchem™ was used. This device is equipped with a fan to maintain room temperature during the irradiation process.

A 10 ml teflon lined hydrothermal synthesis autoclave was used for the reaction at 150 °C.

2. Synthesis of starting compounds

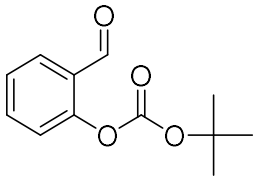
2.1 Synthesis of tert-butyl (2-formylphenyl) carbonates (1)



General method

A mixture of the corresponding 2-hydroxybenzaldehyde (10 mmol), Boc_2O (2.62 g, 12 mmol) and DMAP (0.49 g, 4 mmol) in freshly distilled CH_3CN (50 mL) was stirred at room temperature for 15 h. EtOAc (200 mL) was added and the resulted mixture was washed with brine (3×50 mL). Organic layer was dried over anhydrous Na_2SO_4, all volatiles were removed in vacuo and the residue was purified with flash chromatography (eluent – mixture of hexane and EtOAc, v/v 50:1).

Tert-butyl (2-formylphenyl) carbonate (1a)

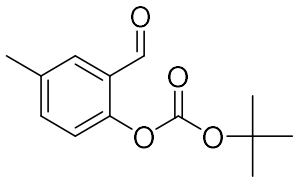


Yield 2.04 g (92%).

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 10.03 (1H, s), 7.95 (1H, dd, *J*=7.6, 1.7 Hz), 7.70 - 7.79 (1H, m), 7.53 (1H, td, *J*=7.5, 1.0 Hz), 7.36 (1H, dd, *J*=8.1, 1.0 Hz), 1.50 (9H, s).

The spectral properties corresponded to the literature data.[1]

Tert-butyl (2-formyl-4-methylphenyl) carbonate (1b)

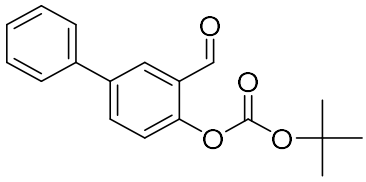


Yield 2.03 g (86%).

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 9.98 (1H, s), 7.73 (1H, d, *J*=1.9 Hz), 7.55 (1H, ddd, *J*=8.2, 2.3, 0.6 Hz), 7.23 (1H, d, *J*=8.2 Hz), 2.39 (3H, s), 1.49 (9H, s).

The spectral properties corresponded to the literature data.[2]

Tert-butyl (3-formyl-[1,1'-biphenyl]-4-yl) carbonate (1c)



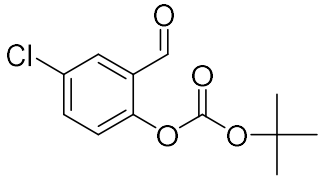
Yield 2.89 g (97%), white solid, 70-72 ℃.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.10 (1H, s), 8.24 (1H, d, *J*=2.4 Hz), 8.04 (1H, dd, *J*=8.4, 2.4 Hz), 7.74 - 7.77 (2H, m), 7.52 (2H, t, *J*=7.7 Hz), 7.43 - 7.46 (2H, m), 1.52 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm 190.1, 150.6, 149.5, 138.6, 138.1, 133.4, 130.9, 129.1, 128.3, 128.1, 126.8, 124.0, 83.8, 27.2.

HRMS (ESI) m/z: 299.1280 found (calcd for $C_{18}H_{19}O_4^+$, $[M+H]^+$ 299.1278).

Tert-butyl (4-chloro-2-formylphenyl) carbonate (1d)



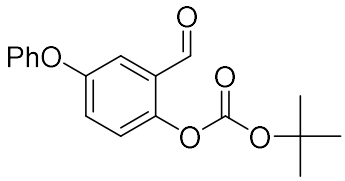
Yield 2.05 g (80%), white oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 9.98 (1H, s), 7.98 (1H, d, *J*=2.7 Hz), 7.81 (1H, dd, *J*=8.7, 2.7 Hz), 7.43 (1H, d, *J*=8.6 Hz), 1.49 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 188.9, 150.3, 149.1, 135.1, 131.5, 130.9, 129.3, 125.5, 84.1, 27.2.

HRMS (ESI) m/z: 257.0577 found (calcd for $C_{12}H_{14}ClO_4^+$, $[M+H]^+$ 257.0575).

Tert-butyl (2-formyl-4-phenoxyphenyl) carbonate (1e)



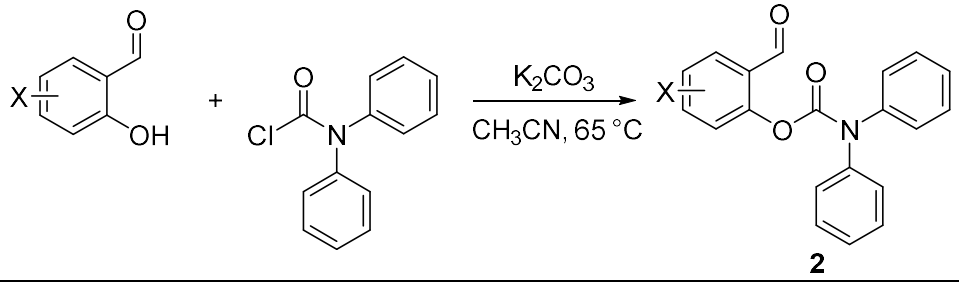
Yield 2.67 g (85%), white solid, m.p. 78-80 °C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.97 (1H, s), 7.47 (1H, s), 7.45 (2H, t, *J*=7.7 Hz), 7.39 (2H, d, *J*=1.3 Hz), 7.22 (1H, td, *J*=7.4, 0.6 Hz), 7.07 - 7.12 (2H, m), 1.50 (9H, s).

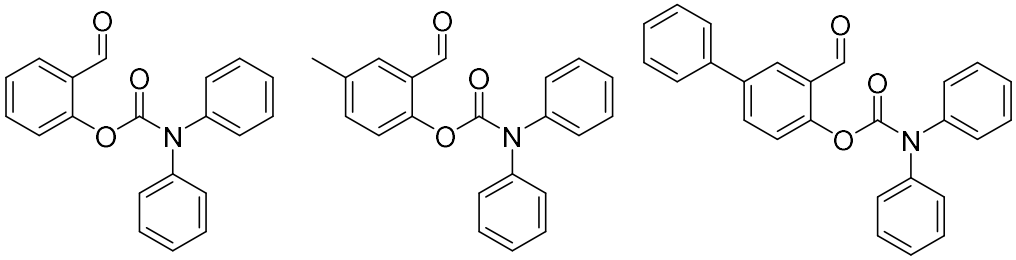
^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.2, 155.9, 154.9, 150.9, 146.0, 130.3, 129.0, 125.3, 125.2, 124.3, 120.0, 119.1, 83.8, 27.2.

HRMS (ESI) m/z: 315.1230 found (calcd for $C_{18}H_{19}O_5^+$, $[M+H]^+$ 315.1227).

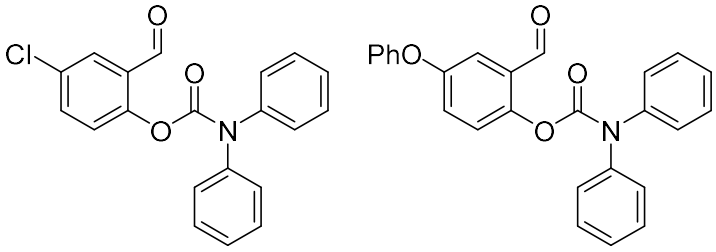
2.2. Synthesis of 2-formylphenyl diphenylcarbamates (2)



2



2a **2b** **2c**

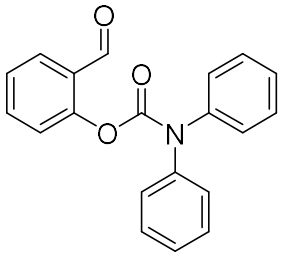


2d **2e**

General method

A mixture of the corresponding 2-hydroxybenzaldehyde (10 mmol), diphenylcarbamoyl chloride (13 mmol) and K_2CO_3 (2.76 g, 20 mmol), in freshly distilled CH_3CN (50 mL) was heated at 65℃ for 10 h. EtOAc (200 mL) was added and the resulted mixture was washed with brine (3×50 mL). Organic layer was dried over anhydrous Na_2SO_4, all volatiles were removed in vacuo and the residue was purified with flash chromatography (eluent – mixture of hexane and EtOAc, v/v 50:1).

2-Formylphenyl diphenylcarbamate (2a)



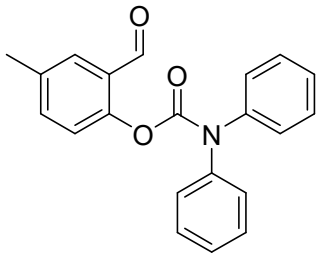
Yield 3.05 g (96%), yellow solid, m.p. 98-100°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.06 (1H, s), 7.89 (1H, dd, *J*=7.6, 1.6 Hz), 7.72 - 7.74 (1H, m), 7.49 - 7.60 (3H, m), 7.45 - 7.48 (2H, m), 7.39 - 7.45 (5H, m), 7.30 (2H, br. s.).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.9, 151.9, 151.2, 142.1, 135.5, 131.0, 129.2, 128.2, 127.3 (br. s.), 126.9 (br. s.), 126.4, 123.6.

HRMS (ESI) m/z: 318.1130 found (calcd for $C_{20}H_{16}NO_3^+$, $[M+H]^+$ 318.1125).

2-Formyl-4-methylphenyl diphenylcarbamate (2b)



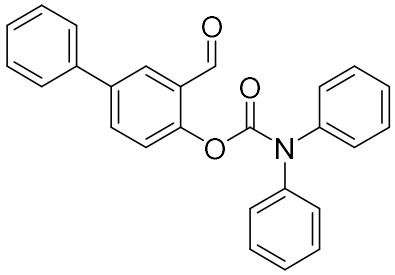
Yield 3.25 g (98%), yellow solid, m.p. 100-102°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.02 (1H, s), 7.68 (1H, d, *J*=1.7 Hz), 7.47 - 7.58 (4H, m), 7.43 (5H, t, *J*=6.9 Hz), 7.26 - 7.32 (3H, m), 2.37 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.9, 152.0, 149.2, 142.1, 136.0, 135.9, 131.0, 129.2, 127.9, 127.3 (br. s.), 126.9 (br. s.), 123.4, 20.1.

HRMS (ESI) m/z: 332.1283 found (calcd for $C_{21}H_{18}NO_3^+$, $[M+H]^+$ 332.1281).

3-Formyl-[1,1'-biphenyl]-4-yl diphenylcarbamate (2c)



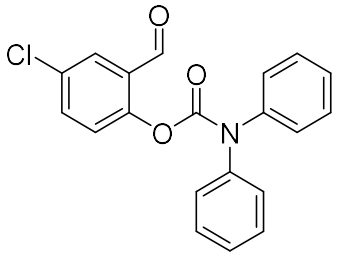
Yield 3.54 g (90%), yellow solid, m.p. 106-108°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.11 (1H, s), 8.17 (1H, d, *J*=2.5 Hz), 8.03 (1H, dd, *J*=8.4, 2.5 Hz), 7.72 - 7.75 (2H, m), 7.47 - 7.61 (7H, m), 7.36 - 7.47 (6H, m), 7.31 (2H, br. s.).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 190.1, 151.9, 150.3, 142.1, 138.2, 138.1, 133.3, 129.5, 129.2, 129.1, 128.5, 128.0, 126.9 (br. s.), 126.7, 124.3.

HRMS (ESI) m/z: 394.1442 found (calcd for $C_{26}H_{20}NO_3^+$, $[M+H]^+$ 394.1438).

4-Chloro-2-formylphenyl diphenylcarbamate (2d)



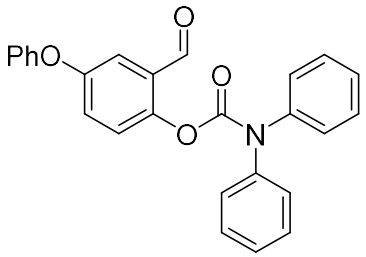
Yield 3.41 g (97%), beige solid, m.p. 100-102°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.99 (1H, s), 7.90 (1H, d, *J*=2.7 Hz), 7.79 (1H, dd, *J*=8.7, 2.7 Hz), 7.45 - 7.60 (5H, m), 7.43 (5H, br. s.), 7.30 (2H, br. s.).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 188.8, 151.6, 149.9, 141.9, 134.9, 130.6, 130.1, 129.5, 129.2, 127.0 (br. s.), 125.8.

HRMS (ESI) m/z: 352.0739 found (calcd for $C_{20}H_{15}ClNO_3^+$, $[M+H]^+$ 352.0735).

2-Formyl-4-phenoxyphenyl diphenylcarbamate (2e)



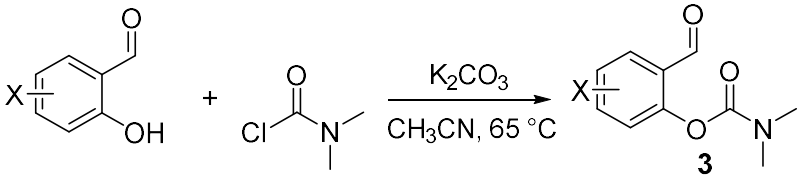
Yield 3.77 g (92%), beige solid, m.p. 102-104°C.

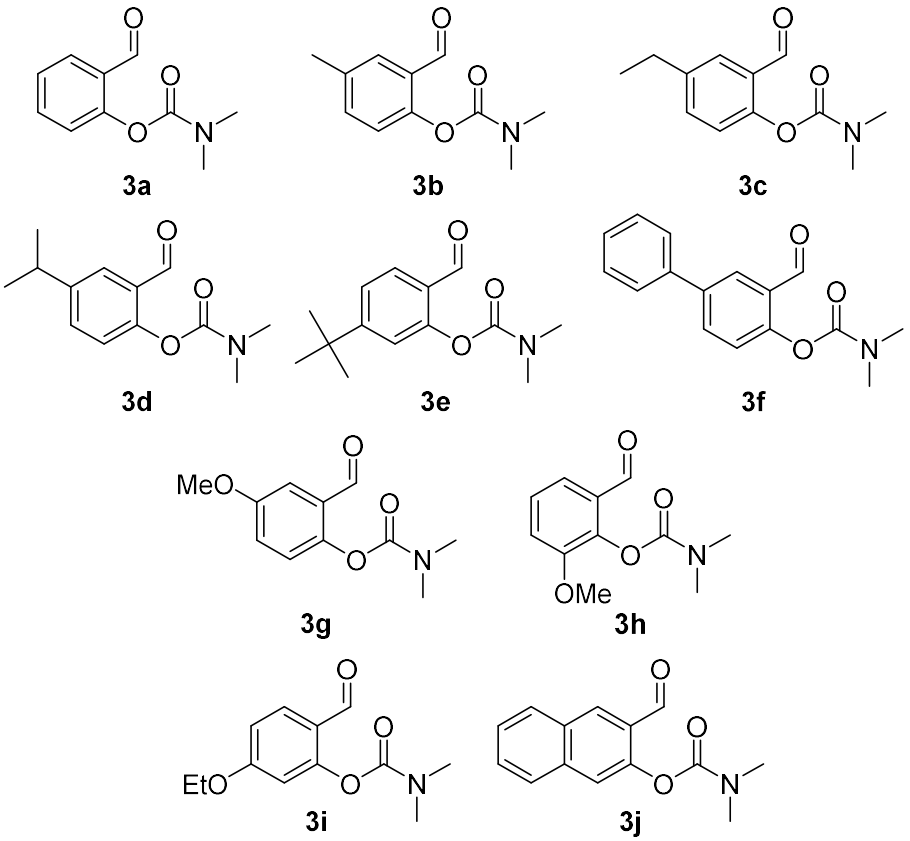
^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.99 (1H, s), 7.41 - 7.56 (10H, m), 7.34 - 7.41 (3H, m), 7.31 (2H, d, *J*=5.6 Hz), 7.21 (1H, t, *J*=7.4 Hz), 7.05 - 7.10 (2H, m).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.3, 155.9, 154.7, 152.0, 146.8, 142.0, 130.3, 129.2, 126.9 (br. s.), 125.6, 125.2, 124.2, 119.1, 118.6.

HRMS (ESI) m/z: 410.3266 found (calcd for $C_{26}H_{20}NO_4^+$, $[M+H]^+$ 410.1387).

2.3. Synthesis of 2-formylphenyl dimethylcarbamates (3)

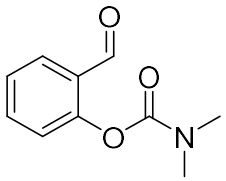




General method

A mixture of the corresponding 2-hydroxybenzaldehyde (10 mmol), dimethylcarbamoyl chloride (1.4 g, 13 mmol) and K_2CO_3 (2.76 g, 20 mmol), in freshly distilled CH_3CN (50 mL) was heated at 65℃ for 10 h. EtOAc (200 mL) was added and the resulted mixture was washed with brine (3×50 mL). Organic layer was dried over anhydrous Na_2SO_4, all volatiles were removed in vacuo and the residue was purified with flash chromatography (eluent – mixture of hexane and EtOAc, v/v 50:1).

2-Formylphenyl dimethylcarbamate (3a)

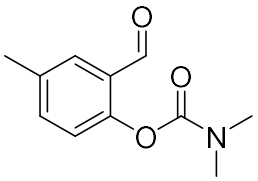


Yield 1.74 g (90%).

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.10 (1H, s), 7.86 (1H, dd, *J*=7.6, 1.6 Hz), 7.72 (1H, td, *J*=7.7, 1.7 Hz), 7.44 (1H, t, *J*=7.5 Hz), 7.30 (1H, d, *J*=8.1 Hz), 3.11 (3H, s), 2.93 (3H, s).

The spectral properties corresponded to the literature data.[4]

2-Formyl-4-methylphenyl dimethylcarbamate (3b)



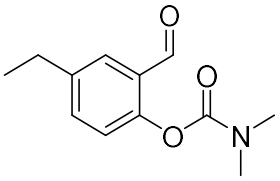
Yield 1.65 g (80%), white oil.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.05 (1H, s), 7.65 (1H, d, *J*=1.9 Hz), 7.52 (1H, dd, *J*=8.2, 2.2 Hz), 7.18 (1H, d, *J*=8.2 Hz), 3.09 (3H, s), 2.92 (3H, s), 2.37 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.5, 153.8, 150.4, 135.9, 135.3, 129.6, 128.0, 123.7, 36.4, 36.2, 20.1.

HRMS (ESI) m/z: 208.0963 found (calcd for $C_{11}H_{14}NO_3^+$, $[M+H]^+$ 208.0968).

4-Ethyl-2-formylphenyl dimethylcarbamate (3c)



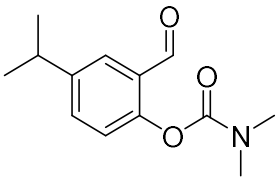
Yield 2.01 g (91%), orange solid, m.p. 48-50℃.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.06 (1H, s), 7.68 (1H, d, *J*=2.1 Hz), 7.56 (1H, dd, *J*=8.3, 2.2 Hz), 7.20 (1H, d, *J*=8.3 Hz), 3.10 (3H, s), 2.92 (3H, s), 2.68 (2H, q, *J*=7.6 Hz), 1.21 (3H, t, *J*=7.6 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.5, 153.8, 150.6, 141.5, 134.8, 128.4, 128.1, 123.8, 36.4, 36.2, 27.3, 15.3.

HRMS (ESI) m/z: 222.1125 found (calcd for $C_{12}H_{16}NO_3^+$, $[M+H]^+$ 222.1125).

2-Formyl-4-isopropylphenyl dimethylcarbamate (3d)



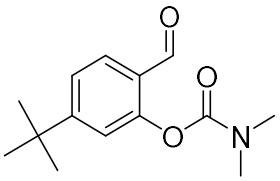
Yield 2.18 g (93%), beige solid, m.p. 46-48℃.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.07 (1H, s), 7.71 (1H, d, *J*=2.2 Hz), 7.60 (1H, dd, *J*=8.4, 2.3 Hz), 7.21 (1H, d, *J*=8.3 Hz), 3.09 (3H, s), 2.98 - 3.01 (1H, m), 2.92 (3H, s), 1.23 (6H, d, *J*=6.9 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.6, 153.8, 150.6, 146.0, 133.5, 128.1, 127.0, 123.8, 36.4, 36.2, 32.7, 23.6.

HRMS (ESI) m/z: 236.1284 found (calcd for $C_{13}H_{18}NO_3^+$, $[M+H]^+$ 236.1281).

5-(Tert-butyl)-2-formylphenyl dimethylcarbamate (3e)



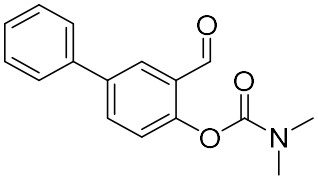
Yield 2.29 g (92%), beige solid, m.p. 54-56°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.01 (1H, s), 7.79 (1H, d, *J*=8.1 Hz), 7.47 (1H, dd, *J*=8.1, 1.4 Hz), 7.26 (1H, d, *J*=1.6 Hz), 3.10 (3H, s), 2.93 (3H, s), 1.30 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.0, 159.3, 153.8, 152.3, 129.7, 126.1, 123.0, 120.7, 36.4, 36.2, 35.1, 30.6.

HRMS (ESI) m/z: 250.1439 found (calcd for $C_{14}H_{20}NO_3^+$, $[M+H]^+$ 250.1438).

3-Formyl-[1,1'-biphenyl]-4-yl dimethylcarbamate (3f)



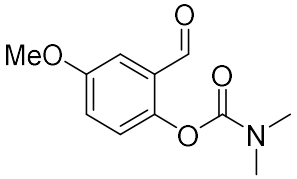
Yield 2.37 g (88%), yellow solid, m.p. 78-80°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.15 (1H, s), 8.12 (1H, d, *J*=2.5 Hz), 8.01 (1H, dd, *J*=8.4, 2.5 Hz), 7.70 - 7.76 (2H, m), 7.51 (2H, t, *J*=7.7 Hz), 7.37 - 7.45 (2H, m), 3.13 (3H, s), 2.95 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.7, 153.6, 151.6, 138.3, 137.8, 133.3, 129.1, 128.6, 128.0, 127.9, 126.7, 124.5, 36.5, 36.3.

HRMS (ESI) m/z: 270.1127 found (calcd for $C_{16}H_{16}NO_3^+$, $[M+H]^+$ 270.1125).

2-Formyl-4-methoxyphenyl dimethylcarbamate (3g)

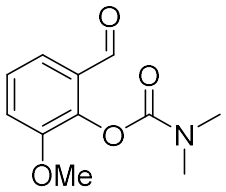


Yield 1.94 g (87%).

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.04 (1H, s), 7.32 (1H, d, *J*=3.2 Hz), 7.26 - 7.28 (1H, m), 7.21 - 7.24 (1H, m), 3.82 (3H, s), 3.09 (3H, s), 2.92 (3H, s).

The spectral properties corresponded to the literature data.[4]

2-Formyl-6-methoxyphenyl dimethylcarbamate (3h)

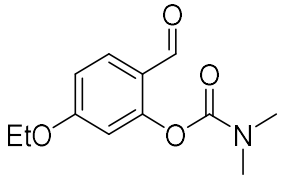


Yield 1.98 g (89%).

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.09 (1H, s), 7.45 (1H, dd, *J*=6.5, 3.2 Hz), 7.38 - 7.40 (2H, m), 3.83 (3H, s), 3.10 (3H, s), 2.92 (3H, s).

The spectral properties corresponded to the literature data.[5]

5-Ethoxy-2-formylphenyl dimethylcarbamate (3i)



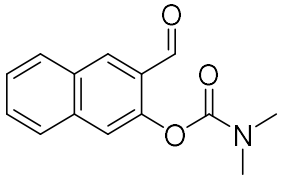
Yield 2.19 g (92%), beige solid, m.p. 50-52°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.92 (1H, s), 7.78 (1H, d, *J*=8.6 Hz), 6.97 (1H, dd, *J*=8.6, 2.2 Hz), 6.84 (1H, d, *J*=2.3 Hz), 4.14 (2H, q, *J*=7.0 Hz), 3.09 (3H, s), 2.92 (3H, s), 1.34 (3H, t, *J*=7.0 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 187.9, 164.0, 154.3, 153.5, 131.7, 121.9, 112.5, 109.3, 64.2, 36.4, 36.3, 14.4 (s).

HRMS (ESI) m/z: 238.1075 found (calcd for $C_{12}H_{16}NO_4^+$, $[M+H]^+$ 238.1074).

3-Formylnaphthalen-2-yl dimethylcarbamate (3j)



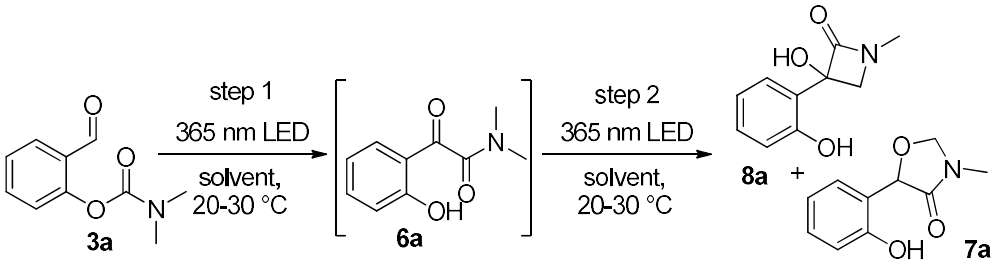
Yield 2.06 g (85%), brown solid, m.p. 52-54°C.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.61 (1H, s), 9.06 (1H, d, *J*=8.6 Hz), 8.30 (1H, d, *J*=8.8 Hz), 8.05 (1H, d, *J*=8.2 Hz), 7.73 (1H, ddd, *J*=8.4, 7.0, 1.3 Hz), 7.62 (1H, ddd, *J*=8.0, 6.9, 1.1 Hz), 7.46 (1H, d, *J*=8.9 Hz), 3.16 (3H, s), 2.97 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 191.0, 155.4, 153.6, 136.4, 131.0, 130.1, 129.5, 128.6, 126.4, 124.3, 122.7, 121.2, 36.6, 36.3.

HRMS (ESI) m/z: 244.0962 found (calcd for $C_{14}H_{14}NO_3^+$, $[M+H]^+$ 244.0968).

3. Phototransformation of 2-Formylphenyl dimethylcarbamate 3a in various solvents



2-Formylphenyl dimethylcarbamate **3a** (19 mg, 0.1 mmol) was dissolved in freshly distilled solvent under argon atmosphere (2.0 mL). Vials, prefilled with argon, with the obtained solutions were irradiated with 365 nm LED lamp in Evoluchem™ PhotoRedOx box with stirring. After 6 hours of irradiation, solvents were removed in vacuum. For solutions in DMSO, DMF, DMAC and NMP reaction mixtures were dissolved in 10 mL of EtOAc and washed with saturated KCl solution (10×3 mL). Next, organic solutions were dried over Na_2SO_4 and similarly evaporated. Finally, all the residues were analyzed by ^{1}H NMR in DMSO-d_6.

The overall aromatic signal from 6.75 ppm to 7.48 ppm (4H) was used as a standard. The quantity of components was assessed using the following signals:

- singlet from 10.02 ppm to 10.12 ppm (1H) for the starting aldehyde **3a**.
- singlet from 10.70 ppm to 10.90 ppm (1H) for product **6a**.
- singlet from 5.31 ppm to 5.33 ppm (1H) for product **7a**
- multiplet signal from 3.38 ppm to 3.62 ppm (2H) for product **8a**

Results are presented in Table S1.

Table S1. Solvents screening results for **1a**.

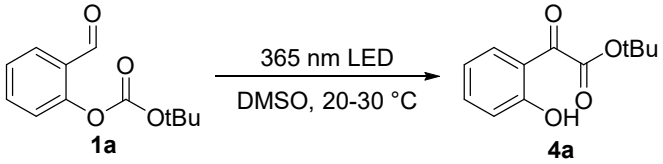
№	Solvent	**7a**, %	**8a**, %	Unreacted **3a**, %	6a, %
1	$C_2H_4Cl_2$	0	0	10	0
2	CH_2Cl_2	0	0	15	0
3	$CHCl_3$	0	0	0	0
4	CCl_4	0	0	0	0
5	MeOH	0	0	0	0
6	EtOH	0	0	0	0
7	*i*-PrOH	0	0	0	0
8	Acetone	0	0	0	0
9	CF_3CH_2OH	0	0	20	0
10	$(CF_3)_2CHOH$	0	0	85	0
11	THF	0	0	0	0
12	1,4-Dioxane	0	0	0	0
13	Et_2O	0	0	0	0
14	EtOAc	0	0	15	0
15	CH_3CN	0	0	5	0
16	C_6H_6	0	0	13	0
17	Toluene	0	0	5	0
18	PhCl	0	0	10	0
19	Hexane	0	0	0	0
20	DMSO	62	35	0	0
21	DMF	0	0	0	0
22	DMAC	60	31	0	0
23	NMP	0	0	0	0
24	Pyridine	0	0	0	0
25	DIPEA	0	0	2	0
26	NEt_3	0	0	0	0

Figure S1. Photochemical set-up. Solvent screening.

4. Kinetic study

Compound **1a**, **2a** or **3a** (0.03 mmol) was dissolved in 1 mL of DMSO-d_6 in a Schlenk vessel. The mixture was degassed under vacuum and filled with argon three times. Next, 0.65 mL portion of the solution were transferred to argon fused NMR tube and sealed. NMR tube with these solution were irradiated with 365 nm LED lamp in Evoluchem™ PhotoRedOx box for 4 h. The process was carried out strictly with two samples at a time. This approach allowed us to claim approximately identical irradiation conditions for all samples, since the tubes were installed symmetrically into the reactor each time. The mixture was analyzed by ^{1}H NMR. TMS signal was used as an internal standard - for all spectra its integral area was set on equal value. Initial integral area of aldehyde singlet signal from 10.00 ppm to 10.20 ppm was determined as 100%. The yields of component were determined using the following integral areas:

- singlet from 10.00 ppm to 10.20 ppm (1H) for the starting aldehyde **1a-3a**.
- multiplet from 6.90 ppm to 7.05 ppm (2H) for product **4a**
- triplet from 6.83 ppm to 6.94 ppm (1H) for product **5a**
- singlet from 10.70 ppm to 10.90 ppm (1H) for product **6a**
- singlet from 5.27 ppm to 5.35 ppm (1H) for product **7a**
- doublet from 3.55 ppm to 3.65 ppm (1H) for product **8a**



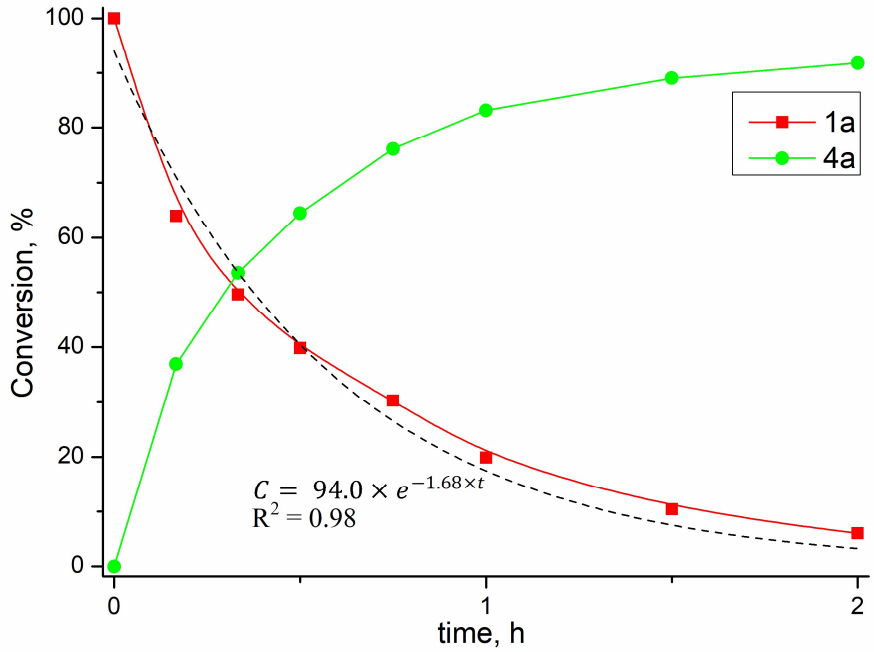


Figure S2. Kinetic study of compound **1a**.

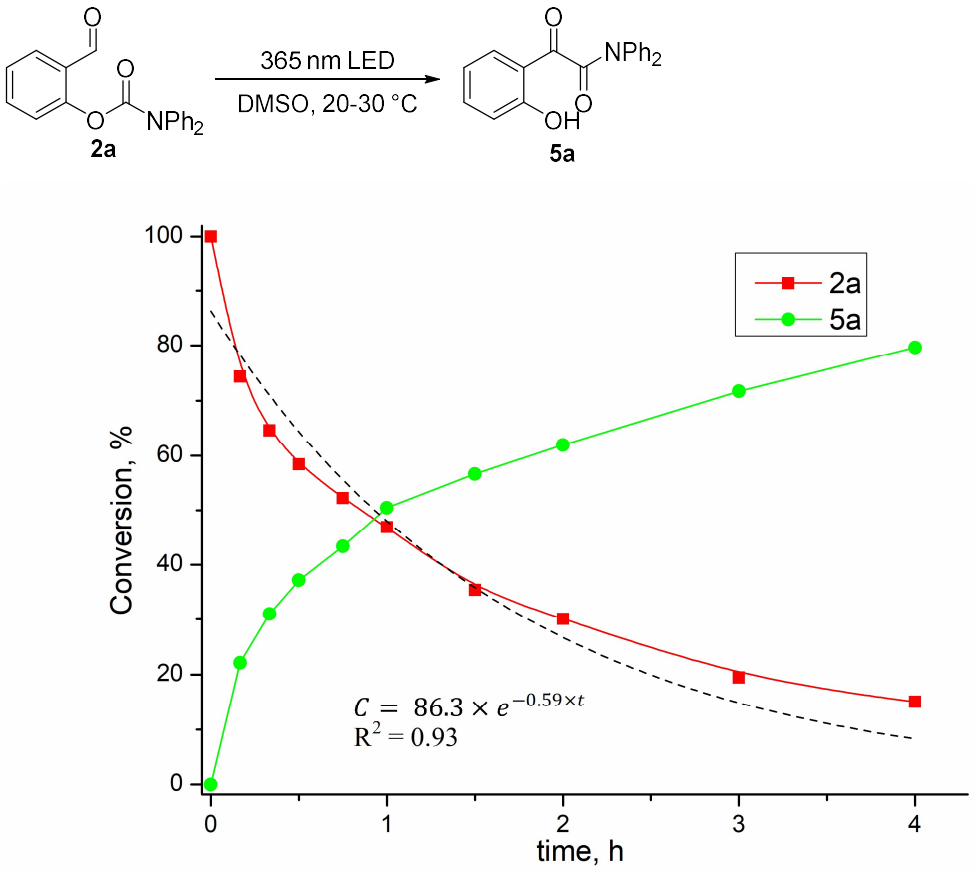


Figure S3. Kinetic study of compound **2a**.

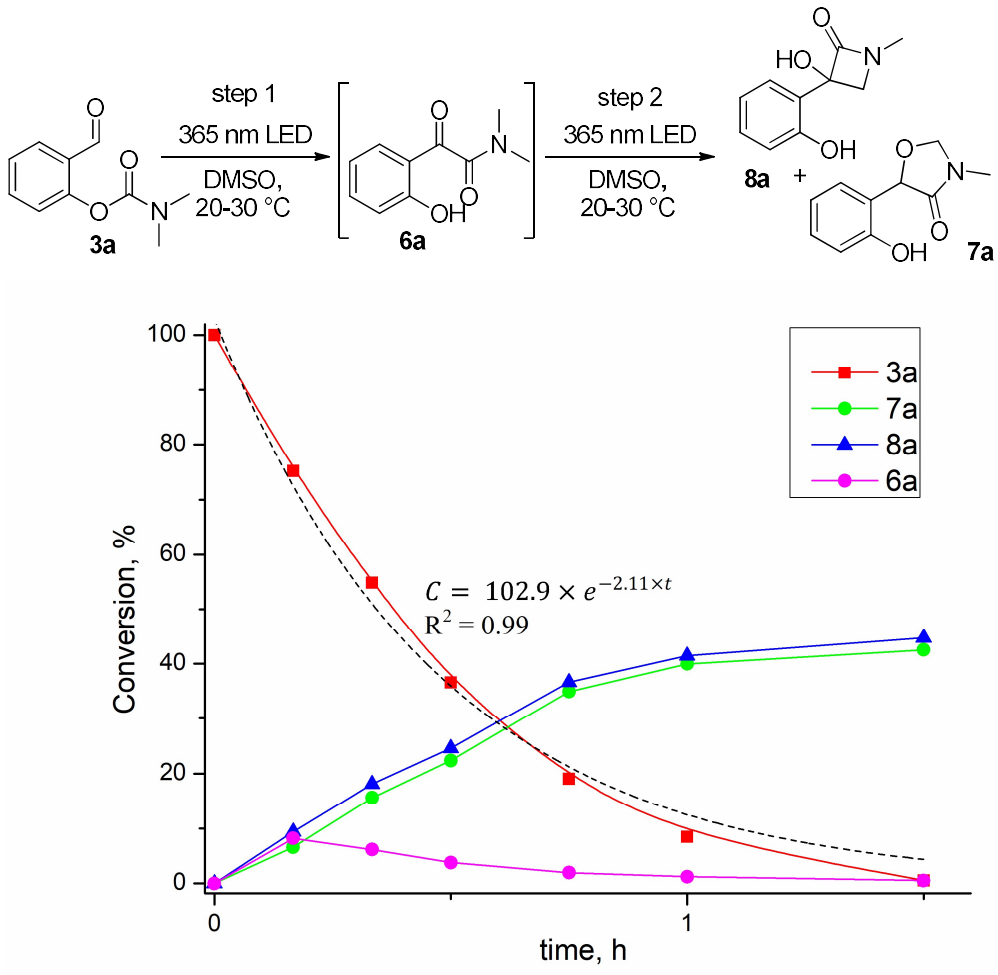


Figure S4. Kinetic study of compound **3a**.

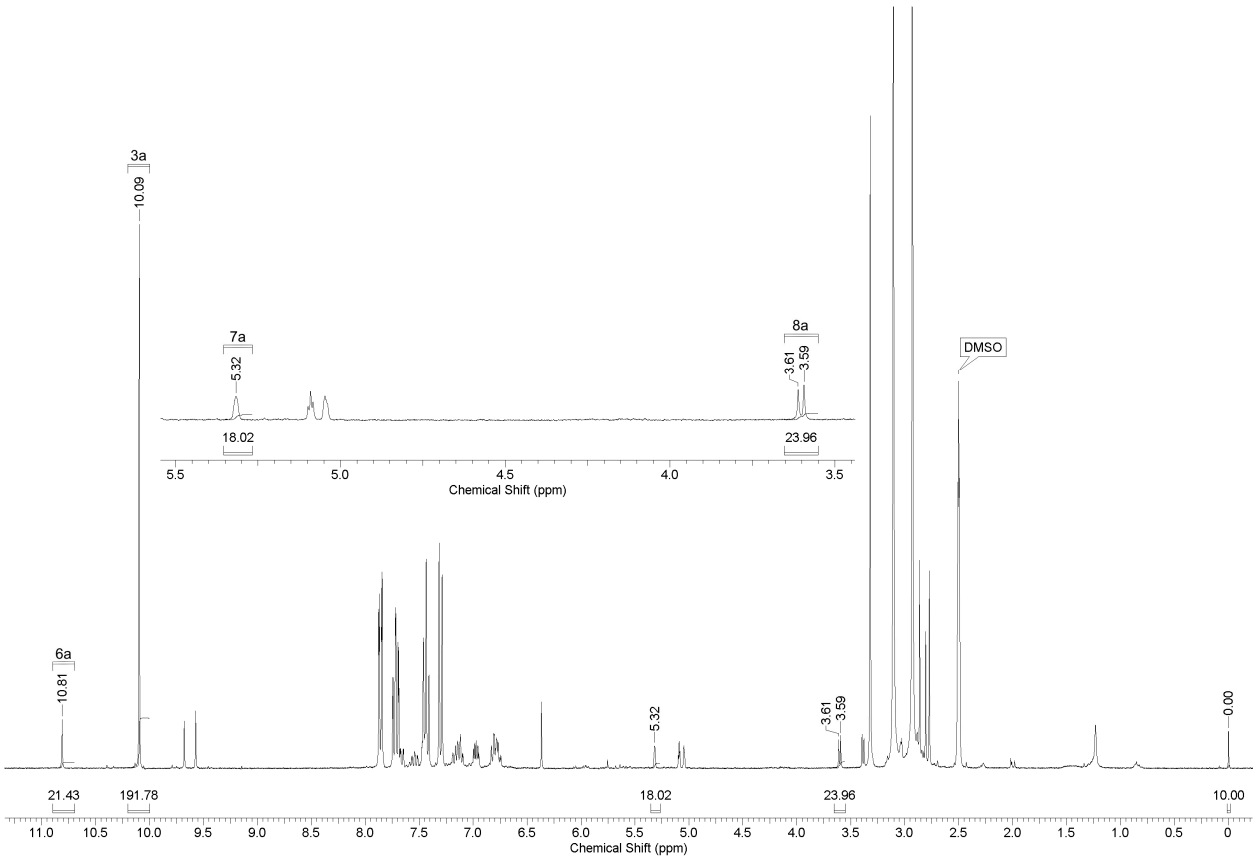


Figure S5. Example of ^{1}H NMR after 10 minutes of **3a** irradiation.

Figure S6. Photochemical set-up. Kinetic study.

5. Phototransformation of various compounds 1-3

General method

Corresponding compound **1-3** (1.0 mmol) was dissolved in freshly distilled DMSO (20 mL) in a Schlenk vessel. The mixtures were degassed under vacuum and filled with argon three times. Obtained solutions were irradiated with 365 nm LED lamp in Evoluchem™ PhotoRedOx box with stirring.

The process was carried out strictly with two samples at a time. This approach allowed us to claim approximately identical irradiation conditions for all samples, since the Schlenk vessels were installed symmetrically into the reactor each time.

The progress of the reaction was monitored by TLC and ^{1}H NMR. After the reaction completion, reaction mixtures were dissolved in 200 mL of EtOAc, washed with saturated KCl solution (10×30 mL) and dried over Na_2SO_4. All volatiles were removed in vacuo and the residue was purified with flash chromatography (eluent – mixture of hexane and EtOAc, v/v 5:1).

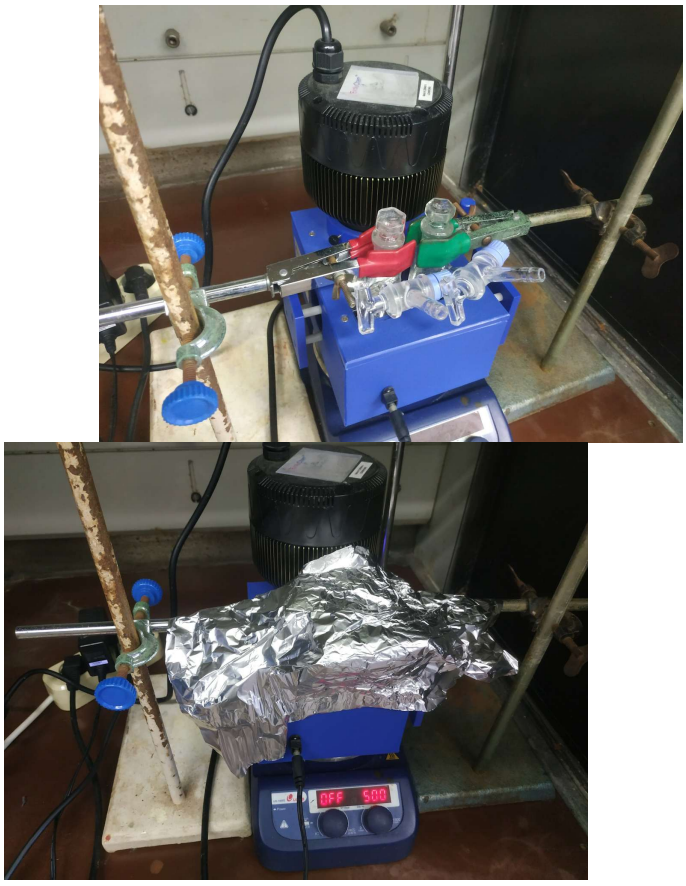
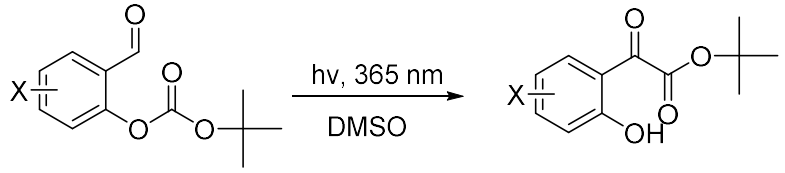


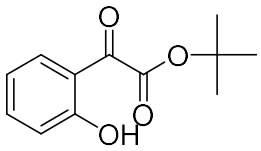
Figure S7. Photochemical set-up. 1.0 mmol scale.

5.1. Synthesis of tert-butyl 2-(2-hydroxyphenyl)-2-oxoacetates (4)



1 4

Tert-butyl 2-(2-hydroxyphenyl)-2-oxoacetate (4a)



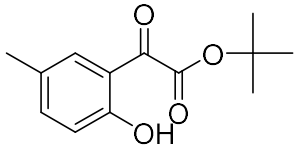
Yield 178 mg (80%), yellow solid, m.p. 32-34°C.

Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.95 (1H, s), 7.68 (1H, dd, *J*=7.8, 1.6 Hz), 7.52 - 7.56 (1H, m), 6.94 - 7.00 (2H, m), 1.52 (9H, s).

The spectral properties corresponded to the literature data.[3]

Tert-butyl 2-(2-hydroxy-4-methylphenyl)-2-oxoacetate (4b)



Yield 187 mg (79%), yellow solid, m.p. 38-40°C.

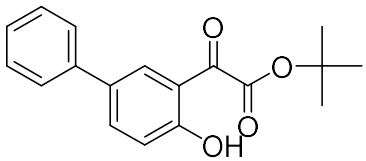
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.69 (1H, s), 7.46 (1H, d, *J*=1.6 Hz), 7.36 (1H, dd, *J*=8.4, 2.2 Hz), 6.88 (1H, d, *J*=8.4 Hz), 2.24 (3H, s), 1.51 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 186.8, 164.5, 157.6, 137.7, 129.2, 128.5, 119.1, 117.1, 82.9, 27.5, 19.8.

HRMS (ESI) m/z: 237.1117 found (calcd for $C_{13}H_{17}O_4^+$, $[M+H]^+$ 237.1121).

Tert-butyl 2-(3-hydroxy-[1,1'-biphenyl]-4-yl)-2-oxoacetate (4c)



Yield 248 mg (83%), yellow oil.

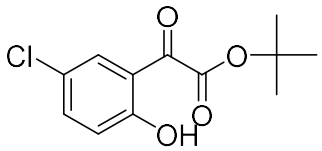
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.15 (1H, s), 7.91 (1H, d, *J*=2.5 Hz), 7.87 (1H, dd, *J*=8.6, 2.5 Hz), 7.60 - 7.63 (2H, m), 7.45 (2H, t, *J*=7.7 Hz), 7.33 - 7.36 (1H, m), 7.09 (1H, d, *J*=8.5 Hz), 1.54 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 186.6, 164.4, 159.1, 138.8, 135.1, 131.7, 129.0, 127.2, 127.2, 126.1, 119.9, 117.9, 83.1, 27.5.

HRMS (ESI) m/z: 299.1282 found (calcd for $C_{18}H_{19}O_4^+$, $[M+H]^+$ 299.1278).

Tert-butyl 2-(4-chloro-2-hydroxyphenyl)-2-oxoacetate (4d)



Yield 218 mg (85%), yellow solid, m.p. 50-52°C.

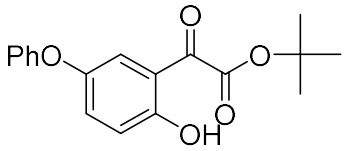
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.34 (1H, s), 7.62 (1H, d, *J*=2.7 Hz), 7.58 (1H, dd, *J*=8.8, 2.8 Hz), 7.01 (1H, d, *J*=8.8 Hz), 1.52 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 185.5, 163.8, 158.3, 136.3, 128.4, 123.5, 121.0, 119.3, 83.4, 27.4.

HRMS (ESI) m/z: 255.0441 found (calcd for $C_{12}H_{12}ClO_4^-$, $[M-H]^-$ 255.0430).

Tert-butyl 2-(2-hydroxy-4-phenoxyphenyl)-2-oxoacetate (4e)



Yield 273 mg (87%), yellow solid, m.p. 48-50°C.

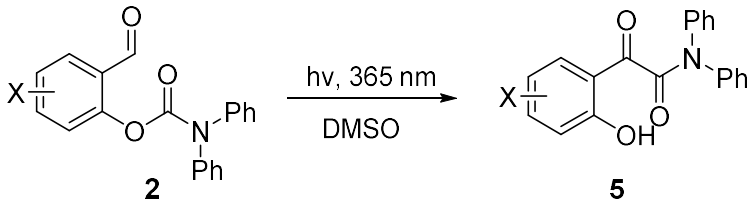
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.95 (1H, s), 7.38 (2H, t, *J*=8.0 Hz), 7.33 (1H, dd, *J*=8.9, 3.1 Hz), 7.21 (1H, d, *J*=3.1 Hz), 7.12 (1H, t, *J*=7.4 Hz), 7.03 (1H, d, *J*=8.9 Hz), 6.98 (2H, d, *J*=7.7 Hz), 1.51 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 186.0, 164.2, 157.2, 156.0, 148.9, 130.1, 128.8, 123.3, 119.8, 119.1, 118.2, 117.9, 83.2, 27.5.

HRMS (ESI) m/z: 315.1230 found (calcd for $C_{18}H_{19}O_5^+$, $[M+H]^+$ 315.1227).

5.2. Synthesis of 2-(2-hydroxyphenyl)-2-oxo-N,N-diphenylacetamides (5)



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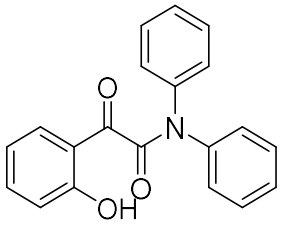
2-(2-Hydroxyphenyl)-2-oxo-N,N-diphenylacetamide (5a)



Yield 279 mg (88%), yellow solid, m.p. 144-146°C.

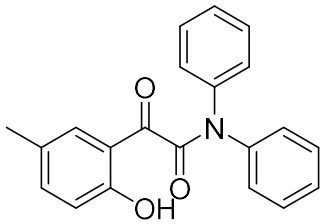
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.16 (1H, s), 7.57 (1H, dd, *J*=7.9, 1.6 Hz), 7.44 - 7.51 (3H, m), 7.40 (2H, d, *J*=7.5 Hz), 7.36 (2H, d, *J*=7.6 Hz), 7.29 - 7.33 (3H, m), 7.23 (1H, t, *J*=7.3 Hz), 6.97 (1H, d, *J*=8.2 Hz), 6.88 (1H, t, *J*=7.5 Hz).

^{13}C NMR (201 MHz, DMSO-d_6) δ ppm: 189.5, 166.6, 158.9, 141.3, 140.6, 136.4, 129.8, 129.5, 129.1, 128.2, 128.0, 126.7, 126.2, 120.5, 119.6, 117.4.

HRMS (ESI) m/z: 318.1129 found (calcd for $C_{20}H_{16}NO_3^+$, $[M+H]^+$ 318.1125).

2-(2-Hydroxy-4-methylphenyl)-2-oxo-N,N-diphenylacetamide (5b)



Yield 295 mg (89%), yellow solid, m.p. 128-130°C.

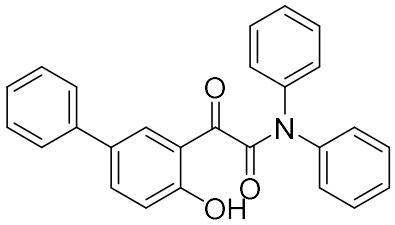
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.92 (1H, s), 7.45 (2H, t, *J*=7.8 Hz), 7.37 - 7.40 (2H, m), 7.36 (3H, d, *J*=7.4 Hz), 7.29 - 7.33 (4H, m), 7.24 (1H, t, *J*=7.4 Hz), 6.87 (1H, d, *J*=8.4 Hz), 2.19 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.4, 166.8, 156.9, 141.4, 140.7, 137.4, 129.5, 129.3, 129.1, 128.4, 128.1, 128.0, 126.7, 126.3, 120.0, 117.4, 19.7.

HRMS (ESI) m/z: 332.1283 found (calcd for $C_{21}H_{18}NO_3^+$, $[M+H]^+$ 332.1281).

2-(3-Hydroxy-[1,1'-biphenyl]-4-yl)-2-oxo-N,N-diphenylacetamide (5c)



Yield 354 mg (90%), yellow solid, m.p. 122-124°C.

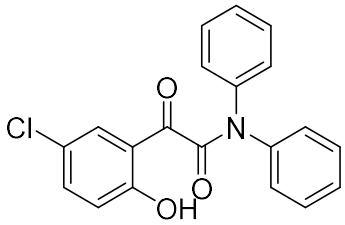
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.39 (1H, br. s.), 7.81 (1H, dd, *J*=8.6, 2.4 Hz), 7.75 (1H, d, *J*=2.5 Hz), 7.55 (2H, d, *J*=7.4 Hz), 7.47 (2H, t, *J*=7.8 Hz), 7.37 - 7.44 (6H, m), 7.33 (4H, q, *J*=7.0 Hz), 7.24 (1H, t, *J*=7.4 Hz), 7.08 (1H, d, *J*=8.6 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 189.1, 166.7, 158.4, 141.3, 140.7, 138.8, 134.7, 131.6, 129.6, 129.1, 129.0, 128.1, 128.0, 127.2, 127.1, 126.8, 126.3, 126.1, 120.9, 118.2.

HRMS (ESI) m/z: 394.1439 found (calcd for $C_{26}H_{20}NO_3^+$, $[M+H]^+$ 394.1438).

2-(4-Chloro-2-hydroxyphenyl)-2-oxo-N,N-diphenylacetamide (5d)



Yield 303 mg (86%), yellow solid, m.p. 116-118°C.

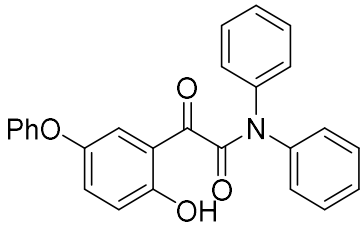
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.59 (1H, br. s.), 7.52 (1H, dd, *J*=8.8, 2.8 Hz), 7.44 - 7.47 (3H, m), 7.37 - 7.40 (2H, m), 7.30 - 7.35 (5H, m), 7.24 - 7.27 (1H, m), 7.01 (1H, d, *J*=8.8 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 188.0, 166.2, 157.6, 141.2, 140.4, 135.9, 129.6, 129.1, 128.3, 128.1, 128.1, 126.8, 126.1, 123.4, 121.9, 119.6.

HRMS (ESI) m/z: 352.0740 found (calcd for $C_{20}H_{15}ClNO_3^+$, $[M+H]^+$ 352.0735).

2-(2-Hydroxy-4-phenoxyphenyl)-2-oxo-N,N-diphenylacetamide (5e)



Yield 356 mg (87%), yellow solid, m.p. 148-150°C.

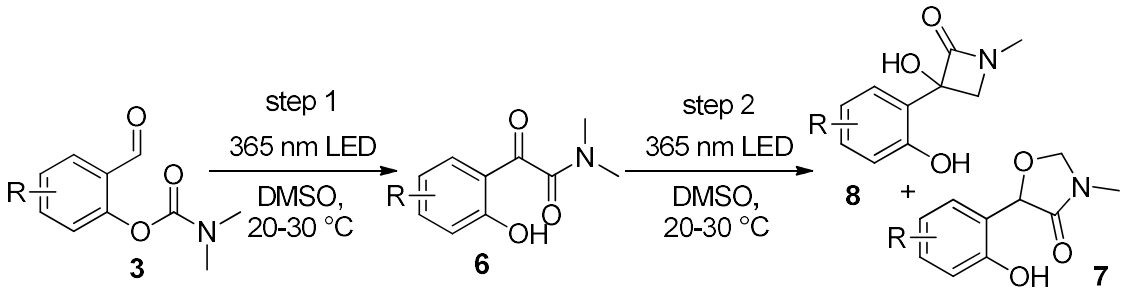
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 11.17 (1H, br. s.), 7.45 (2H, t, *J*=7.8 Hz), 7.35 - 7.39 (4H, m), 7.29 - 7.34 (5H, m), 7.26 - 7.29 (1H, m), 7.25 (1H, dd, *J*=8.9, 3.1 Hz), 7.11 (1H, t, *J*=7.4 Hz), 6.96 - 7.04 (2H, m), 6.87 (2H, d, *J*=7.8 Hz).

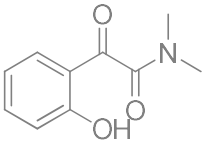
^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 188.9, 166.3, 157.2, 155.2, 148.7, 141.2, 140.4, 130.0, 129.5, 129.1, 128.3, 128.2, 128.1, 126.8, 126.2, 123.2, 121.1, 119.2, 118.3, 117.8.

HRMS (ESI) m/z: 410.1386 found (calcd for $C_{26}H_{20}NO_4^+$, $[M+H]^+$ 410.1387).

5.3. Phototransformation of 2-formylphenyl dimethylcarbamates (3)

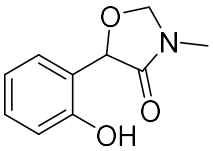


2-(2-hydroxyphenyl)-N,N-dimethyl-2-oxoacetamide (6a)



Not revealed in reaction mixture.

4-(2-Hydroxyphenyl)-2-methyl-1,2-oxazetidin-3-one (7a)



Yield 106 mg (55%), yellow solid, m.p. 110-112°C.

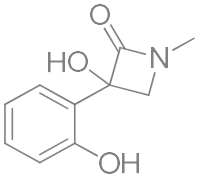
Reaction time ~ 7 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.66 (1H, s), 7.16 (1H, td, *J*=7.7, 1.6 Hz), 7.13 (1H, dd, *J*=7.5, 1.3 Hz), 6.82 (1H, d, *J*=8.0 Hz), 6.78 (1H, t, *J*=7.4 Hz), 5.32 (1H, s), 5.09 (1H, t, *J*=2.3 Hz), 5.05 (1H, d, *J*=1.3 Hz), 2.81 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.6, 155.8, 130.0, 129.8, 123.7, 118.8, 115.6, 81.4, 75.4, 26.2.

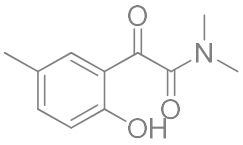
HRMS (ESI) m/z: 194.0814 found (calcd for $C_{10}H_{12}NO_3^+$, $[M+H]^+$ 194.0812).

3-Hydroxy-3-(2-hydroxylphenyl)-1-methylazetidin-2-one (8a)



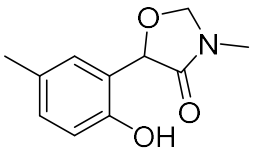
Not isolated. Decomposed on silica.

2-(2-hydroxy-5-methylphenyl)-N,N-dimethyl-2-oxoacetamide (6b)



Not revealed in reaction mixture.

5-(2-Hydroxy-5-methylphenyl)-3-methyloxazolidin-4-one (7b)



Yield 104 mg (50%), yellow solid, m.p. 130-132℃.

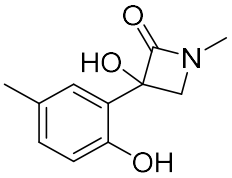
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.39 (1H, s), 6.96 (1H, d, *J*=8.1 Hz), 6.92 (1H, s), 6.71 (1H, d, *J*=8.2 Hz), 5.29 (1H, s), 5.09 (1H, s), 5.03 (1H, s), 2.80 (3H, s), 2.18 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.7, 153.5, 130.2, 130.1, 127.3, 123.3, 115.4, 81.3, 75.3, 26.3, 20.0.

HRMS (ESI) m/z: 208.0966 found (calcd for $C_{11}H_{14}NO_3^+$, $[M+H]^+$ 208.0968).

3-Hydroxy-3-(2-hydroxy-5-methylphenyl)-1-methylazetidin-2-one (8b)



Yield 85 mg (40%), yellow solid, m.p. 70-72℃.

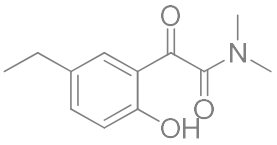
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.29 (1H, s), 7.26 (1H, d, *J*=1.9 Hz), 6.92 (1H, dd, *J*=8.1, 2.0 Hz), 6.68 (1H, d, *J*=8.1 Hz), 6.31 (1H, s), 3.59 (1H, d, *J*=5.3 Hz), 3.38 (1H, d, *J*=5.3 Hz), 2.76 (3H, s), 2.18 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.9, 152.7, 129.1, 127.9, 126.7, 125.1, 115.4, 83.9, 57.8, 28.0, 20.2.

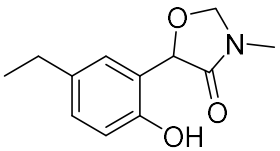
HRMS (ESI) m/z: 208.0966 found (calcd for $C_{11}H_{14}NO_3^+$, $[M+H]^+$ 208.0968).

2-(5-ethyl-2-hydroxyphenyl)-N,N-dimethyl-2-oxoacetamide (6c)



Not revealed in reaction mixture.

5-(5-Ethyl-2-hydroxyphenyl)-3-methyloxazolidin-4-one (7c)



Yield 115 mg (52%), yellow solid, m.p. 128-130°C.

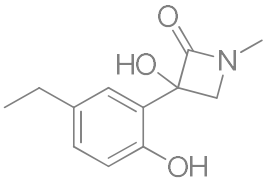
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.41 (1H, s), 7.00 (1H, d, *J*=7.1 Hz), 6.95 (1H, s), 6.73 (1H, d, *J*=8.1 Hz), 5.28 (1H, s), 5.09 (1H, br. s.), 5.04 (1H, s), 2.80 (3H, s), CH_2 (intersect with DMSO), 1.12 (3H, t, *J*=7.5 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.7, 153.7, 133.9, 129.2, 128.9, 123.3, 115.5, 81.4, 75.6, 27.2, 26.3, 15.9.

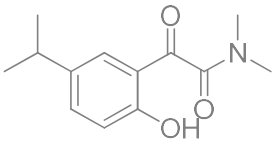
HRMS (ESI) m/z: 222.1122 found (calcd for $C_{12}H_{16}NO_3^+$, $[M+H]^+$ 222.1125).

3-(5-ethyl-2-hydroxyphenyl)-3-hydroxy-1-methylazetidin-2-one (8c)



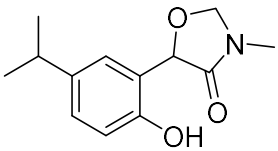
Not isolated. Decomposed on silica.

2-(2-hydroxy-5-isopropylphenyl)-N,N-dimethyl-2-oxoacetamide (6d)



Not revealed in reaction mixture.

5-(2-Hydroxy-5-isopropylphenyl)-3-methyloxazolidin-4-one (7d)



Yield 139 mg (59%), yellow solid, m.p. 118-120°C.

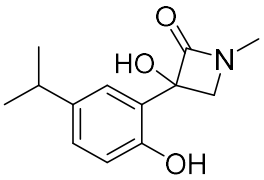
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.41 (1H, s), 7.04 (1H, dd, *J*=8.2, 2.1 Hz), 6.98 (1H, d, *J*=2.1 Hz), 6.73 (1H, d, *J*=8.3 Hz), 5.27 (1H, s), 5.09 (1H, t, *J*=2.2 Hz), 5.04 (1H, d, *J*=1.1 Hz), 2.80 (3H, s), 2.75 - 2.79 (1H, m), 1.14 (6H, dd, *J*=6.9, 2.3 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.7, 153.8, 138.6, 127.9, 127.4, 123.2, 115.4, 81.4, 75.9, 32.5, 26.3, 24.2, 24.1.

HRMS (ESI) m/z: 236.1280 found (calcd for $C_{13}H_{18}NO_3^+$, $[M+H]^+$ 236.1281).

3-Hydroxy-3-(2-hydroxy-5-isopropylphenyl)-1-methylazetidin-2-one (8d)



Yield 89 mg (38%), yellow solid, m.p. 78-80°C.

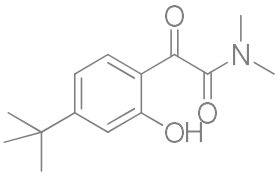
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.32 (1H, s), 7.34 (1H, d, *J*=1.9 Hz), 6.99 (1H, dd, *J*=8.1, 2.0 Hz), 6.71 (1H, d, *J*=8.2 Hz), 6.31 (1H, s), 3.61 (1H, d, *J*=5.3 Hz), 3.37 (1H, d, *J*=5.3 Hz), 2.78 - 2.82 (1H, m), 2.77 (3H, s), 1.14 (6H, d, *J*=6.6 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 170.0, 152.9, 138.0, 126.4, 125.3, 124.9, 115.3, 84.0, 57.7, 32.7, 28.1, 24.2, 24.2.

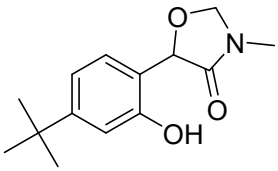
HRMS (ESI) m/z: 236.1278 found (calcd for $C_{13}H_{18}NO_3^+$, $[M+H]^+$ 236.1281).

2-(4-(tert-butyl)-2-hydroxyphenyl)-N,N-dimethyl-2-oxoacetamide (6e)



Not revealed in reaction mixture.

5-(5-(Tert-butyl)-2-hydroxyphenyl)-3-methyloxazolidin-4-one (7e)



Yield 135 mg (54%), yellow solid, m.p. 100-102°C.

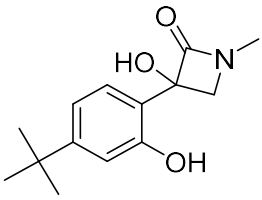
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.48 (1H, s), 7.03 (1H, d, *J*=8.0 Hz), 6.84 (1H, d, *J*=1.7 Hz), 6.81 (1H, dd, *J*=8.0, 1.8 Hz), 5.27 (1H, s), 5.07 (1H, t, *J*=2.2 Hz), 4.99 - 5.05 (1H, m), 2.80 (3H, s), 1.24 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.7, 155.5, 152.7, 129.7, 120.7, 115.8, 112.5, 81.3, 75.3, 34.2, 31.0, 26.3.

HRMS (ESI) m/z: 250.1438 found (calcd for $C_{14}H_{20}NO_3^+$, $[M+H]^+$ 250.1438).

3-(5-(Tert-butyl)-2-hydroxyphenyl)-3-hydroxy-1-methylazetidin-2-one (8e)



Yield 112 mg (45%), yellow solid, m.p. 140-142°C.

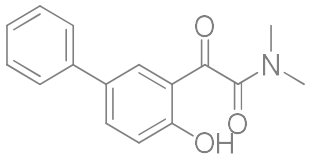
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.37 (1H, s), 7.36 (1H, d, *J*=8.1 Hz), 6.82 (1H, d, *J*=1.8 Hz), 6.80 (1H, dd, *J*=8.1, 1.9 Hz), 6.27 (1H, s), 3.60 (1H, d, *J*=5.3 Hz), 3.37 (1H, d, *J*=5.3 Hz), 2.76 (3H, s), 1.23 (9H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.9, 154.7, 151.8, 127.1, 122.4, 115.3, 112.5, 83.8, 57.7, 34.1, 31.1, 28.0.

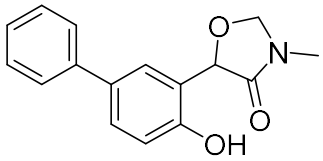
HRMS (ESI) m/z: 250.1436 found (calcd for $C_{14}H_{20}NO_3^+$, $[M+H]^+$ 250.1438).

2-(4-hydroxy-[1,1'-biphenyl]-3-yl)-N,N-dimethyl-2-oxoacetamide (6f)



Not revealed in reaction mixture.

5-(4-Hydroxy-[1,1'-biphenyl]-3-yl)-3-methyloxazolidin-4-one (7f)



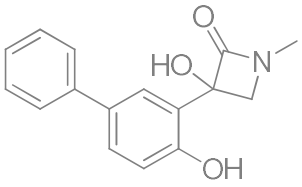
Yield 143 mg (53%), yellow solid, m.p. 140-142°C.

Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.86 (1H, s), 7.56 (2H, d, *J*=8.1 Hz), 7.49 (1H, dd, *J*=8.3, 2.0 Hz), 7.44 (1H, d, *J*=2.2 Hz), 7.42 (2H, t, *J*=7.5 Hz), 7.29 (1H, td, *J*=7.3, 0.7 Hz), 6.91 (1H, d, *J*=8.3 Hz), 5.39 (1H, s), 5.15 (1H, t, *J*=2.0 Hz), 5.08 (1H, d, *J*=1.0 Hz), 2.83 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.5, 155.6, 139.8, 130.9, 128.8, 128.4, 128.1, 126.5, 126.0, 124.1, 116.1, 81.5, 75.7, 26.3.

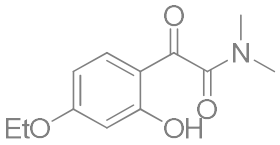
HRMS (ESI) m/z: 270.1131 found (calcd for $C_{16}H_{16}NO_3^+$, $[M+H]^+$ 270.1125).



3-hydroxy-3-(4-hydroxy-[1,1'-biphenyl]-3-yl)-1-methylazetidin-2-one (8f)

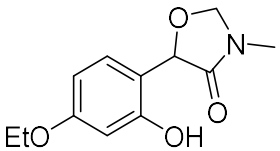
Not isolated. Decomposed on silica.

2-(4-ethoxy-2-hydroxyphenyl)-N,N-dimethyl-2-oxoacetamide (6g)



Not revealed in reaction mixture.

5-(4-Ethoxy-2-hydroxyphenyl)-3-methyloxazolidin-4-one (7g)



Yield 109 mg (46%), yellow solid, m.p. 116-118°C.

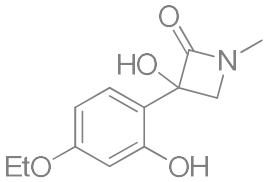
Reaction time ~ 24 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 9.67 (1H, s), 6.99 (1H, d, *J*=8.1 Hz), 6.33 - 6.36 (2H, m), 5.22 (1H, s), 5.04 (1H, t, *J*=2.2 Hz), 5.00 (1H, s), 3.95 (2H, q, *J*=6.9 Hz), 2.79 (3H, s), 1.30 (3H, t, *J*=6.9 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 169.8, 159.9, 157.0, 131.0, 116.1, 104.9, 101.7, 81.1, 75.3, 62.9, 26.3, 14.6.

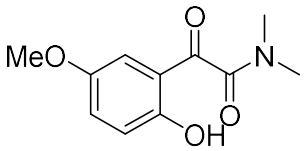
HRMS (ESI) m/z: 238.1070 found (calcd for $C_{12}H_{16}NO_4^+$, $[M+H]^+$ 238.1074).

3-(4-ethoxy-2-hydroxyphenyl)-3-hydroxy-1-methylazetidin-2-one (8g)



Not isolated. Decomposed on silica.

2-(2-Hydroxy-5-methoxyphenyl)-N,N-dimethyl-2-oxoacetamide (6h)



Yield 145 mg (65%), yellow oil.

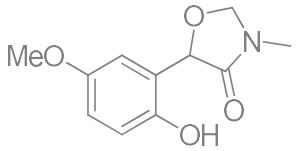
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.36 (1H, s), 7.18 (1H, dd, J=8.9, 3.3 Hz), 7.13 (1H, d, J=3.2 Hz), 6.91 (1H, d, J=8.9 Hz), 3.73 (3H, s), 2.91 (3H, s), 2.85 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 191.6, 168.0, 154.1, 152.2, 124.6, 120.0, 118.8, 111.3, 55.5, 36.1, 33.3.

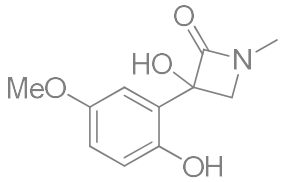
HRMS (ESI) m/z: 224.0921 found (calcd for $C_{11}H_{14}NO_4^+$, $[M+H]^+$ 224.0917).

5-(2-hydroxy-5-methoxyphenyl)-3-methyloxazolidin-4-one (7h)



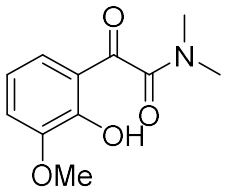
Not revealed in reaction mixture.

3-hydroxy-3-(2-hydroxy-5-methoxyphenyl)-1-methylazetidin-2-one (8h)



Not revealed in reaction mixture.

2-(2-Hydroxy-3-methoxyphenyl)-N,N-dimethyl-2-oxoacetamide (6i)



Yield 127 mg (57%), yellow oil.

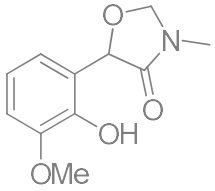
Reaction time ~ 48 h.

^{1}H NMR (800 MHz, DMSO-d_6) δ ppm: 10.35 (1H, s), 7.27 (1H, dd, *J*=7.9, 1.3 Hz), 7.22 (1H, dd, *J*=8.1, 1.4 Hz), 6.93 (1H, t, *J*=8.0 Hz), 3.83 (3H, s), 2.93 (3H, s), 2.85 (3H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 192.8, 167.4, 150.0, 148.5, 121.0, 119.9, 119.4, 117.7, 56.1, 36.2, 33.3.

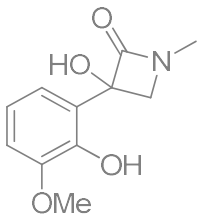
HRMS (ESI) m/z: 224.0916 found (calcd for $C_{11}H_{14}NO_4^+$, $[M+H]^+$ 224.0917).

5-(2-hydroxy-3-methoxyphenyl)-3-methyloxazolidin-4-one (7i)



Not revealed in reaction mixture.

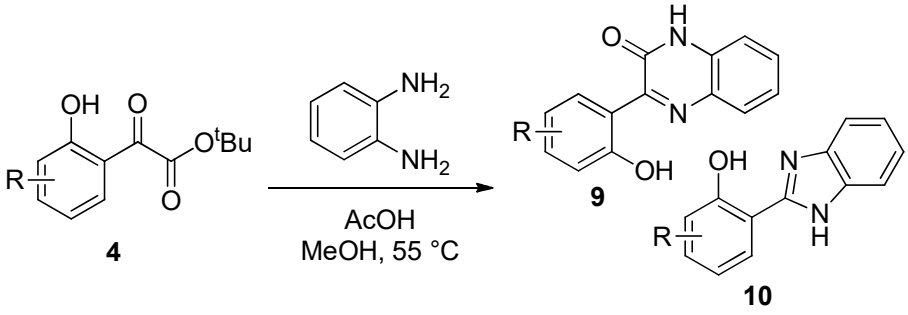
3-hydroxy-3-(2-hydroxy-3-methoxyphenyl)-1-methylazetidin-2-one (8i)



Not revealed in reaction mixture.

6. Post-transformations of 1,2-dicarbonyl compounds 4 and 5.

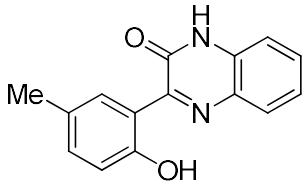
6.1. Synthesis of quinoxalinone (9) and benzimidazole (10) derivatives



General method

A mixture of corresponding α-ketoester **4** (0.05 mmol), *o*-phenylenediamine (6 mg, 0.05 mmol) and AcOH (10 mg, 0.16 mmol) in freshly distilled MeOH (1 mL) was stirred at 55 °C for 5 h. The reaction mixture was evaporated and the residue was purified with flash chromatography (elient – mixture of hexane and EtOAc, v/v 4:1). Corresponding products **9** (Rf = 0.25) and **10** (Rf = 0.50) were isolated in separate fractions.\

3-(2-Hydroxy-5-methylphenyl)quinoxalin-2(1*H*)-one (9b)



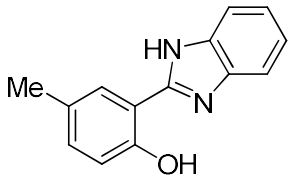
Yield 5 mg (60%), orange oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 12.74 (1 H, br. s.), 12.32 (1 H, s), 8.40 (1 H, d, *J*=1.2 Hz), 7.84 (1 H, d, *J*=7.7 Hz), 7.49 - 7.62 (1 H, m), 7.29 - 7.42 (2 H, m), 7.20 (1 H, dd, *J*=8.5, 1.7 Hz), 6.85 (1 H, d, *J*=8.3 Hz), 2.27 (3 H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 156.9, 155.0, 154.8, 133.0, 131.8, 131.0, 130.4, 130.1, 127.5, 126.8, 123.7, 119.7, 117.0, 115.2, 20.3.

HRMS (ESI) m/z: 253.0972 found (calcd for $C_{15}H_{13}N_2O_2^+$, $[M+H]^+$ 253.0972).

2-(1H-benzo[d]imidazol-2-yl)-4-methylphenol (10b)



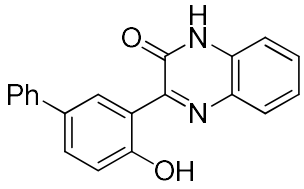
Yield 7 mg, (39%), colorless oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 13.15 (1 H, br. s.), 12.87 (1 H, br. s.), 7.88 (1 H, d, *J*=0.6 Hz), 7.75 – 7.50 (2 H, m), 7.23 - 7.36 (2 H, m), 7.19 (1 H, dd, *J*=8.4, 1.3 Hz), 6.93 (1 H, d, *J*=8.3 Hz), 2.33 (3 H, s).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 155.9, 151.7, 141.0, 133.3, 132.4, 127.7, 126.2, 123.2, 122.4, 117.9, 117.0, 112.2, 111.5, 20.2.

HRMS (ESI) m/z: 225.1026 found (calcd for $C_{14}H_{13}N_2O^+$, $[M+H]^+$ 225.1022).

3-(4-Hydroxy-[1,1'-biphenyl]-3-yl)quinoxalin-2(1*H*)-one (9c)



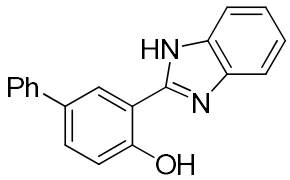
Yield 4 mg (63%), orange oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 12.77 (1 H, br. s.), 12.62 (1 H, s), 8.91 (1 H, d, *J*=2.2 Hz), 7.88 (1 H, d, *J*=8.0 Hz), 7.70 (1 H, dd, *J*=8.4, 1.9 Hz), 7.54 - 7.66 (3 H, m), 7.46 (2 H, t, *J*=7.6 Hz), 7.27 - 7.42 (3 H, m), 7.05 (1 H, d, *J*=8.5 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 158.7, 154.9, 140.0, 132.0, 130.6, 130.5, 130.5, 130.2, 129.3, 129.0, 127.7, 126.7, 126.1, 123.8, 120.4, 117.7, 115.3.

HRMS (ESI) m/z: 313.0982 found (calcd for $C_{20}H_{13}N_2O_2^-$, $[M-H]^-$ 313.0983).

3-(1H-benzo[d]imidazol-2-yl)-[1,1'-biphenyl]-4-ol (10c)



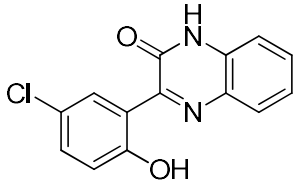
Yield 10 mg (25%), colorless oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 13.35 (1 H, br. s.), 13.26 (1 H, br. s.), 8.45 (1 H, d, *J*=2.0 Hz), 7.68 - 7.81 (4 H, m), 7.64 (1 H, d, *J*=7.5 Hz), 7.51 (2 H, t, *J*=7.5 Hz), 7.24 - 7.42 (3 H, m), 7.13 (1 H, d, *J*=8.6 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 157.6, 151.7, 140.9, 139.4, 133.2, 131.2, 129.9, 128.9, 127.0, 126.2, 124.3, 123.4, 122.5, 118.0, 117.8, 112.9, 111.6.

HRMS (ESI) m/z: 287.1180 found (calcd for $C_{19}H_{15}N_2O^+$, $[M+H]^+$ 287.1179).

3-(5-Chloro-2-hydroxyphenyl)quinoxalin-2(1*H*)-one (9d)



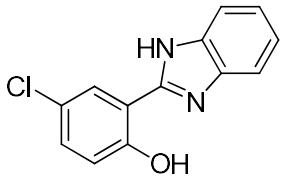
Yield 5 mg (51%), orange oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 12.82 (1 H, br. s.), 12.66 (1 H, s), 8.63 (1 H, d, *J*=2.7 Hz), 7.88 (1 H, d, *J*=7.7 Hz), 7.53 - 7.65 (1 H, m), 7.32 - 7.46 (3 H, m), 6.98 (1 H, d, *J*=8.8 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 157.9, 154.7, 153.7, 132.0, 131.7, 130.9, 130.1, 130.0, 127.8, 123.8, 121.9, 121.2, 118.9, 115.3.

HRMS (ESI) m/z: 271.0278 found (calcd for $C_{14}H_8ClN_2O_2^+$, $[M-H]^-$ 271.0280).

2-(1H-benzo[d]imidazol-2-yl)-4-chlorophenol (10d)



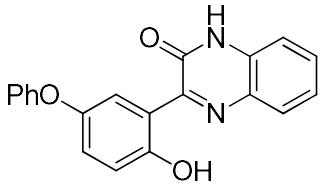
Yield 7 mg (37%), colorless oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 13.11 - 13.49 (2 H, m), 8.17 (1 H, d, *J*=2.6 Hz) 7.80 – 7.57 (2 H, m), 7.41 (1 H, dd, *J*=8.8, 2.6 Hz), 7.22 - 7.37 (2 H, m), 7.08 (1 H, d, *J*=8.9 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 156.7, 150.4, 133.1, 131.3, 125.6, 123.7, 122.8, 122.7, 119.1, 118.2, 114.1, 111.7.

HRMS (ESI) m/z: 245.0478 found (calcd for $C_{13}H_{10}ClN_2O^+$, $[M+H]^+$ 245.0476).

3-(2-Hydroxy-5-phenoxyphenyl)quinoxalin-2(1*H*)-one (9e)



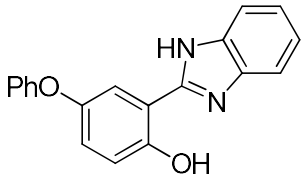
Yield 5 mg (61%), orange oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 12.58 - 12.97 (2 H, m), 8.47 (1 H, d, *J*=2.8 Hz), 7.87 (1 H, d, *J*=8.0 Hz), 7.63 – 7.53 (1 H, m), 7.45 – 7.27 (4 H, m), 7.15 (1 H, dd, *J*=8.8, 2.9 Hz), 6.91 - 7.11 (4 H, m).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 158.7, 154.9, 140.0, 132.0, 130.6, 130.5, 130.5, 130.2, 129.3, 129.0, 127.7, 126.7, 126.1, 123.8, 120.4, 117.7, 115.3.

HRMS (ESI) m/z: 329.0930 found (calcd for $C_{20}H_{13}N_2O_3^-$, $[M-H]^-$ 329.0932).

2-(1H-benzo[d]imidazol-2-yl)-4-phenoxyphenol (10e)



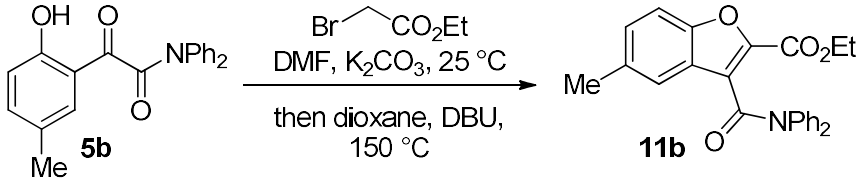
Yield 10 mg (30%), colorless oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 13.18 (1 H, br. s.), 13.01 (1 H, br. s.), 7.87 (1 H, d, *J*=2.6 Hz), 7.73 (1 H, d, *J*=7.3 Hz), 7.57 (1 H, d, *J*=7.0 Hz), 7.33 - 7.43 (2 H, m), 7.22 - 7.33 (2 H, m), 7.05 - 7.18 (3 H, m), 7.00 (2 H, d, *J*=7.7 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 158.3, 154.6, 151.0, 147.8, 140.8, 133.2, 130.0, 123.9, 123.5, 122.6, 122.5, 118.6, 118.1, 117.5, 117.0, 113.2, 111.6.

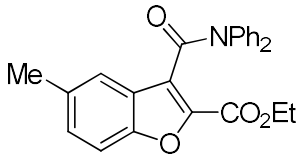
HRMS (ESI) m/z: 303.1132 found (calcd for $C_{19}H_{15}N_2O_2^+$, $[M+H]^+$ 303.1128).

6.2. Synthesis of ethyl 3-(diphenylcarbamoyl)-5-methylbenzofuran-2-carboxylate (11b)



A mixture of 2-(2-Hydroxy-4-methylphenyl)-2-oxo-N,N-diphenylacetamide **5b** (17 mg, 0.05 mmol), ethyl bromoacetate (11 mg, 0.06 mmol) and K_2CO_3 (11 mg, 0.07 mmol) in freshly distilled DMF (1 mL) was stirred at room temperature for 1 h. EtOAc (30 mL) was added and the resulted mixture was washed with brine (3×30 mL). Organic layer was dried over anhydrous Na_2SO_4, all volatiles were removed in vacuo. The residue was dissolved in dioxane (1 mL) and DBU (16 mg, 0.10 mmol) was added. The mixture was heated in an autoclave at 150 °C for 10 h. The reaction mixture was evaporated and the residue was purified with flash chromatography (elient – mixture of hexane and EtOAc, v/v 4:1).

Ethyl 3-(diphenylcarbamoyl)-5-methylbenzofuran-2-carboxylate (11b)



Yield 11 mg (54%), colorless oil.

^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 7.76 (1 H, s), 7.41 - 7.56 (5 H, m), 7.28 - 7.40 (2 H, m), 7.01 - 7.24 (5 H, m), 4.40 (2 H, q, *J*=7.1 Hz), 2.46 (3 H, s), 1.35 (3 H, t, *J*=7.1 Hz).

^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 162.2, 157.9, 152.0, 142.0, 141.9, 140.1, 134.2, 130.1, 129.1, 129.1, 127.9, 127.6, 126.8, 126.7, 125.6, 124.0, 121.3, 111.8, 61.8, 20.8, 14.2.

HRMS (ESI) m/z: 400.1548 found (calcd for $C_{25}H_{22}NO_4^+$, $[M+H]^+$ 400.1543).

7. References

[1] Sessions, E. H.; Jacobi, P. A., *Org. Lett.* **2006**, *8*, 4125–4128.

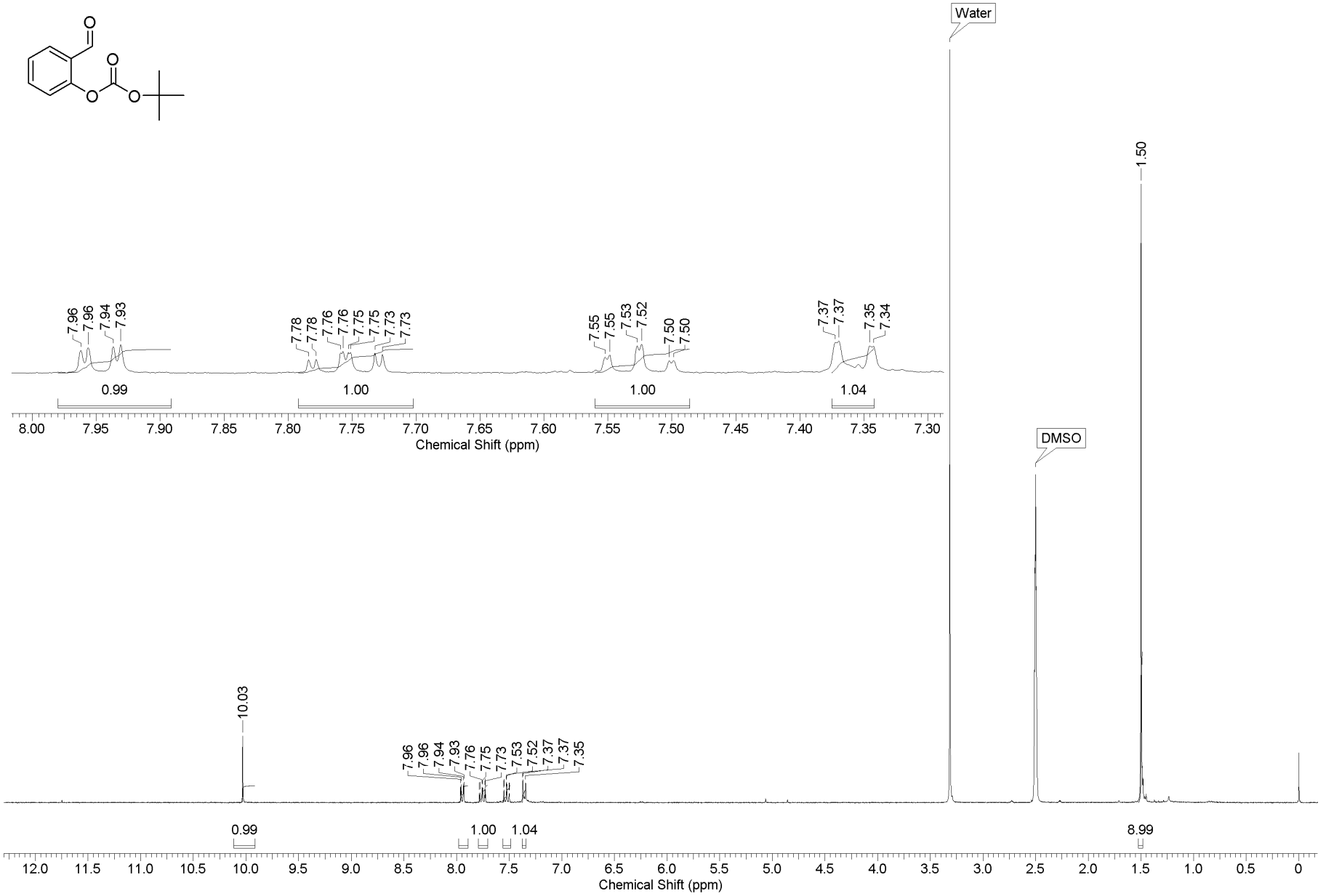
[2] Selenski, C.; Pettus, T. R. R., *J. Org. Chem.* **2004**, *69*, 9196–9203.

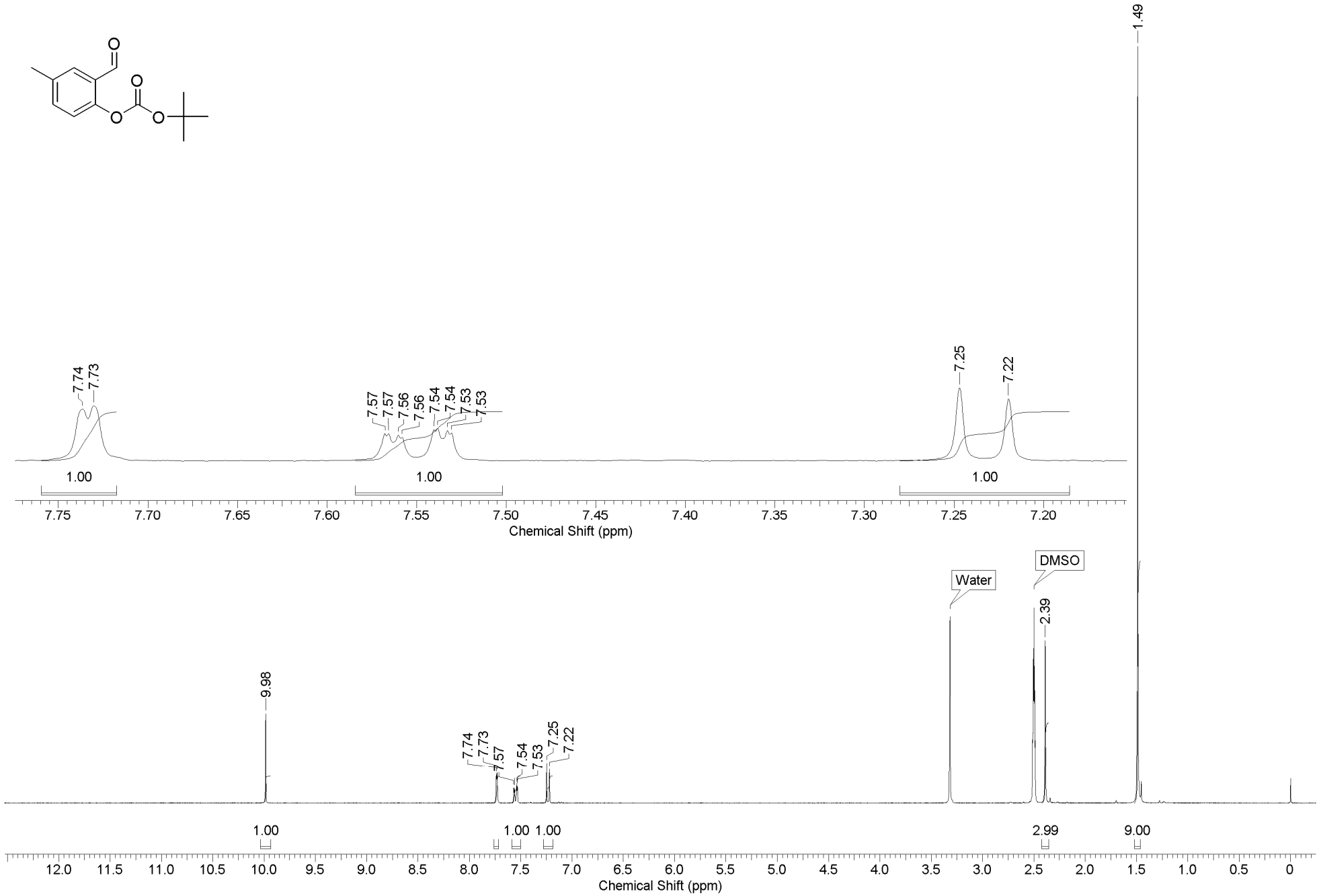
[3] Hari, Y.; Kondo, R.; Date, K.; Aoyama, T., *Tetrahedron* **2009**, *65*, 8708–8713.

[4] Ali, W.; Rout, S. K.; Guin, S.; Modi, A.; Banerjee, A.; Patel, B. K., *Adv. Synth. Catal.* **2015**, *357*, 515–522.

[5] Barve, B. D.; Wu, Y.-C.; El-Shazly, M.; Chuang, D.-W.; Cheng, Y.-B.; Wang, J.-J.; Chang, F.-R., *J. Org. Chem.* **2014**, *79*, 3206–3214.

8. Copies of ^{1}H and ^{13}C NMR spectra

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7.74
7.73
7.57
7.57
7.56
7.56
7.54
7.54
7.53
7.53
7.25
7.22
1.00
1.00
1.00
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
Chemical Shift (ppm)
Water
DMSO
2.39
9.98
7.74
7.73
7.57
7.54
7.53
7.25
7.22
1.00
1.00
1.00
2.99
9.00
12.0
11.5
11.0
10.5
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
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2.5
2.0
1.5
1.0
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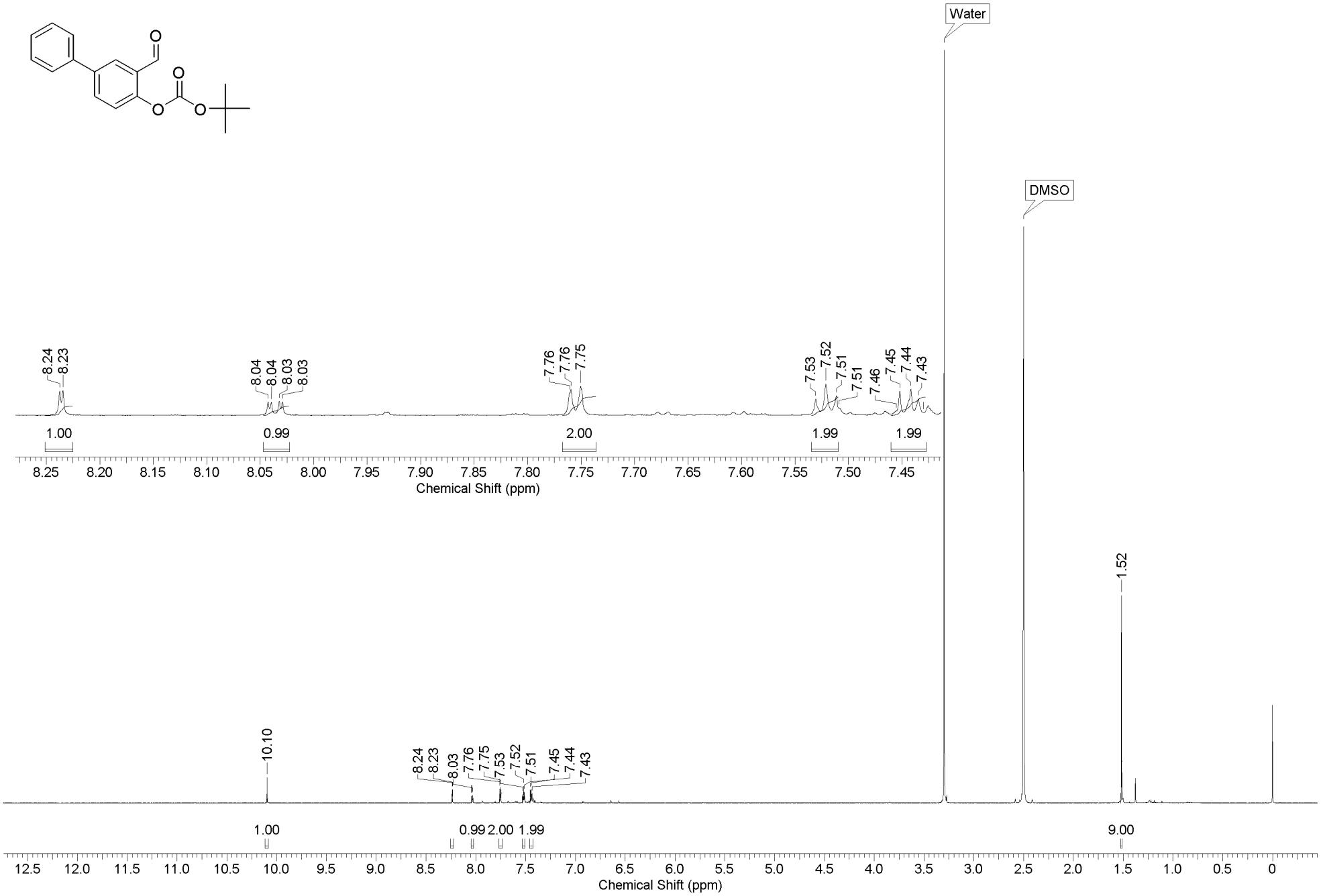
0
Chemical Shift (ppm)


Water
DMSO
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8.23
8.04
8.04
8.03
8.03
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7.76
7.75
7.53
7.52
7.51
7.51
7.46
7.45
7.44
7.43
1.00
0.99
2.00
1.99
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Chemical Shift (ppm)
1.52
10.10
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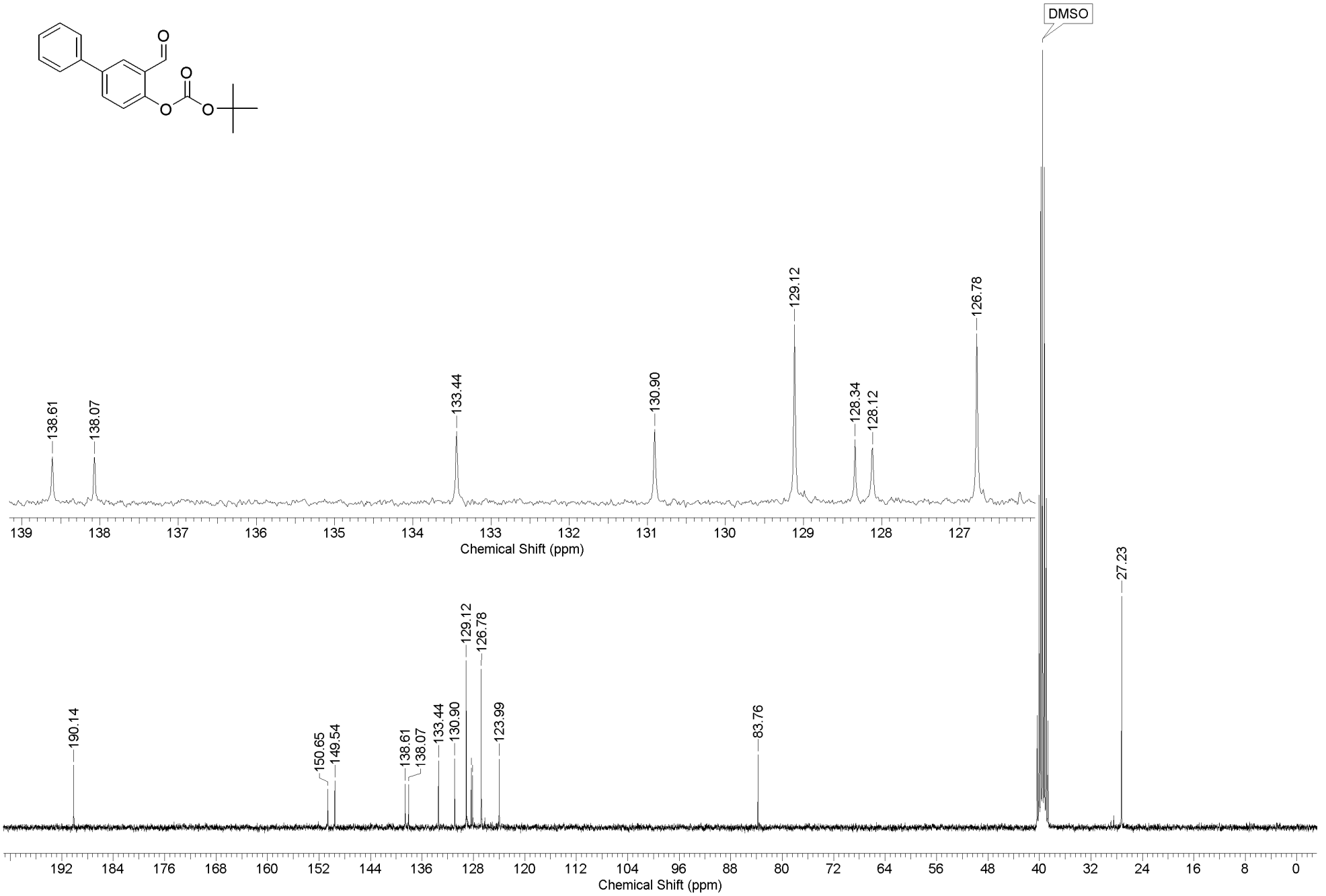
2.00
1.99
9.00
Chemical Shift (ppm)


DMSO
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138.07
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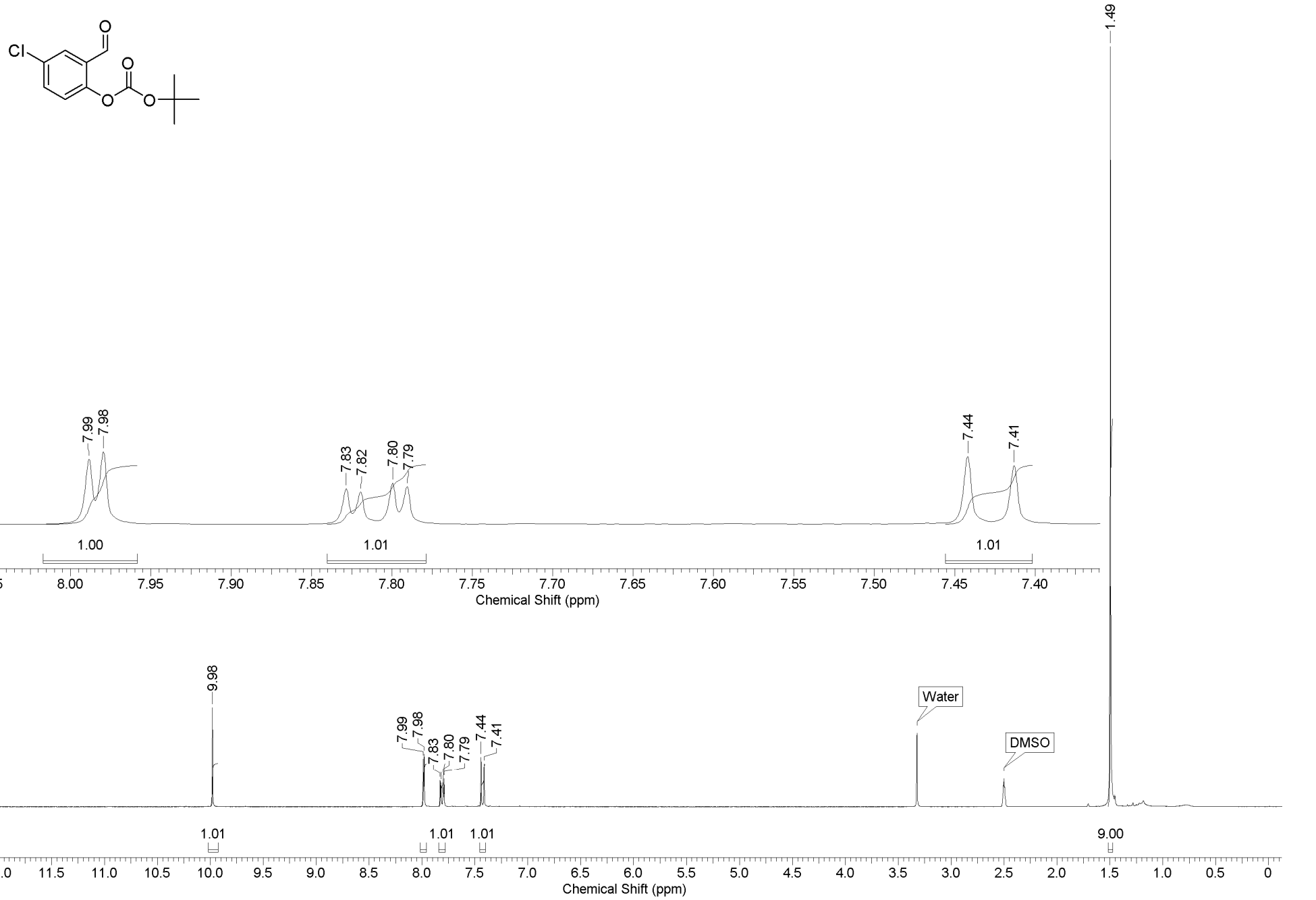
130.90
129.12
128.34
128.12
126.78
139
138
137
136
135
134
133
132
131
130
129
128
127
Chemical Shift (ppm)
27.23
190.14
150.65
149.54
138.61
138.07
133.44
130.90
129.12
126.78
123.99
83.76
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)


Cl
O
O
O
O
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7.99
7.98
7.83
7.82
7.80
7.79
7.44
7.41
1.00
1.01
1.01
8.05
8.00
7.95
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
Chemical Shift (ppm)
9.98
7.99
7.98
7.83
7.80
7.79
7.44
7.41
Water
DMSO
1.01
1.01
1.01
9.00
12.0
11.5
11.0
10.5
10.0
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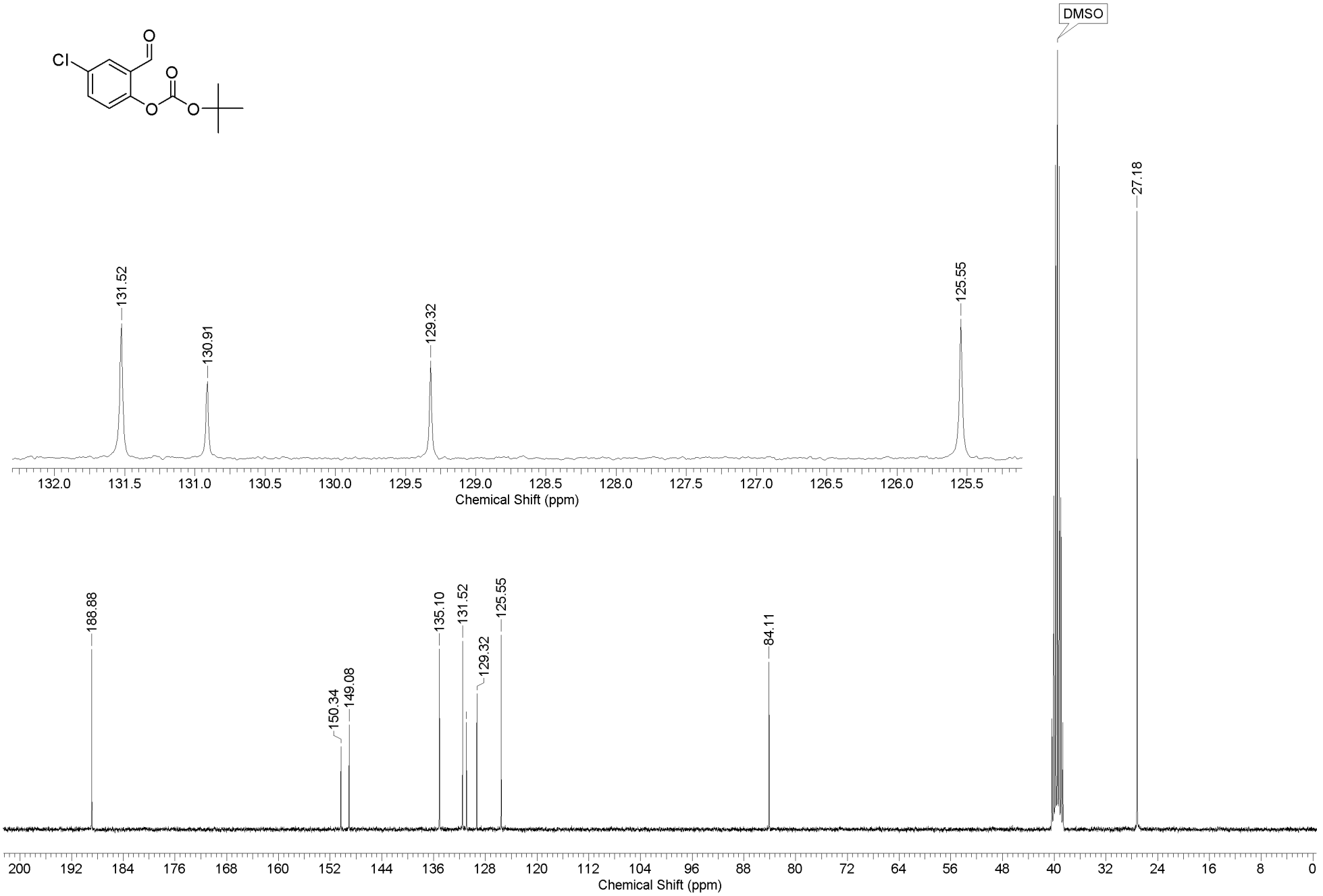
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)


Cl
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O
O
O
DMSO
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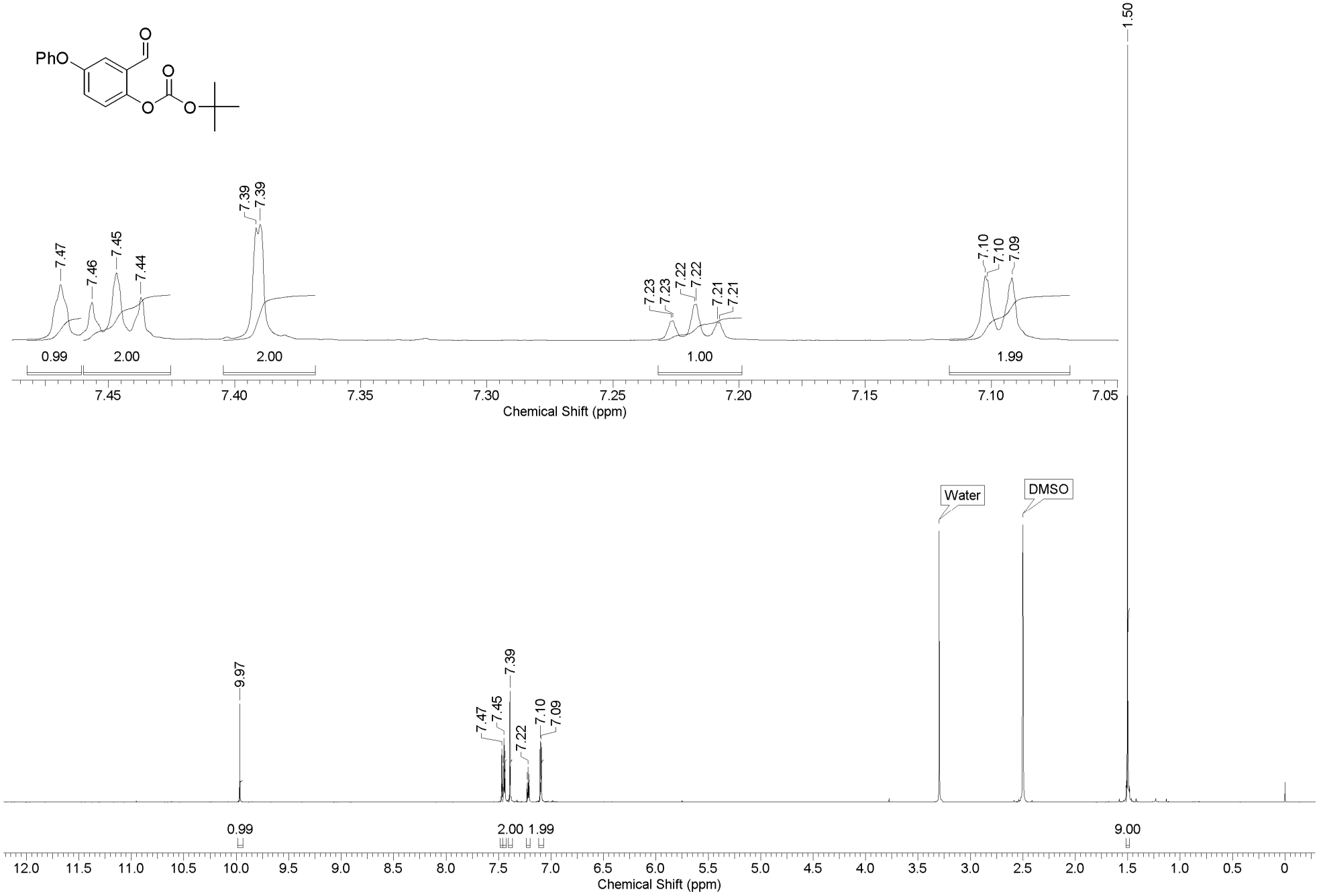
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
125.5
Chemical Shift (ppm)
188.88
150.34
149.08
135.10
131.52
129.32
125.55
84.11
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)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O
O
O
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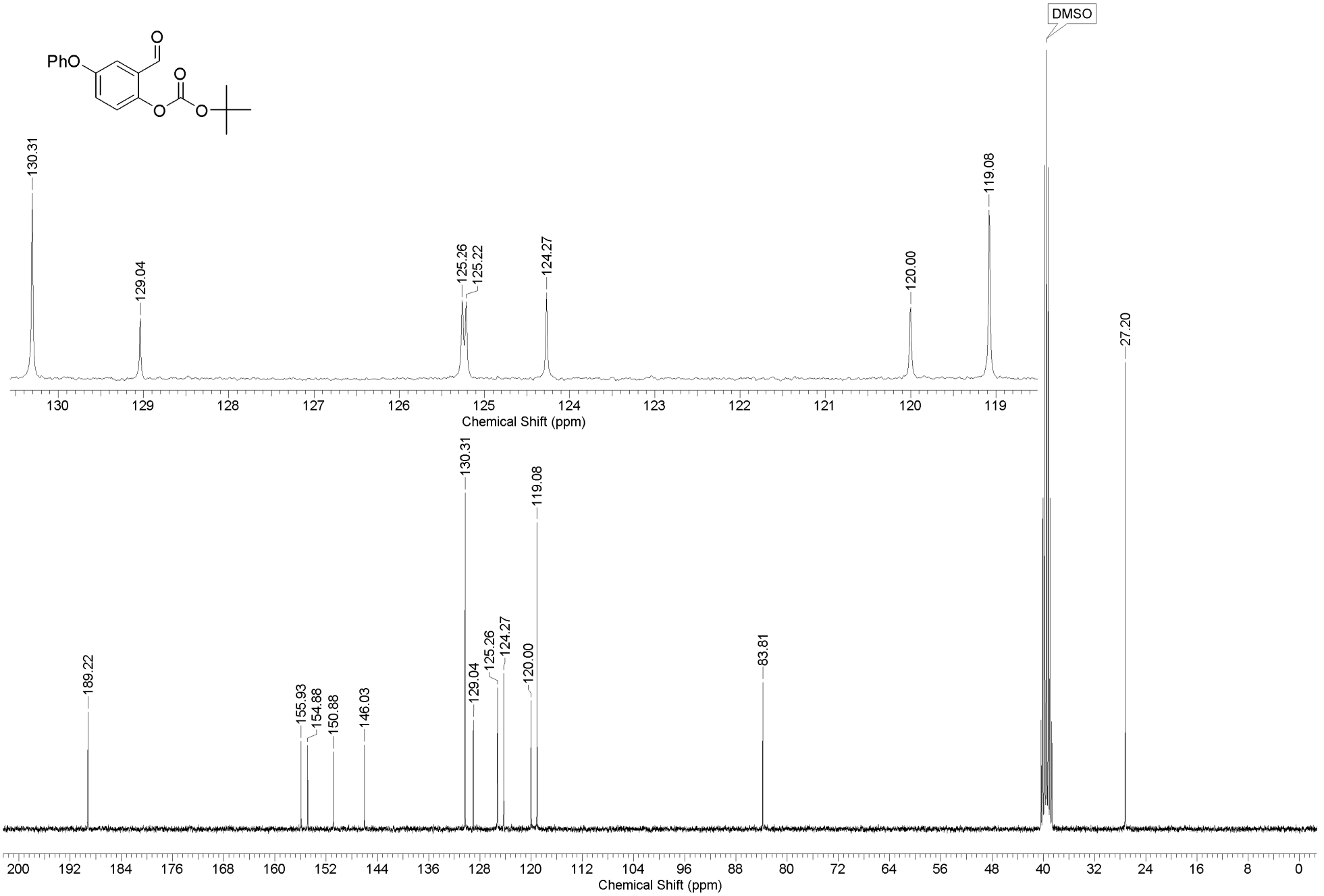
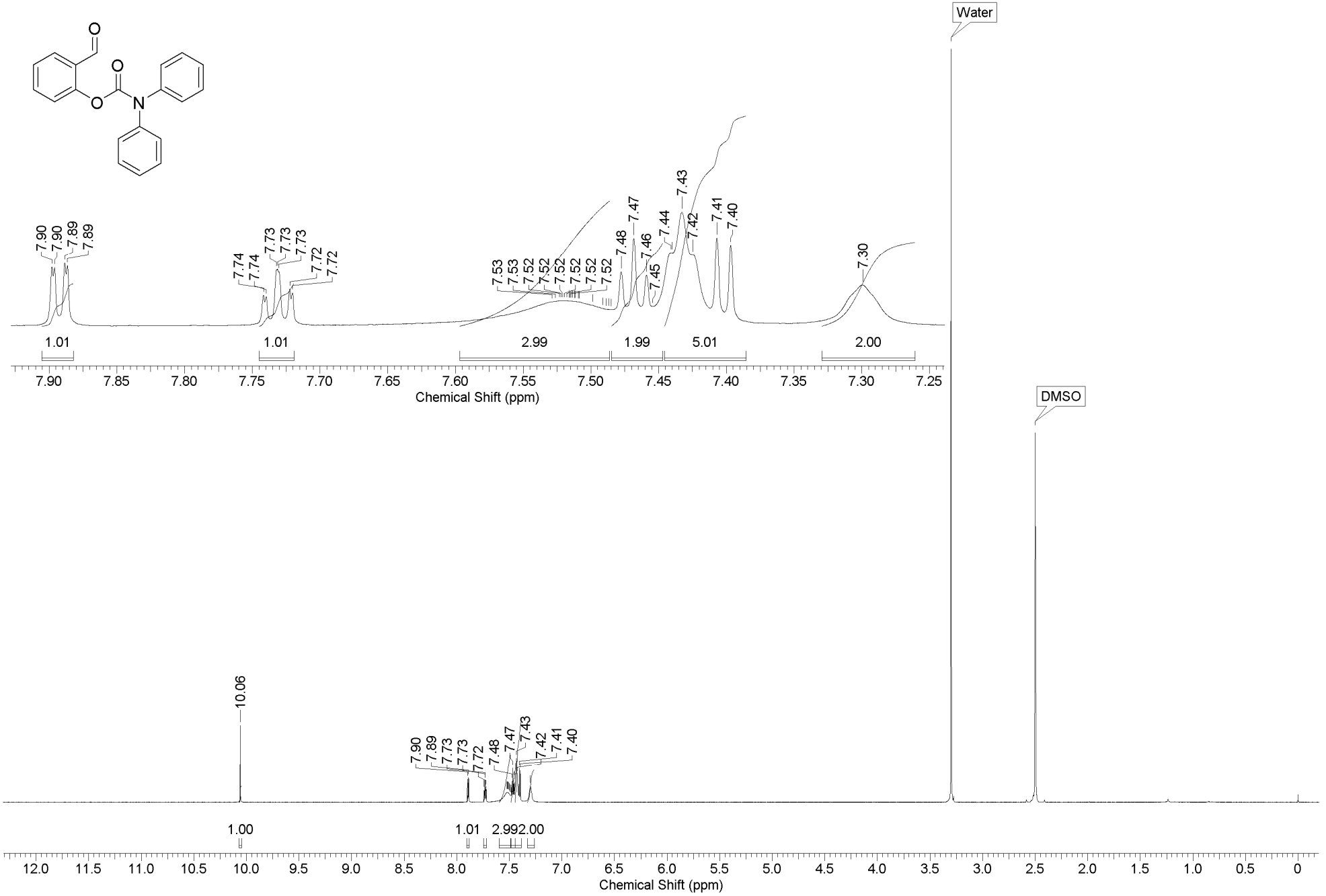
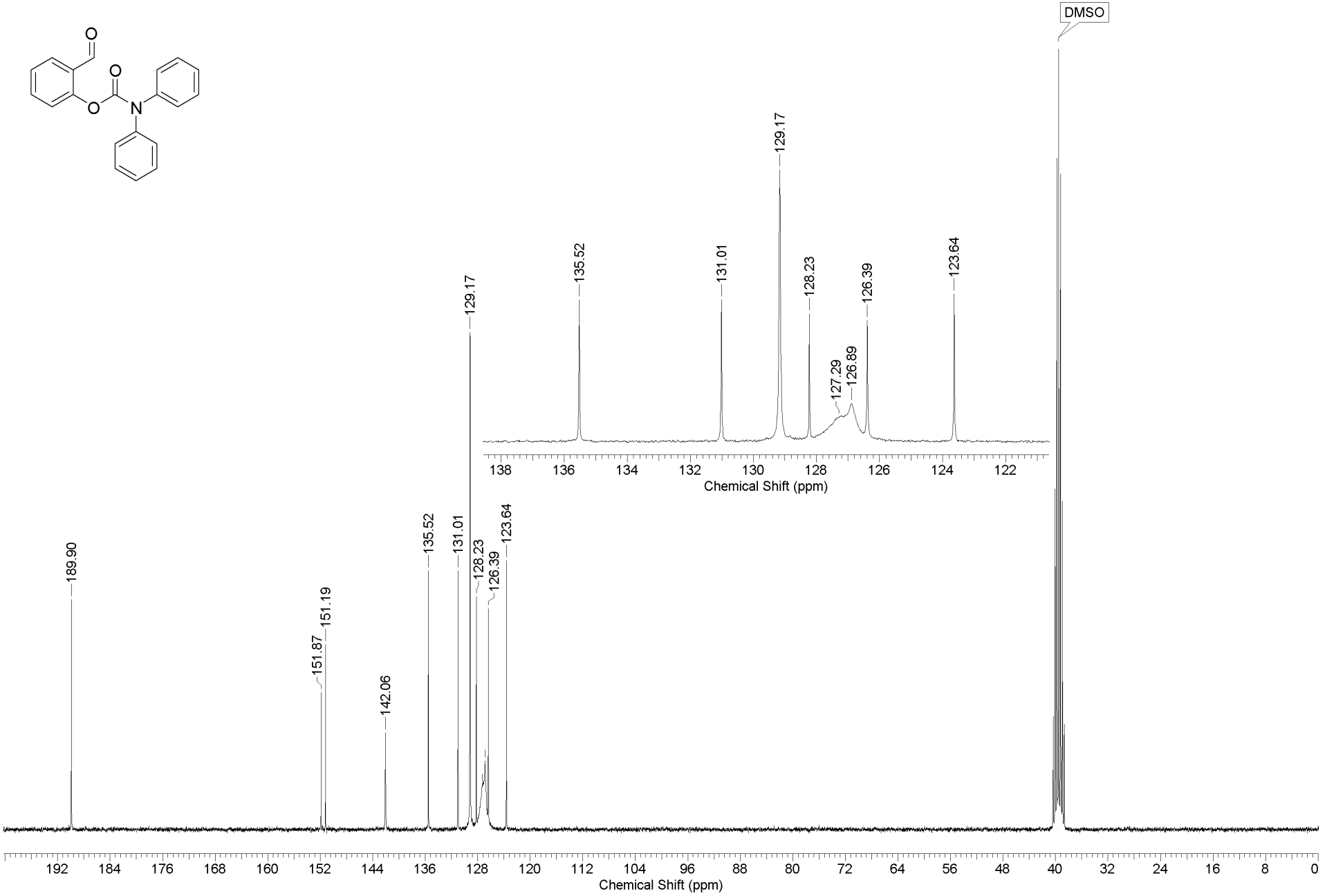
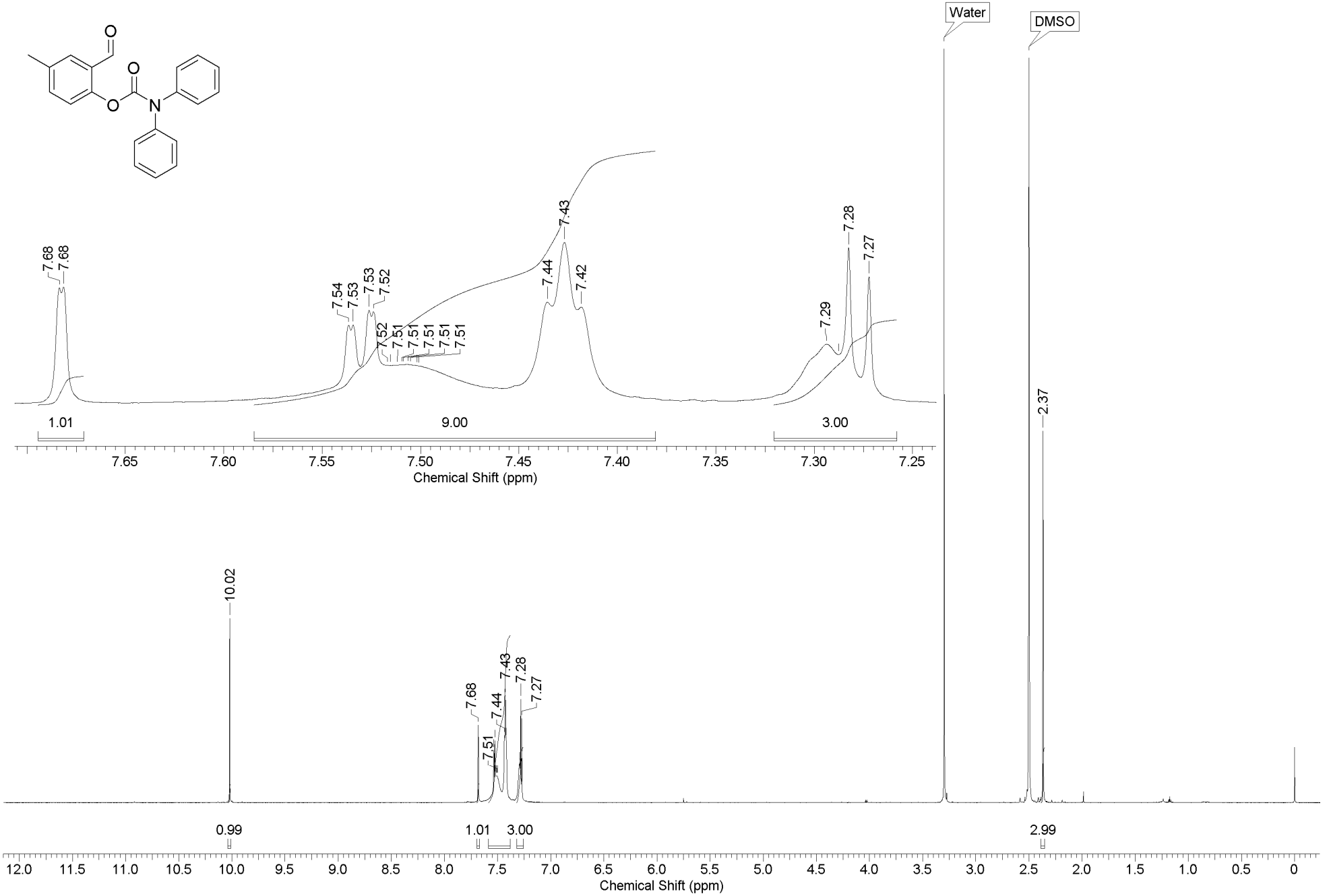
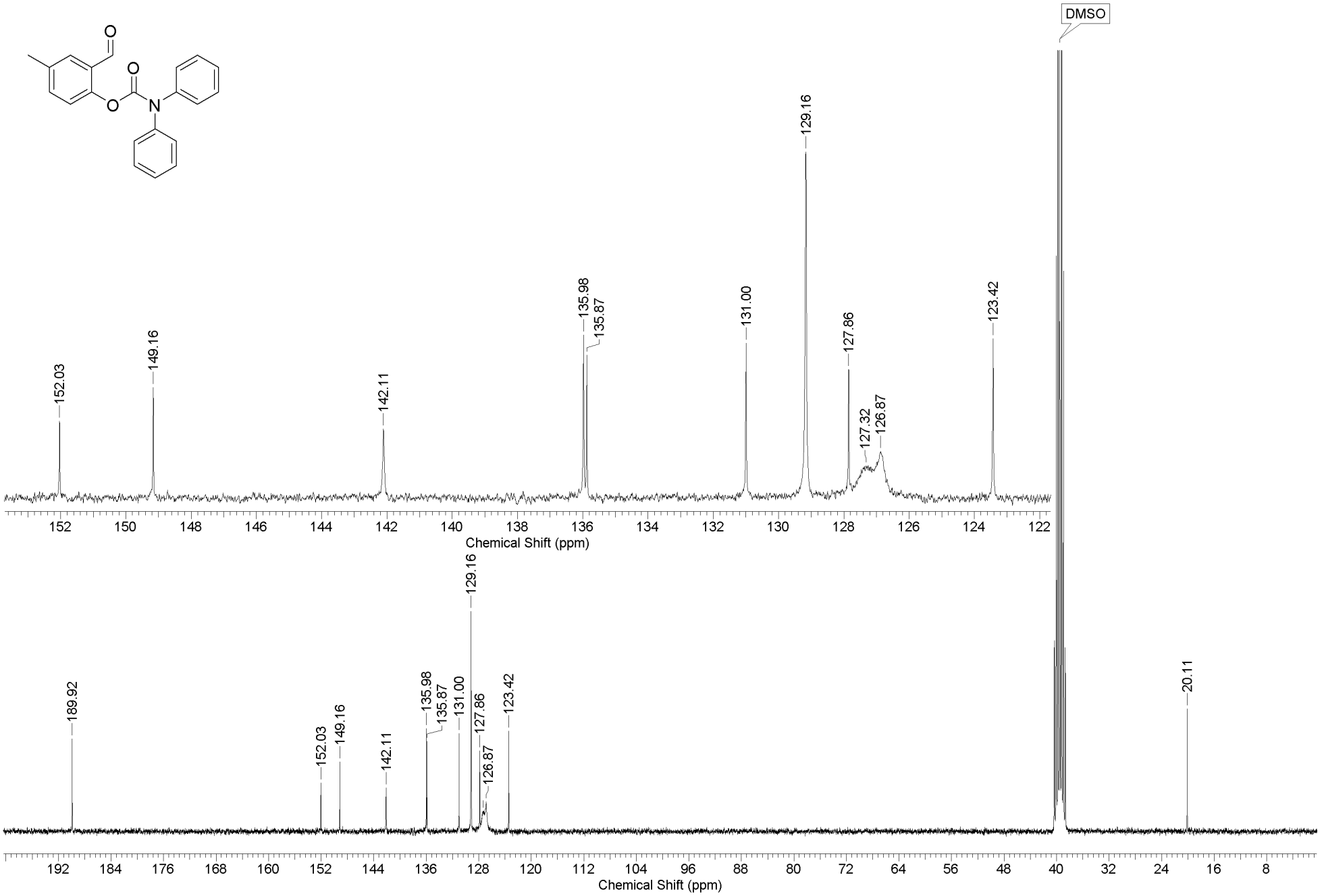
7.39
7.23
7.23
7.22
7.22
7.21
7.21
7.10
7.10
7.09
0.99
2.00
2.00
1.00
1.99
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
Chemical Shift (ppm)
1.50
Water
DMSO
9.97
7.47
7.45
7.39
7.22
7.10
7.09
0.99
2.00
1.99
9.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

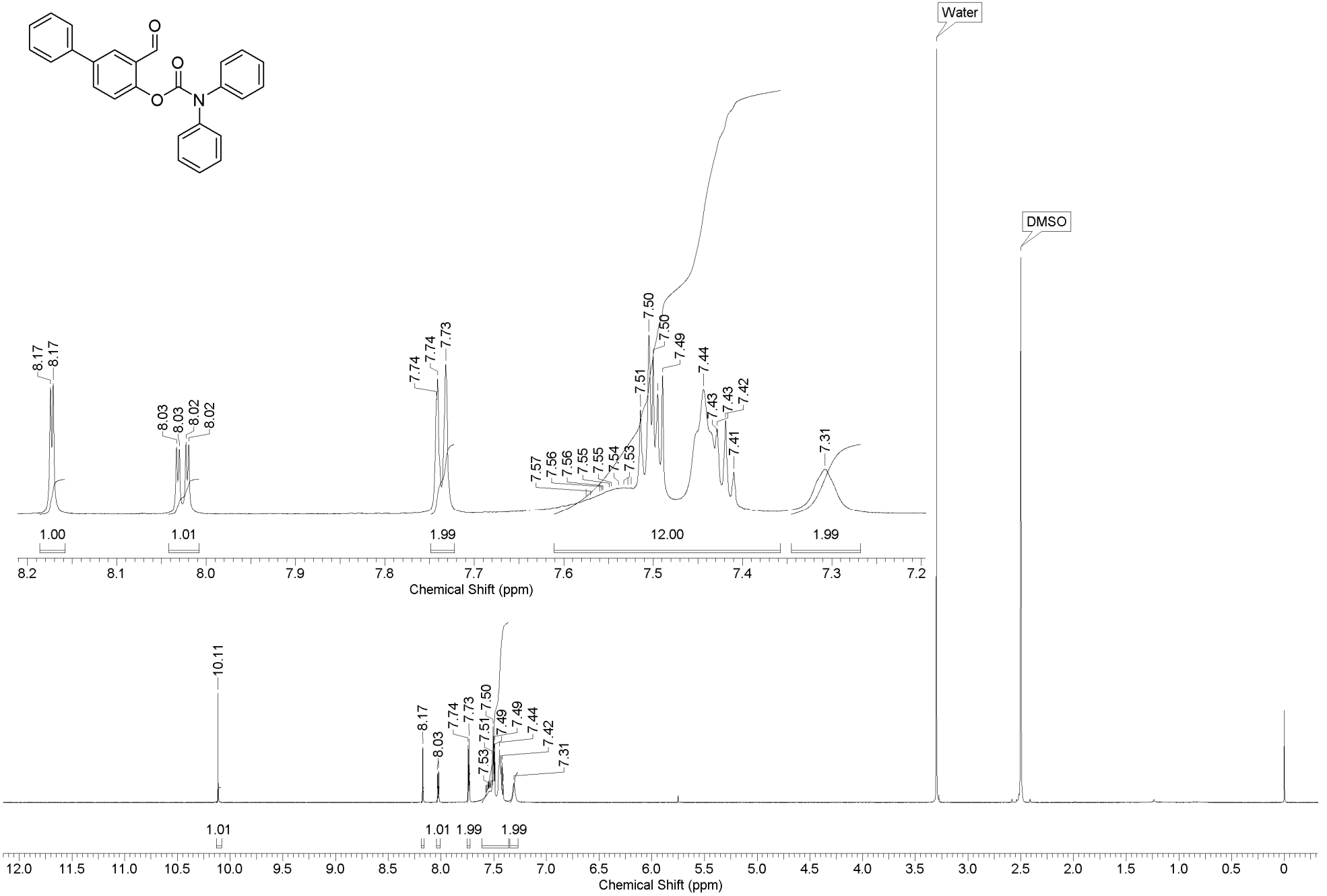

PhO
O
O
O
O
DMSO
130.31
129.04
125.26
125.22
124.27
120.00
119.08
27.20
Chemical Shift (ppm)
189.22
155.93
154.88
150.88
146.03
130.31
129.04
125.26
124.27
120.00
119.08
83.81
27.20
Chemical Shift (ppm)

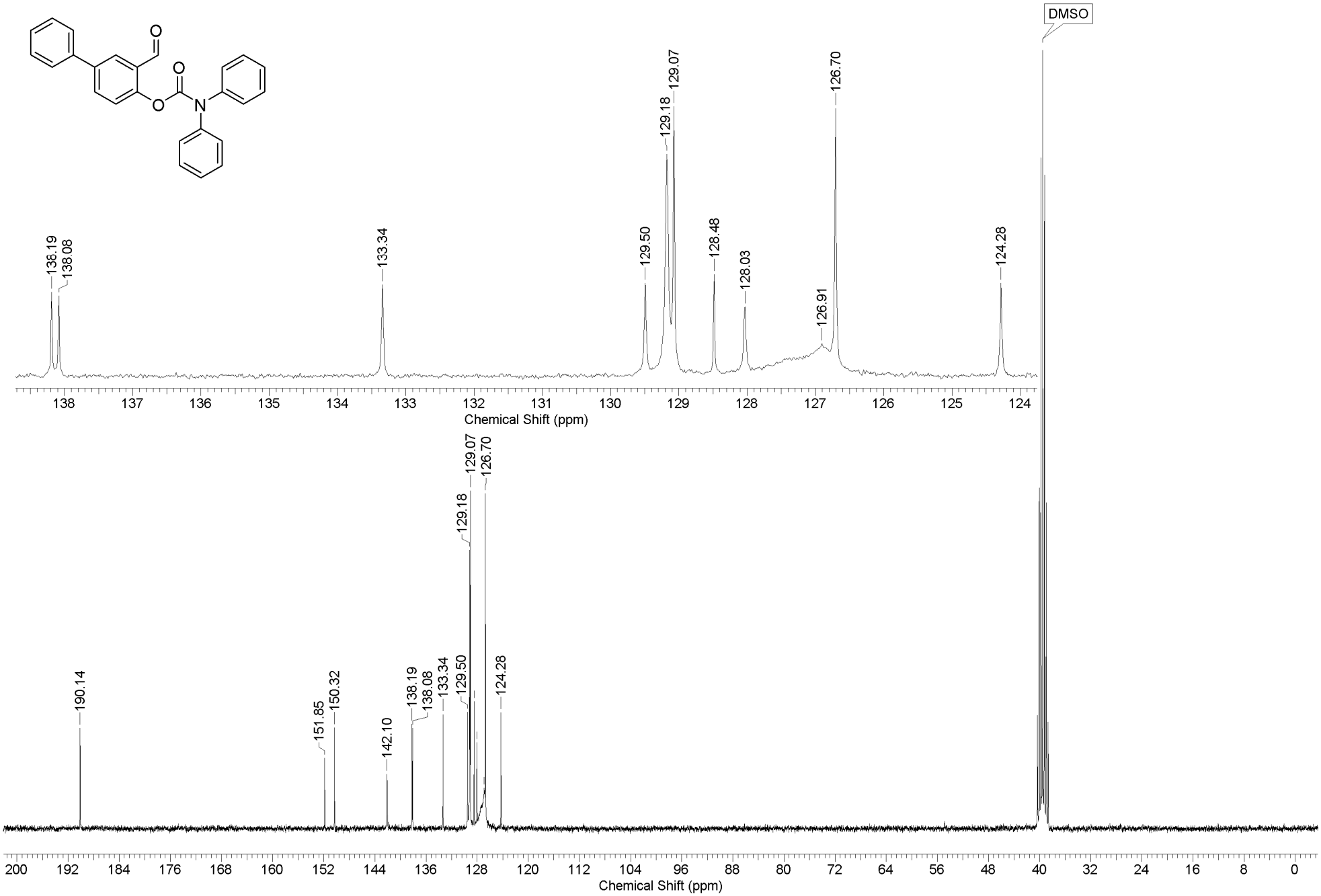

Water
DMSO
7.90
7.90
7.89
7.89
7.74
7.74
7.73
7.73
7.73
7.72
7.72
7.53
7.53
7.52
7.52
7.52
7.52
7.52
7.52
7.48
7.47
7.46
7.45
7.44
7.43
7.42
7.41
7.40
7.30
1.01
1.01
2.99
1.99
5.01
2.00
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
10.06
7.90
7.89
7.73
7.73
7.72
7.48
7.47
7.43
7.42
7.41
7.40
1.00
1.01
2.99
2.00
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

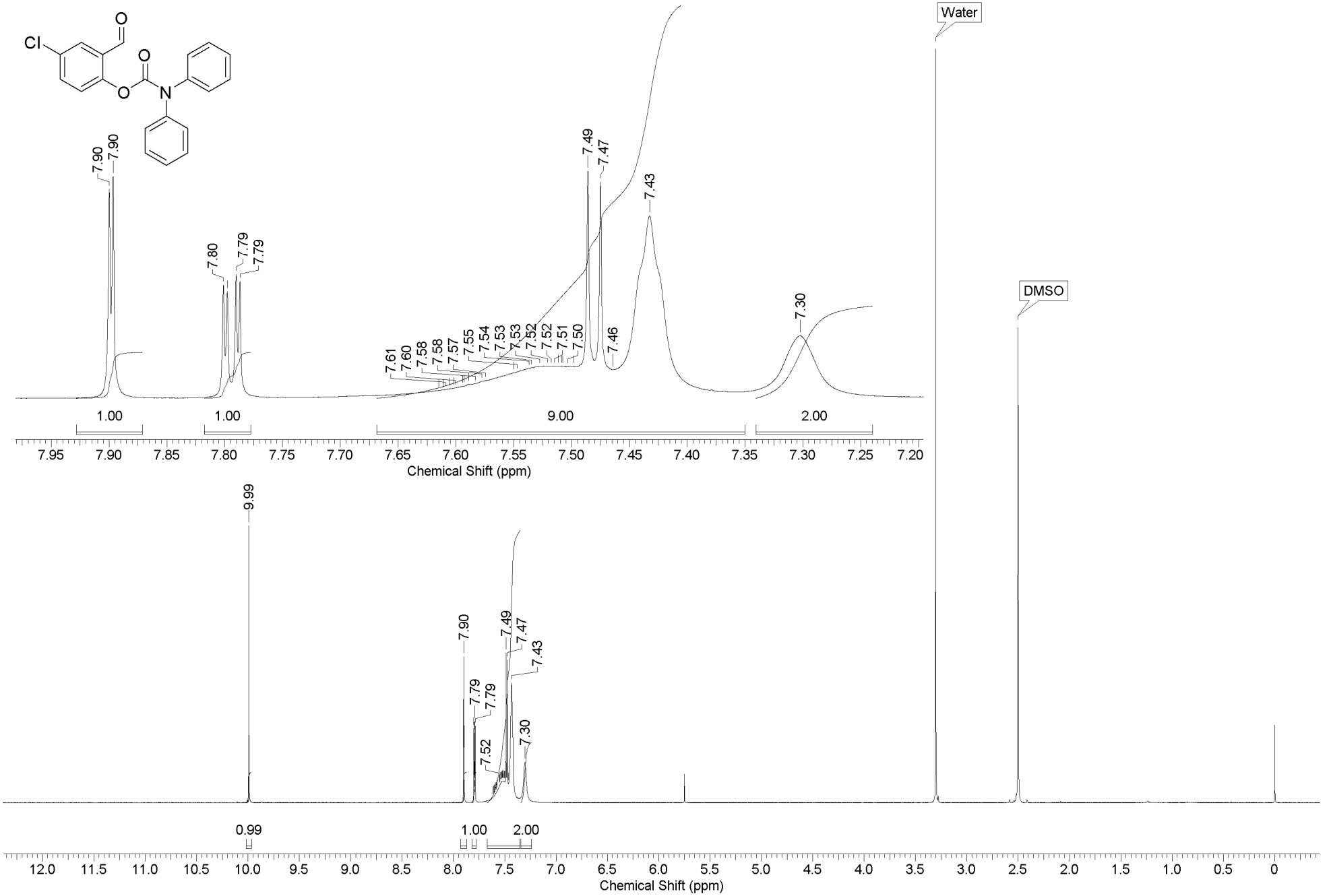

DMSO
189.90
151.87
151.19
142.06
135.52
131.01
129.17
128.23
126.39
123.64
127.29
126.89
Chemical Shift (ppm)
Chemical Shift (ppm)

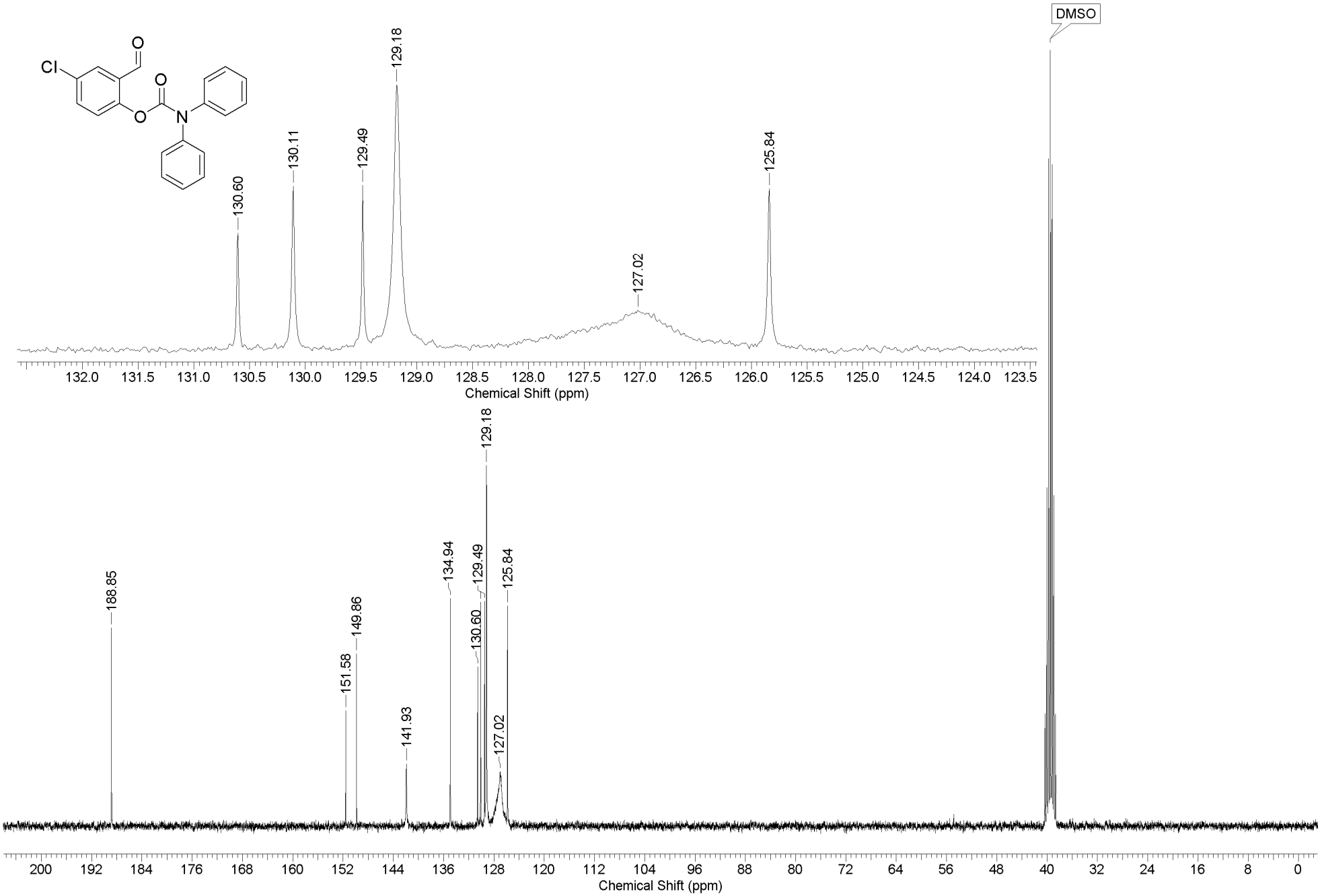

Water
DMSO
10.02
7.68
7.68
7.54
7.53
7.53
7.52
7.52
7.51
7.51
7.51
7.51
7.51
7.44
7.43
7.42
7.29
7.28
7.27
2.37
1.01
9.00
3.00
0.99
1.01
3.00
2.99
Chemical Shift (ppm)
Chemical Shift (ppm)

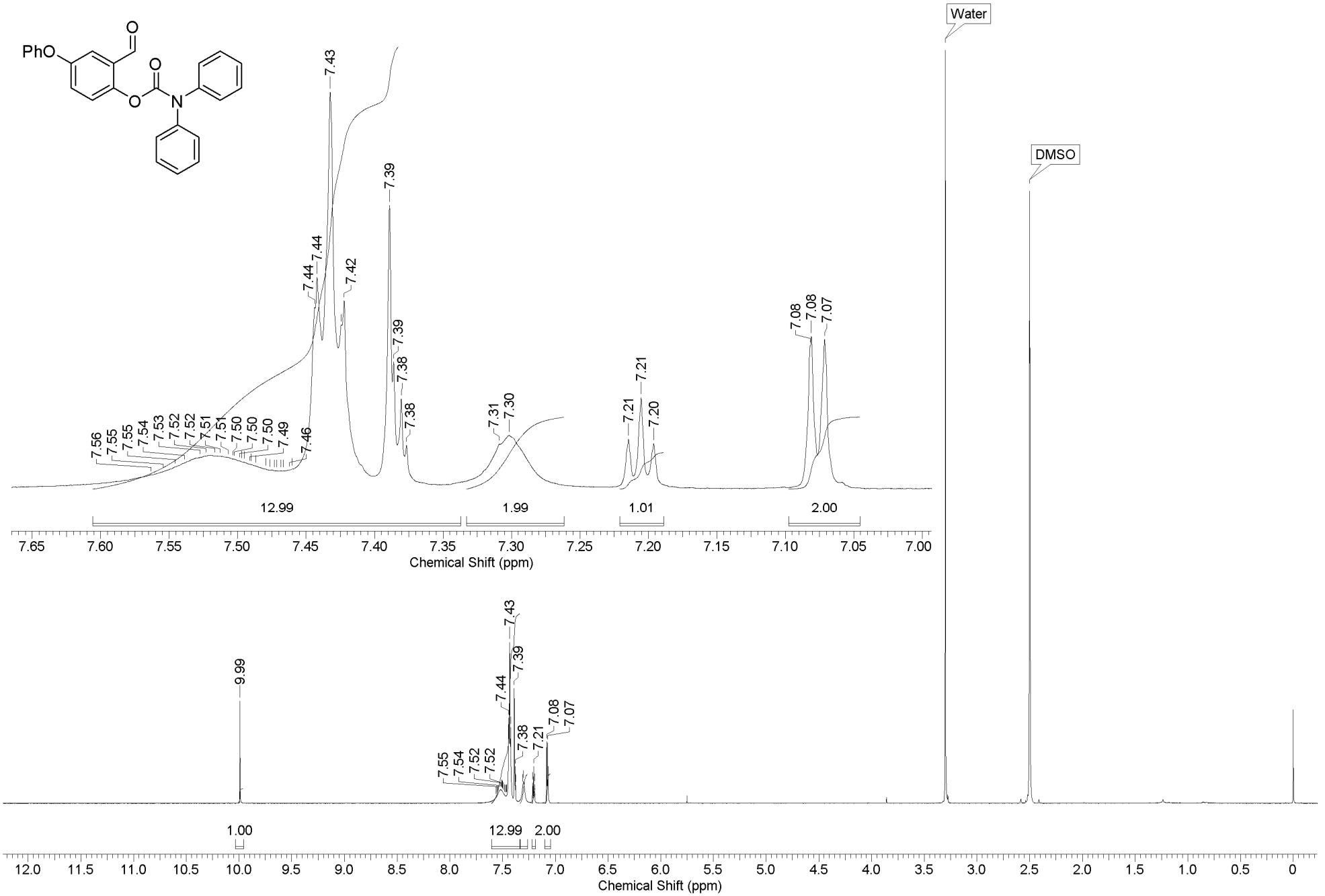

O
O
O
N
DMSO
152.03
149.16
142.11
135.98
135.87
131.00
129.16
127.86
127.32
126.87
123.42
Chemical Shift (ppm)
189.92
152.03
149.16
142.11
135.98
135.87
131.00
129.16
127.86
126.87
123.42
20.11
Chemical Shift (ppm)

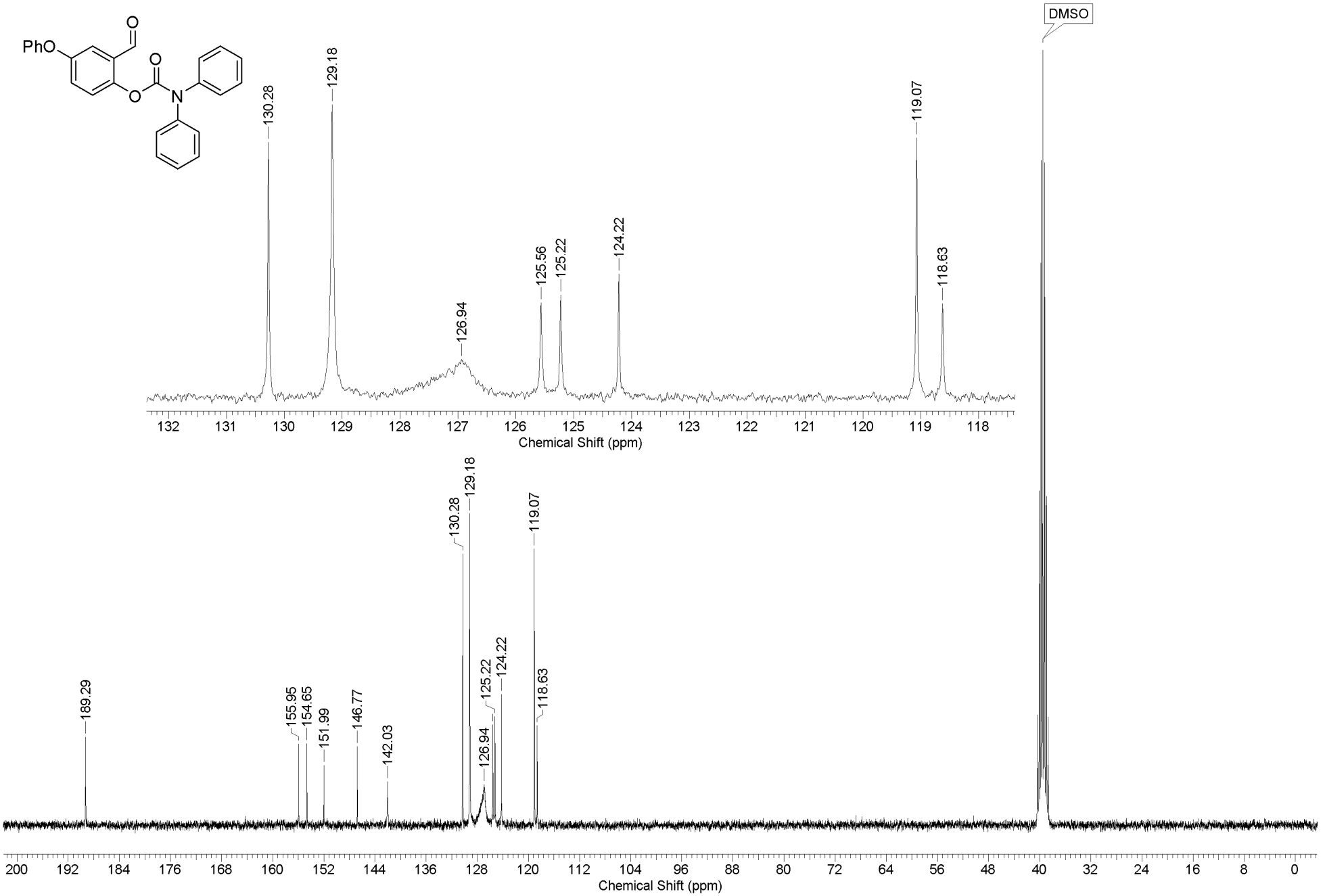

Water
DMSO
8.17
8.17
8.03
8.03
8.02
8.02
7.74
7.74
7.73
7.57
7.56
7.56
7.55
7.55
7.54
7.53
7.50
7.50
7.49
7.51
7.44
7.43
7.43
7.42
7.41
7.31
1.00
1.01
1.99
12.00
1.99
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
Chemical Shift (ppm)
10.11
8.17
8.03
7.74
7.73
7.53
7.51
7.50
7.49
7.49
7.44
7.42
7.31
1.01
1.01
1.99
1.99
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

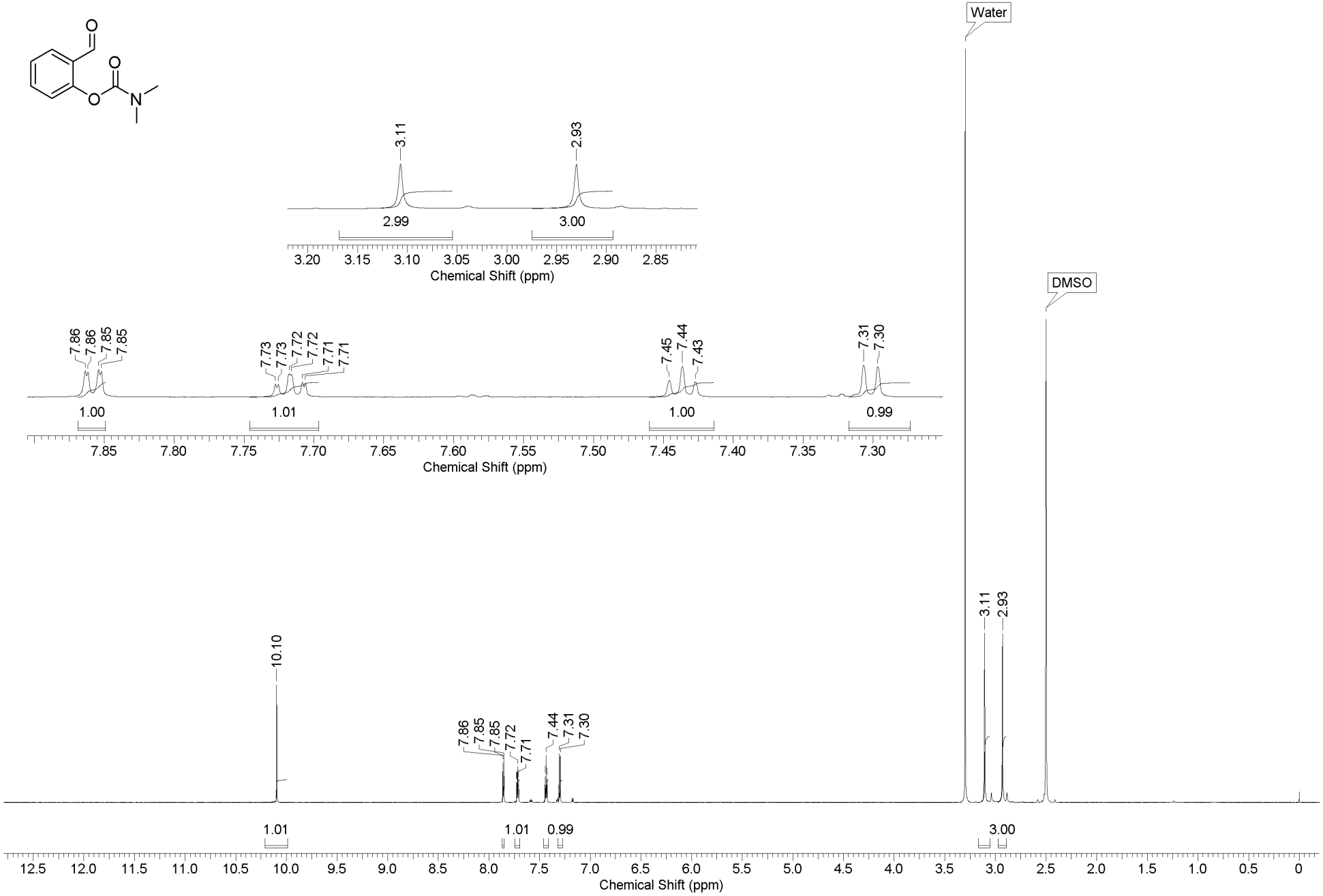

DMSO
138.19
138.08
133.34
129.50
129.18
129.07
128.48
128.03
126.91
126.70
124.28
Chemical Shift (ppm)
190.14
151.85
150.32
142.10
138.19
138.08
133.34
129.50
129.18
129.07
126.70
124.28
Chemical Shift (ppm)

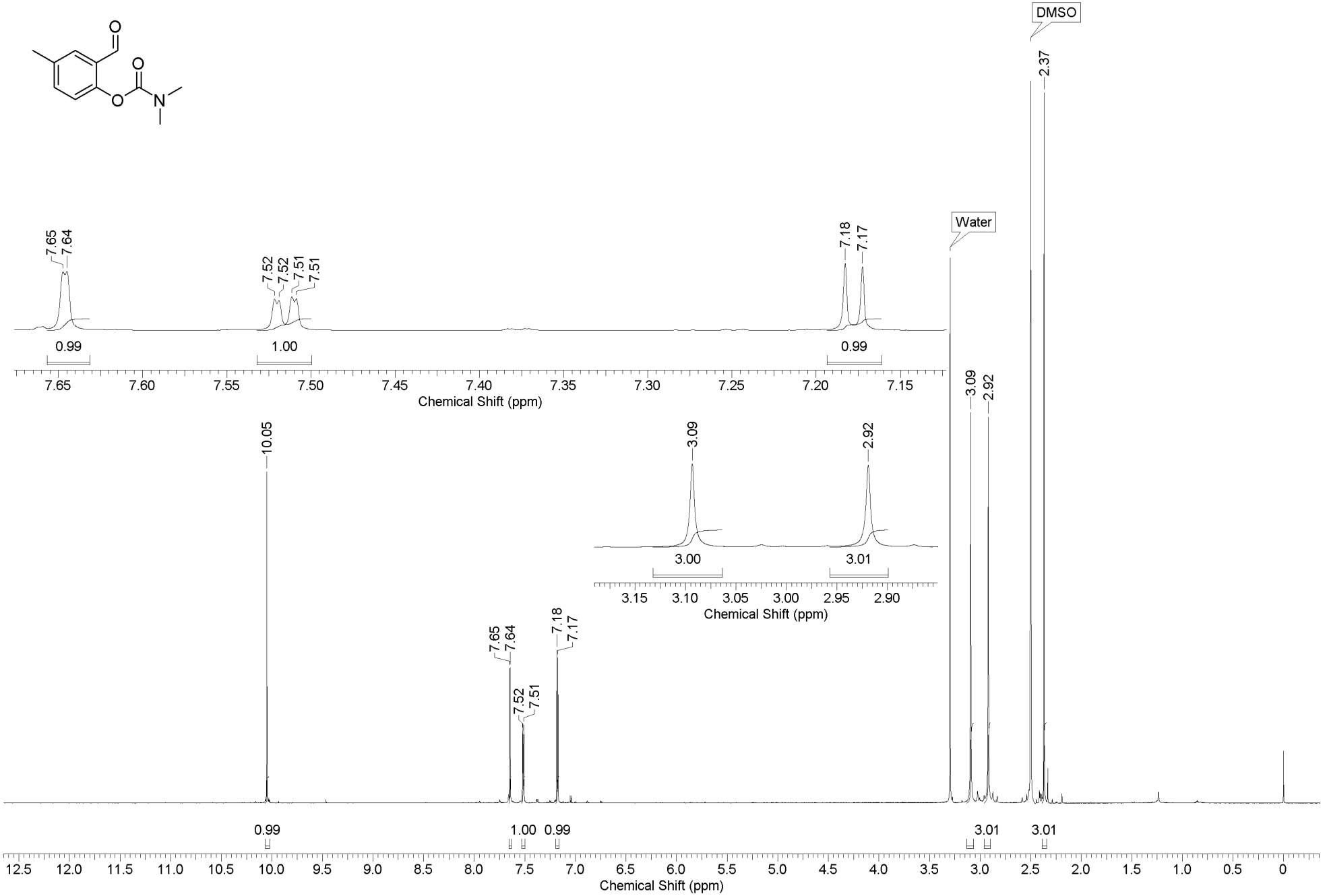

Cl
O
O
O
N
Water
DMSO
7.90
7.90
7.80
7.79
7.79
7.61
7.60
7.58
7.58
7.57
7.55
7.54
7.53
7.53
7.52
7.52
7.51
7.50
7.49
7.47
7.46
7.43
7.30
1.00
1.00
9.00
2.00
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20
Chemical Shift (ppm)
9.99
7.90
7.79
7.79
7.52
7.49
7.47
7.43
7.30
0.99
1.00
2.00
12.0 11.5 11.0 10.5 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
Chemical Shift (ppm)

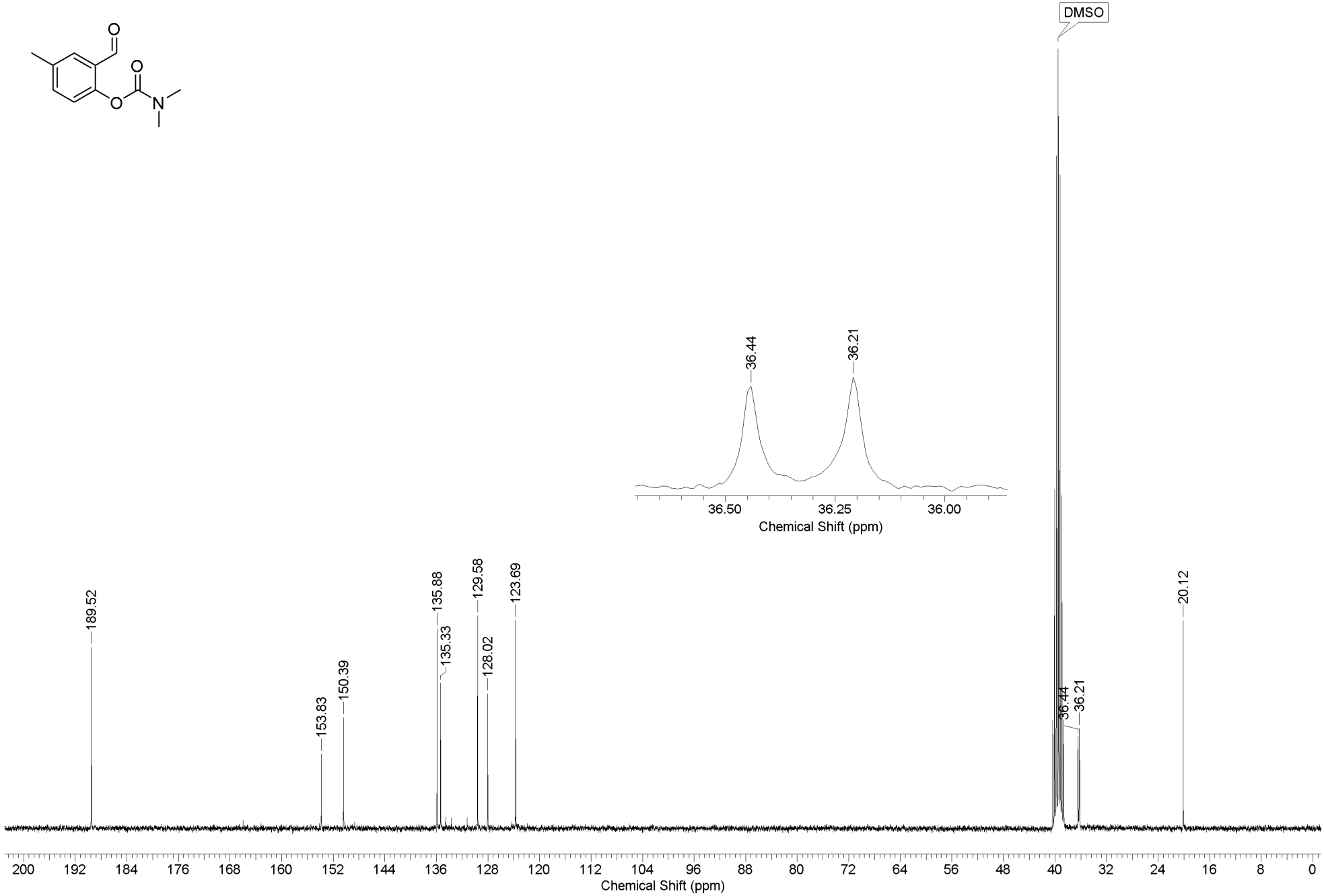

Cl
O
O
O
N
DMSO
129.18
130.11
129.49
130.60
125.84
127.02
132.0
131.5
131.0
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
125.5
125.0
124.5
124.0
123.5
Chemical Shift (ppm)
129.18
188.85
149.86
151.58
134.94
129.49
130.60
125.84
141.93
127.02
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

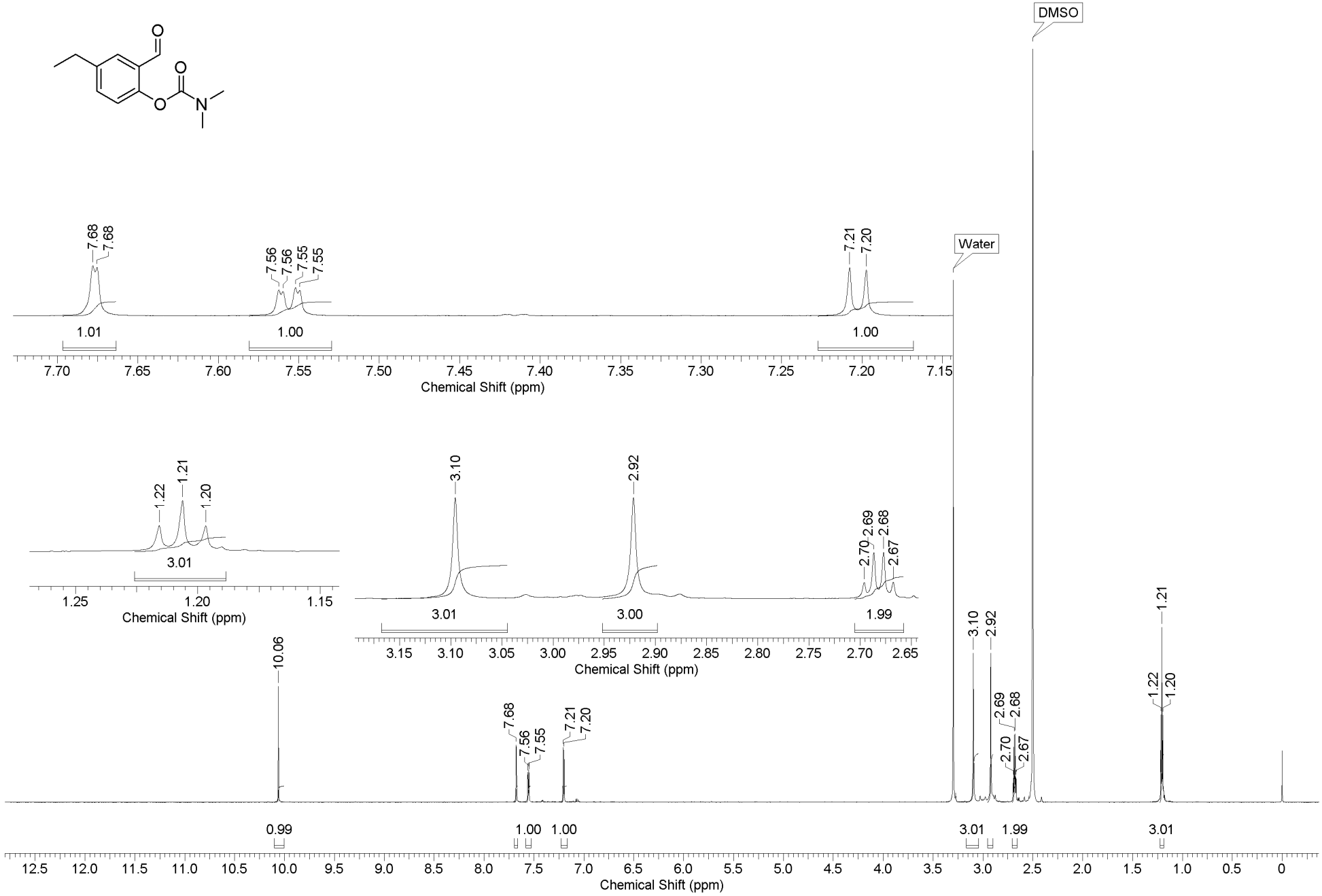

PhO
O
O
O
N
Water
DMSO
7.43
7.44
7.44
7.42
7.39
7.39
7.38
7.38
7.56
7.55
7.55
7.54
7.53
7.52
7.52
7.51
7.51
7.50
7.50
7.50
7.49
7.46
7.31
7.30
7.21
7.21
7.20
7.08
7.08
7.07
12.99
1.99
1.01
2.00
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
7.00
Chemical Shift (ppm)
9.99
7.43
7.39
7.44
7.08
7.07
7.38
7.21
7.55
7.54
7.52
7.52
1.00
12.99
2.00
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

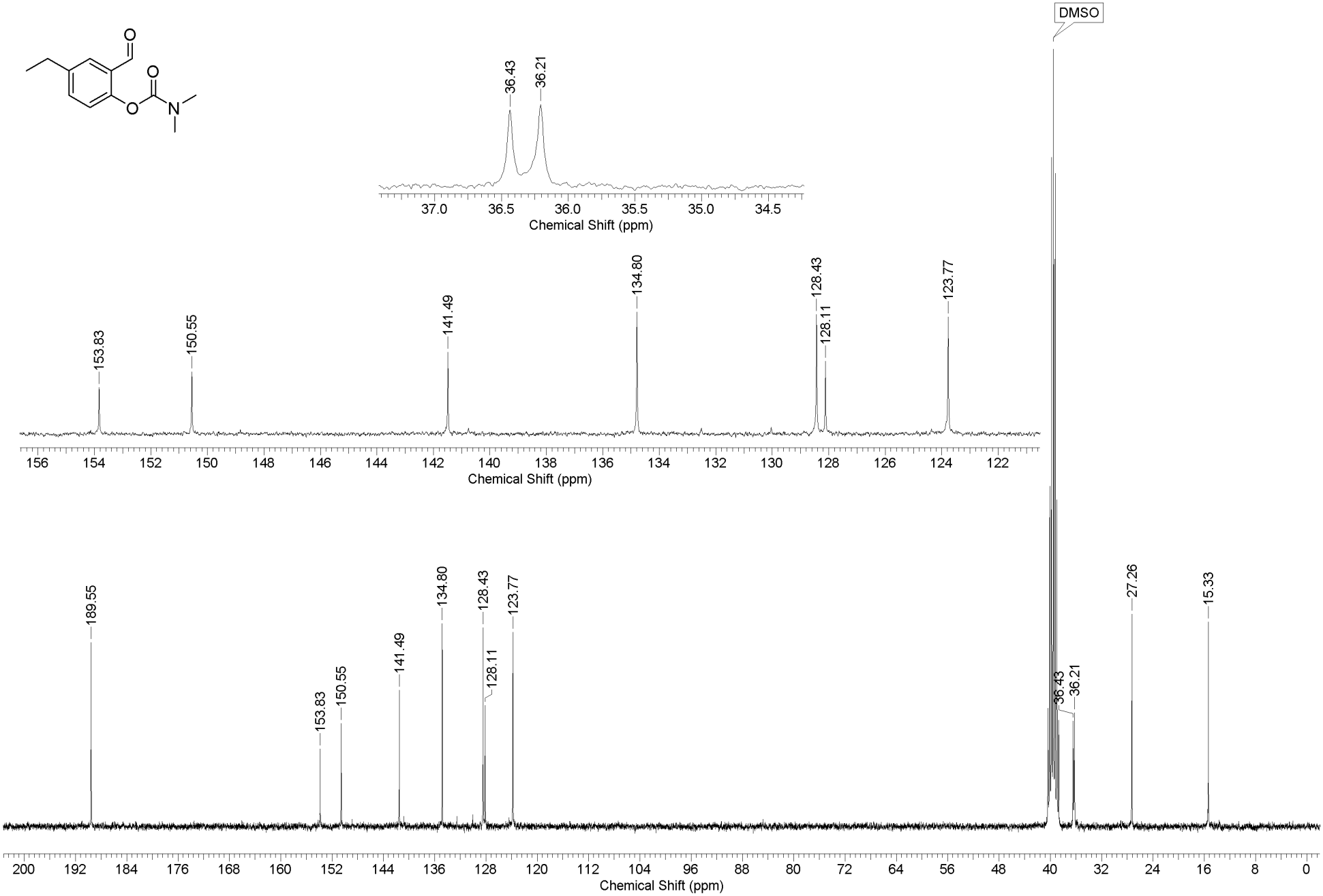

PhO
O
O
O
N
DMSO
130.28
129.18
126.94
125.56
125.22
124.22
119.07
118.63
Chemical Shift (ppm)
189.29
155.95
154.65
151.99
146.77
142.03
130.28
129.18
126.94
125.22
124.22
119.07
118.63
DMSO
Chemical Shift (ppm)

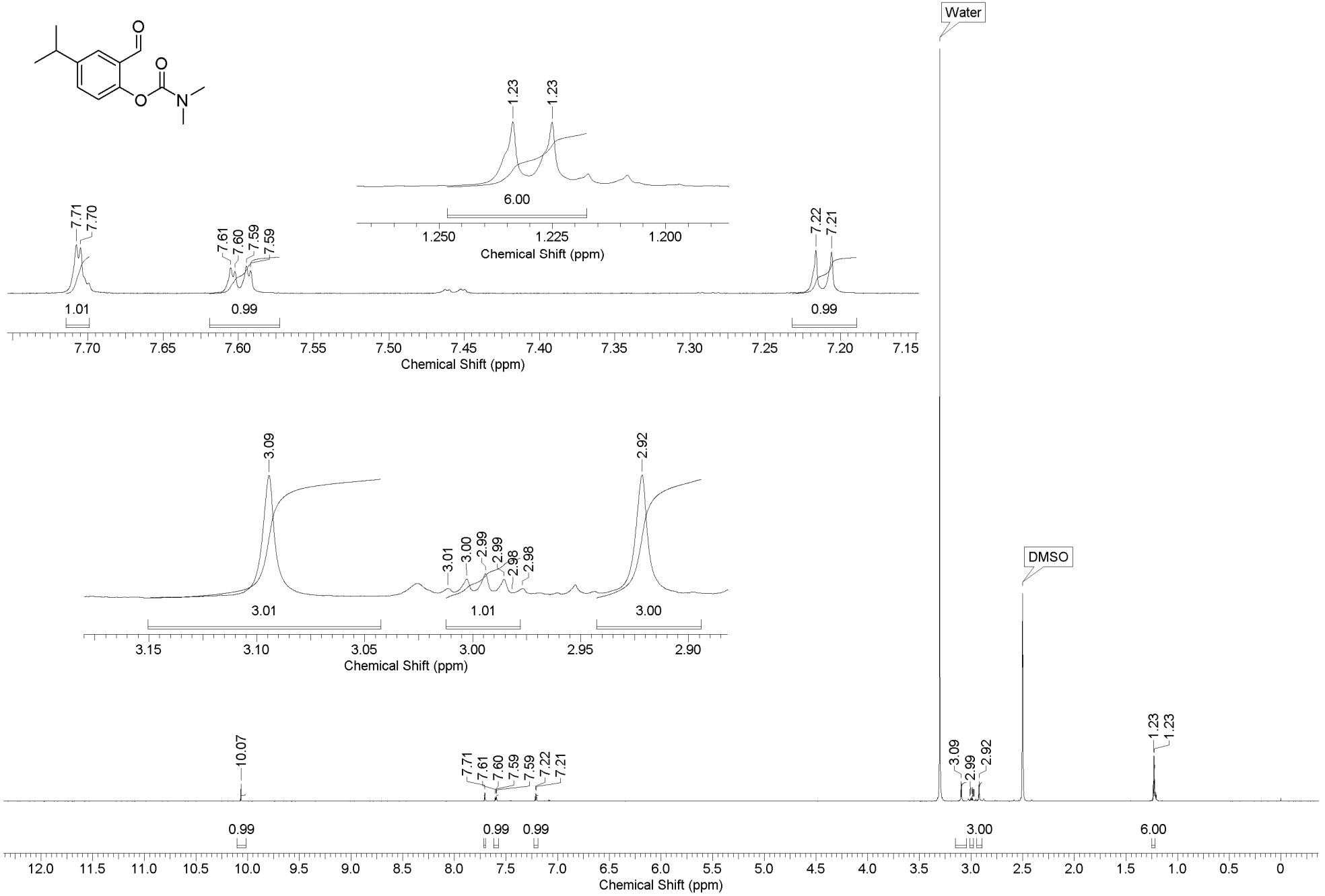

Water
DMSO
3.11
2.93
2.99
3.00
Chemical Shift (ppm)
7.86
7.85
7.73
7.72
7.71
7.45
7.44
7.43
7.31
7.30
1.00
1.01
1.00
0.99
Chemical Shift (ppm)
10.10
1.01
1.01
0.99
3.00
Chemical Shift (ppm)

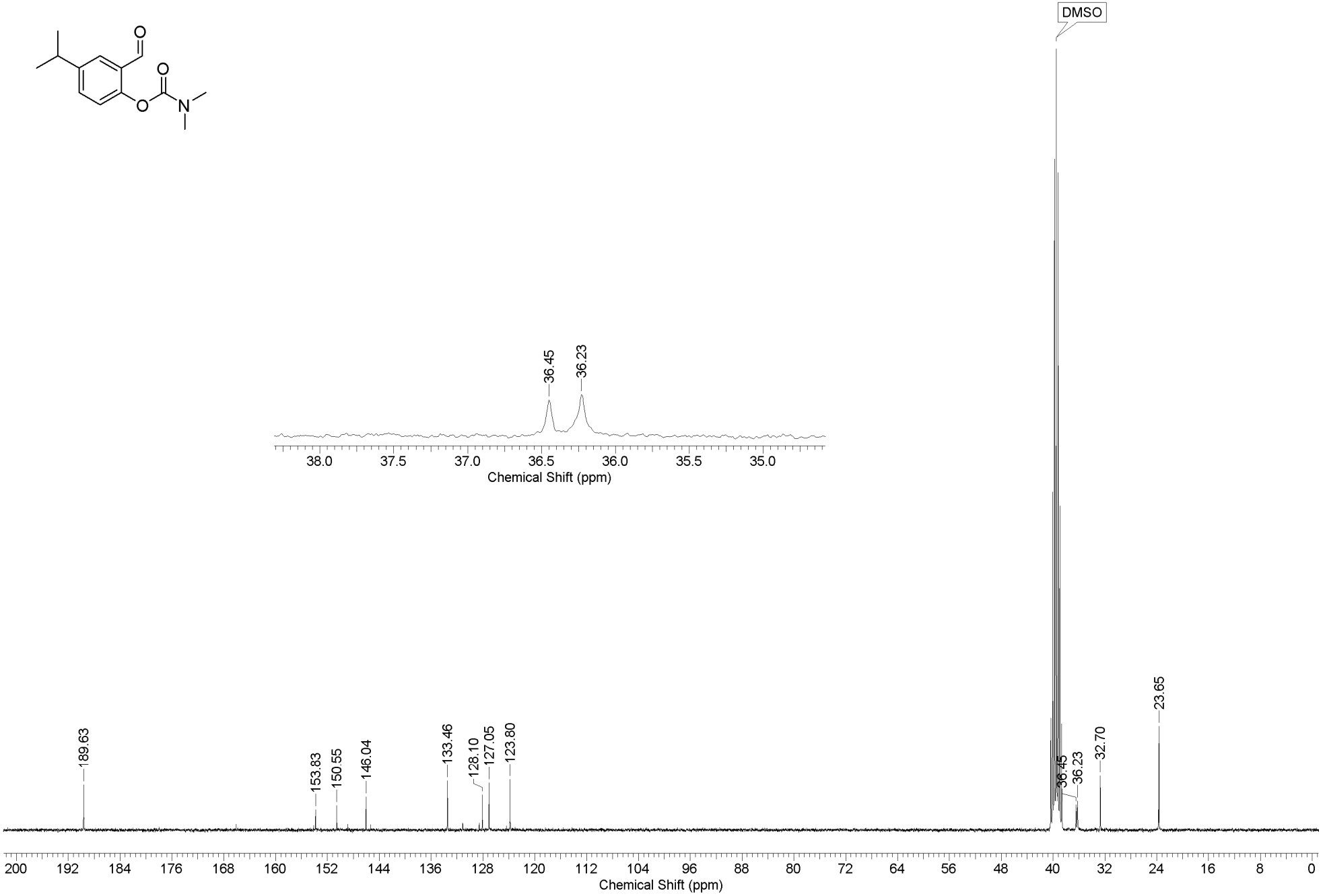

DMSO
2.37
Water
7.65
7.64
7.52
7.52
7.51
7.51
7.18
7.17
0.99
1.00
0.99
7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15
Chemical Shift (ppm)
3.09
2.92
10.05
3.00
3.01
3.15 3.10 3.05 3.00 2.95 2.90
Chemical Shift (ppm)
0.99
1.00
0.99
3.01
3.01
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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
Chemical Shift (ppm)

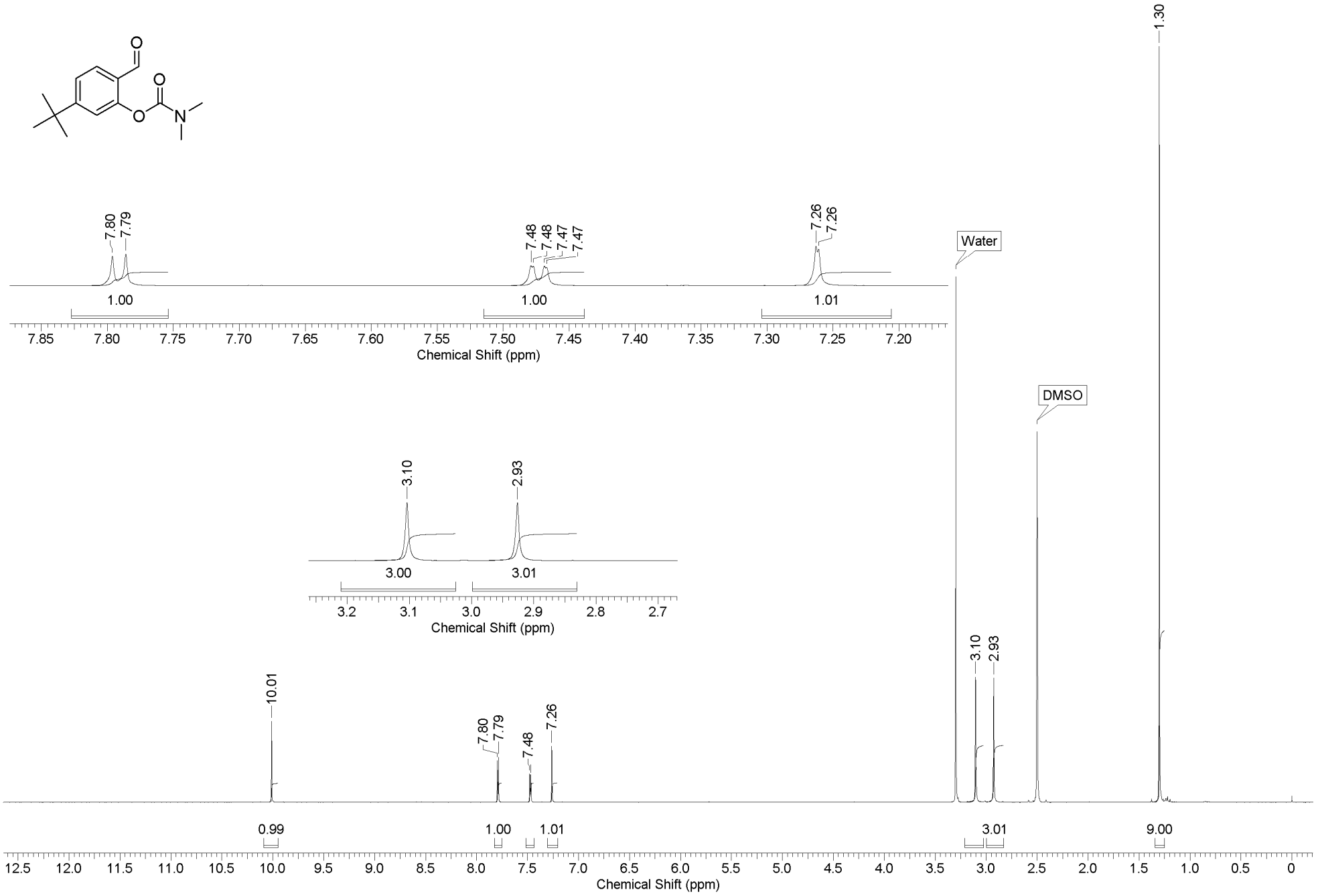

O
O
O
N
DMSO
36.44
36.21
36.50
36.25
36.00
Chemical Shift (ppm)
189.52
153.83
150.39
135.88
135.33
129.58
128.02
123.69
20.12
36.44
36.21
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

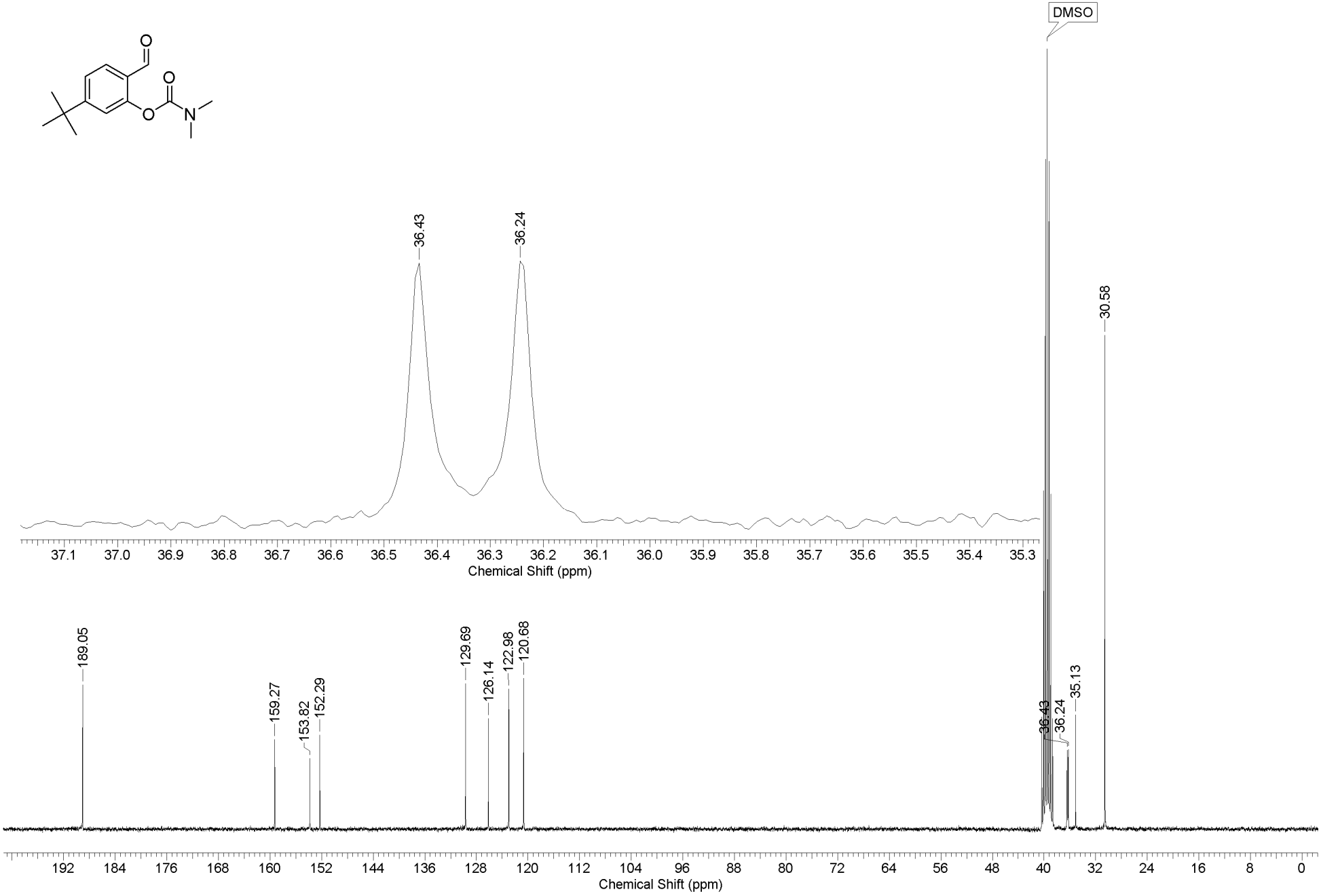

DMSO
Water
7.68
7.68
7.56
7.56
7.55
7.55
7.21
7.20
1.01
1.00
1.00
Chemical Shift (ppm)
1.22
1.21
1.20
3.01
Chemical Shift (ppm)
3.10
2.92
2.70
2.69
2.68
2.67
3.01
3.00
1.99
Chemical Shift (ppm)
10.06
0.99
1.00
1.00
3.01
1.99
3.01
Chemical Shift (ppm)

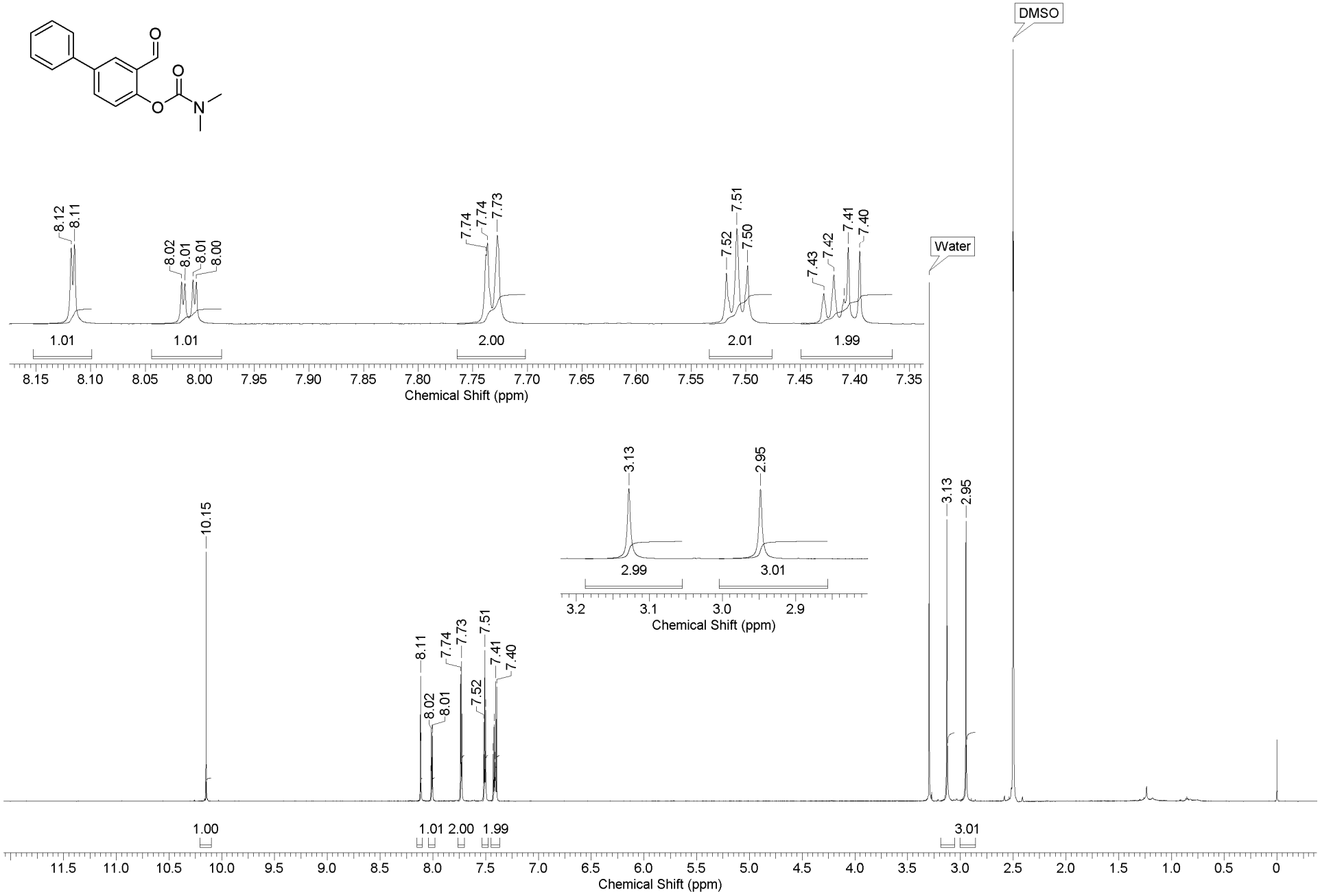

O
O
O
N
DMSO
36.43
36.21
37.0
36.5
36.0
35.5
35.0
34.5
Chemical Shift (ppm)
153.83
150.55
141.49
134.80
128.43
128.11
123.77
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
Chemical Shift (ppm)
189.55
153.83
150.55
141.49
134.80
128.43
128.11
123.77
36.43
36.21
27.26
15.33
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

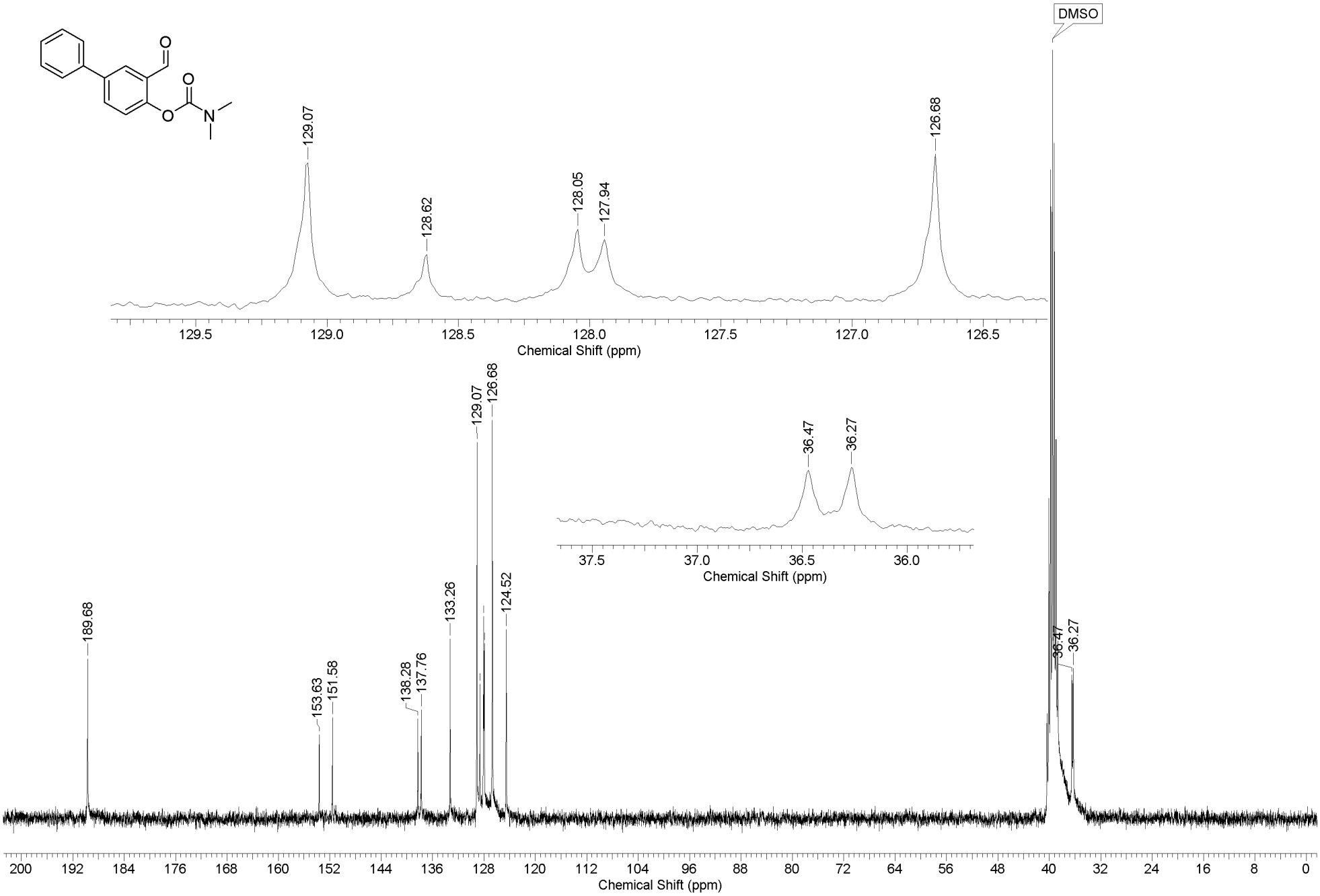

Water
DMSO
Chemical Shift (ppm)

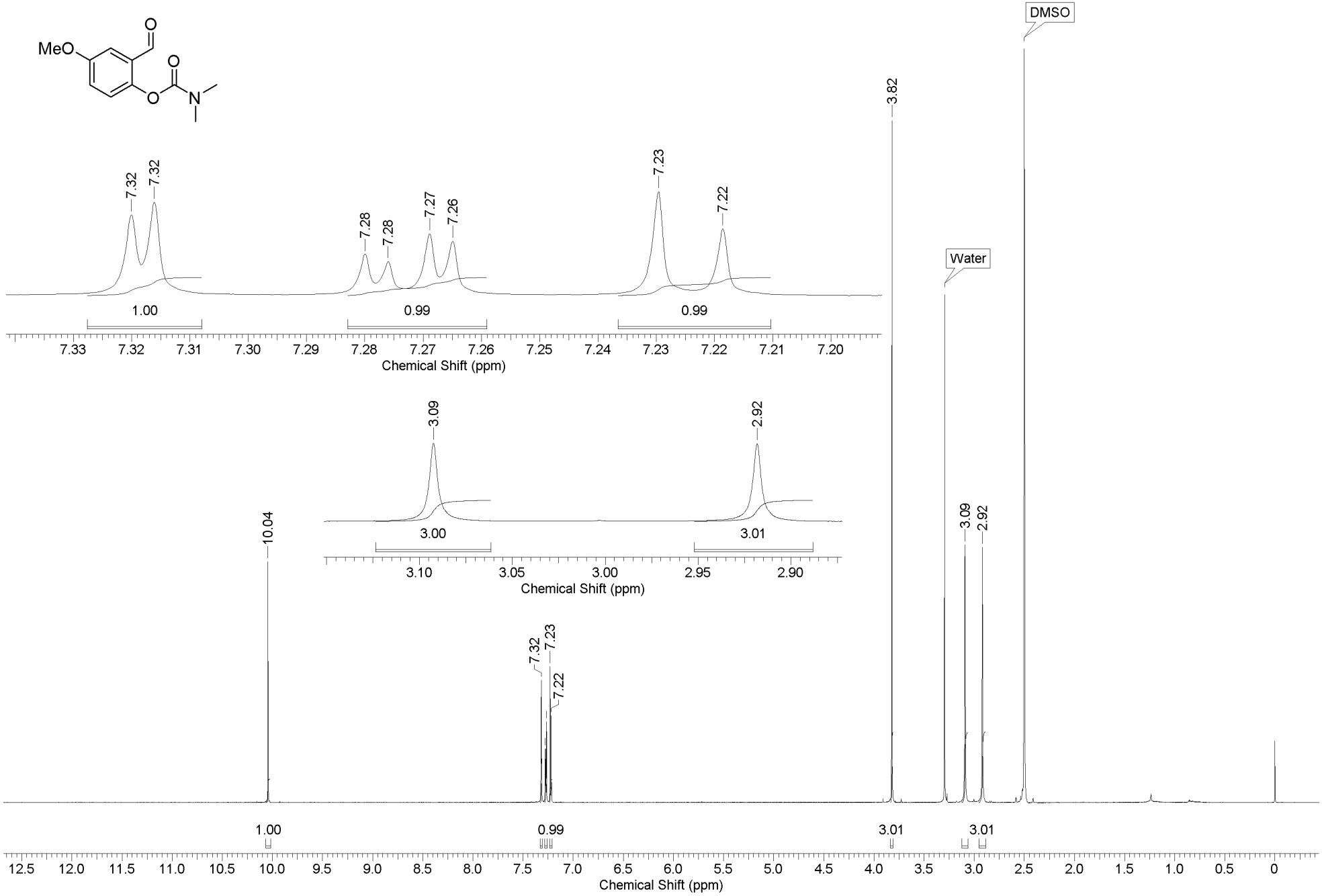

O
O
O
N
DMSO
36.45
36.23
38.0
37.5
37.0
36.5
36.0
35.5
35.0
Chemical Shift (ppm)
189.63
153.83
150.55
146.04
133.46
128.10
127.05
123.80
36.45
36.23
32.70
23.65
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

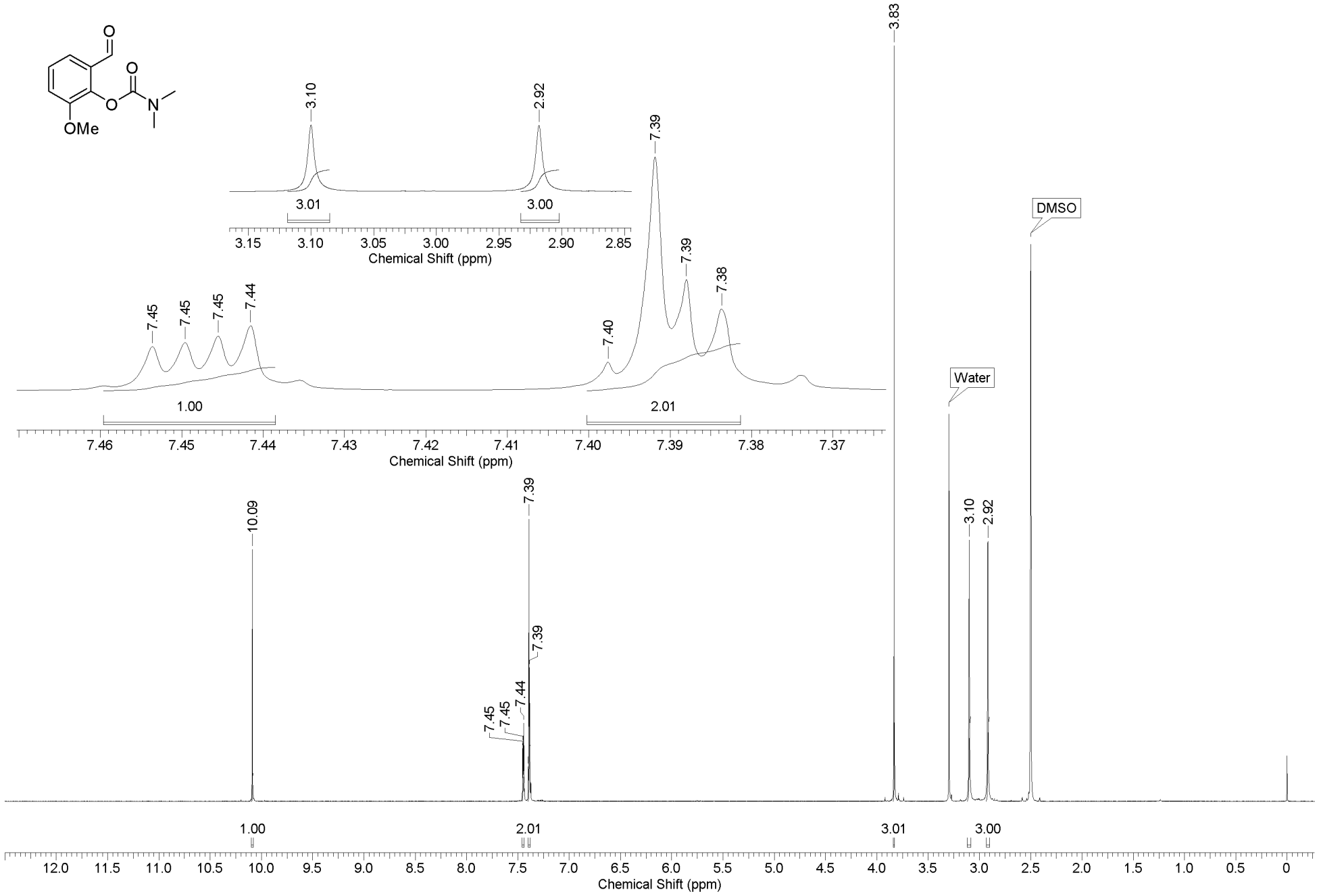

O
O
O
N
7.80
7.79
7.48
7.48
7.47
7.47
7.26
7.26
1.00
1.00
1.01
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20
Chemical Shift (ppm)
Water
DMSO
1.30
3.10
2.93
3.00
3.01
3.2 3.1 3.0 2.9 2.8 2.7
Chemical Shift (ppm)
10.01
7.80
7.79
7.48
7.26
3.10
2.93
0.99
1.00
1.01
3.01
9.00
12.5 12.0 11.5 11.0 10.5 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
Chemical Shift (ppm)

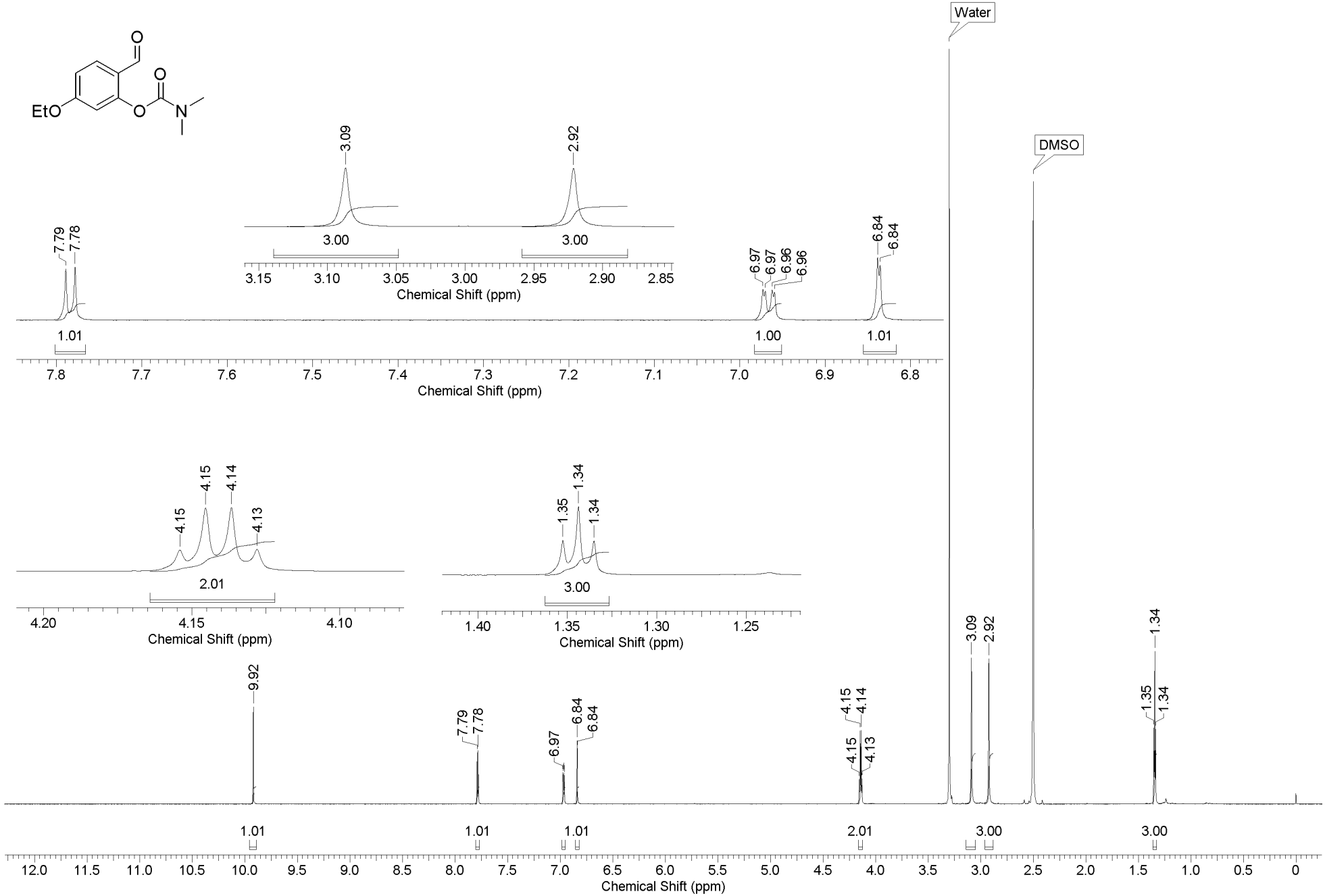

DMSO
36.43
36.24
30.58
189.05
159.27
153.82
152.29
129.69
126.14
122.98
120.68
36.43
36.24
35.13
Chemical Shift (ppm)
Chemical Shift (ppm)

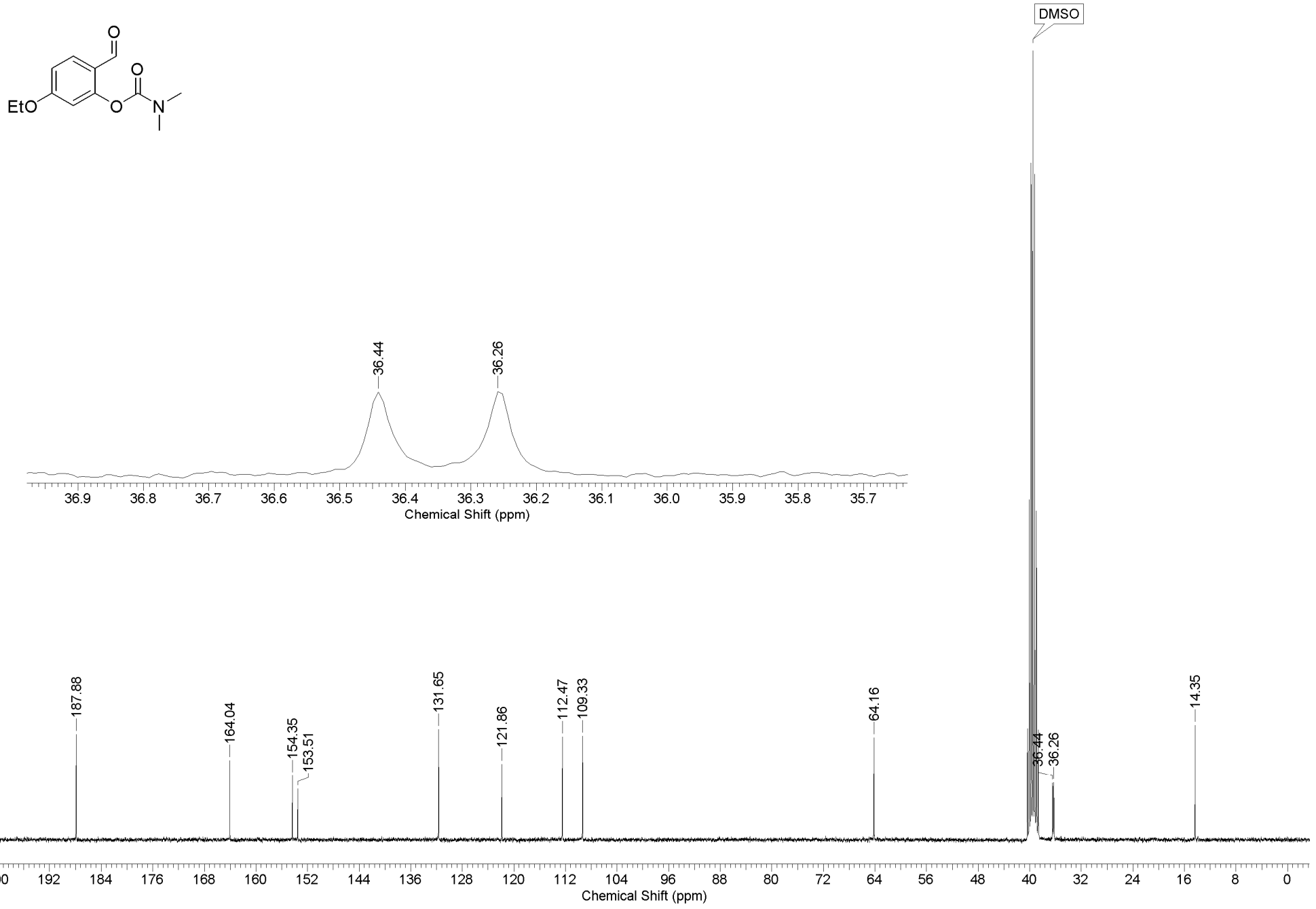

DMSO
Water
10.15
8.12
8.11
8.02
8.01
8.01
8.00
7.74
7.74
7.73
7.52
7.51
7.50
7.43
7.42
7.41
7.40
3.13
2.95
1.00
1.01
1.01
2.00
2.01
1.99
2.99
3.01
3.01
Chemical Shift (ppm)
Chemical Shift (ppm)

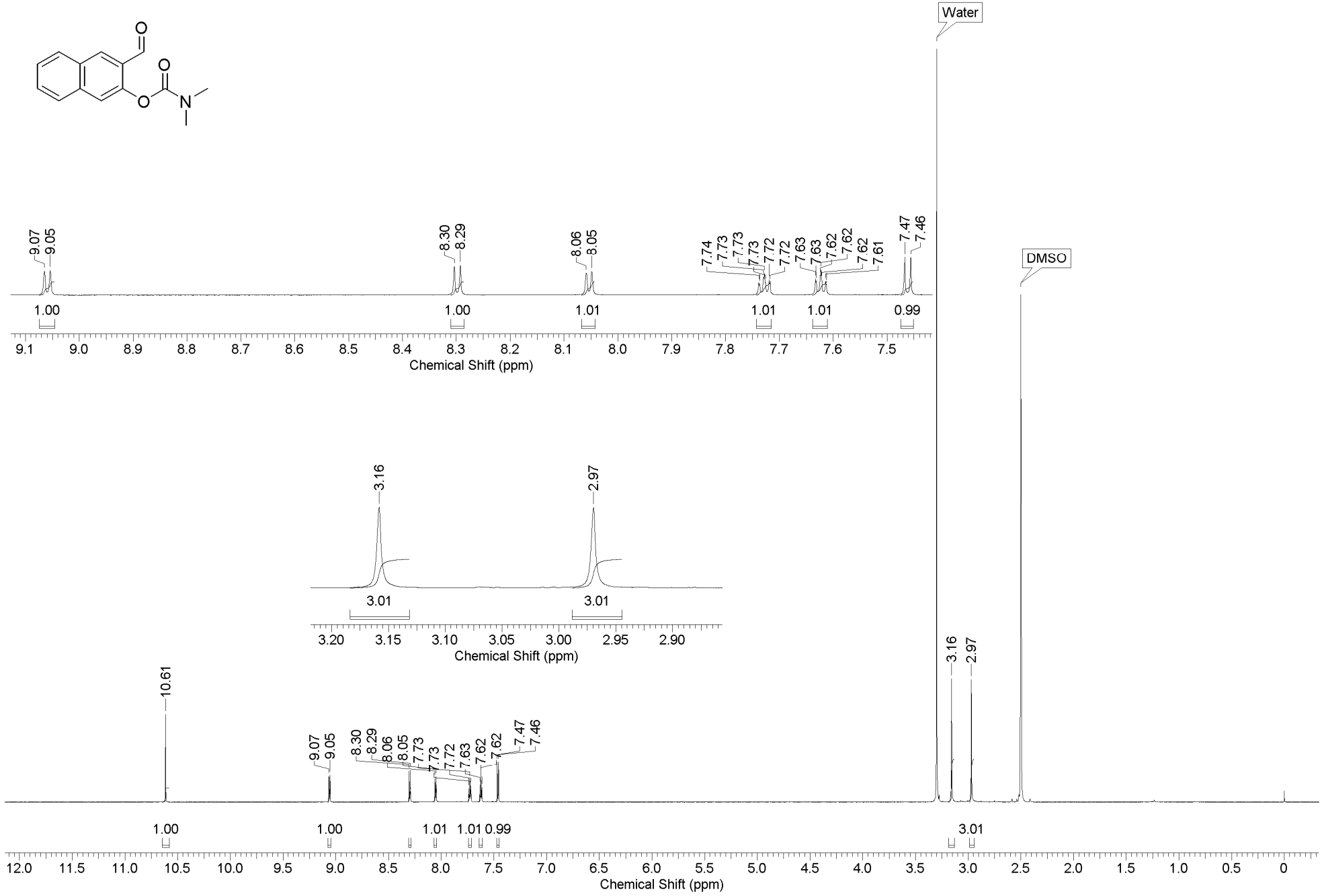

DMSO
129.07
128.62
128.05
127.94
126.68
129.5
129.0
128.5
128.0
127.5
127.0
126.5
Chemical Shift (ppm)
36.47
36.27
37.5
37.0
36.5
36.0
Chemical Shift (ppm)
189.68
153.63
151.58
138.28
137.76
133.26
129.07
126.68
124.52
36.47
36.27
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

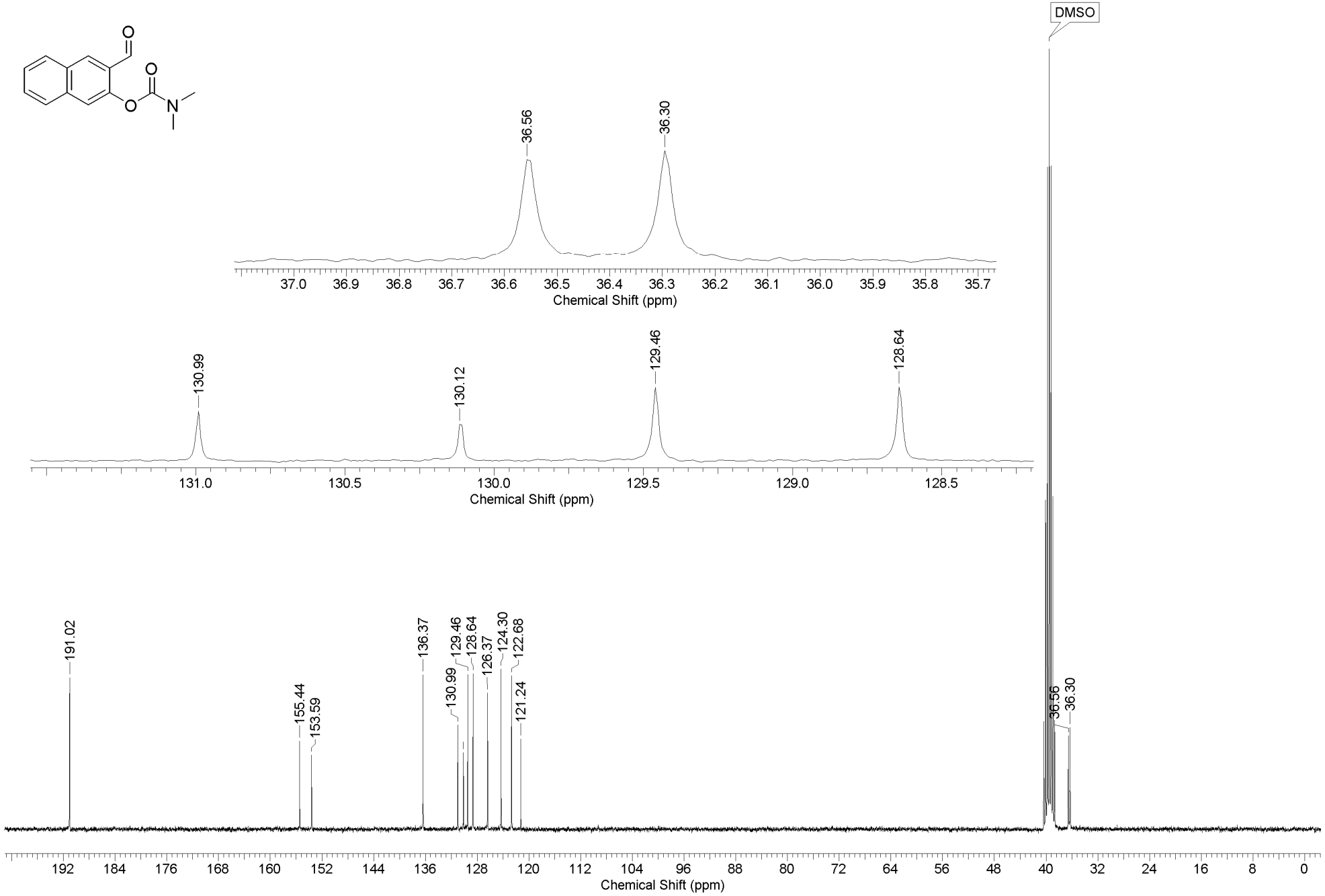

MeO
O
O
O
N
DMSO
Water
3.82
7.32
7.32
7.28
7.28
7.27
7.26
7.23
7.22
1.00
0.99
0.99
7.33 7.32 7.31 7.30 7.29 7.28 7.27 7.26 7.25 7.24 7.23 7.22 7.21 7.20
Chemical Shift (ppm)
3.09
2.92
3.00
3.01
3.10 3.05 3.00 2.95 2.90
Chemical Shift (ppm)
10.04
7.32
7.23
7.22
3.09
2.92
1.00
0.99
3.01
3.01
12.5 12.0 11.5 11.0 10.5 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
Chemical Shift (ppm)

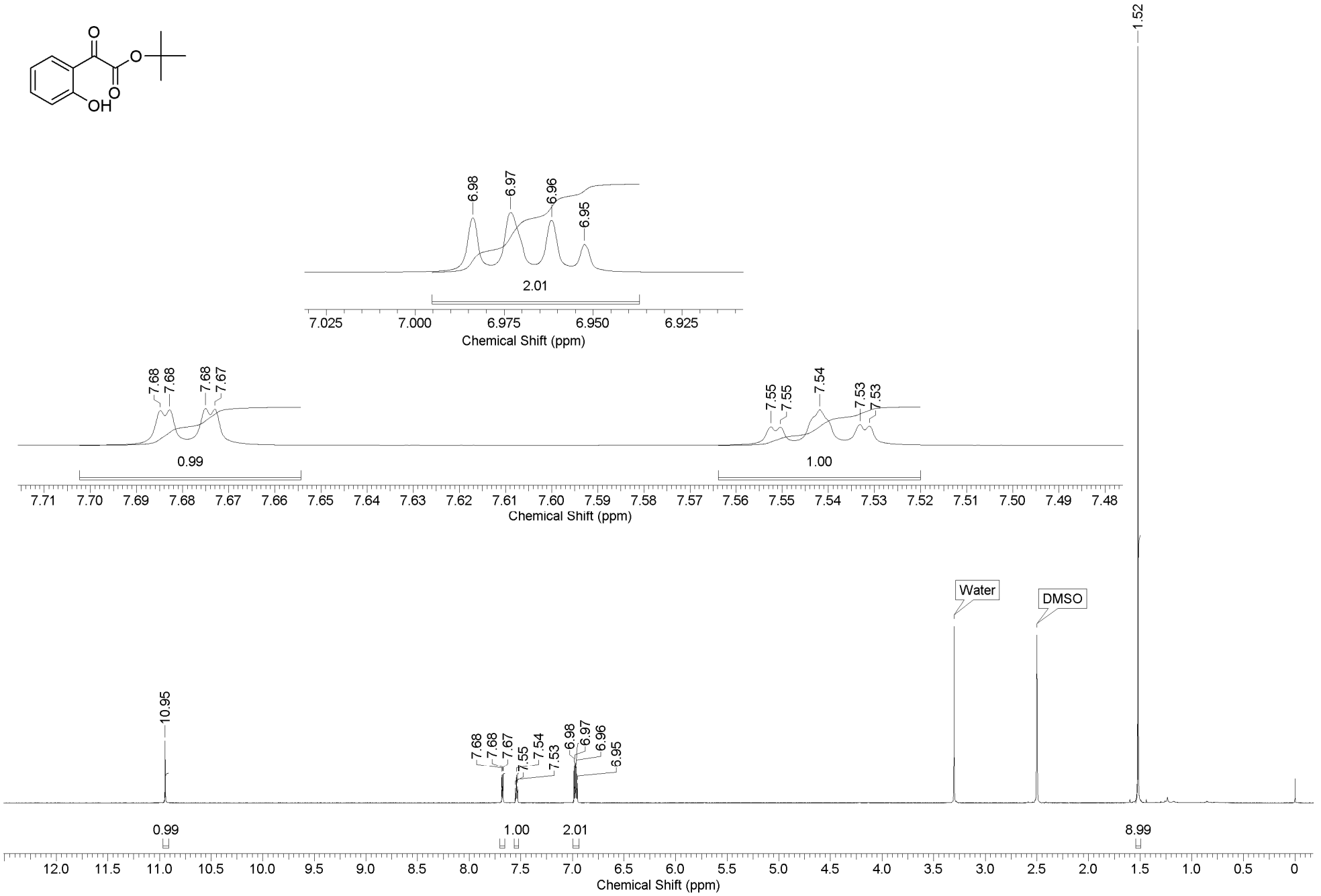

OMe
3.10
2.92
3.01
3.00
Chemical Shift (ppm)
7.45
7.45
7.45
7.44
7.40
7.39
7.39
7.38
1.00
2.01
Chemical Shift (ppm)
3.83
DMSO
Water
10.09
7.39
7.39
7.44
7.45
7.45
3.10
2.92
1.00
2.01
3.01
3.00
Chemical Shift (ppm)

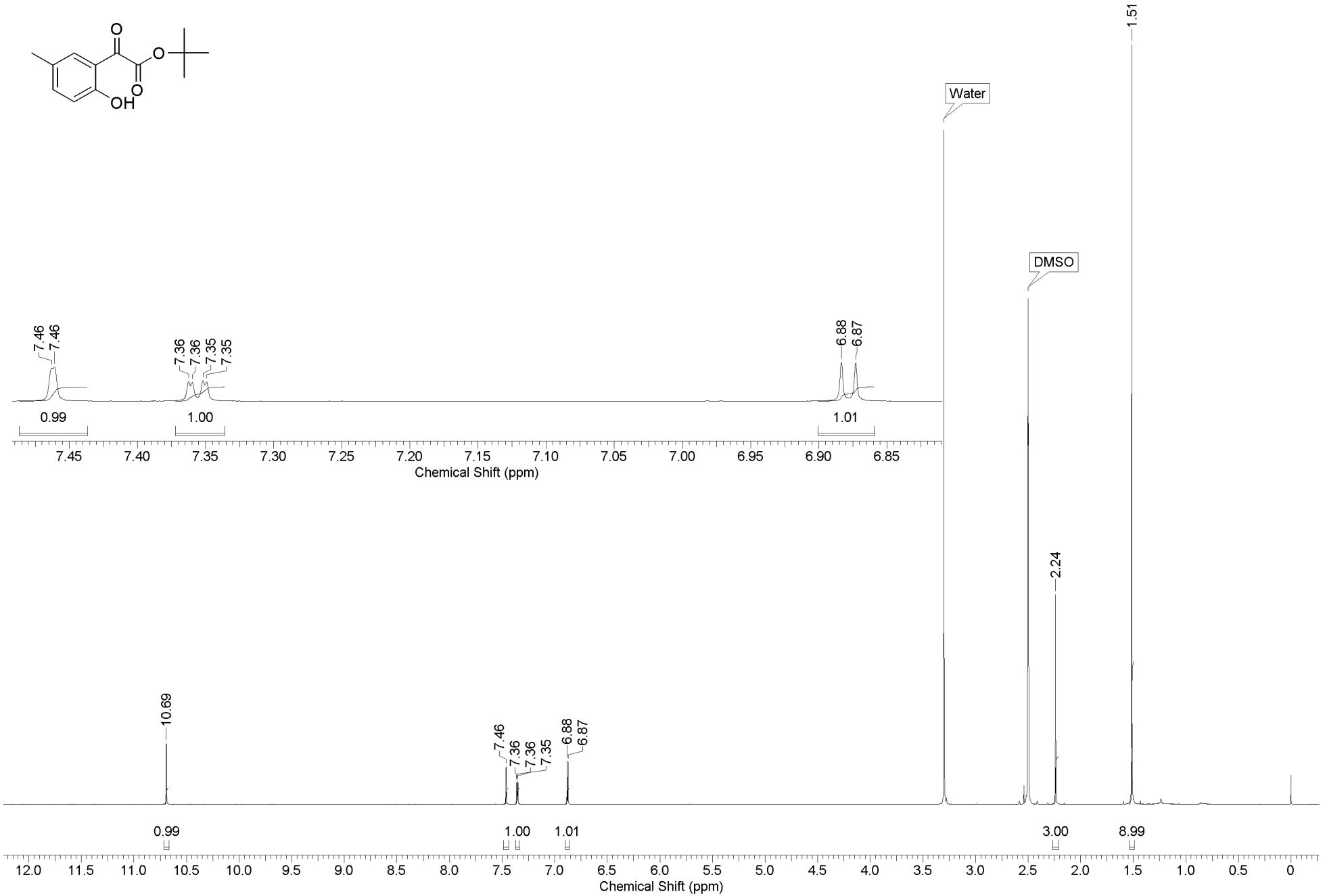

EtO
O
O
O
N
Water
DMSO
9.92
7.79
7.78
6.97
6.97
6.96
6.96
6.84
6.84
4.15
4.14
4.13
3.09
2.92
1.35
1.34
1.01
1.01
1.01
2.01
3.00
3.00
Chemical Shift (ppm)

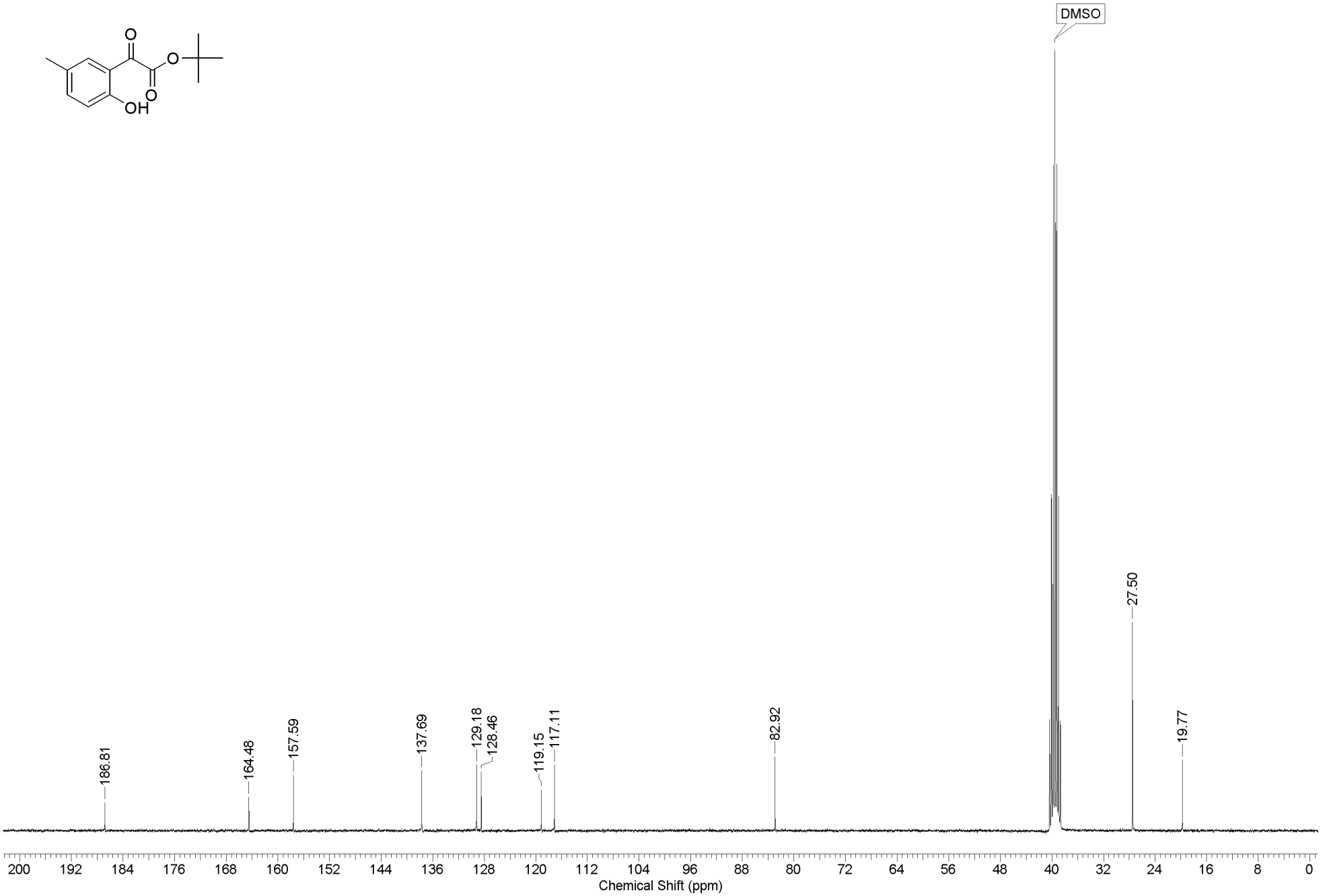

O
O
EtO
O
N
DMSO
36.44
36.26
36.9
36.8
36.7
36.6
36.5
36.4
36.3
36.2
36.1
36.0
35.9
35.8
35.7
Chemical Shift (ppm)
187.88
164.04
154.35
153.51
131.65
121.86
112.47
109.33
64.16
36.44
36.26
14.35
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

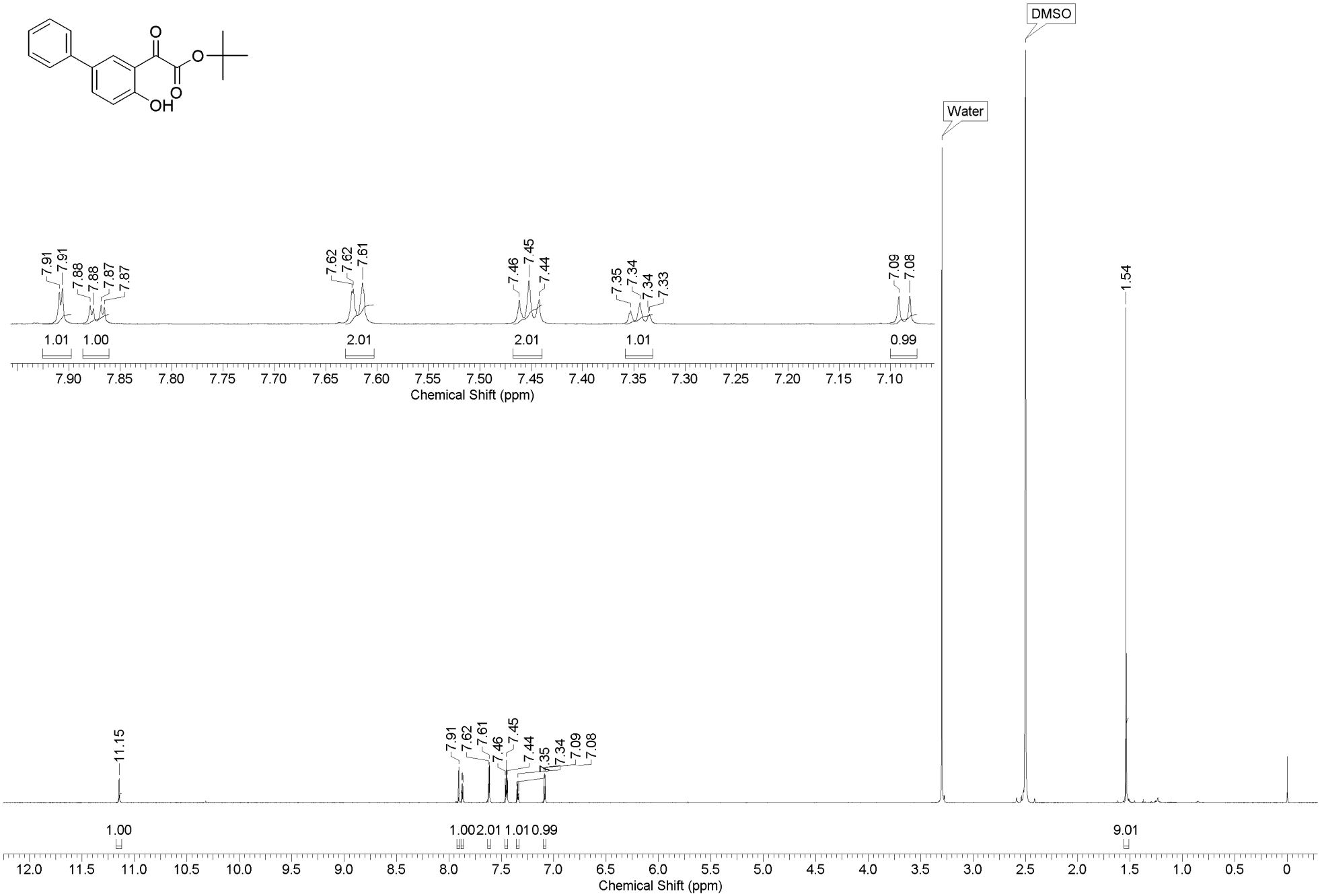

Water
DMSO
9.07
9.05
8.30
8.29
8.06
8.05
7.74
7.73
7.73
7.73
7.72
7.72
7.63
7.63
7.62
7.62
7.62
7.61
7.47
7.46
1.00
1.00
1.01
1.01
1.01
0.99
Chemical Shift (ppm)
3.16
2.97
3.01
3.01
Chemical Shift (ppm)
10.61
3.16
2.97
1.00
1.00
1.01
1.01
0.99
3.01
Chemical Shift (ppm)

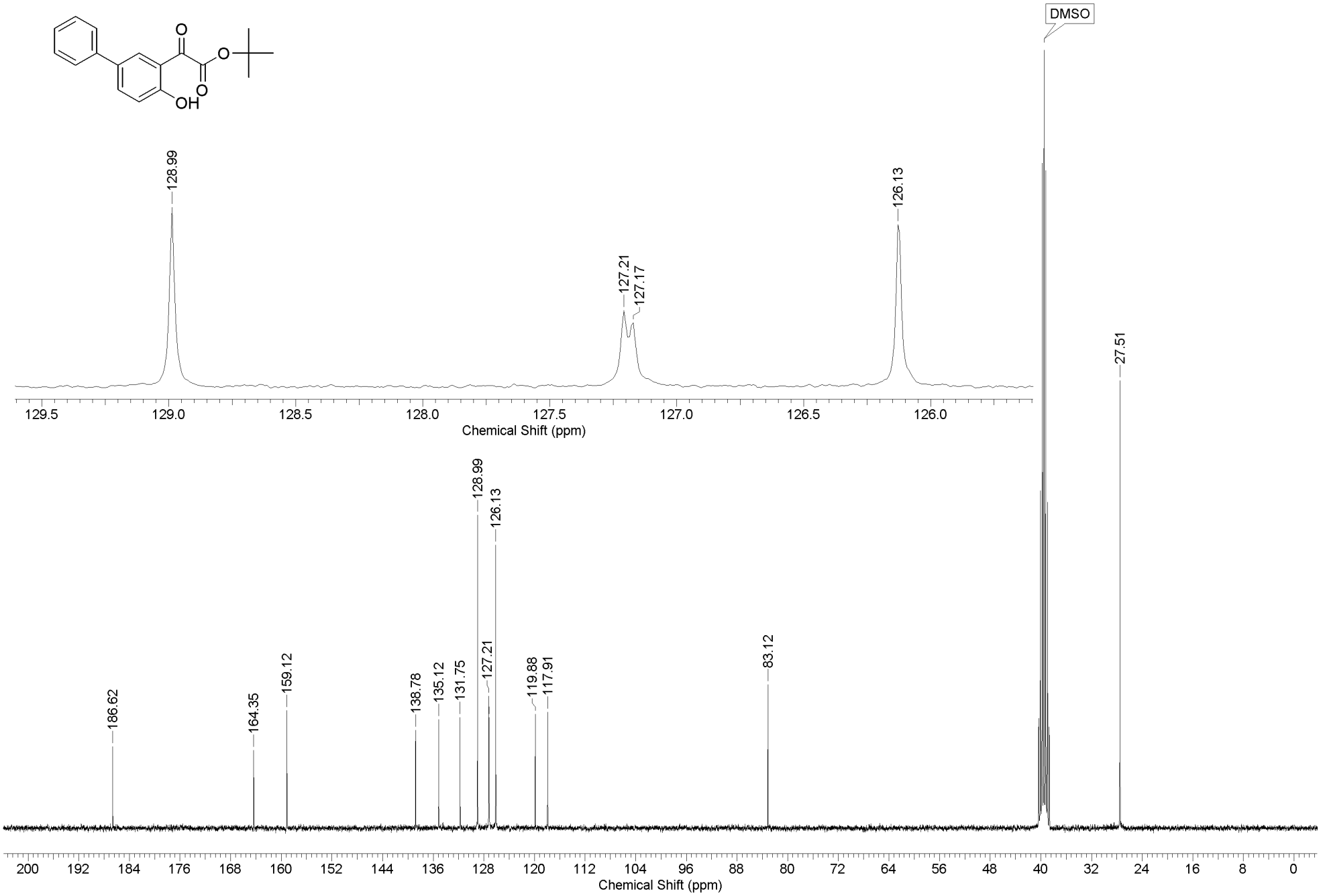

DMSO
36.56
36.30
37.0 36.9 36.8 36.7 36.6 36.5 36.4 36.3 36.2 36.1 36.0 35.9 35.8 35.7
Chemical Shift (ppm)
130.99
130.12
129.46
128.64
131.0 130.5 130.0 129.5 129.0 128.5
Chemical Shift (ppm)
191.02
155.44
153.59
136.37
130.99
129.46
128.64
126.37
124.30
122.68
121.24
36.56
36.30
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

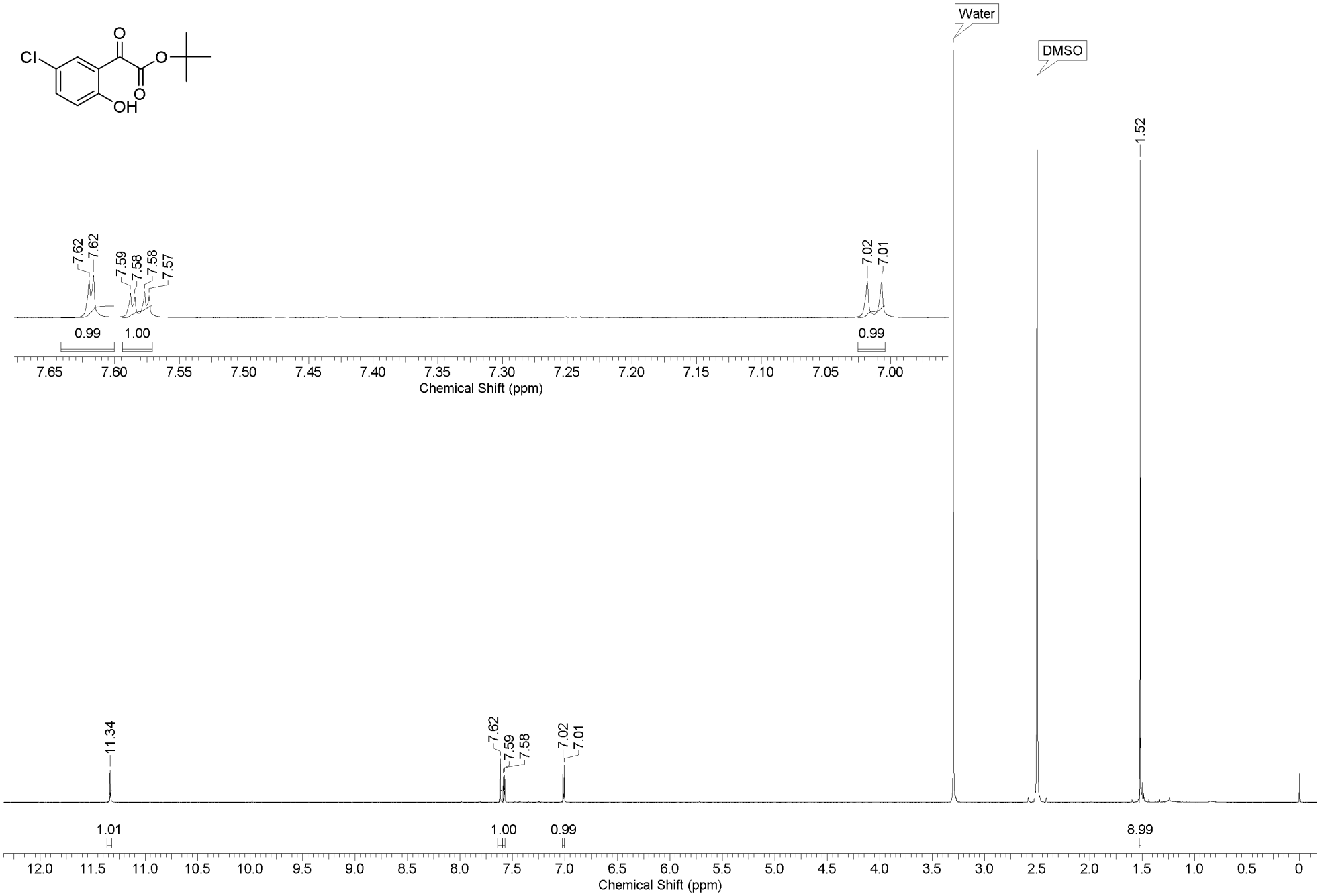

O
O
O
O
OH
6.98
6.97
6.96
6.95
2.01
7.025
7.000
6.975
6.950
6.925
Chemical Shift (ppm)
7.68
7.68
7.68
7.67
7.55
7.55
7.54
7.53
7.53
0.99
1.00
7.71 7.70 7.69 7.68 7.67 7.66 7.65 7.64 7.63 7.62 7.61 7.60 7.59 7.58 7.57 7.56 7.55 7.54 7.53 7.52 7.51 7.50 7.49 7.48
Chemical Shift (ppm)
1.52
Water
DMSO
10.95
7.68
7.68
7.67
7.55
7.54
7.53
6.98
6.97
6.96
6.95
0.99
1.00
2.01
8.99
12.0 11.5 11.0 10.5 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
Chemical Shift (ppm)

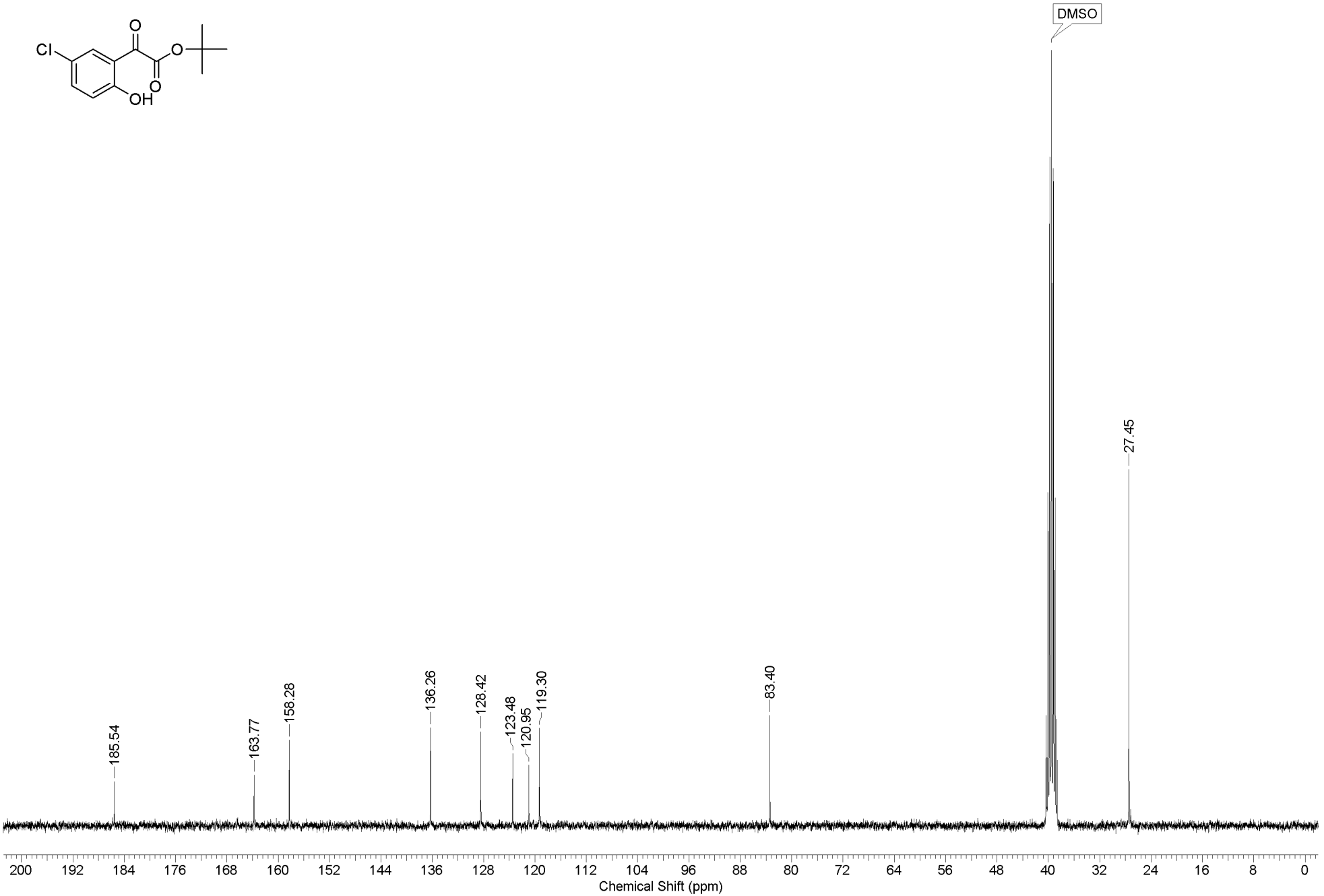

O
O
O
O
OH
Water
DMSO
1.51
2.24
10.69
7.46
7.46
7.36
7.36
7.35
7.35
6.88
6.87
0.99
1.00
1.01
3.00
8.99
Chemical Shift (ppm)

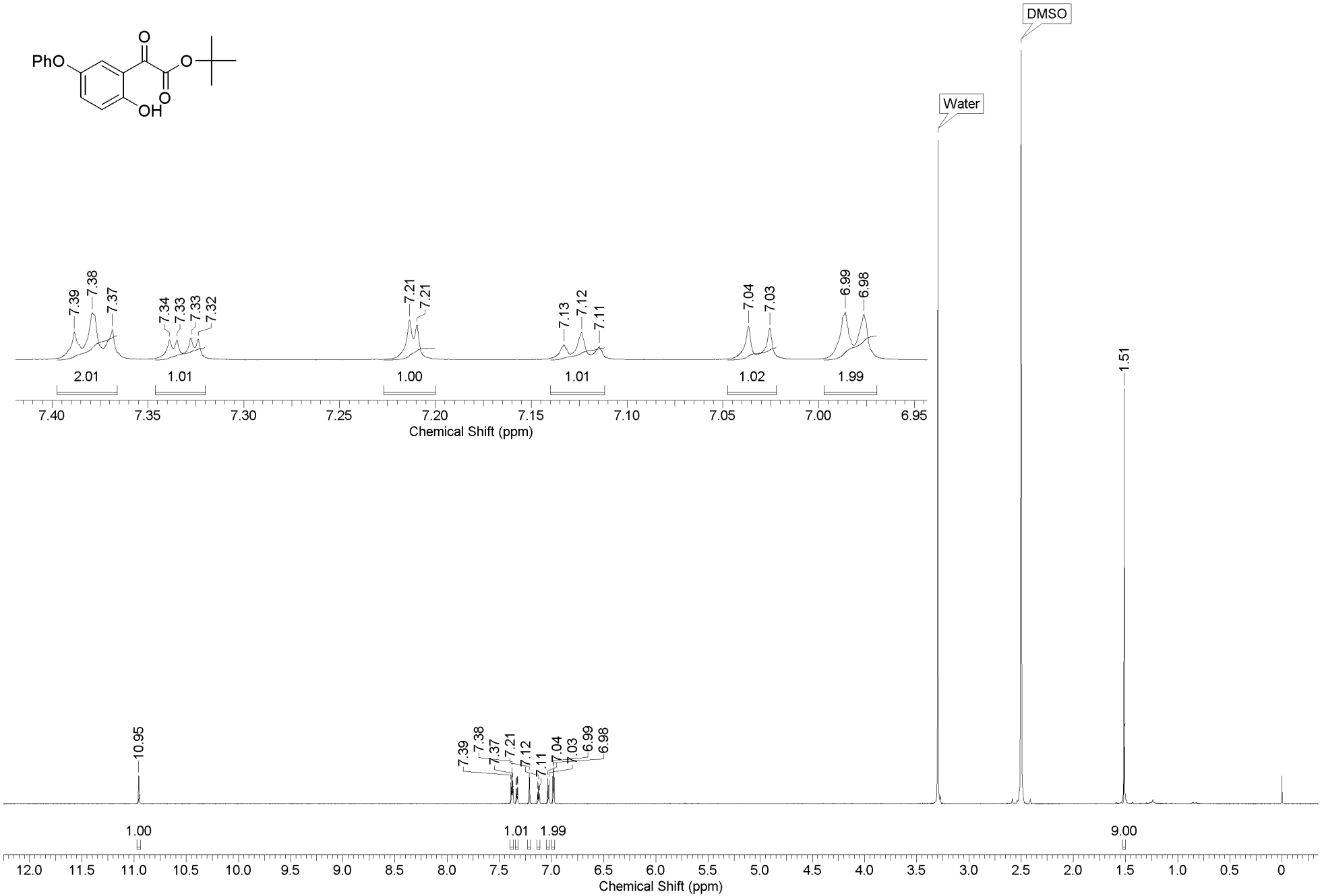

O
O
O
OH
DMSO
186.81
164.48
157.59
137.69
129.18
128.46
119.15
117.11
82.92
27.50
19.77
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

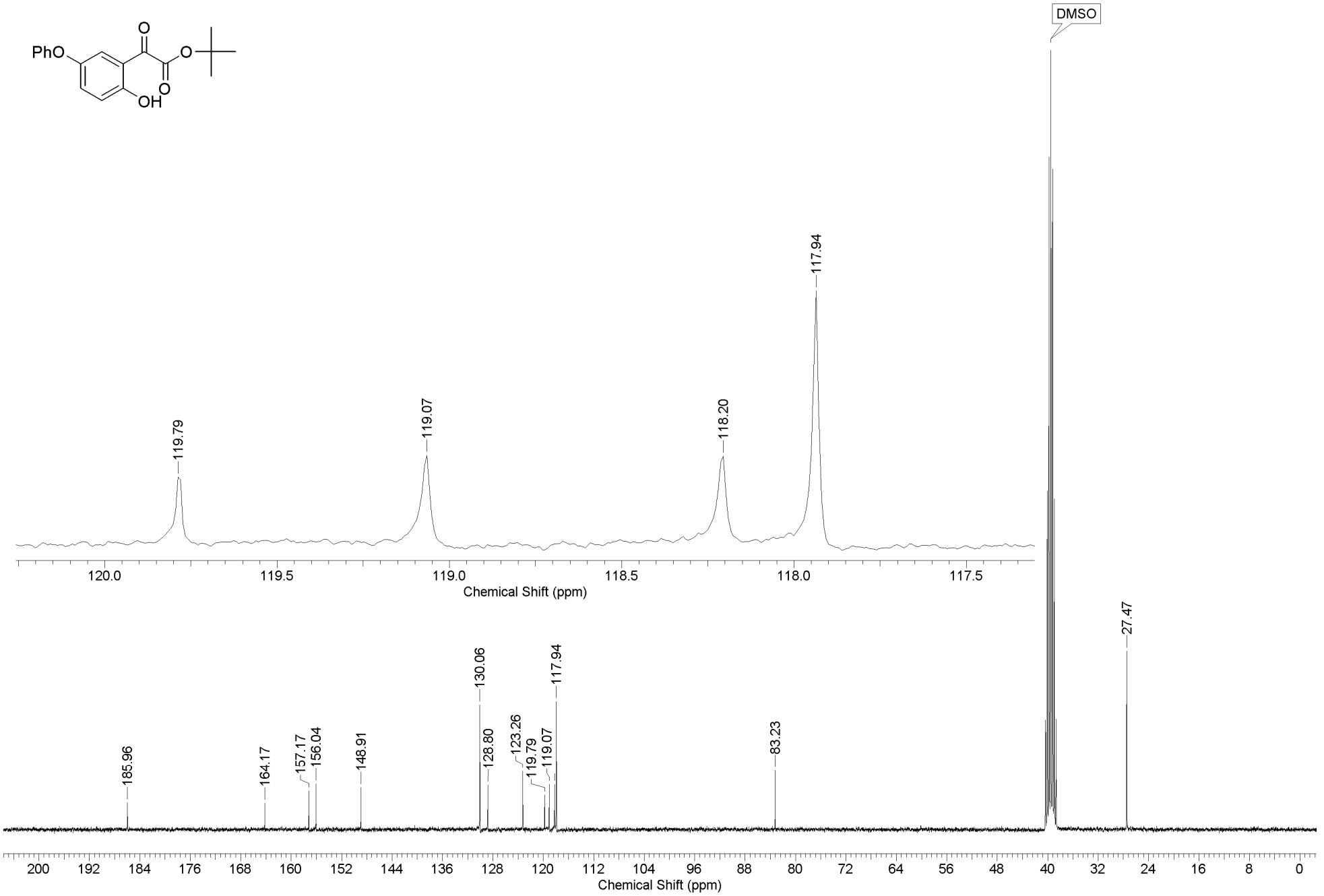

O
O
O
OH
DMSO
Water
7.91
7.91
7.88
7.88
7.87
7.87
7.62
7.62
7.61
7.46
7.45
7.44
7.35
7.34
7.34
7.33
7.09
7.08
1.54
1.01
1.00
2.01
2.01
1.01
0.99
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
Chemical Shift (ppm)
11.15
7.91
7.62
7.61
7.46
7.45
7.44
7.35
7.34
7.09
7.08
1.00
1.00
2.01
1.01
0.99
9.01
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

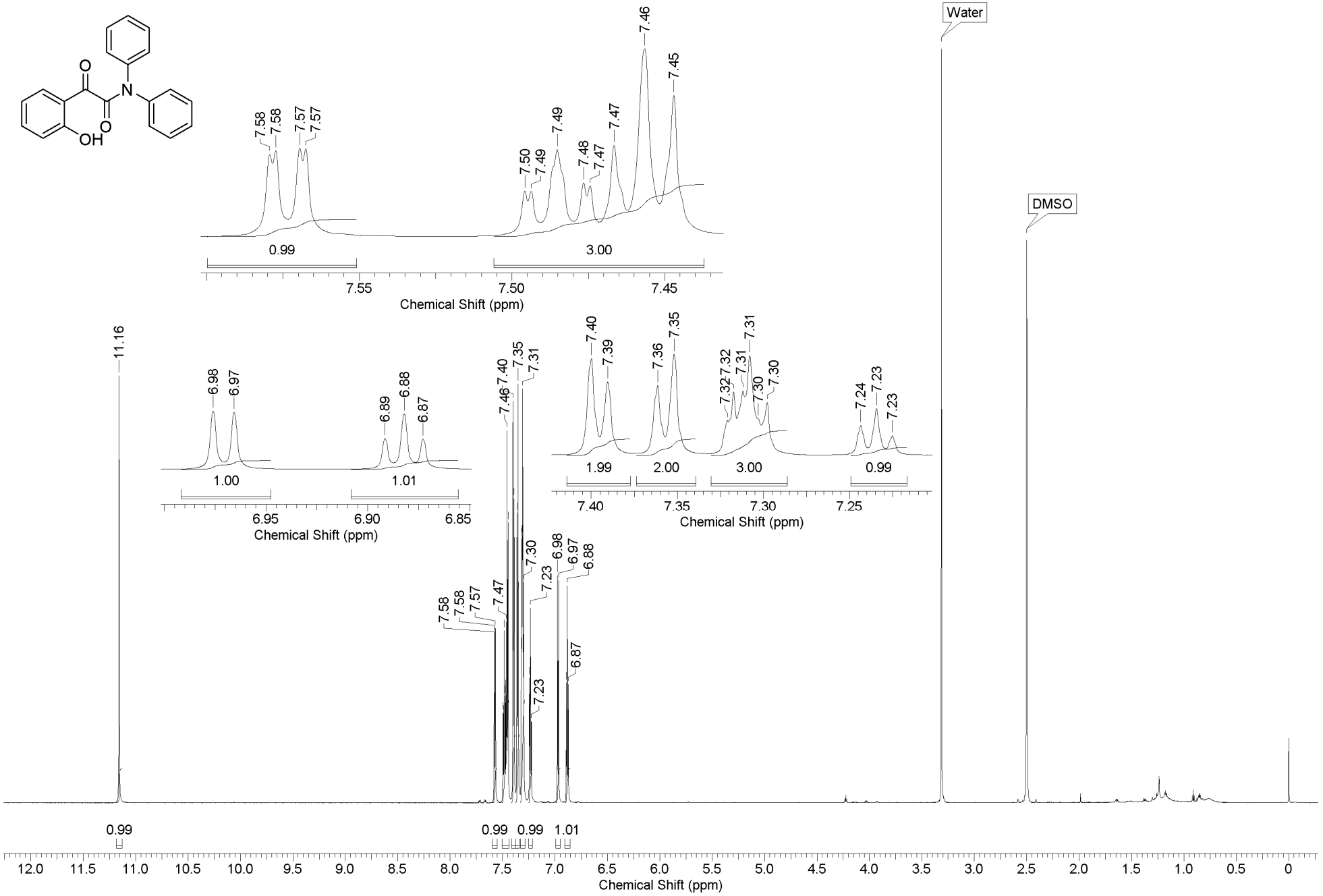

O
O
O
OH
DMSO
128.99
126.13
127.21
127.17
27.51
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
Chemical Shift (ppm)
128.99
126.13
186.62
164.35
159.12
138.78
135.12
131.75
127.21
119.88
117.91
83.12
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

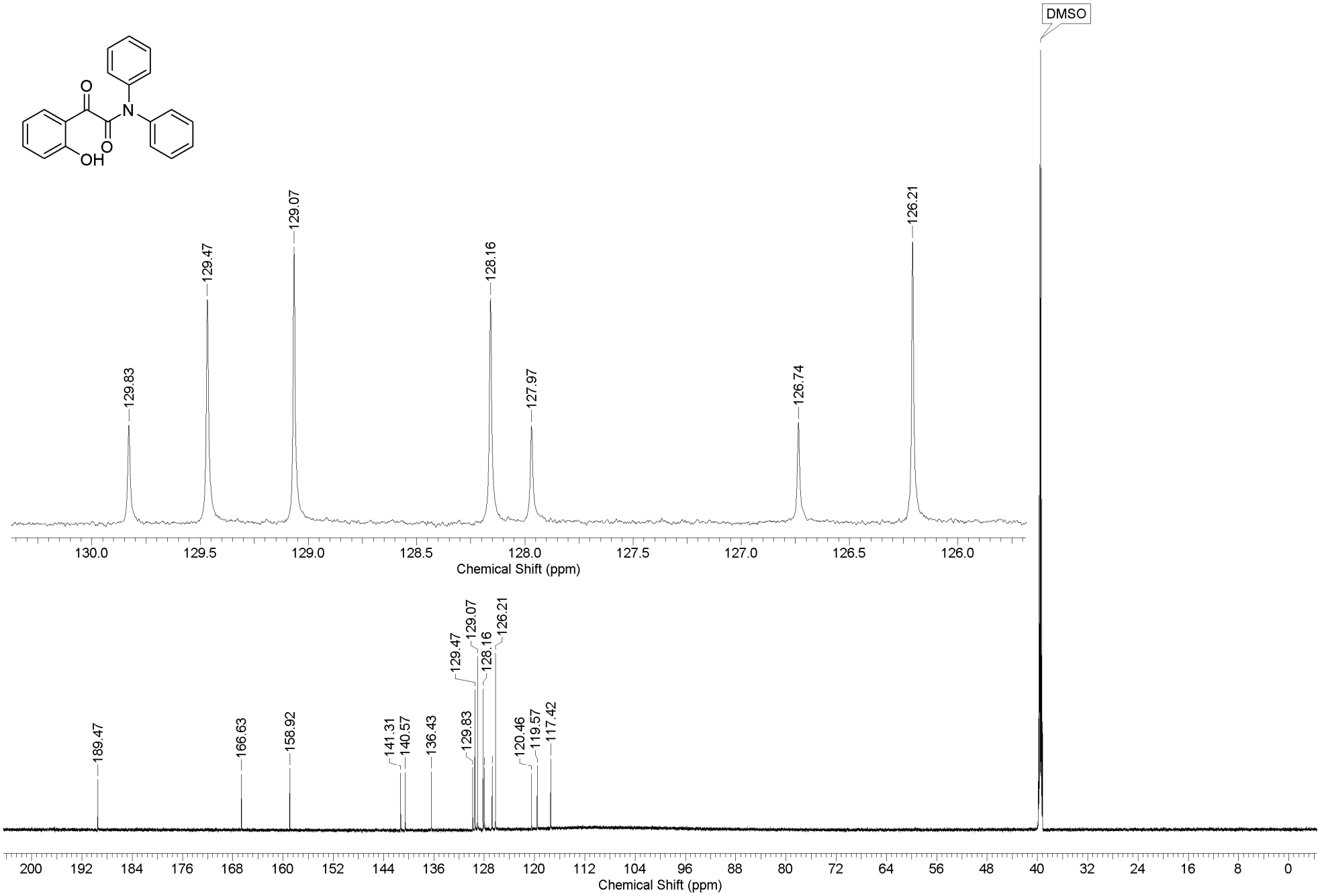

Cl
O
O
O
OH
Water
DMSO
1.52
7.62
7.62
7.59
7.58
7.58
7.57
7.02
7.01
0.99
1.00
0.99
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
7.00
Chemical Shift (ppm)
11.34
7.62
7.59
7.58
7.02
7.01
1.01
1.00
0.99
8.99
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

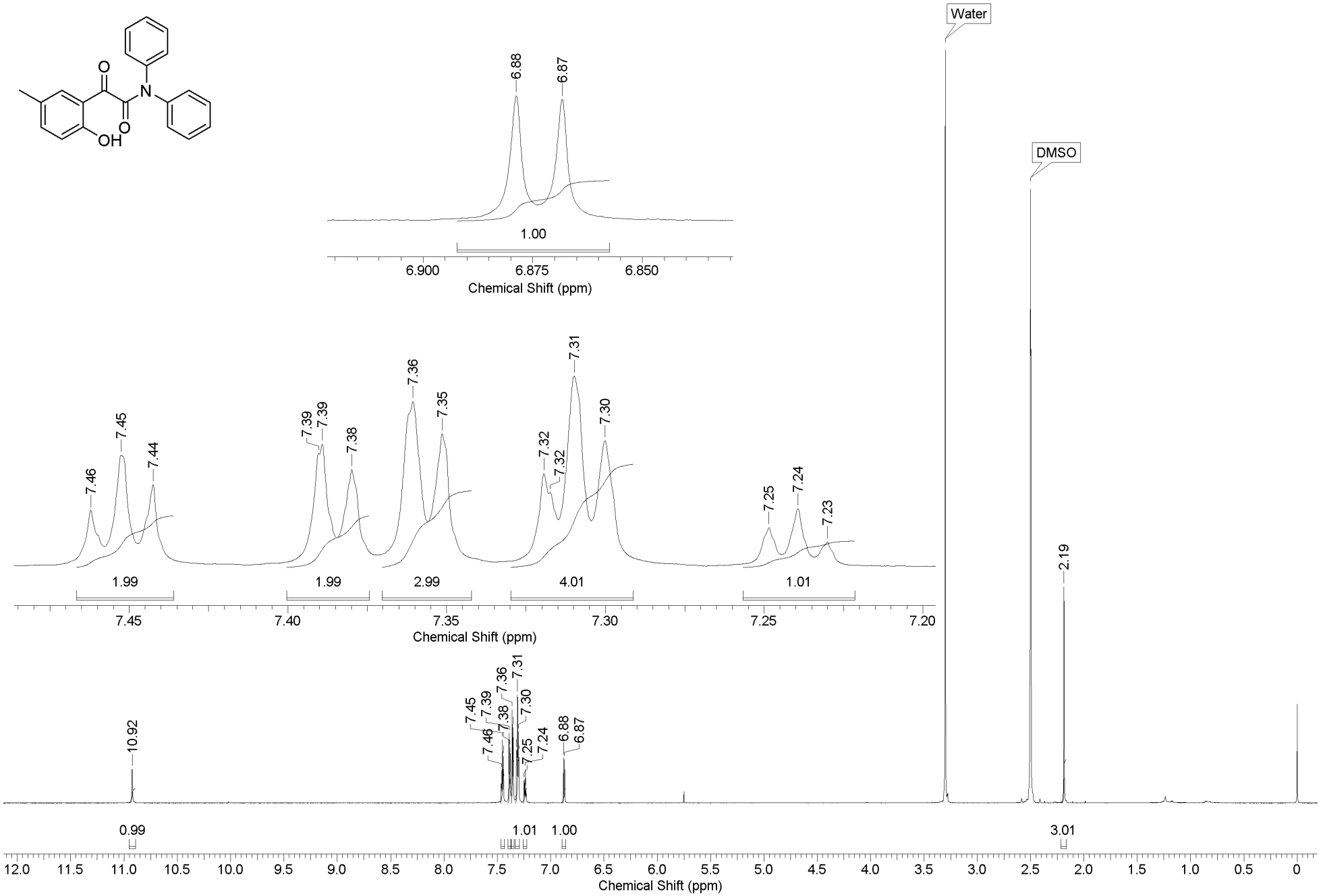

Cl
O
O
O
OH
DMSO
27.45
185.54
163.77
158.28
136.26
128.42
123.48
120.95
119.30
83.40
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

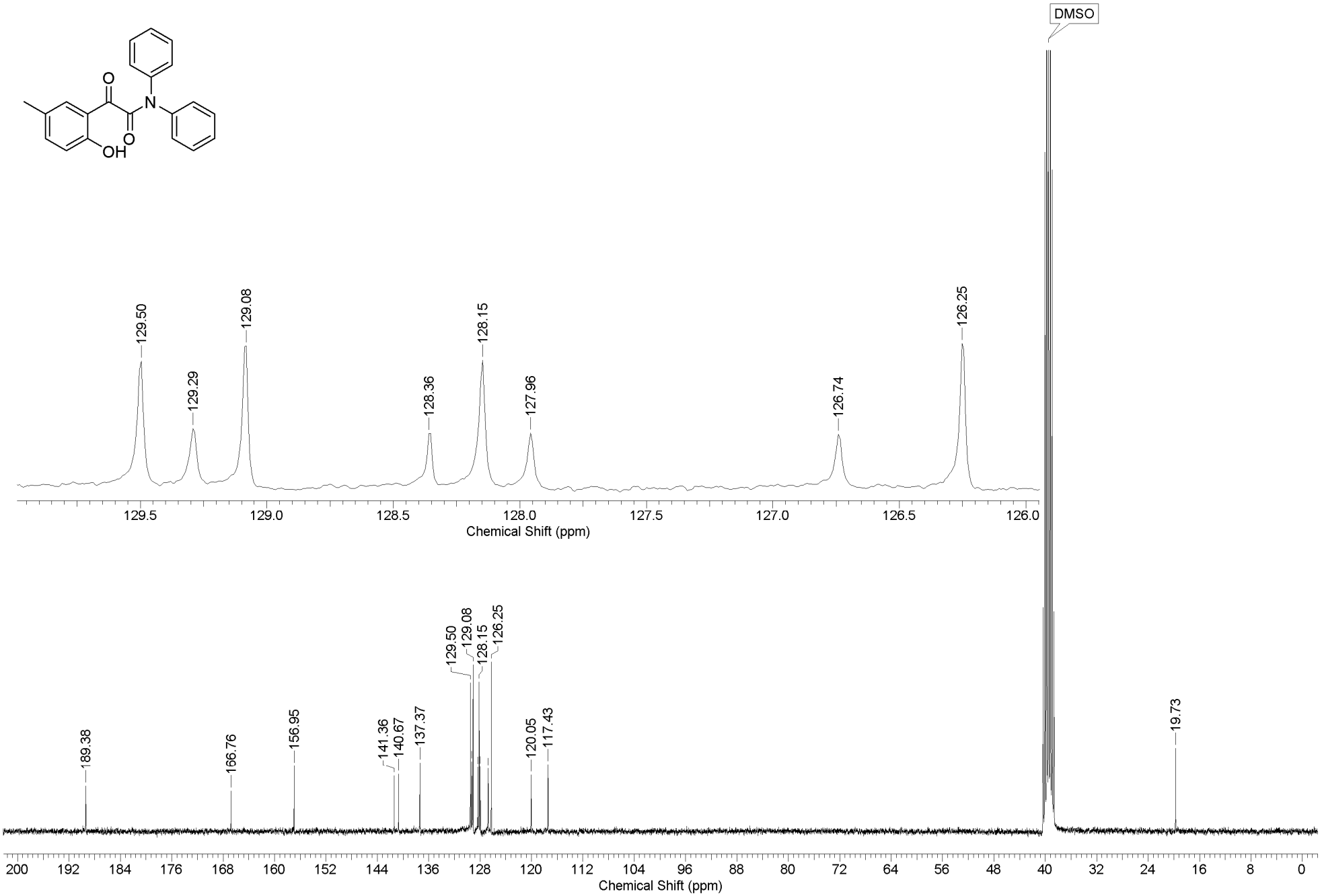

PhO
O
O
O
OH
DMSO
Water
1.51
10.95
7.39
7.38
7.37
7.34
7.33
7.33
7.32
7.21
7.21
7.13
7.12
7.11
7.04
7.03
6.99
6.98
2.01
1.01
1.00
1.01
1.02
1.99
1.00
1.01
1.99
9.00
Chemical Shift (ppm)
Chemical Shift (ppm)

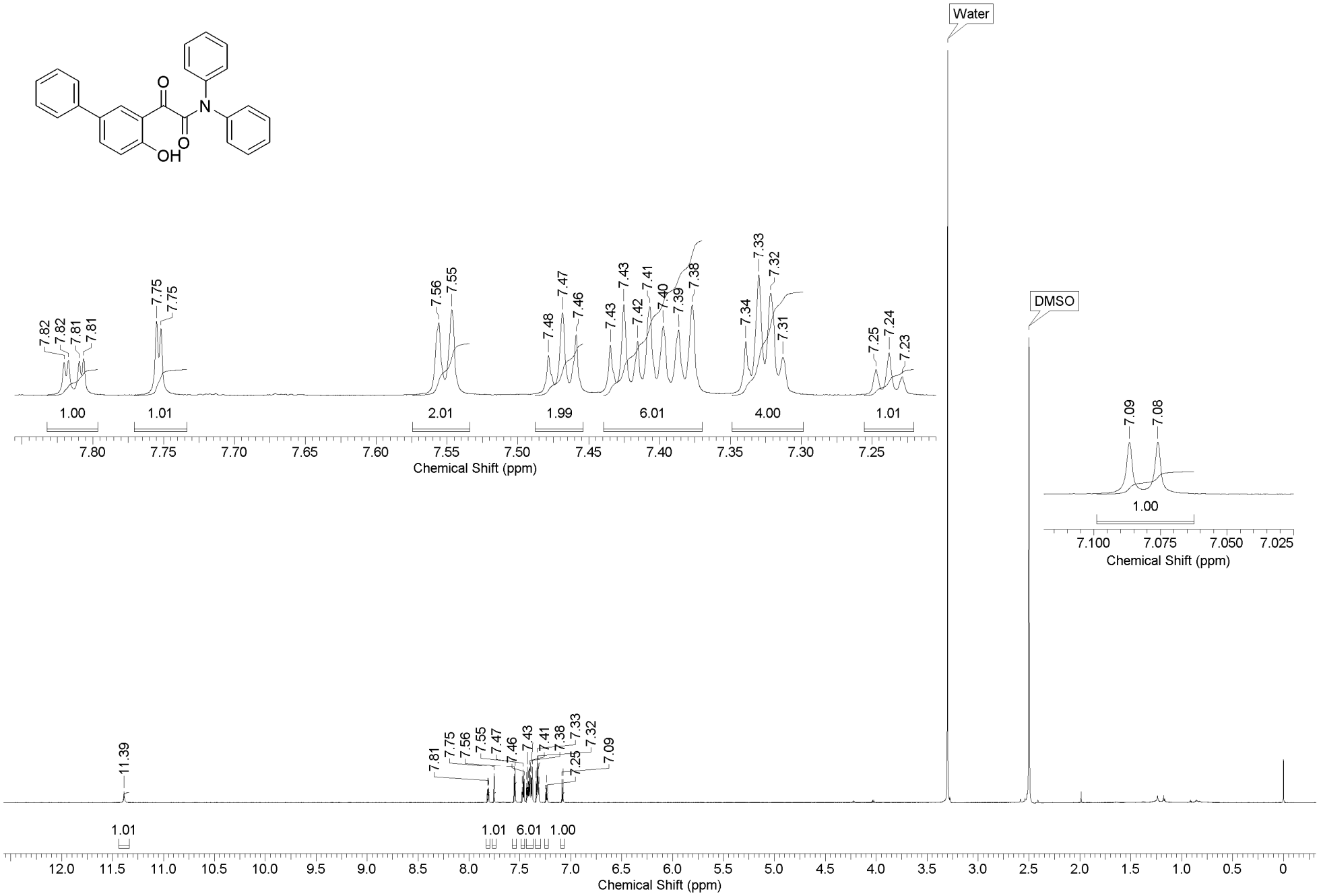

PhO
O
O
O
OH
DMSO
119.79
119.07
118.20
117.94
120.0
119.5
119.0
118.5
118.0
117.5
Chemical Shift (ppm)
27.47
185.96
164.17
157.17
156.04
148.91
130.06
128.80
123.26
119.79
119.07
117.94
83.23
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

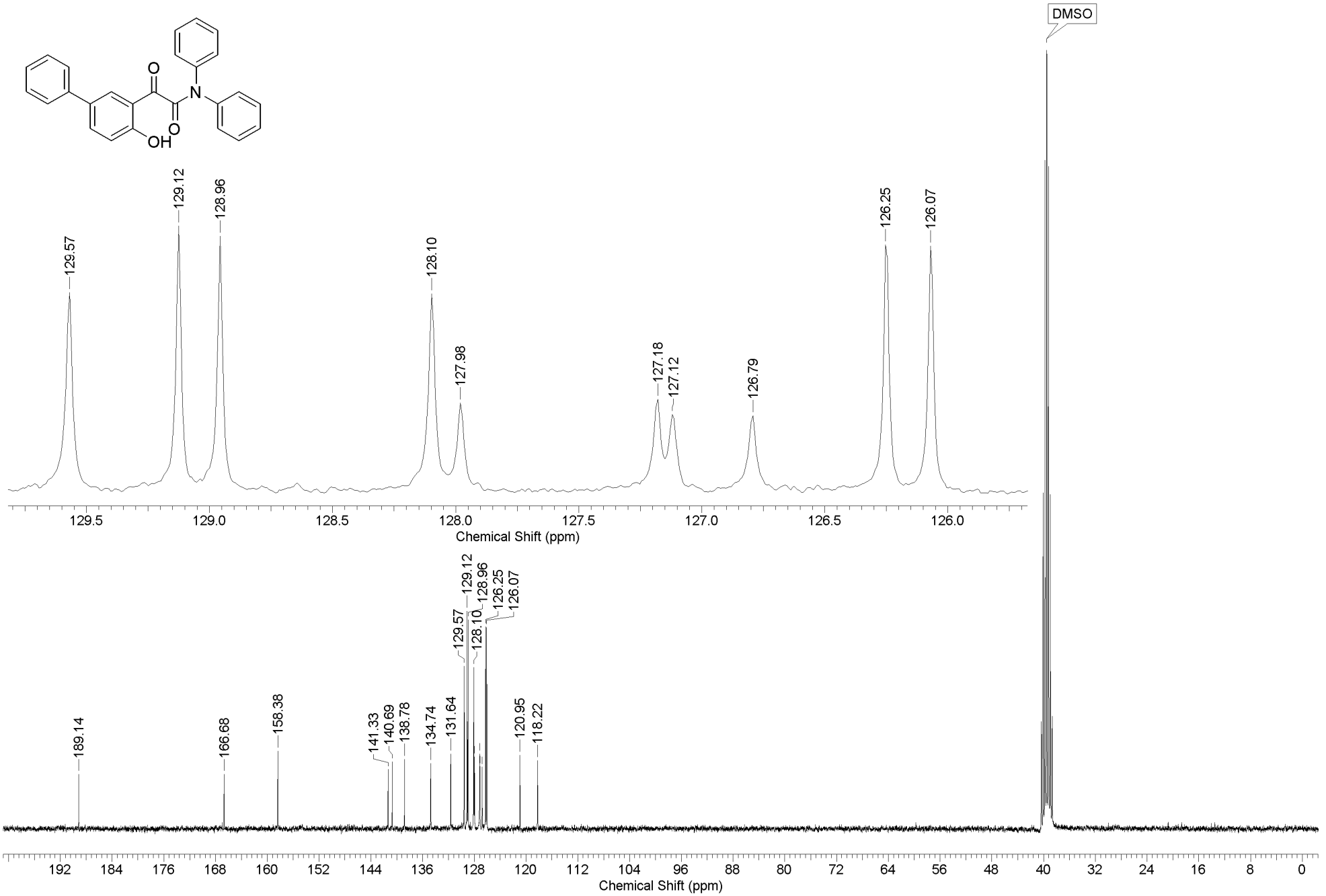

O
N
O
OH
Water
DMSO
11.16
7.58
7.58
7.57
7.57
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.45
0.99
3.00
7.55
7.50
7.45
Chemical Shift (ppm)
6.98
6.97
6.89
6.88
6.87
1.00
1.01
6.95
6.90
6.85
Chemical Shift (ppm)
7.40
7.39
7.36
7.35
7.32
7.31
7.31
7.30
7.30
7.24
7.23
7.23
1.99
2.00
3.00
0.99
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
7.46
7.40
7.35
7.31
7.30
7.23
6.98
6.97
6.88
7.58
7.58
7.57
7.47
6.87
7.23
0.99
0.99
0.99
1.01
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

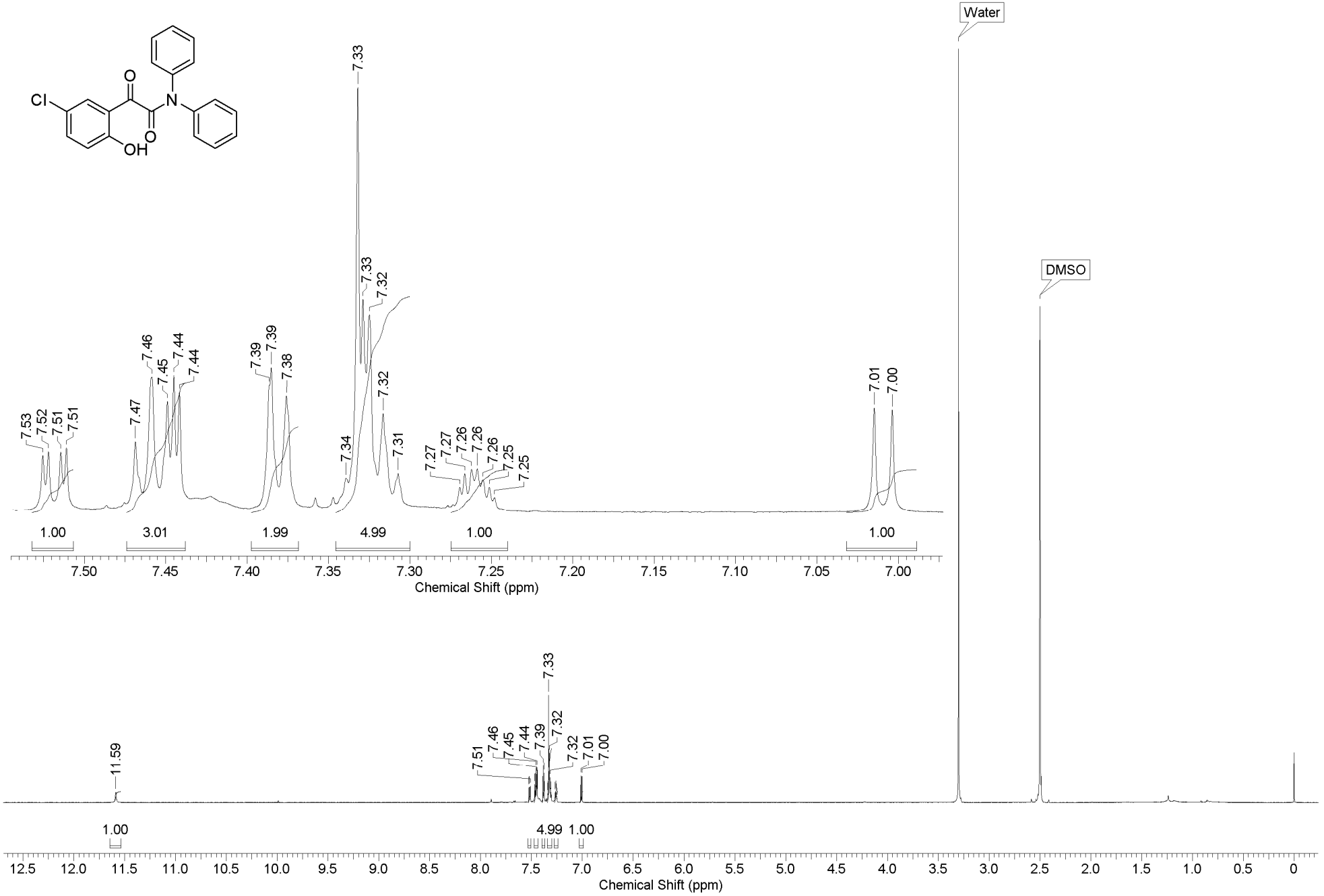

DMSO
129.83
129.47
129.07
128.16
127.97
126.74
126.21
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
Chemical Shift (ppm)
189.47
166.63
158.92
141.31
140.57
136.43
129.83
129.47
129.07
128.16
126.21
120.46
119.57
117.42
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

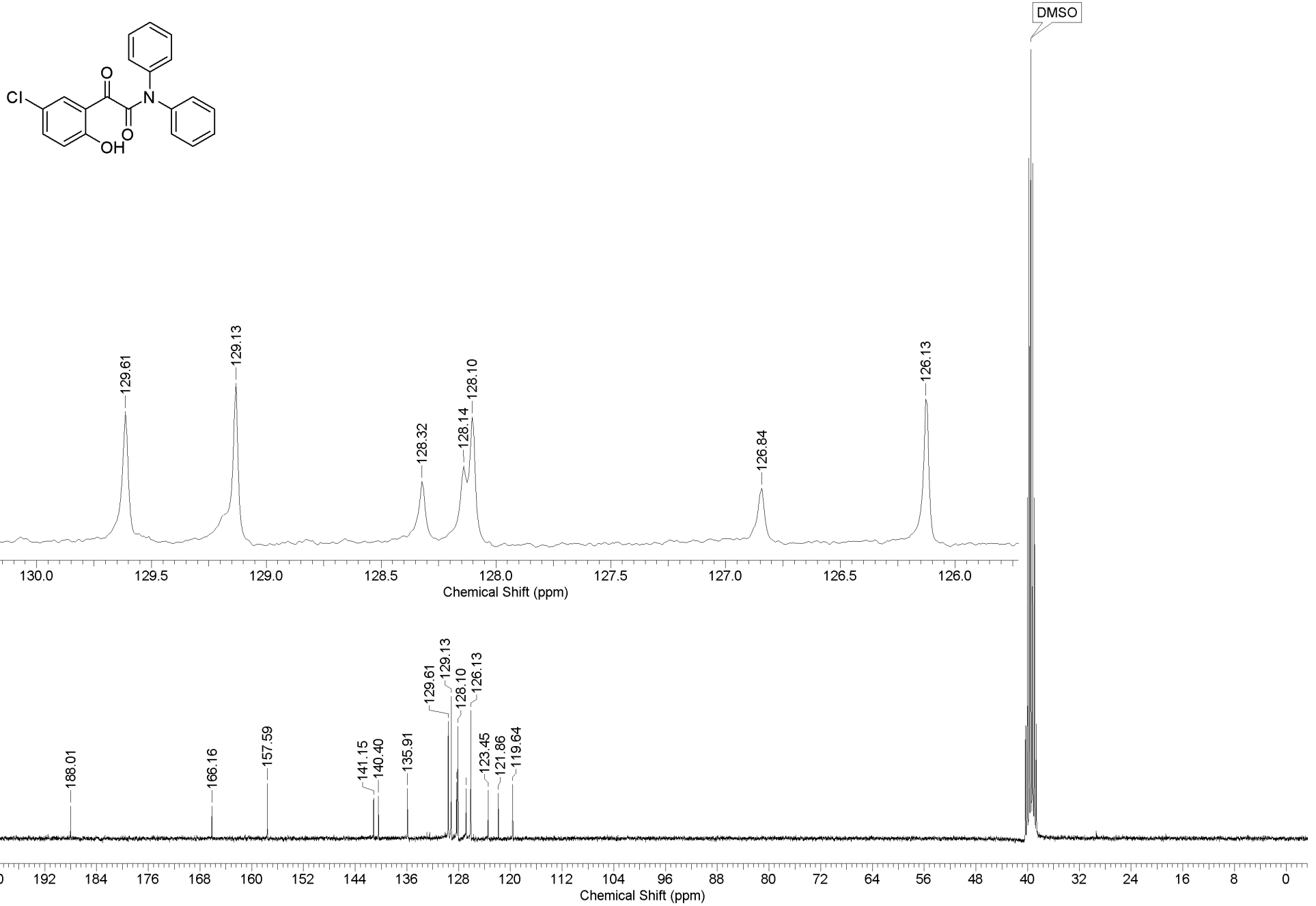

O
N
O
OH
Water
DMSO
6.88
6.87
1.00
6.900
6.875
6.850
Chemical Shift (ppm)
7.46
7.45
7.44
7.39
7.39
7.38
7.36
7.35
7.32
7.32
7.31
7.30
7.25
7.24
7.23
1.99
1.99
2.99
4.01
1.01
7.45
7.40
7.35
7.30
7.25
7.20
Chemical Shift (ppm)
2.19
10.92
7.45
7.39
7.38
7.36
7.31
7.30
7.46
7.25
7.24
6.88
6.87
0.99
1.01
1.00
3.01
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

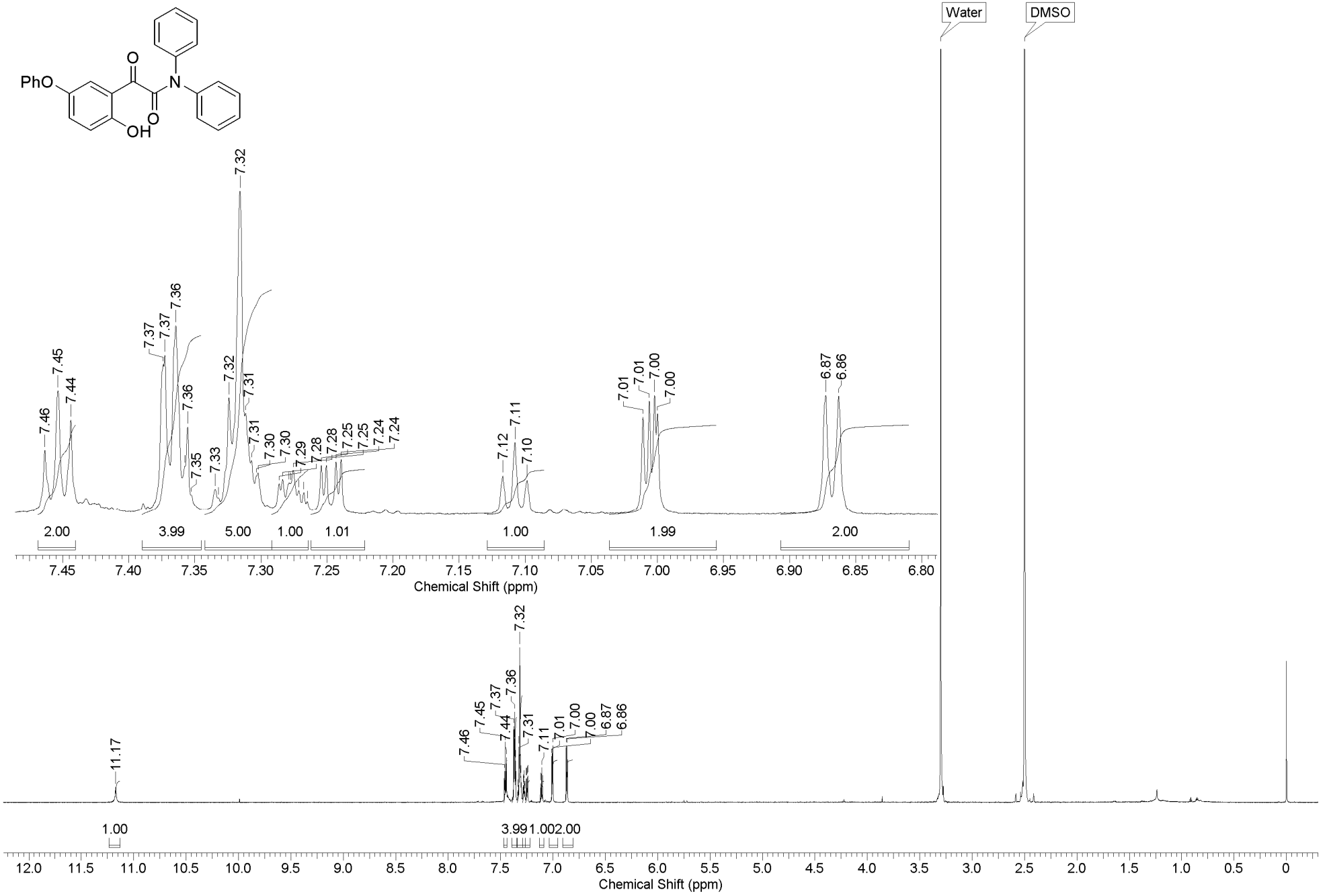

DMSO
129.50
129.29
129.08
128.36
128.15
127.96
126.74
126.25
Chemical Shift (ppm)
189.38
166.76
156.95
141.36
140.67
137.37
129.50
129.08
128.15
126.25
120.05
117.43
19.73
Chemical Shift (ppm)

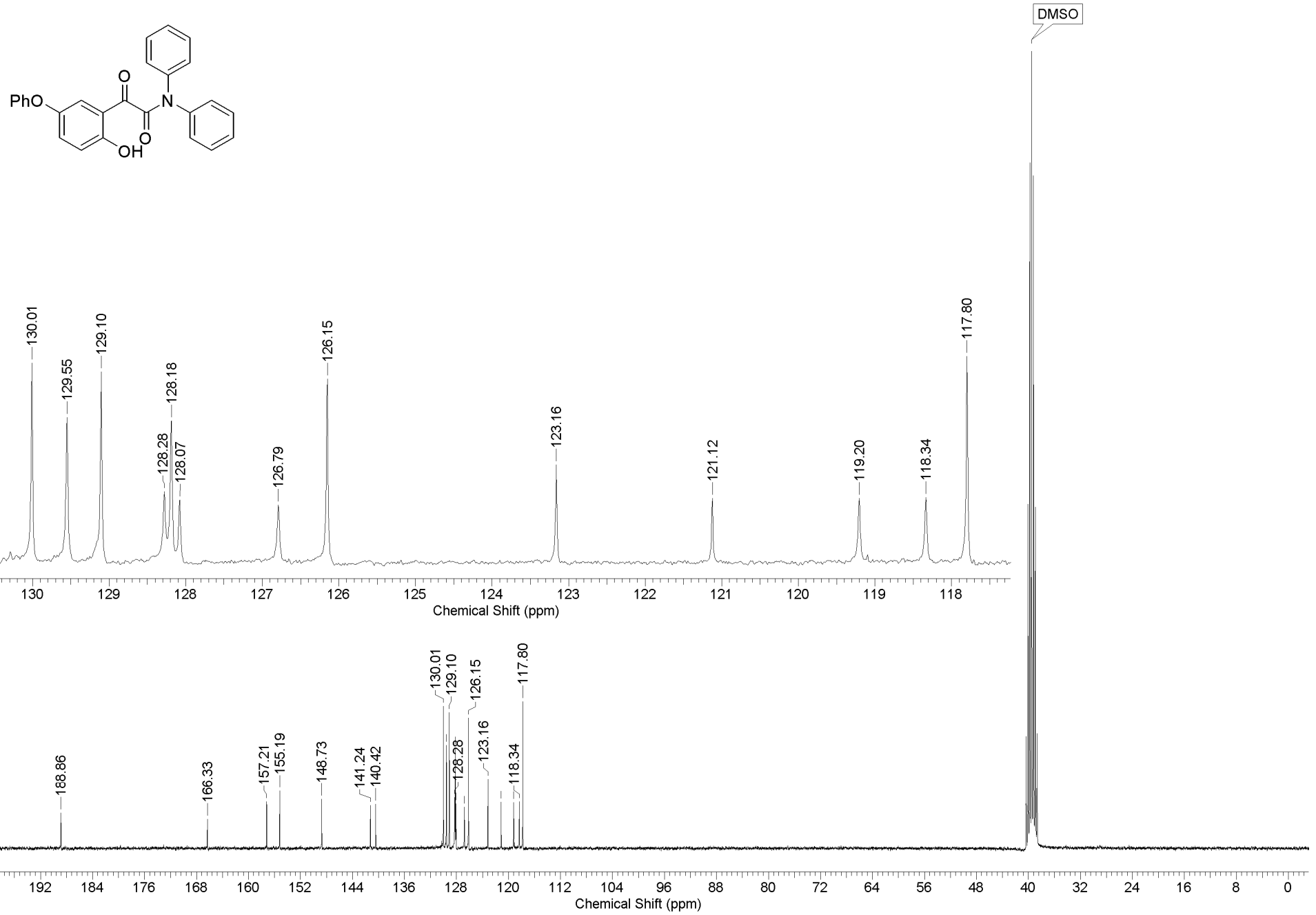

O
N
O
OH
Water
DMSO
7.82
7.82
7.81
7.81
7.75
7.75
7.56
7.55
7.48
7.47
7.46
7.43
7.43
7.42
7.41
7.40
7.39
7.38
7.34
7.33
7.32
7.31
7.25
7.24
7.23
1.00
1.01
2.01
1.99
6.01
4.00
1.01
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
7.09
7.08
1.00
7.100
7.075
7.050
7.025
Chemical Shift (ppm)
11.39
7.81
7.75
7.56
7.55
7.47
7.46
7.43
7.41
7.38
7.33
7.32
7.25
7.09
1.01
1.01
6.01
1.00
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

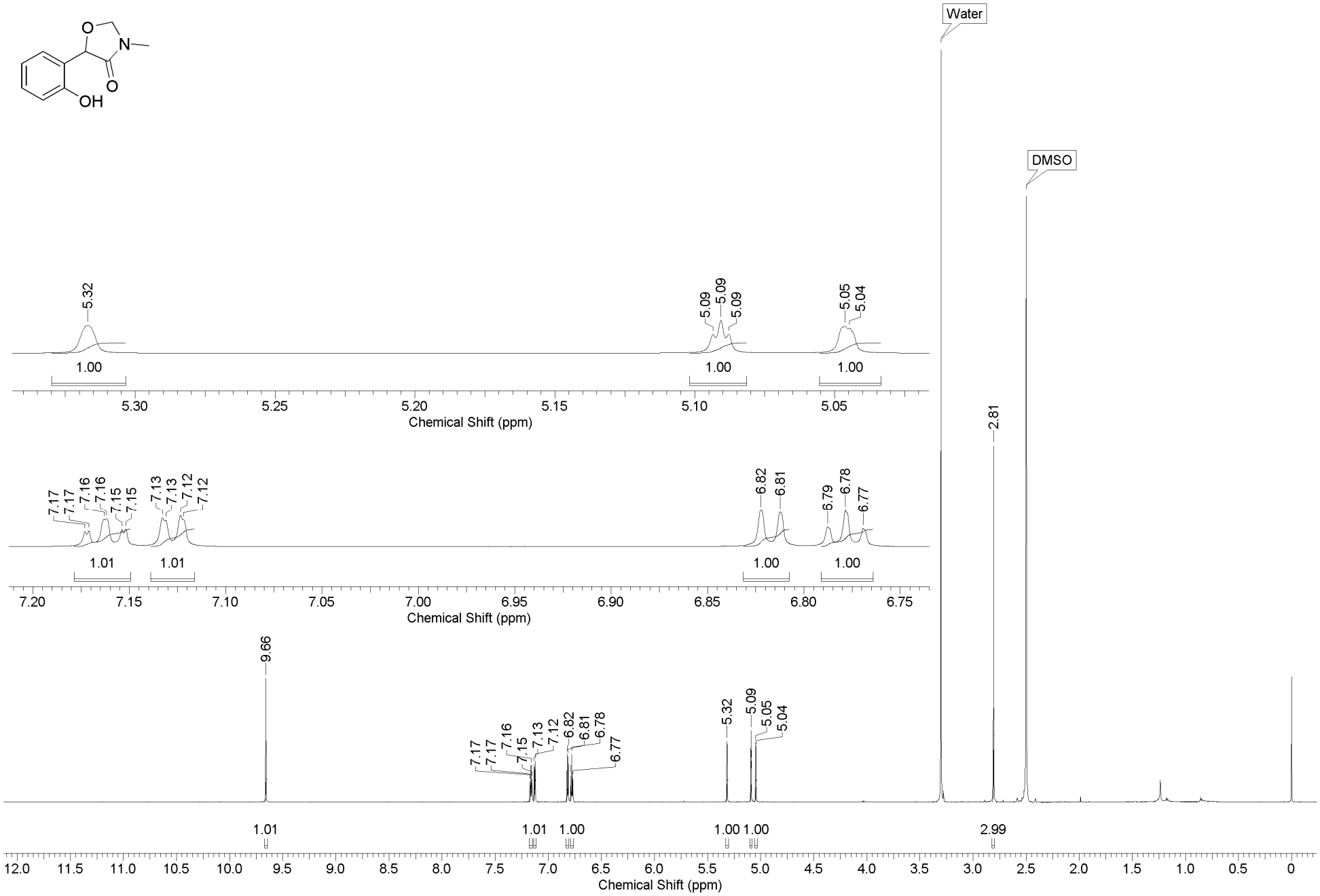

O
N
O
OH
DMSO
129.57
129.12
128.96
128.10
127.98
127.18
127.12
126.79
126.25
126.07
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
Chemical Shift (ppm)
189.14
166.68
158.38
141.33
140.69
138.78
134.74
131.64
129.57
129.12
128.96
128.10
126.25
126.07
120.95
118.22
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

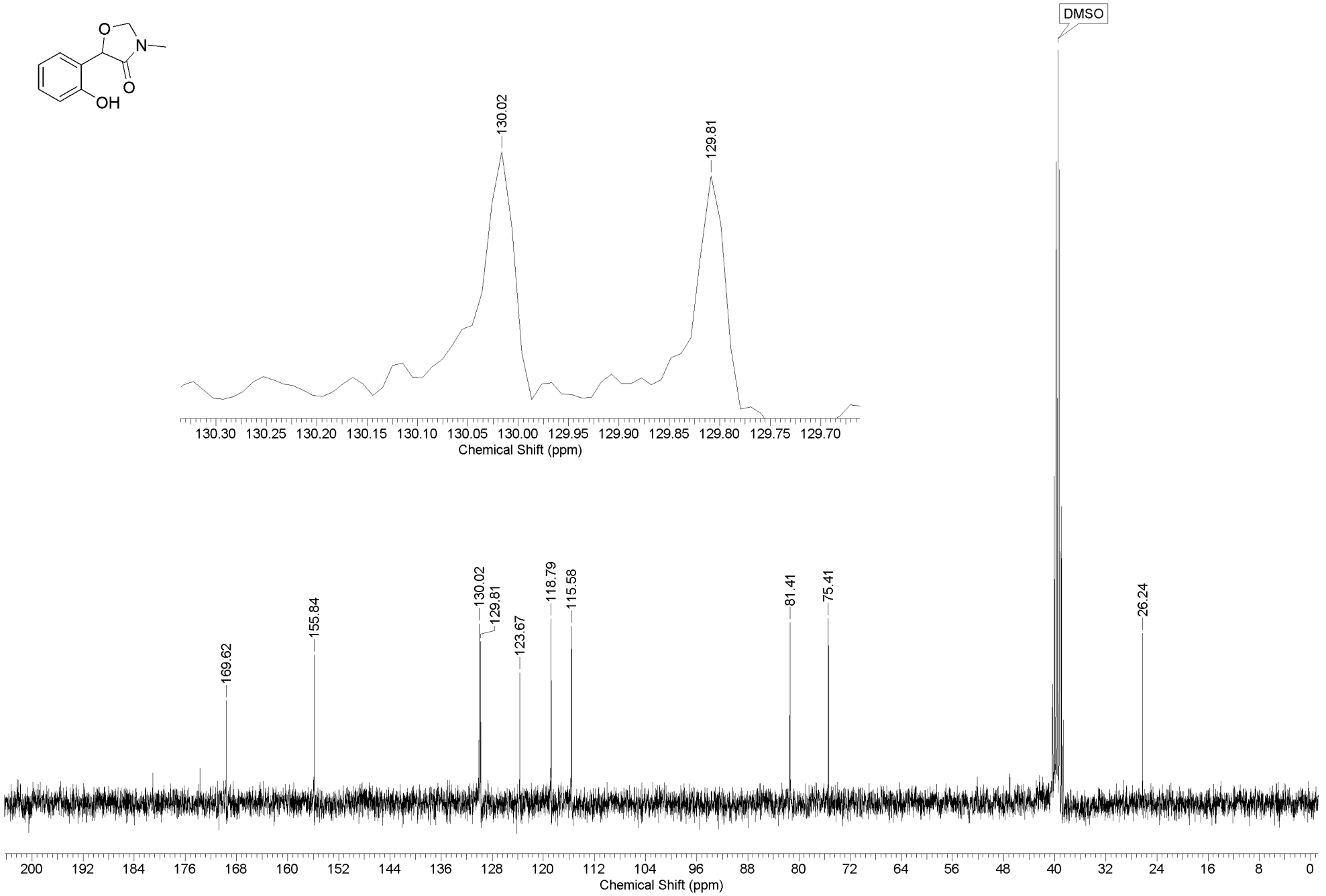

Cl
O
N
O
OH
Water
DMSO
7.33
7.46
7.45
7.44
7.44
7.39
7.39
7.38
7.53
7.52
7.51
7.51
7.47
7.34
7.33
7.32
7.32
7.31
7.27
7.27
7.26
7.26
7.26
7.25
7.25
7.01
7.00
1.00
3.01
1.99
4.99
1.00
1.00
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
7.00
Chemical Shift (ppm)
11.59
7.51
7.46
7.45
7.44
7.39
7.33
7.32
7.32
7.01
7.00
1.00
4.99
1.00
12.5
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

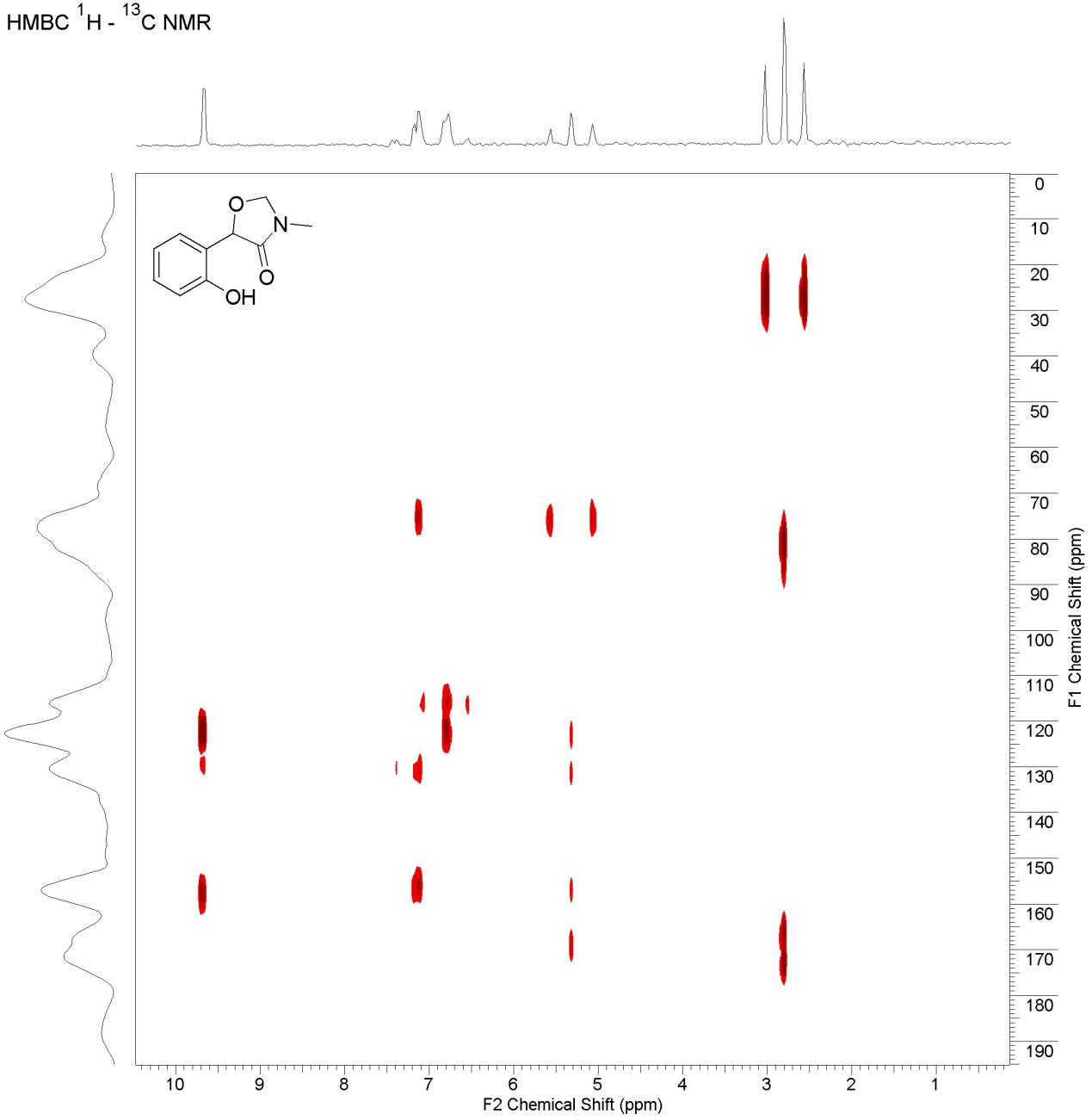

Cl
O
N
O
OH
DMSO
129.61
129.13
128.32
128.14
128.10
126.84
126.13
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
Chemical Shift (ppm)
188.01
166.16
157.59
141.15
140.40
135.91
129.61
129.13
128.10
126.13
123.45
121.86
119.64
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

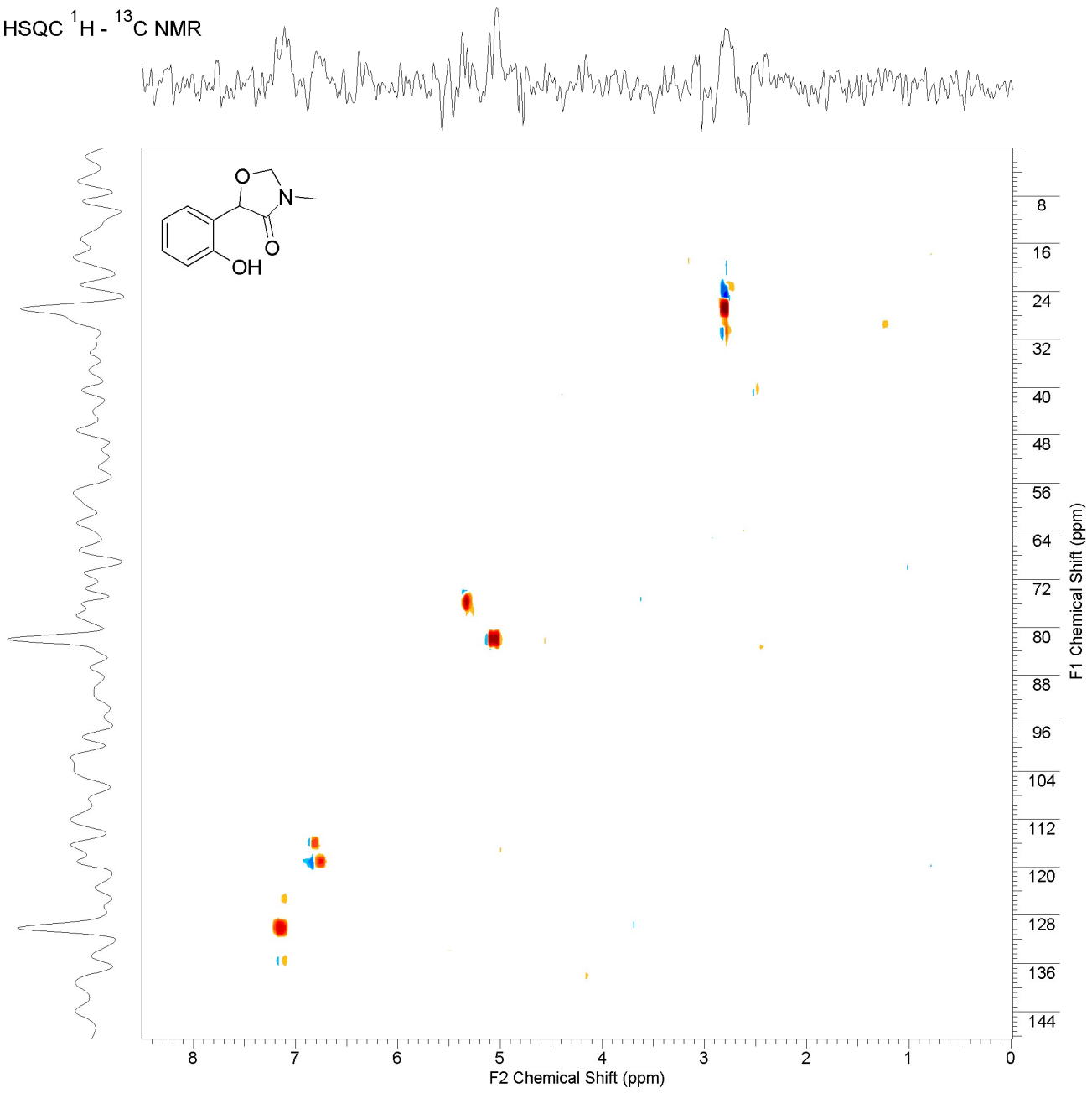

PhO
O
N
O
OH
Water
DMSO
7.46
7.45
7.44
7.37
7.37
7.36
7.36
7.35
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.28
7.25
7.25
7.24
7.24
7.12
7.11
7.10
7.01
7.01
7.00
7.00
6.87
6.86
2.00
3.99
5.00
1.00
1.01
1.00
1.99
2.00
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
7.00
6.95
6.90
6.85
6.80
Chemical Shift (ppm)
11.17
7.46
7.45
7.44
7.37
7.36
7.32
7.31
7.11
7.01
7.00
7.00
6.87
6.86
1.00
3.99
1.00
2.00
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

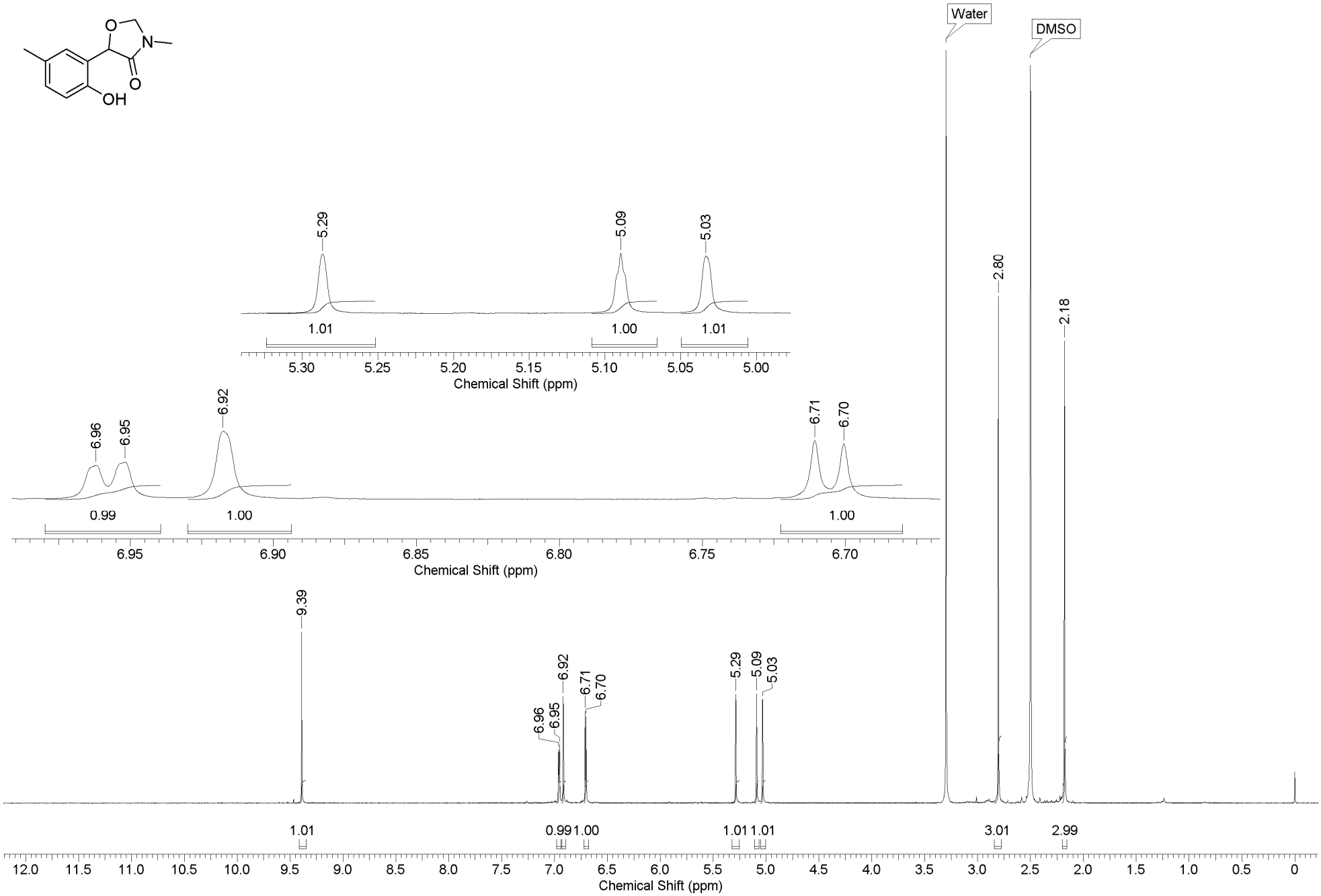

PhO
O
N
O
OH
DMSO
130.01
129.55
129.10
128.18
128.28
128.07
126.79
126.15
123.16
121.12
119.20
118.34
117.80
Chemical Shift (ppm)
188.86
166.33
157.21
155.19
148.73
141.24
140.42
130.01
129.10
128.28
126.15
123.16
118.34
117.80
Chemical Shift (ppm)

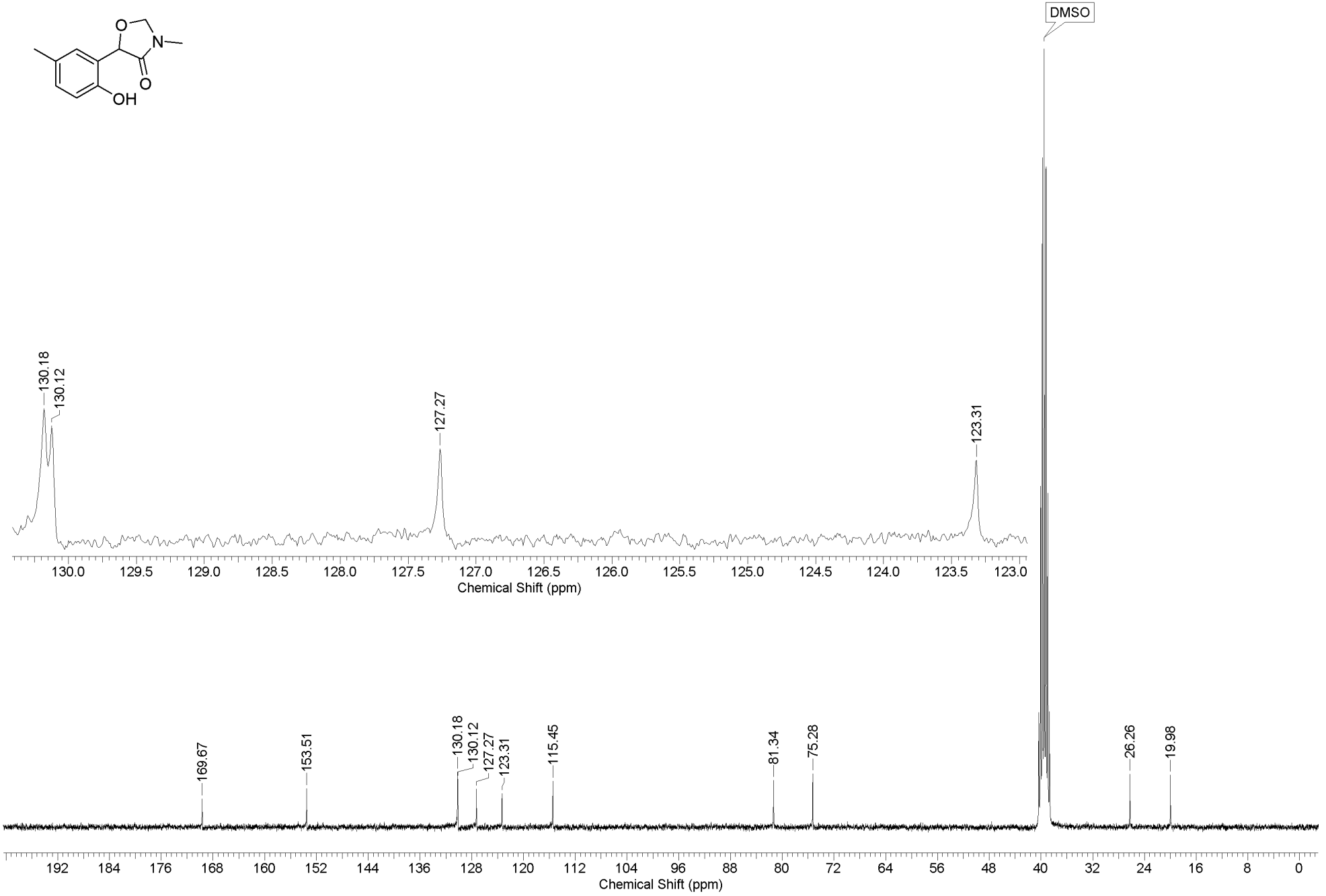

Water
DMSO
2.81
9.66
5.32
5.09
5.09
5.09
5.05
5.04
7.17
7.17
7.16
7.16
7.15
7.15
7.13
7.13
7.12
7.12
6.82
6.81
6.79
6.78
6.77
1.01
1.00
1.00
1.00
1.00
2.99
Chemical Shift (ppm)

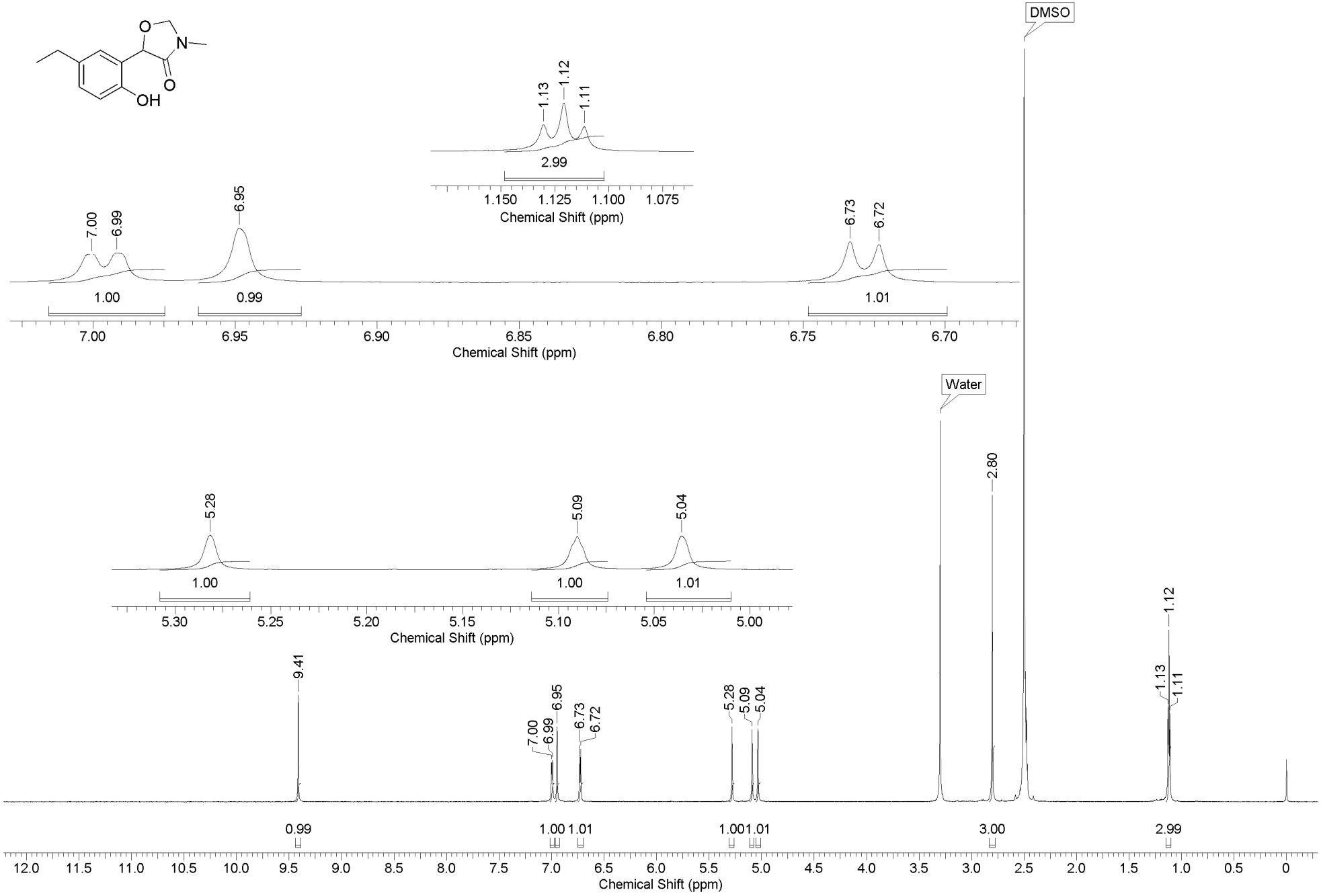

O
N
O
OH
130.02
129.81
130.30 130.25 130.20 130.15 130.10 130.05 130.00 129.95 129.90 129.85 129.80 129.75 129.70
Chemical Shift (ppm)
DMSO
169.62
155.84
130.02
129.81
123.67
118.79
115.58
81.41
75.41
26.24
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

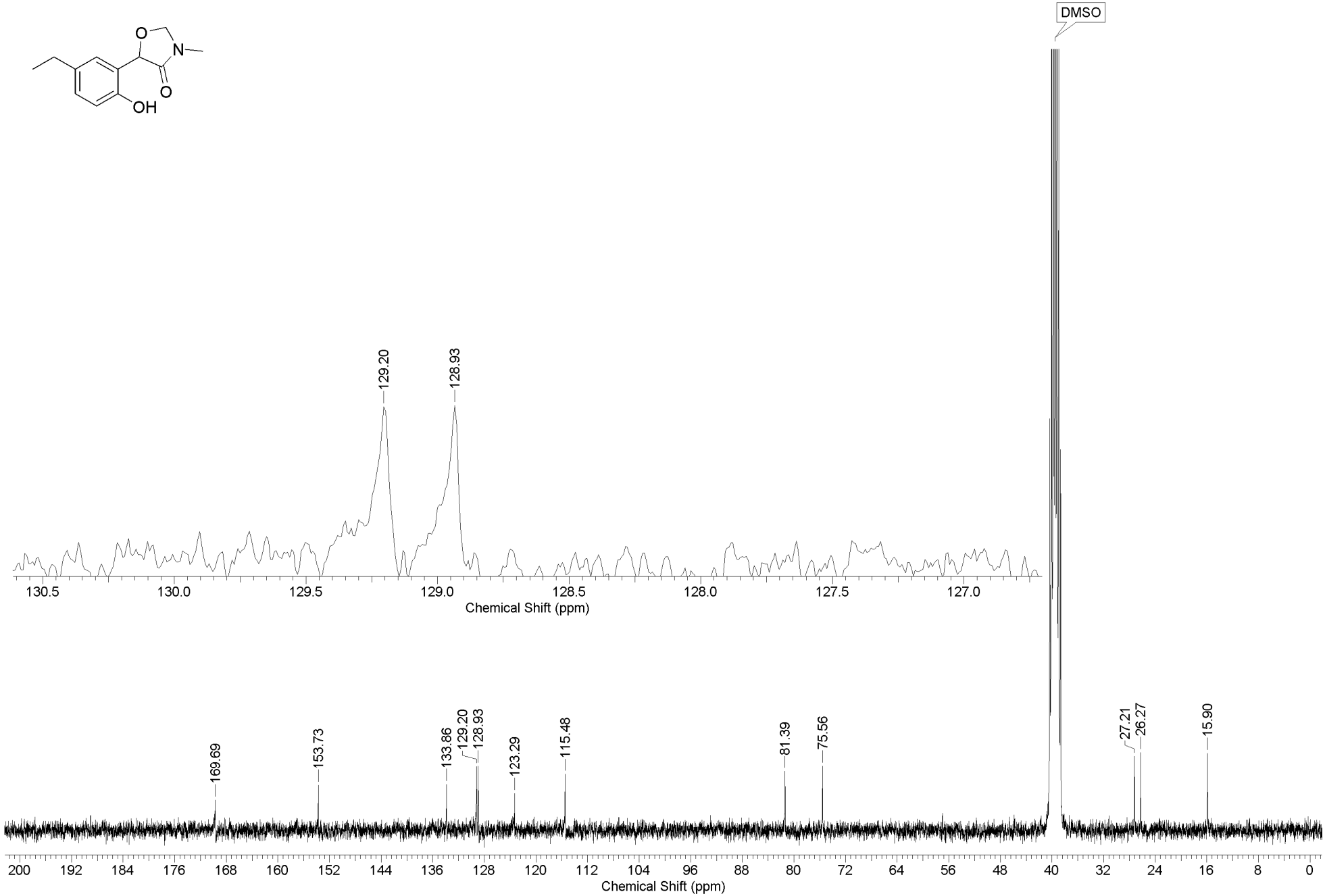

HMBC ^{1}H - ^{13}C NMR
O
N
O
OH
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
F1 Chemical Shift (ppm)
10
9
8
7
6
5
4
3
2
1
F2 Chemical Shift (ppm)

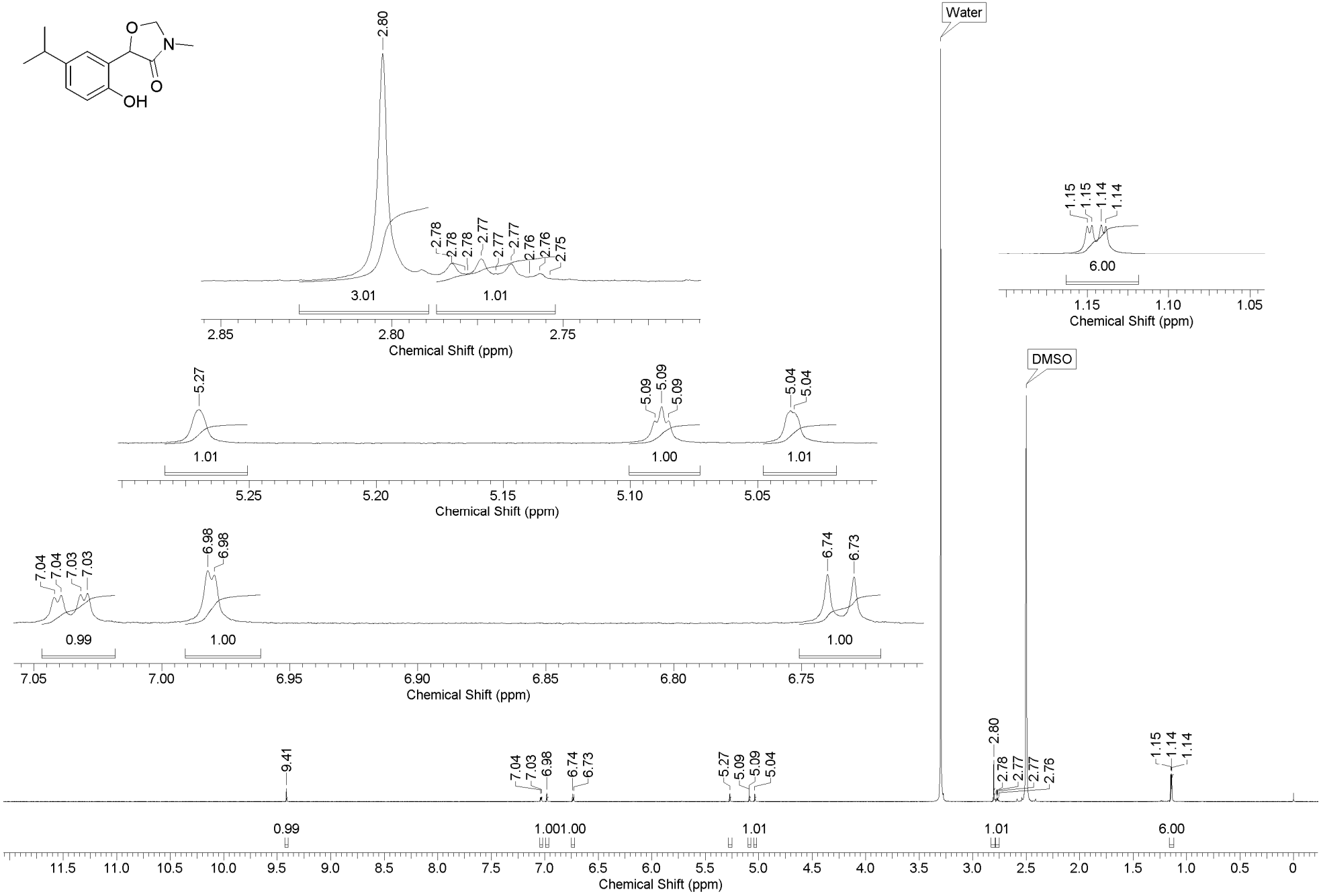

HSQC ^{1}H - ^{13}C NMR
O
N
O
OH
8
16
24
32
40
48
56
64
72
80
88
96
104
112
120
128
136
144
F1 Chemical Shift (ppm)
8
7
6
5
4
3
2
1
0
F2 Chemical Shift (ppm)

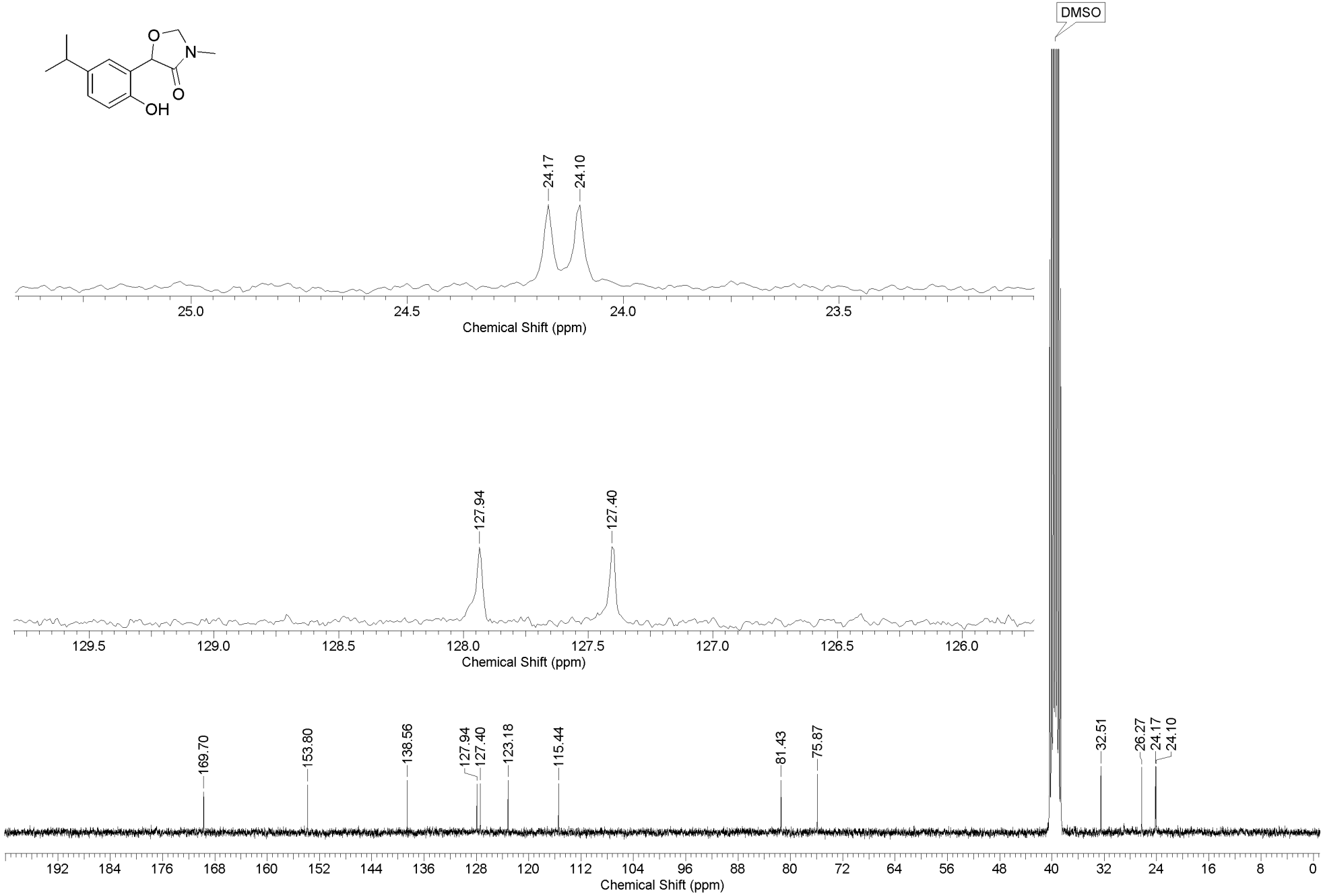

O
N
O
OH
Water
DMSO
2.80
2.18
5.29
5.09
5.03
1.01
1.00
1.01
5.30
5.25
5.20
5.15
5.10
5.05
5.00
Chemical Shift (ppm)
6.96
6.95
6.92
6.71
6.70
0.99
1.00
1.00
6.95
6.90
6.85
6.80
6.75
6.70
Chemical Shift (ppm)
9.39
6.92
6.71
6.70
6.96
6.95
5.29
5.09
5.03
1.01
0.99
1.00
1.01
1.01
3.01
2.99
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

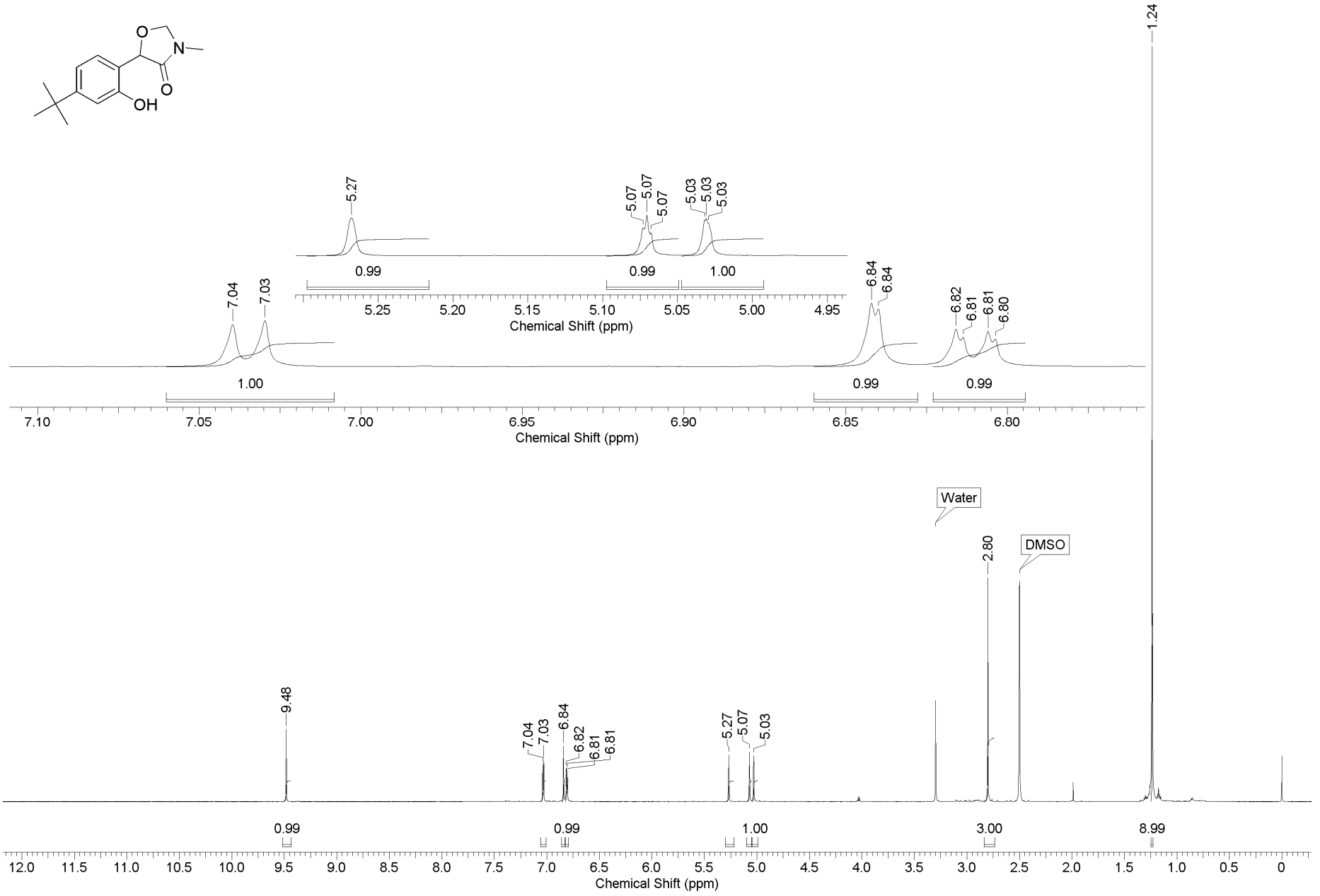

O
N
O
OH
DMSO
130.18
130.12
127.27
123.31
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
125.5
125.0
124.5
124.0
123.5
123.0
Chemical Shift (ppm)
169.67
153.51
130.18
130.12
127.27
123.31
115.45
81.34
75.28
26.26
19.98
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

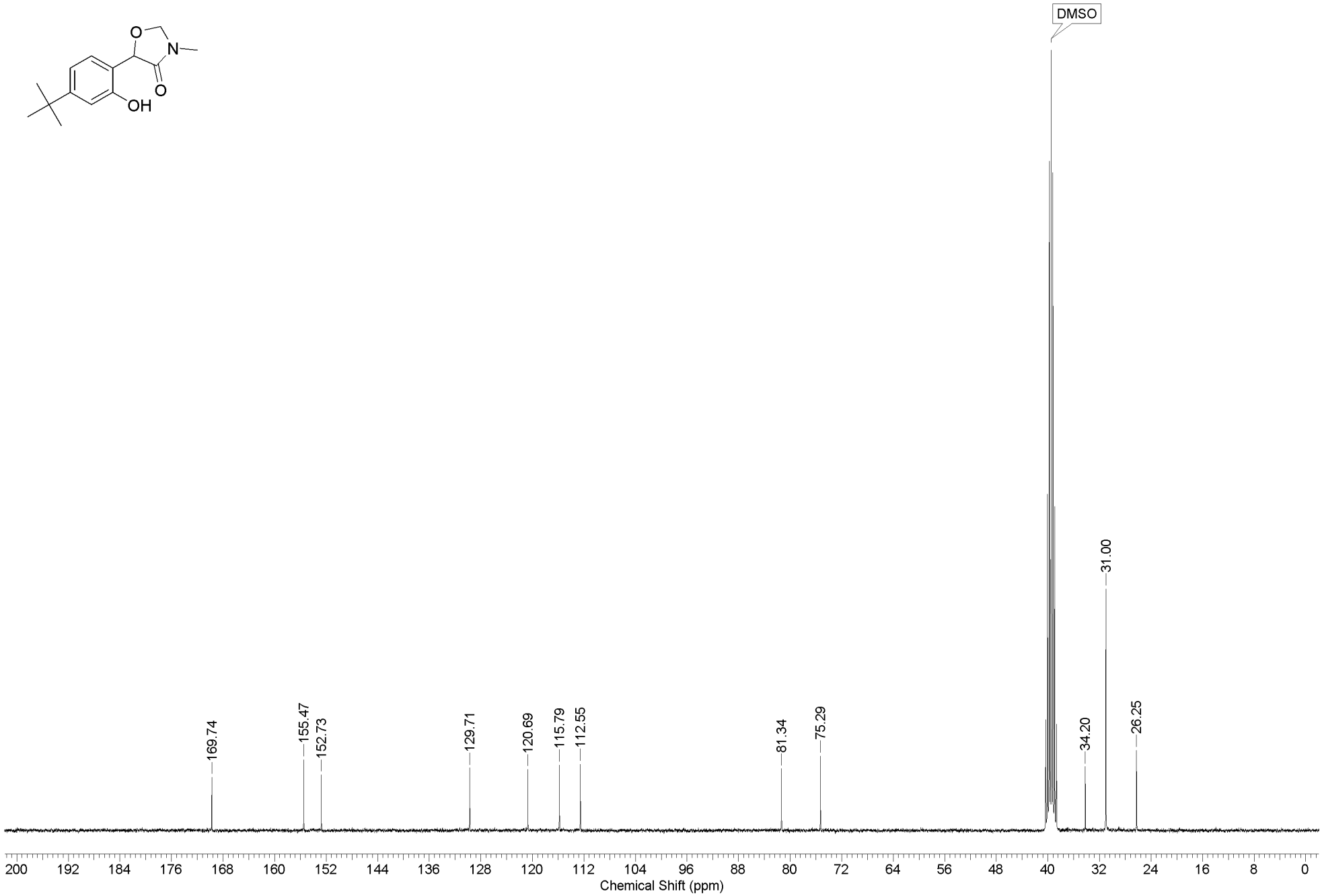

O
N
O
OH
DMSO
Water
2.80
9.41
7.00
6.99
6.95
6.73
6.72
5.28
5.09
5.04
1.13
1.12
1.11
0.99
1.00
1.01
1.00
1.01
3.00
2.99
Chemical Shift (ppm)

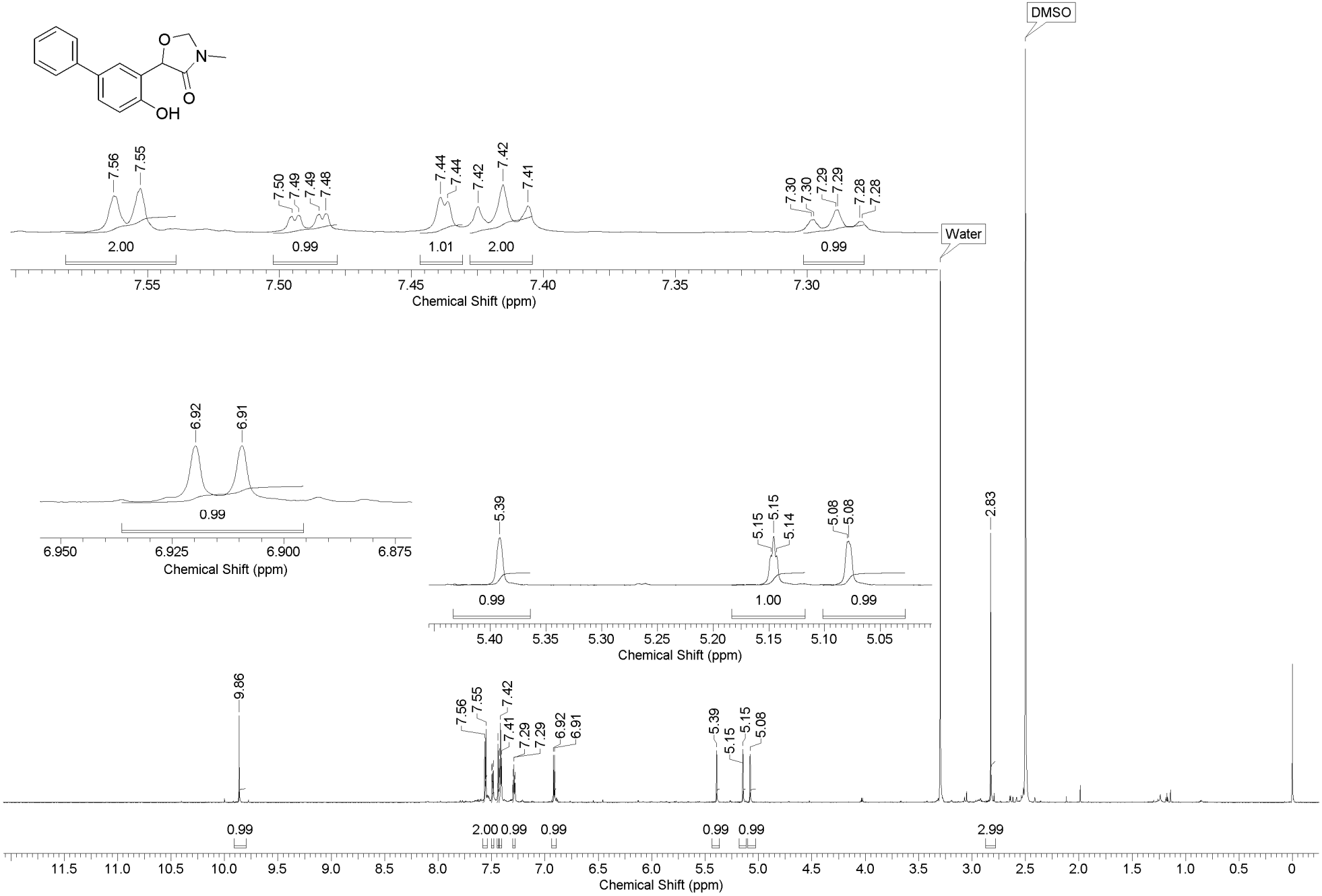

O
N
O
OH
DMSO
129.20
128.93
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
Chemical Shift (ppm)
169.69
153.73
133.86
129.20
128.93
123.29
115.48
81.39
75.56
27.21
26.27
15.90
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

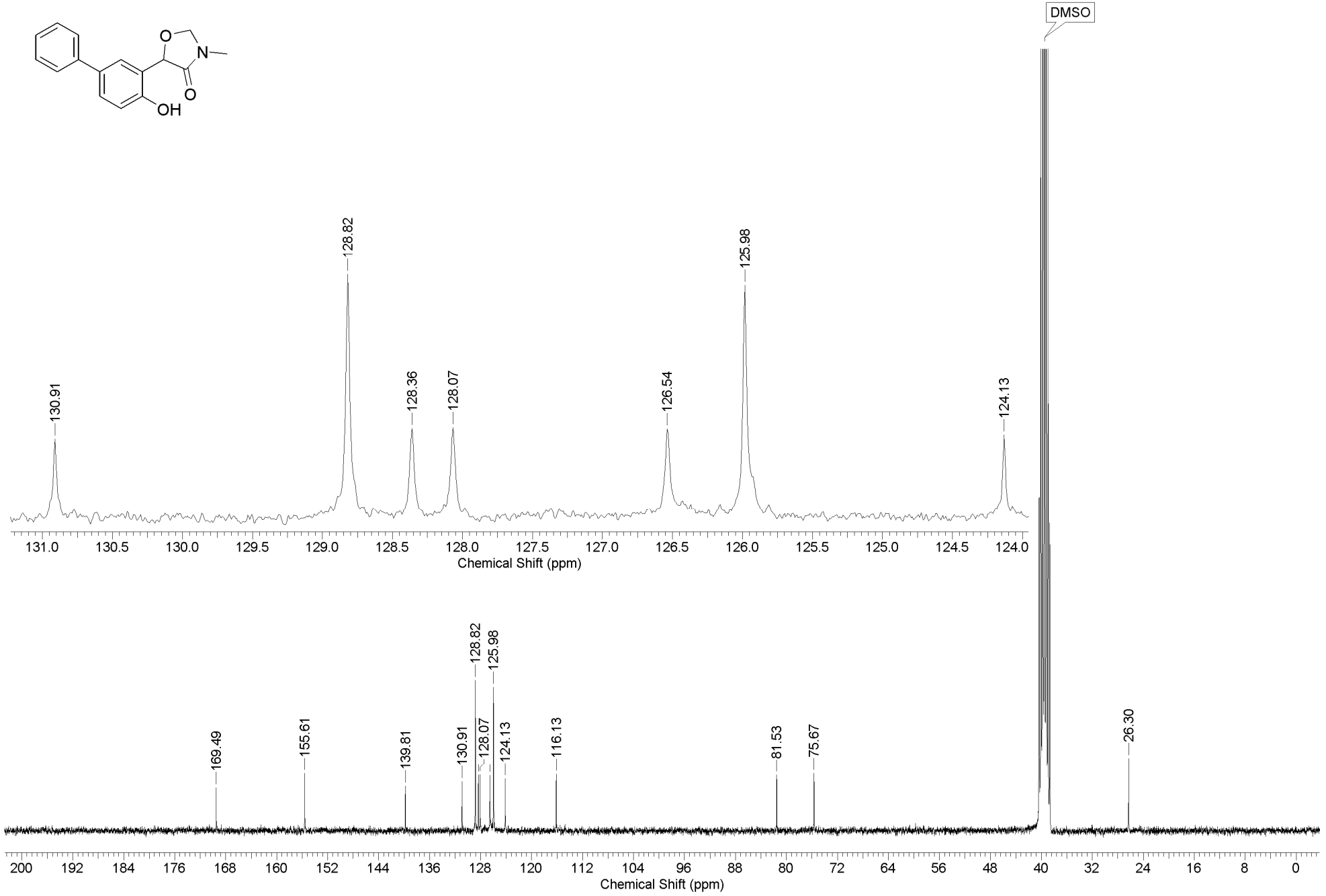

O
N
O
OH
Water
DMSO
2.80
2.78
2.78
2.78
2.77
2.77
2.77
2.76
2.76
2.75
3.01
1.01
2.85
2.80
2.75
Chemical Shift (ppm)
1.15
1.15
1.14
1.14
6.00
1.15
1.10
1.05
Chemical Shift (ppm)
5.27
5.09
5.09
5.09
5.04
5.04
1.01
1.00
1.01
5.25
5.20
5.15
5.10
5.05
Chemical Shift (ppm)
7.04
7.04
7.03
7.03
6.98
6.98
6.74
6.73
0.99
1.00
1.00
7.05
7.00
6.95
6.90
6.85
6.80
6.75
Chemical Shift (ppm)
9.41
7.04
7.03
6.98
6.74
6.73
5.27
5.09
5.09
5.04
2.80
2.78
2.77
2.77
2.76
1.15
1.14
1.14
0.99
1.00
1.00
1.01
1.01
6.00
11.5
11.0
10.5
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
Chemical Shift (ppm)

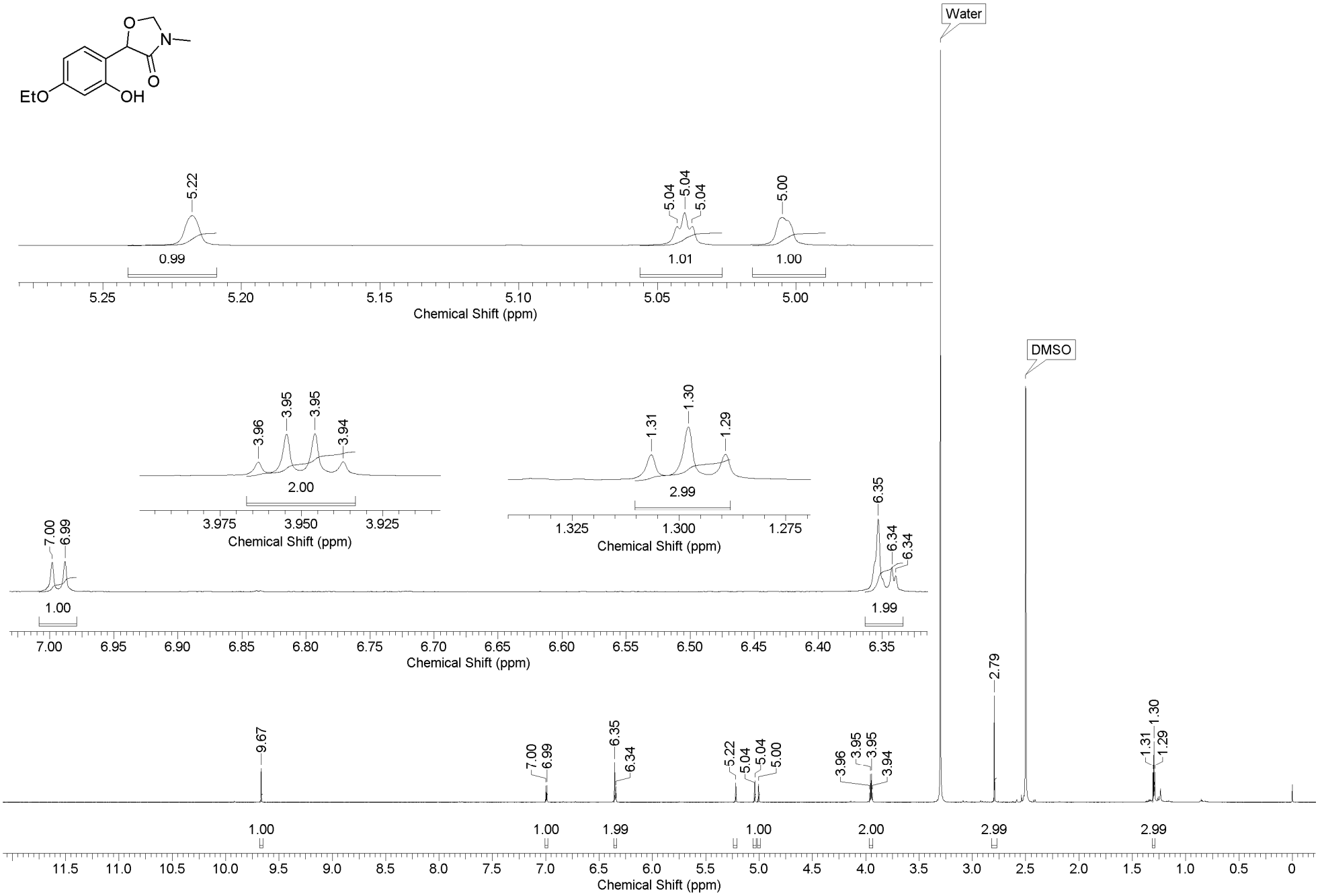

O
N
O
OH
DMSO
24.17
24.10
25.0
24.5
24.0
23.5
Chemical Shift (ppm)
127.94
127.40
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
Chemical Shift (ppm)
169.70
153.80
138.56
127.94
127.40
123.18
115.44
81.43
75.87
32.51
26.27
24.17
24.10
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

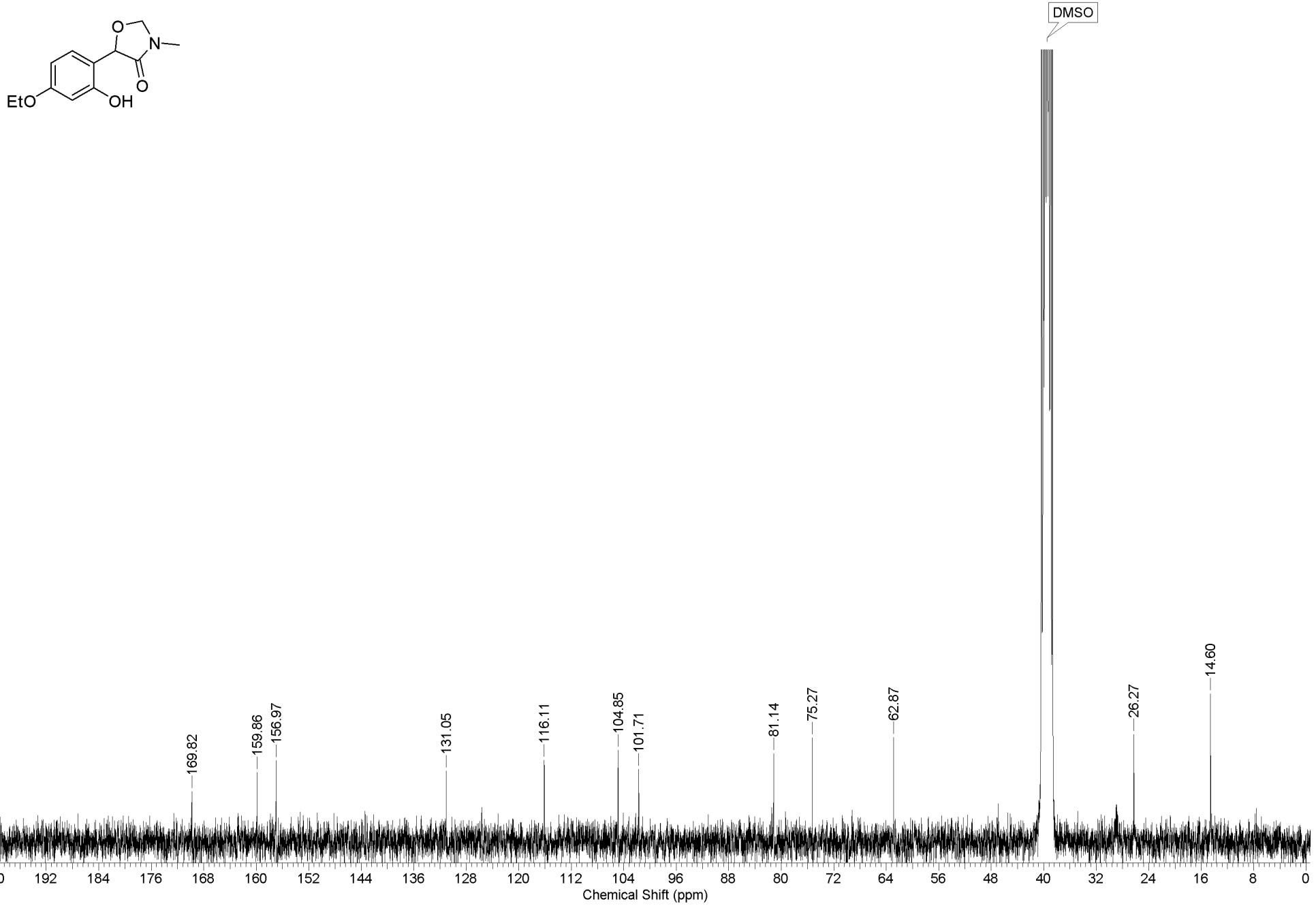

O
N
O
OH
1.24
5.27
5.07
5.07
5.07
5.03
5.03
5.03
0.99
0.99
1.00
5.25
5.20
5.15
5.10
5.05
5.00
4.95
Chemical Shift (ppm)
7.04
7.03
6.84
6.84
6.82
6.81
6.81
6.80
1.00
0.99
0.99
7.10
7.05
7.00
6.95
6.90
6.85
6.80
Chemical Shift (ppm)
Water
2.80
DMSO
9.48
7.04
7.03
6.84
6.82
6.81
6.81
5.27
5.07
5.03
0.99
0.99
1.00
3.00
8.99
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

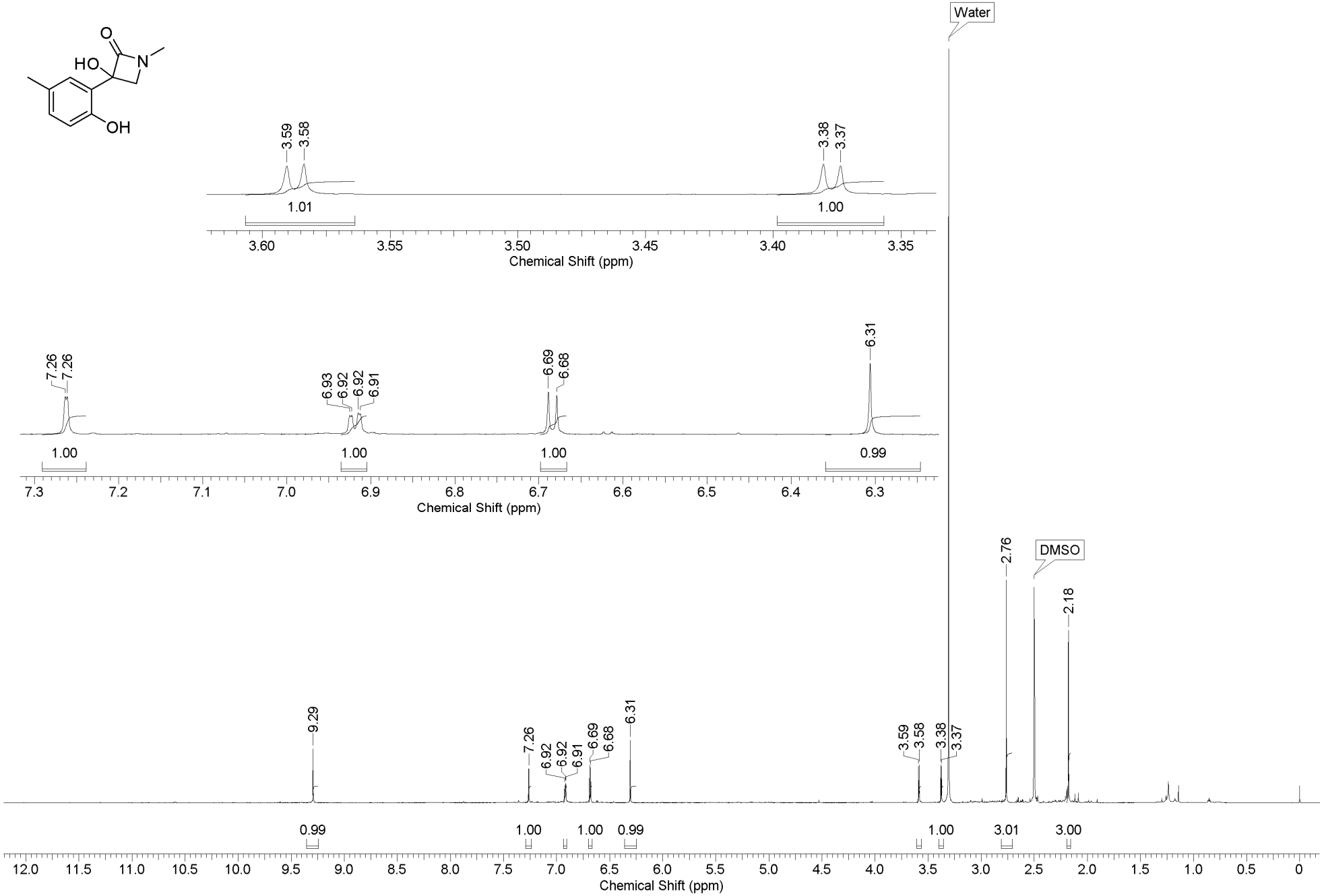

O
N
O
OH
DMSO
169.74
155.47
152.73
129.71
120.69
115.79
112.55
81.34
75.29
34.20
31.00
26.25
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

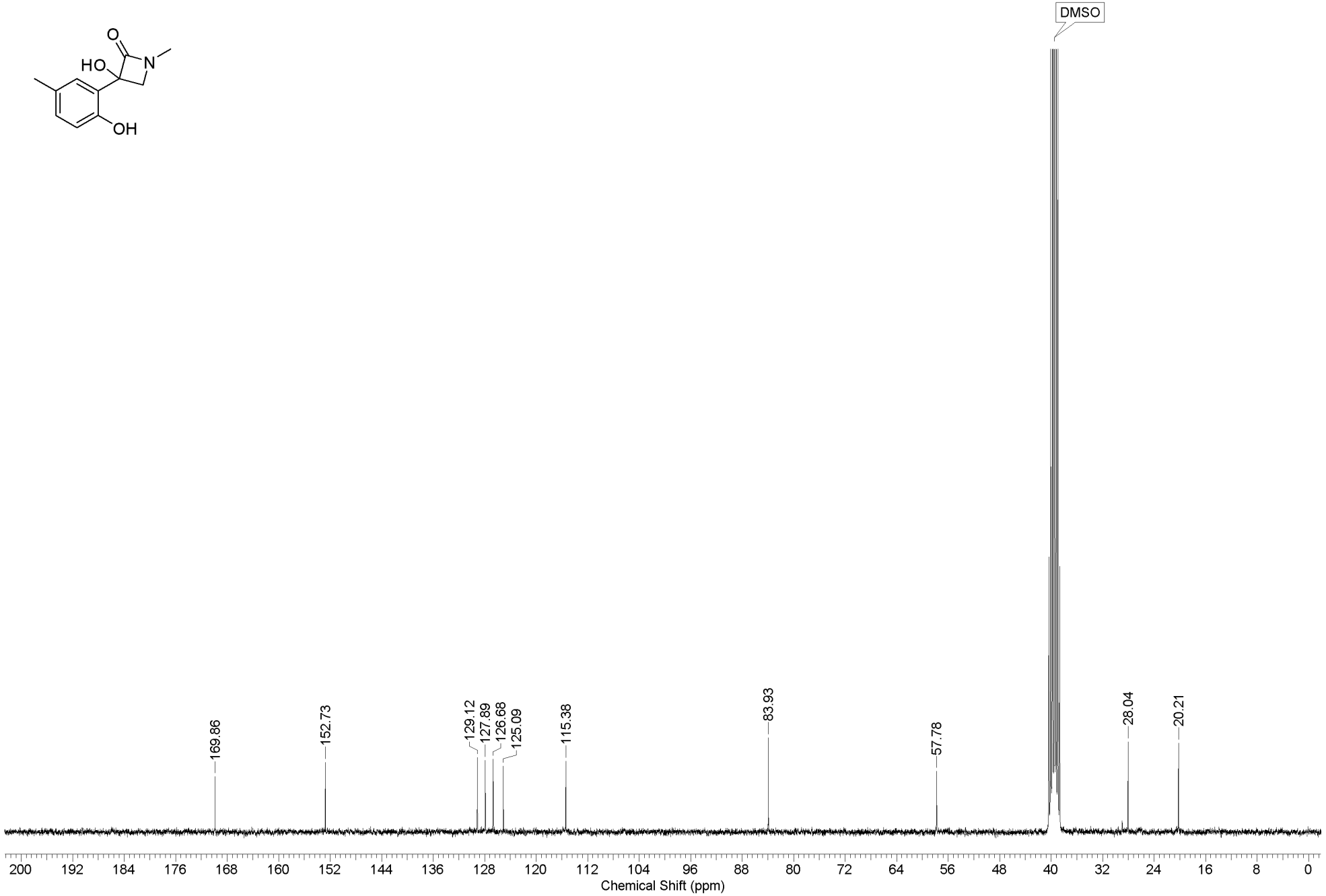

DMSO
Water
9.86
7.56
7.55
7.50
7.49
7.49
7.48
7.44
7.44
7.42
7.42
7.41
7.30
7.30
7.29
7.29
7.28
7.28
6.92
6.91
5.39
5.15
5.15
5.14
5.08
5.08
2.83
2.00
0.99
1.01
2.00
0.99
0.99
1.00
0.99
0.99
2.00
0.99
0.99
0.99
0.99
2.99
Chemical Shift (ppm)

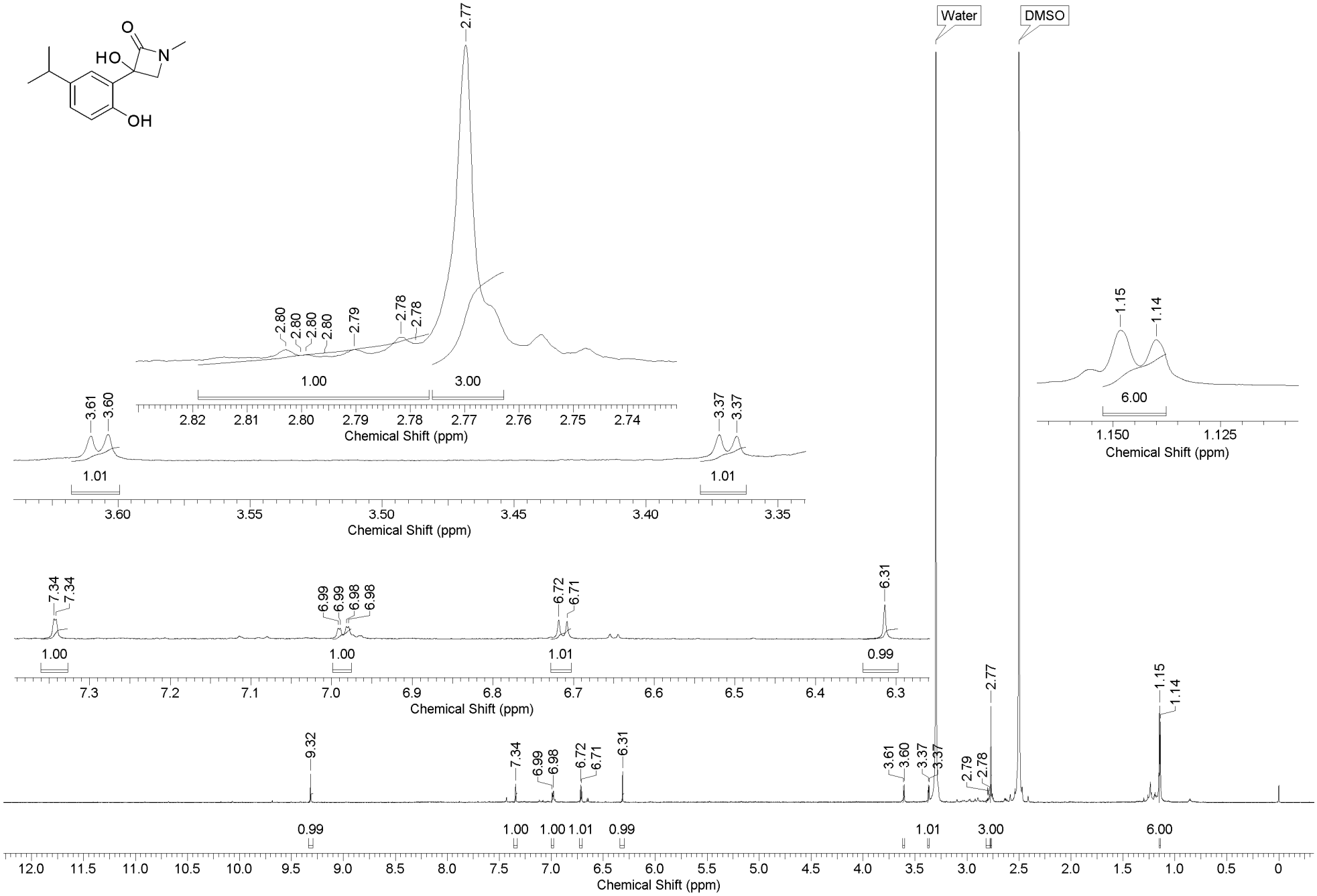

O
N
O
OH
DMSO
130.91
128.82
128.36
128.07
126.54
125.98
124.13
131.0
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
125.5
125.0
124.5
124.0
Chemical Shift (ppm)
169.49
155.61
139.81
130.91
128.82
128.07
125.98
124.13
116.13
81.53
75.67
26.30
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

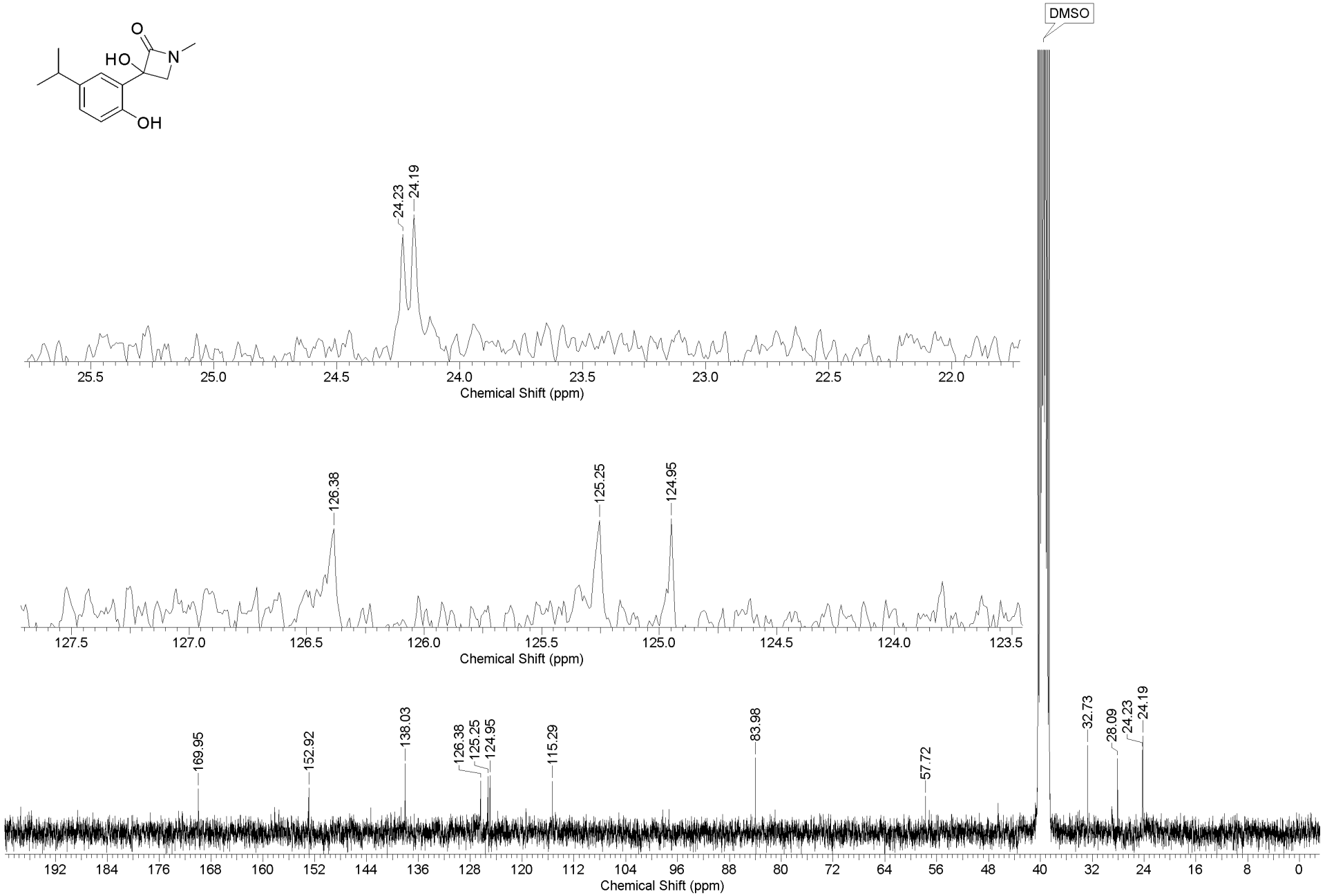

EtO
OH
O
N
O
Water
DMSO
5.22
5.04
5.04
5.04
5.00
0.99
1.01
1.00
5.25
5.20
5.15
5.10
5.05
5.00
Chemical Shift (ppm)
3.96
3.95
3.95
3.94
2.00
3.975
3.950
3.925
Chemical Shift (ppm)
1.31
1.30
1.29
2.99
1.325
1.300
1.275
Chemical Shift (ppm)
7.00
6.99
6.35
6.34
6.34
1.00
1.99
7.00
6.95
6.90
6.85
6.80
6.75
6.70
6.65
6.60
6.55
6.50
6.45
6.40
6.35
Chemical Shift (ppm)
9.67
7.00
6.99
6.35
6.34
5.22
5.04
5.04
5.00
3.96
3.95
3.95
3.94
2.79
1.31
1.30
1.29
1.00
1.00
1.99
1.00
2.00
2.99
2.99
11.5
11.0
10.5
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
Chemical Shift (ppm)

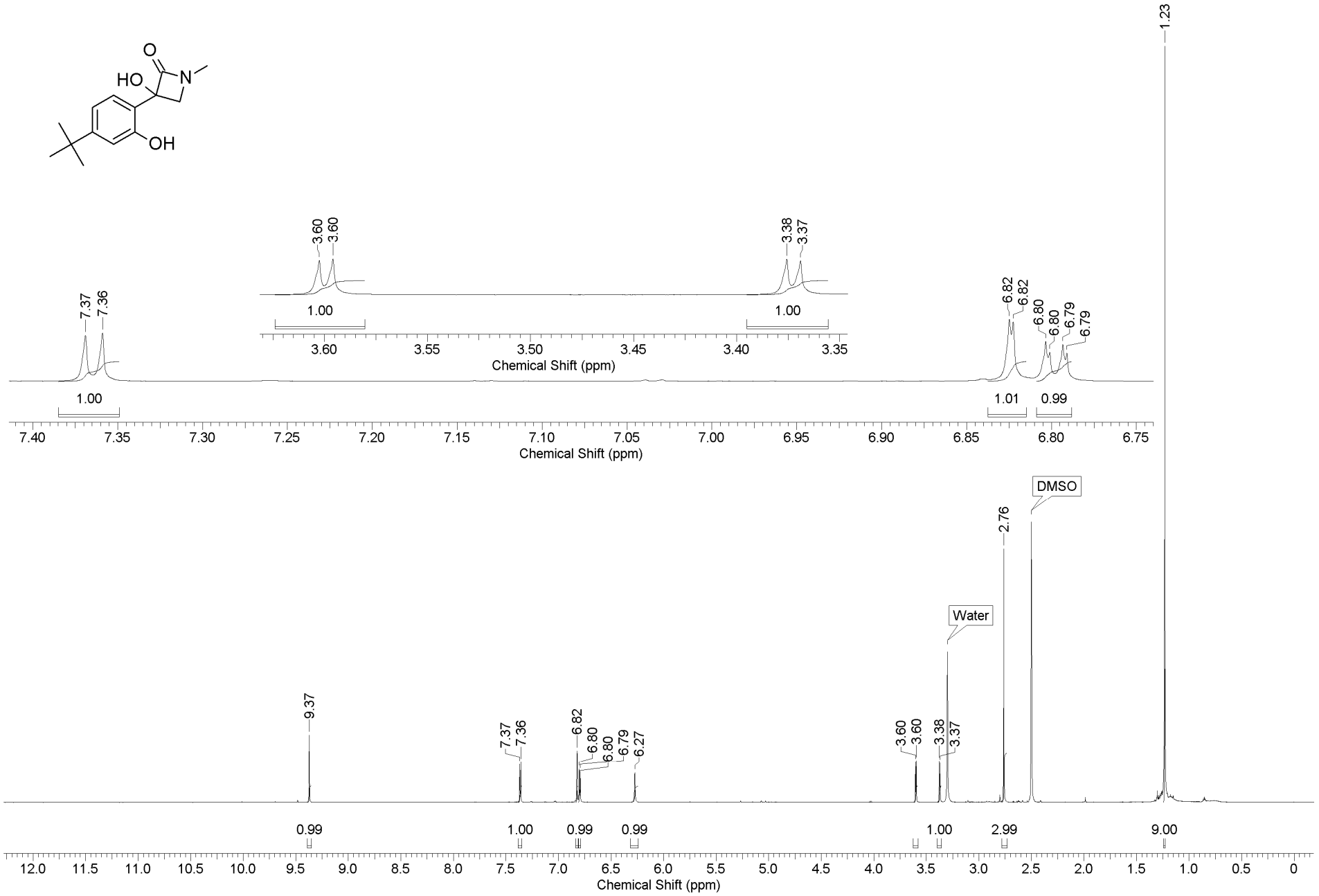

O
N
O
EtO
OH
DMSO
169.82
159.86
156.97
131.05
116.11
104.85
101.71
81.14
75.27
62.87
26.27
14.60
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

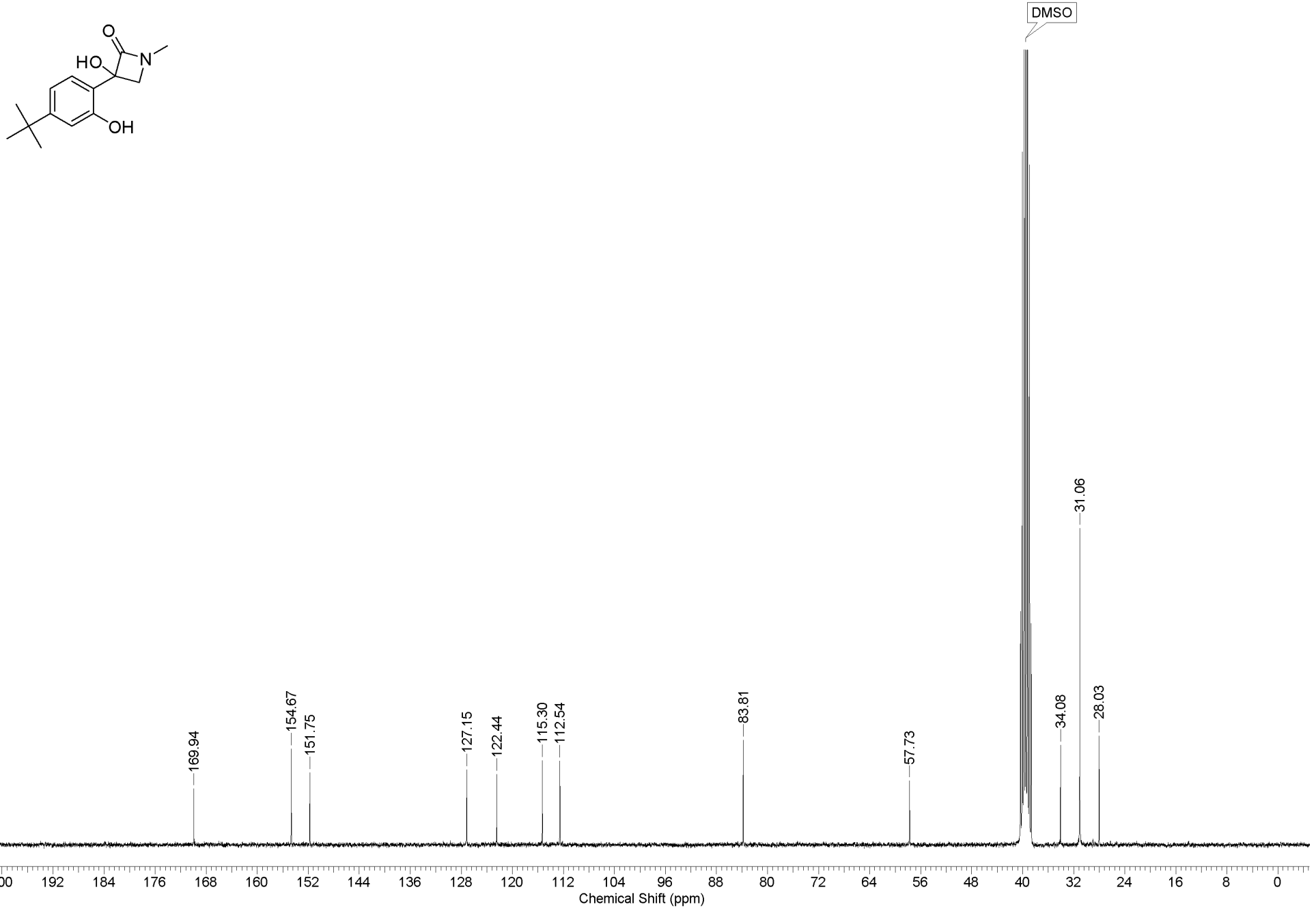

HO
O
N
OH
Water
3.59
3.58
3.38
3.37
1.01
1.00
3.60
3.55
3.50
3.45
3.40
3.35
Chemical Shift (ppm)
7.26
7.26
6.93
6.92
6.92
6.91
6.69
6.68
6.31
1.00
1.00
1.00
0.99
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
6.4
6.3
Chemical Shift (ppm)
2.76
DMSO
2.18
9.29
7.26
6.92
6.92
6.91
6.69
6.68
6.31
3.59
3.58
3.38
3.37
0.99
1.00
1.00
0.99
1.00
3.01
3.00
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)


O
HO
N
OH
DMSO
169.86
152.73
129.12
127.89
126.68
125.09
115.38
83.93
57.78
28.04
20.21
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

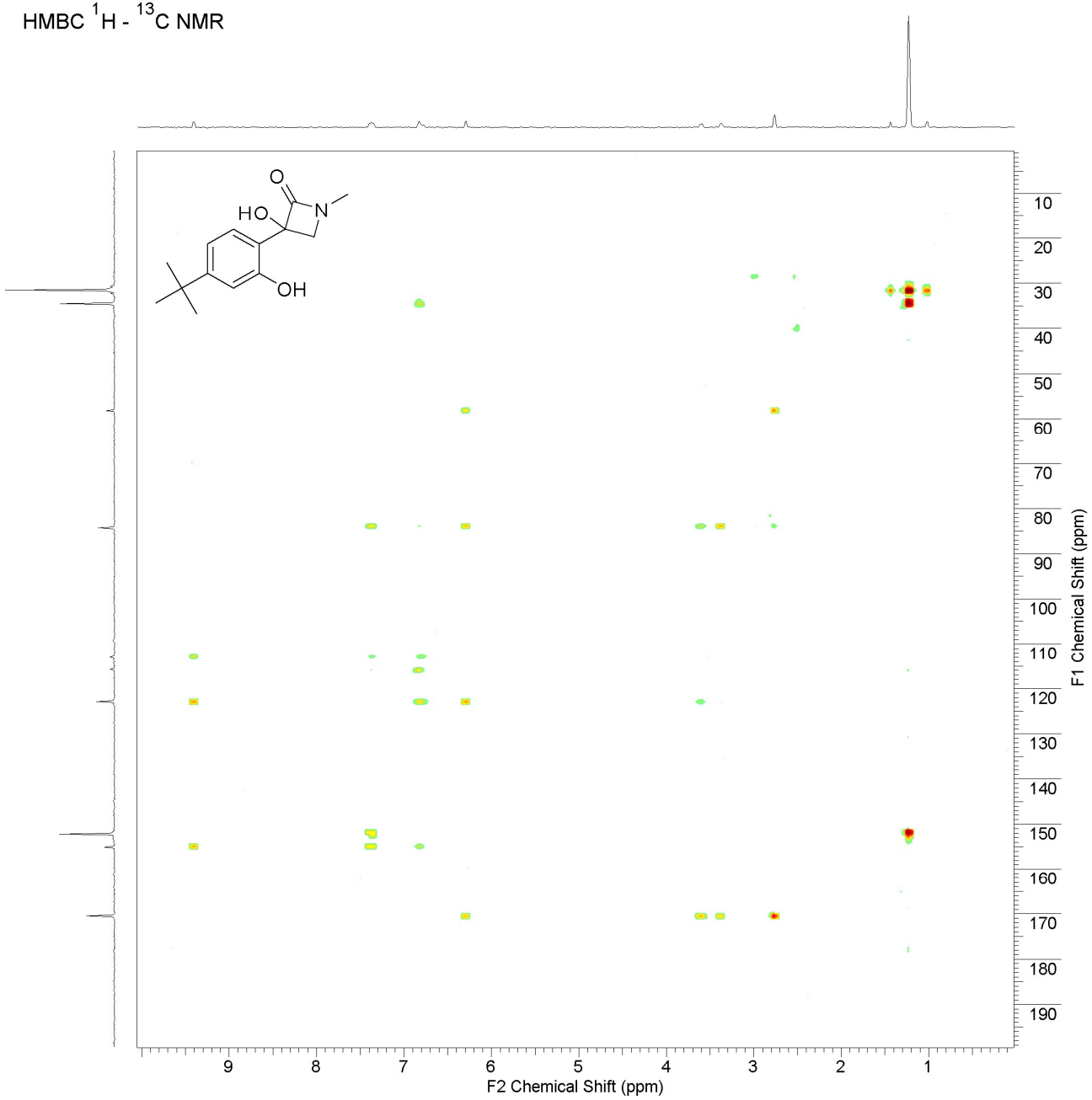

HO
O
N
OH
Water
DMSO
Chemical Shift (ppm)


O
HO
N
OH
DMSO
24.23
24.19
25.5
25.0
24.5
24.0
23.5
23.0
22.5
22.0
Chemical Shift (ppm)
126.38
125.25
124.95
127.5
127.0
126.5
126.0
125.5
125.0
124.5
124.0
123.5
Chemical Shift (ppm)
169.95
152.92
138.03
126.38
125.25
124.95
115.29
83.98
57.72
32.73
28.09
24.23
24.19
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

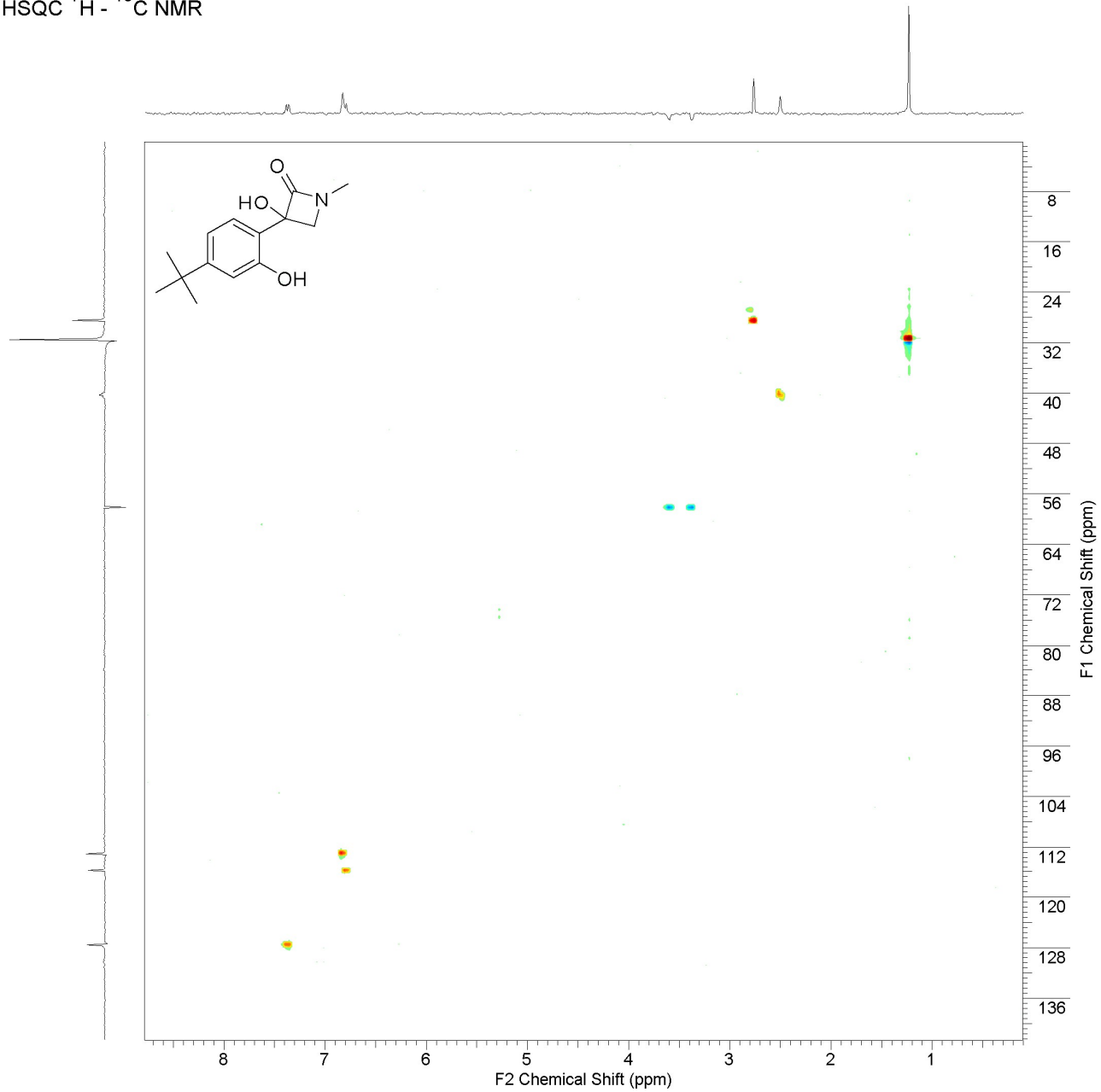

HO
O
N
OH
1.23
DMSO
2.76
Water
9.37
7.37
7.36
6.82
6.80
6.80
6.79
6.27
3.60
3.60
3.38
3.37
0.99
1.00
0.99
0.99
1.00
2.99
9.00
Chemical Shift (ppm)

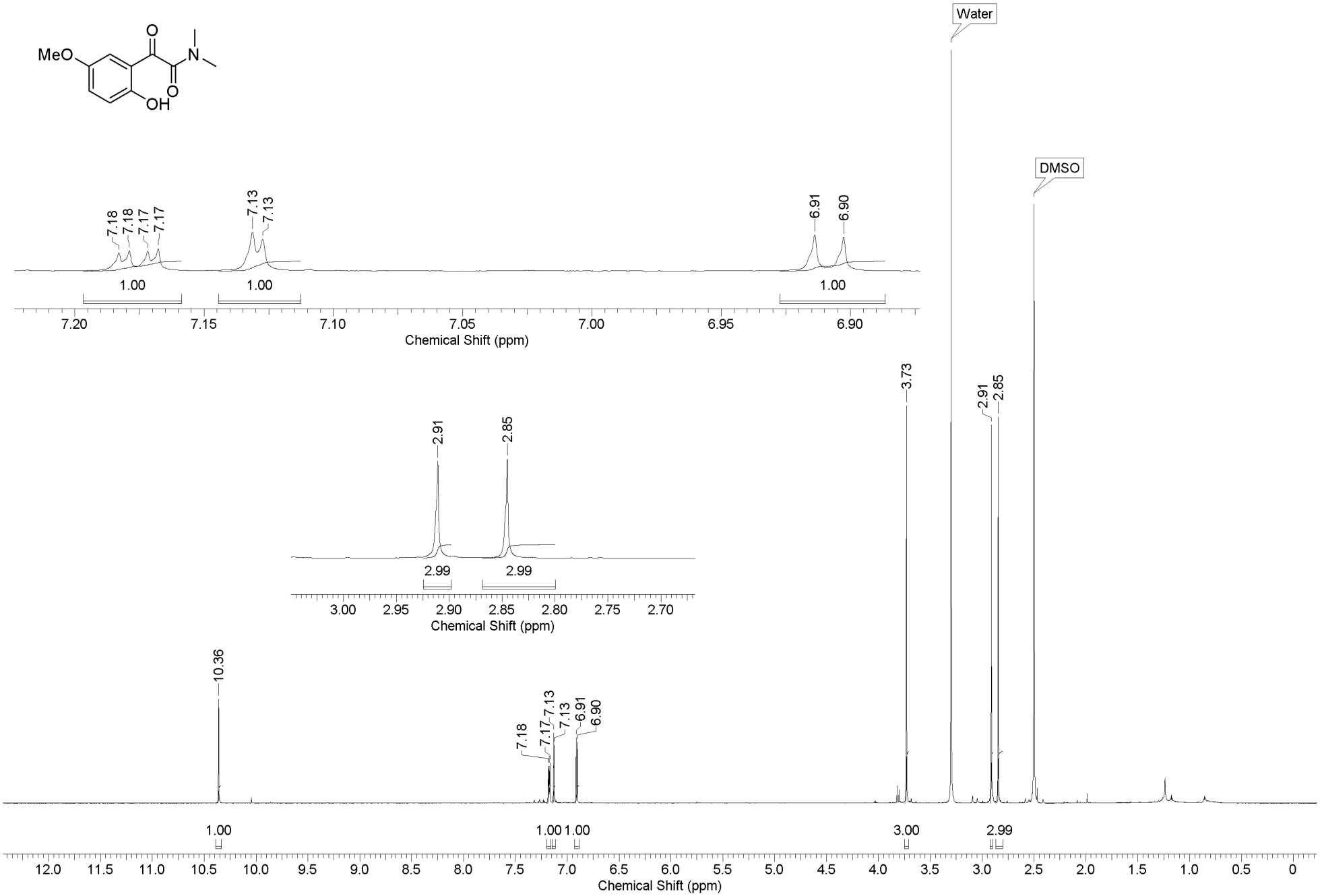

O
HO
N
OH
DMSO
169.94
154.67
151.75
127.15
122.44
115.30
112.54
83.81
57.73
34.08
31.06
28.03
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

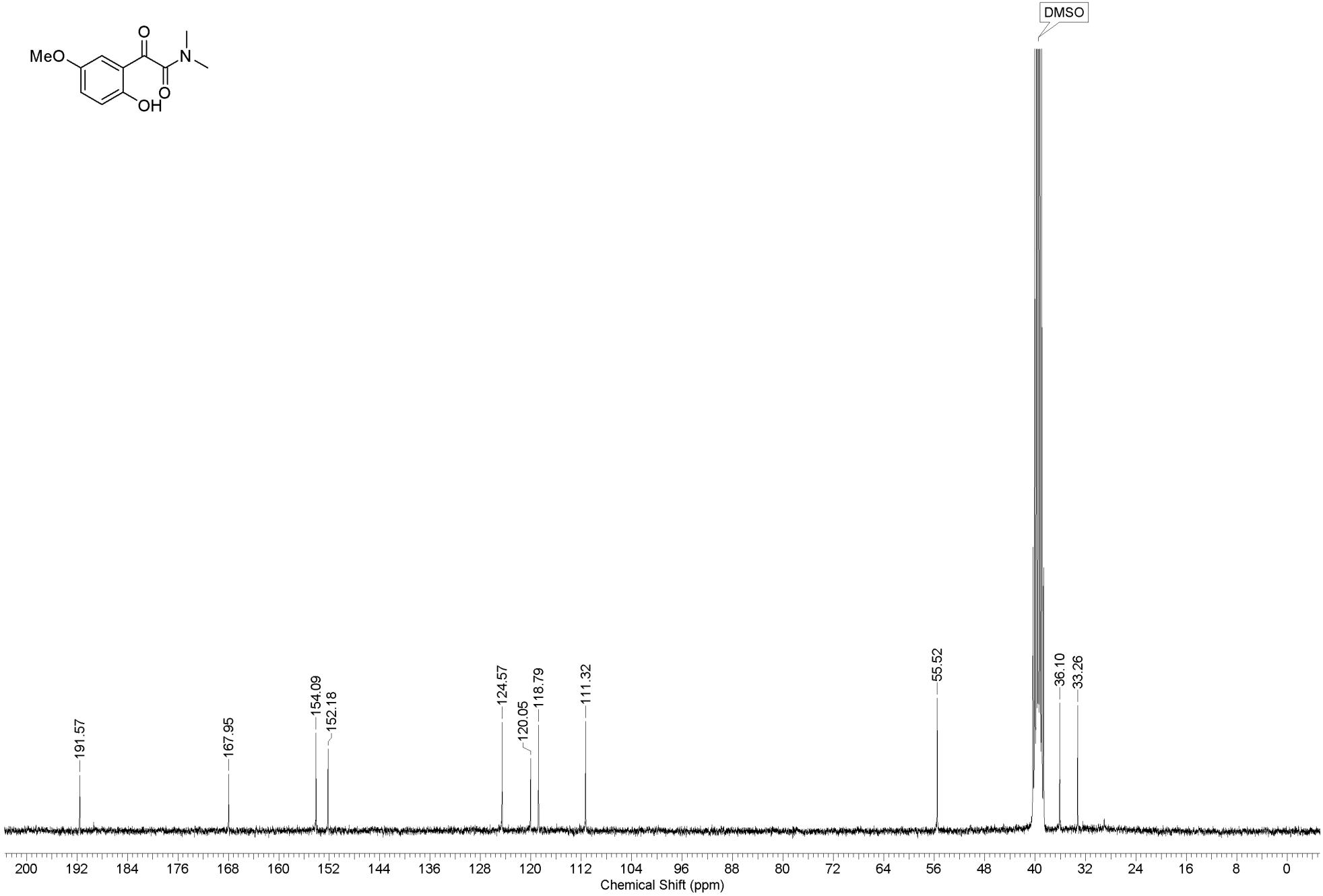
HMBC ^{1}H - ^{13}C NMR

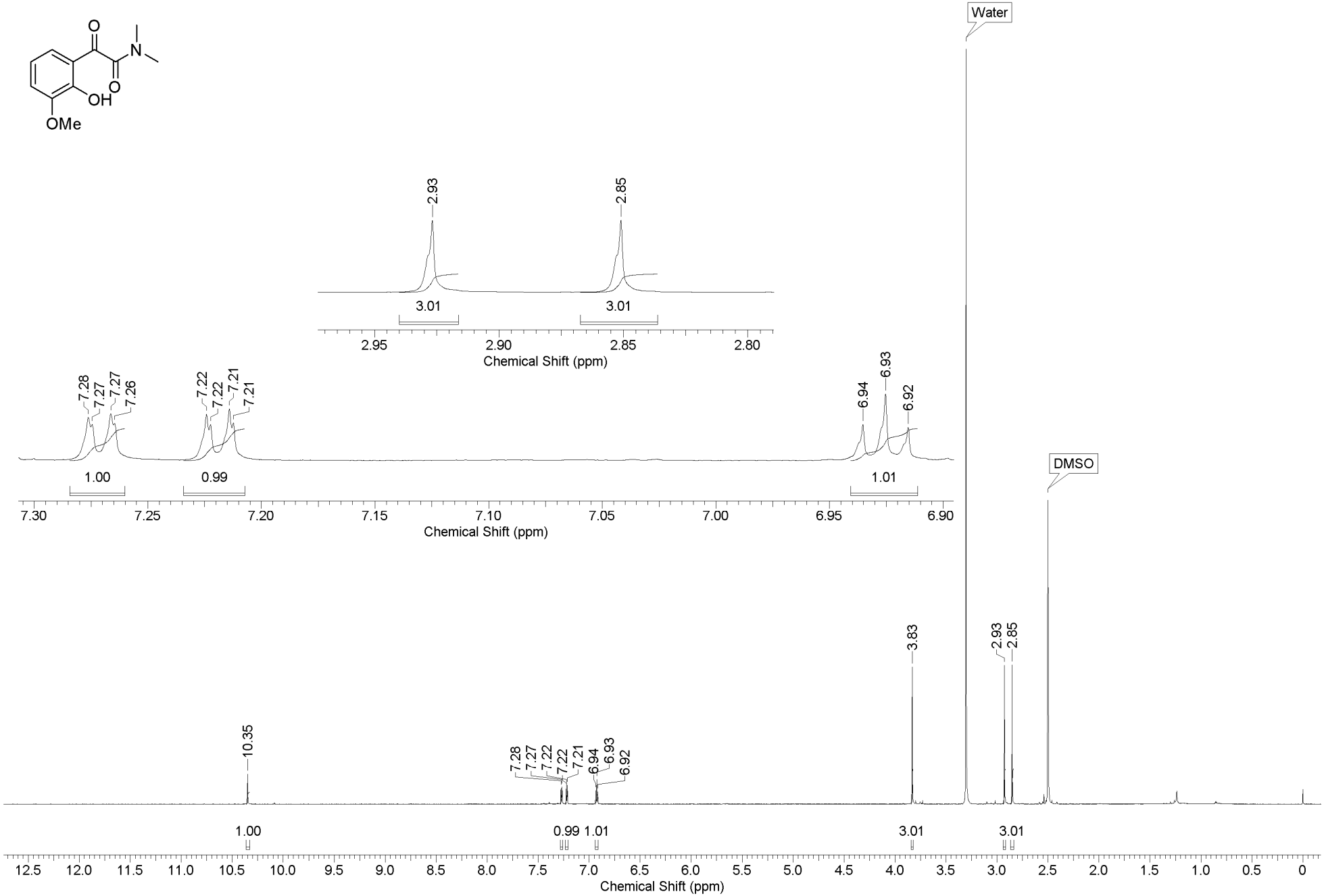


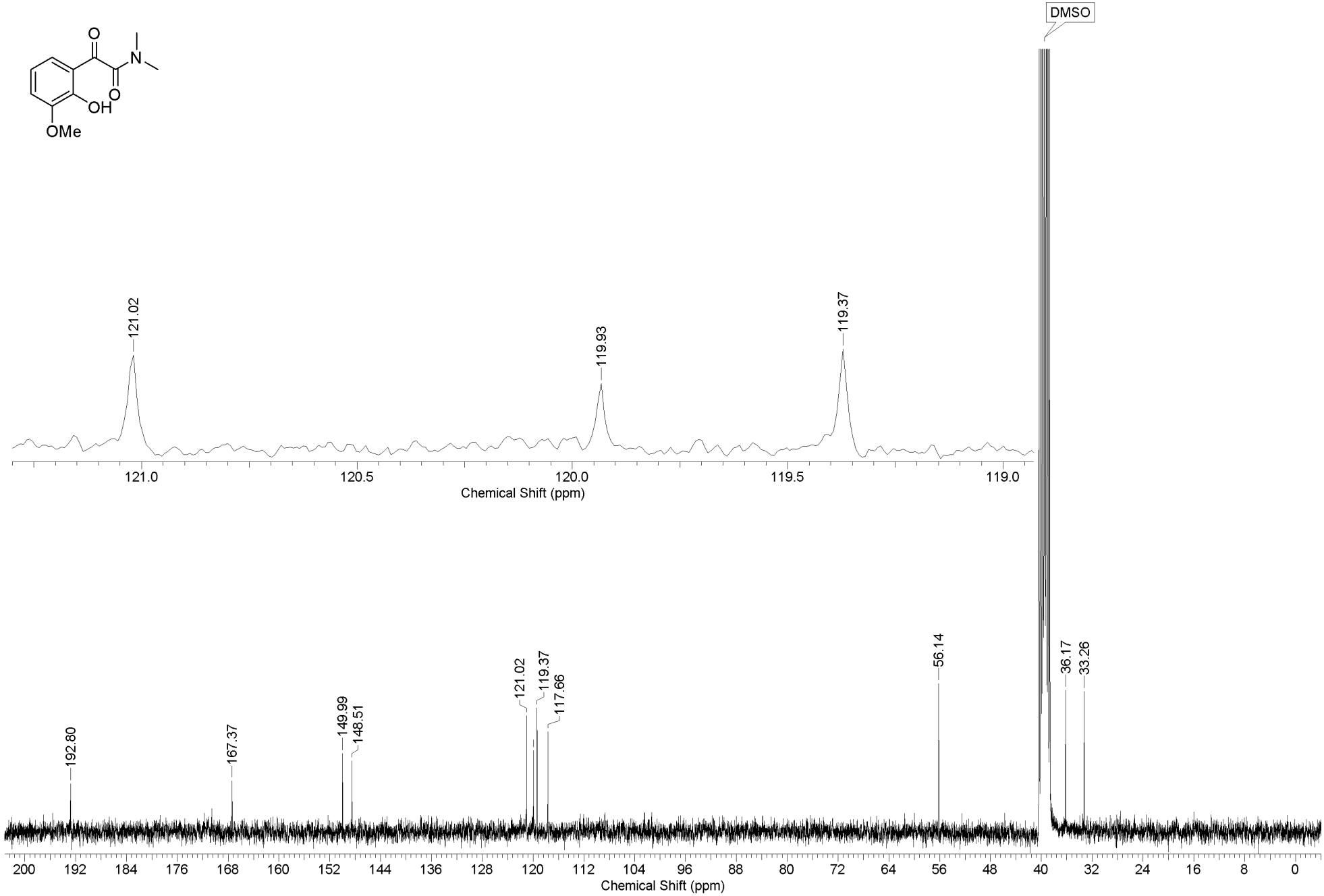
HSQC ^{1}H - ^{13}C NMR

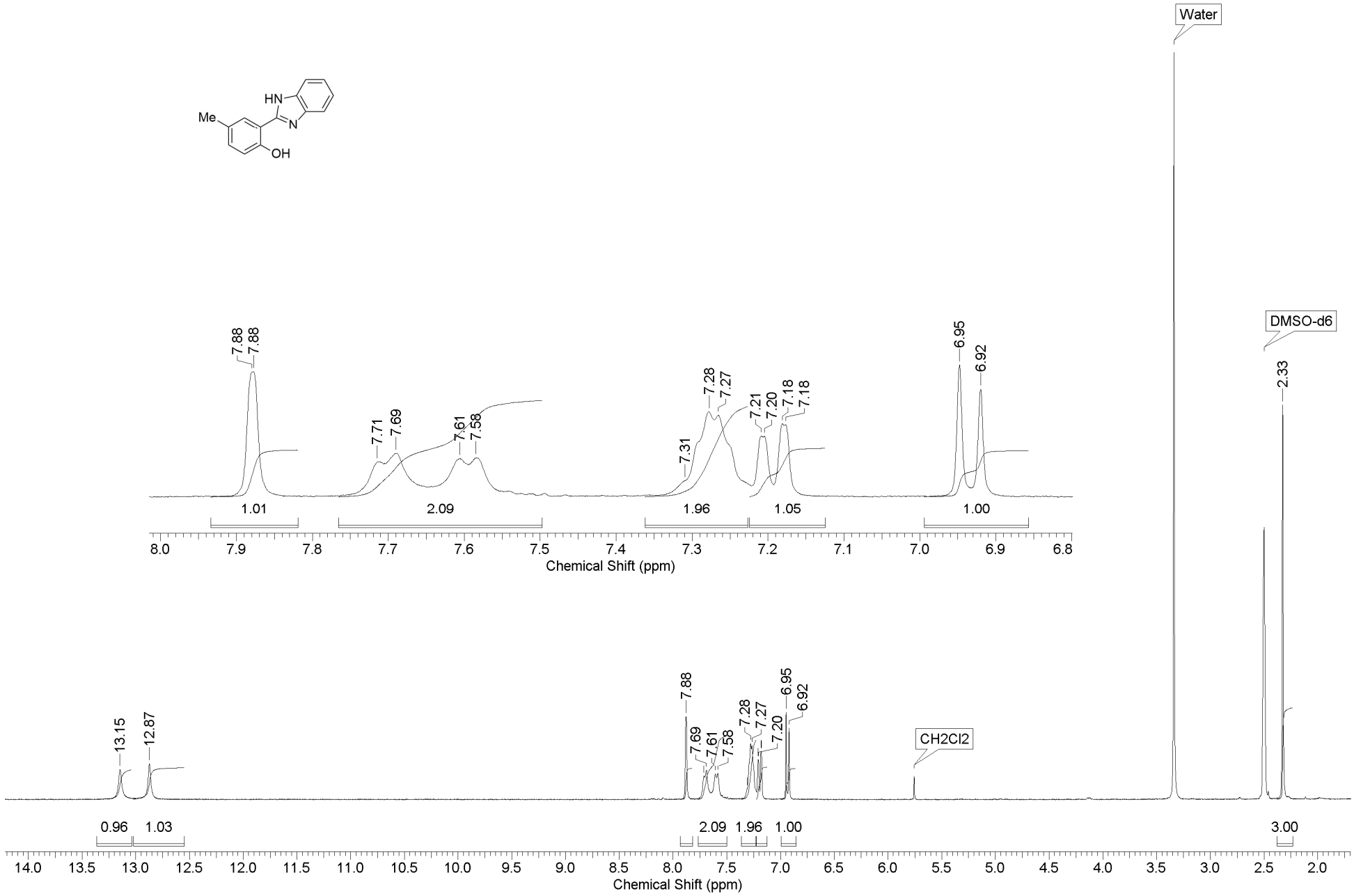


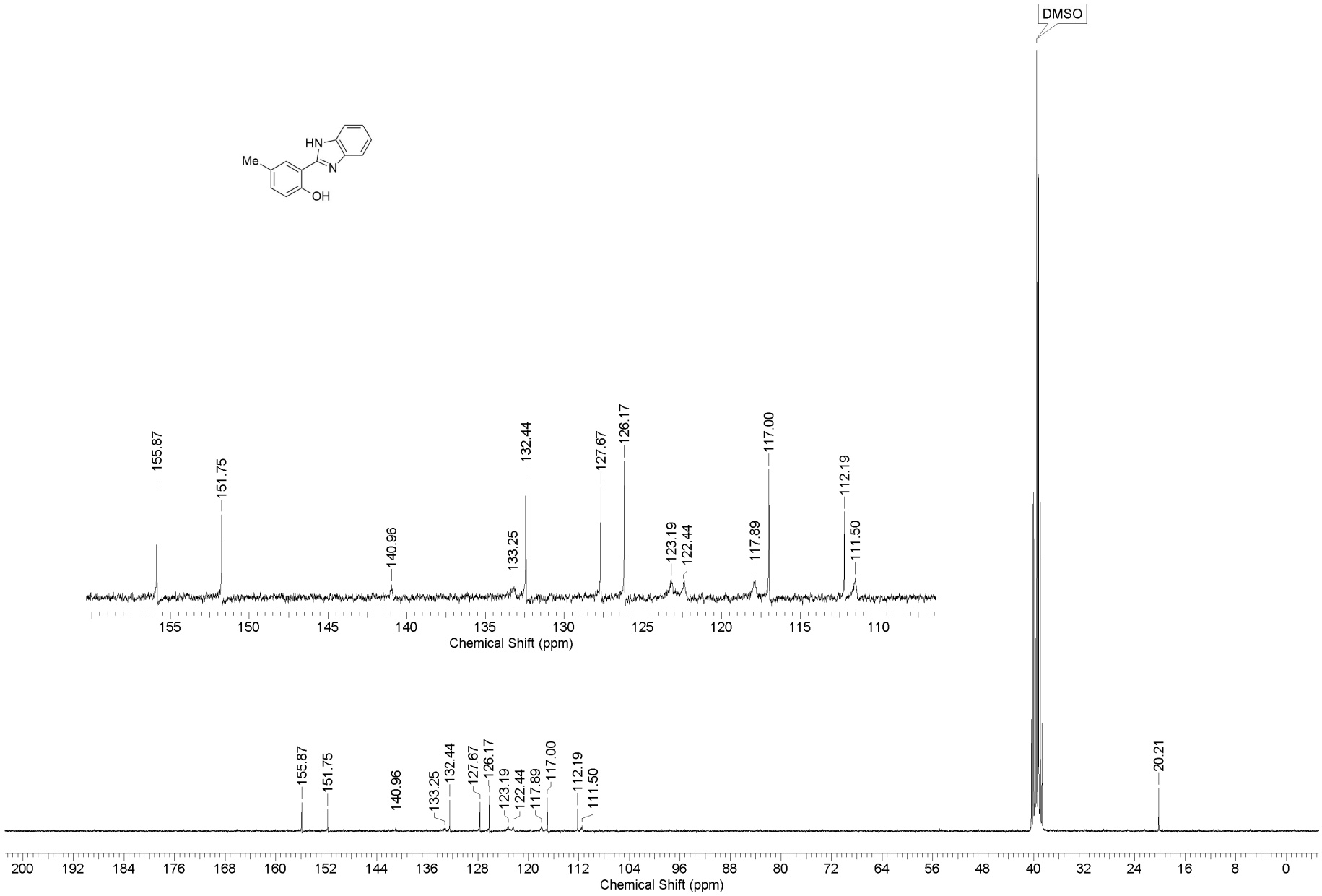

MeO
O
N
O
OH
Water
DMSO
7.18
7.18
7.17
7.17
7.13
7.13
6.91
6.90
1.00
1.00
1.00
7.20
7.15
7.10
7.05
7.00
6.95
6.90
Chemical Shift (ppm)
2.91
2.85
2.99
2.99
3.00
2.95
2.90
2.85
2.80
2.75
2.70
Chemical Shift (ppm)
3.73
2.91
2.85
10.36
7.18
7.17
7.13
7.13
6.91
6.90
1.00
1.00
1.00
3.00
2.99
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

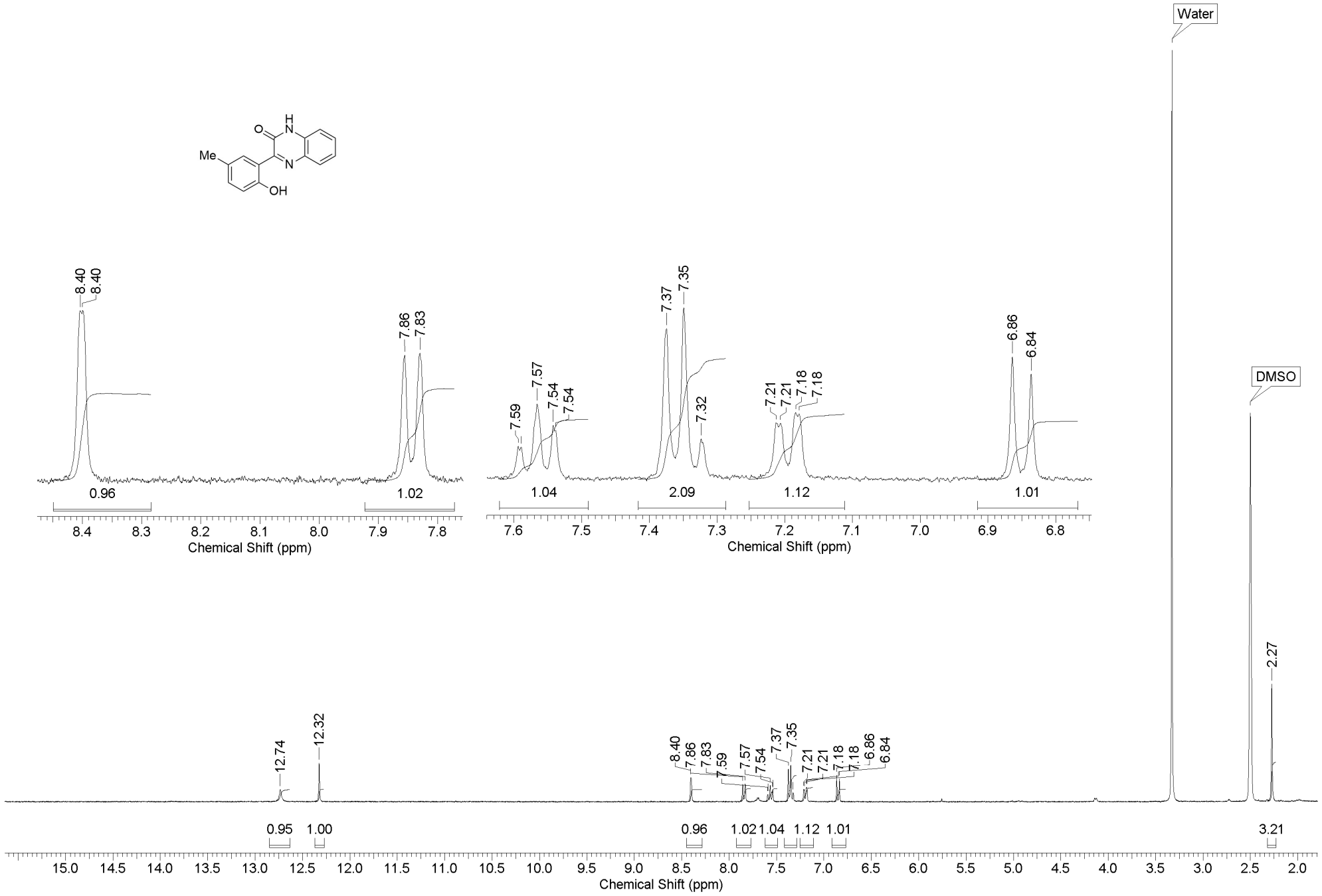

MeO
O
N
O
OH
DMSO
191.57
167.95
154.09
152.18
124.57
120.05
118.79
111.32
55.52
36.10
33.26
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

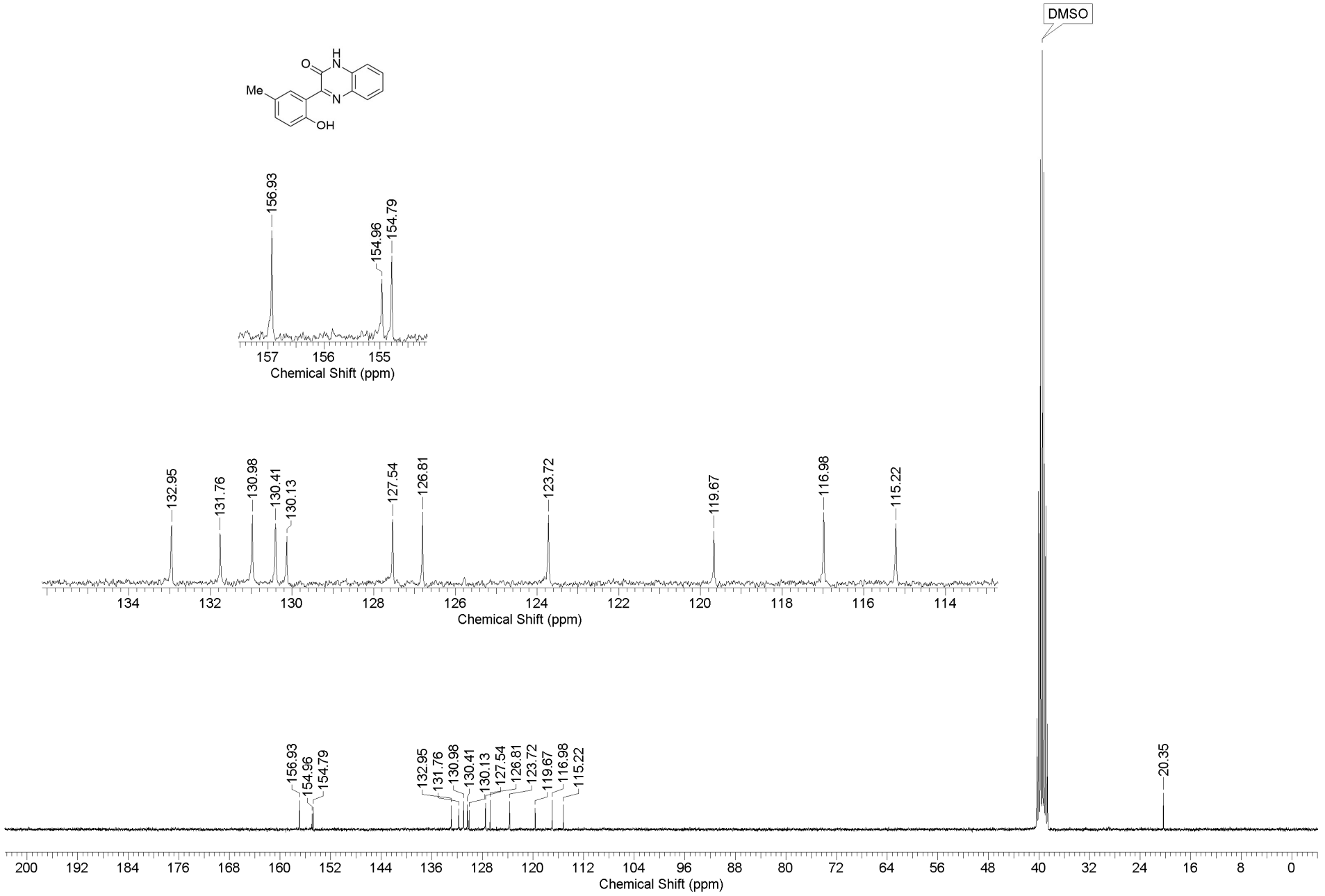

O
N
O
OH
OMe
Water
DMSO
2.93
2.85
3.01
3.01
2.95
2.90
2.85
2.80
Chemical Shift (ppm)
7.28
7.27
7.27
7.26
7.22
7.22
7.21
7.21
6.94
6.93
6.92
1.00
0.99
1.01
7.30
7.25
7.20
7.15
7.10
7.05
7.00
6.95
6.90
Chemical Shift (ppm)
10.35
7.28
7.27
7.22
7.22
7.21
6.94
6.93
6.92
3.83
2.93
2.85
1.00
0.99 1.01
3.01
3.01
12.5
12.0
11.5
11.0
10.5
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
Chemical Shift (ppm)

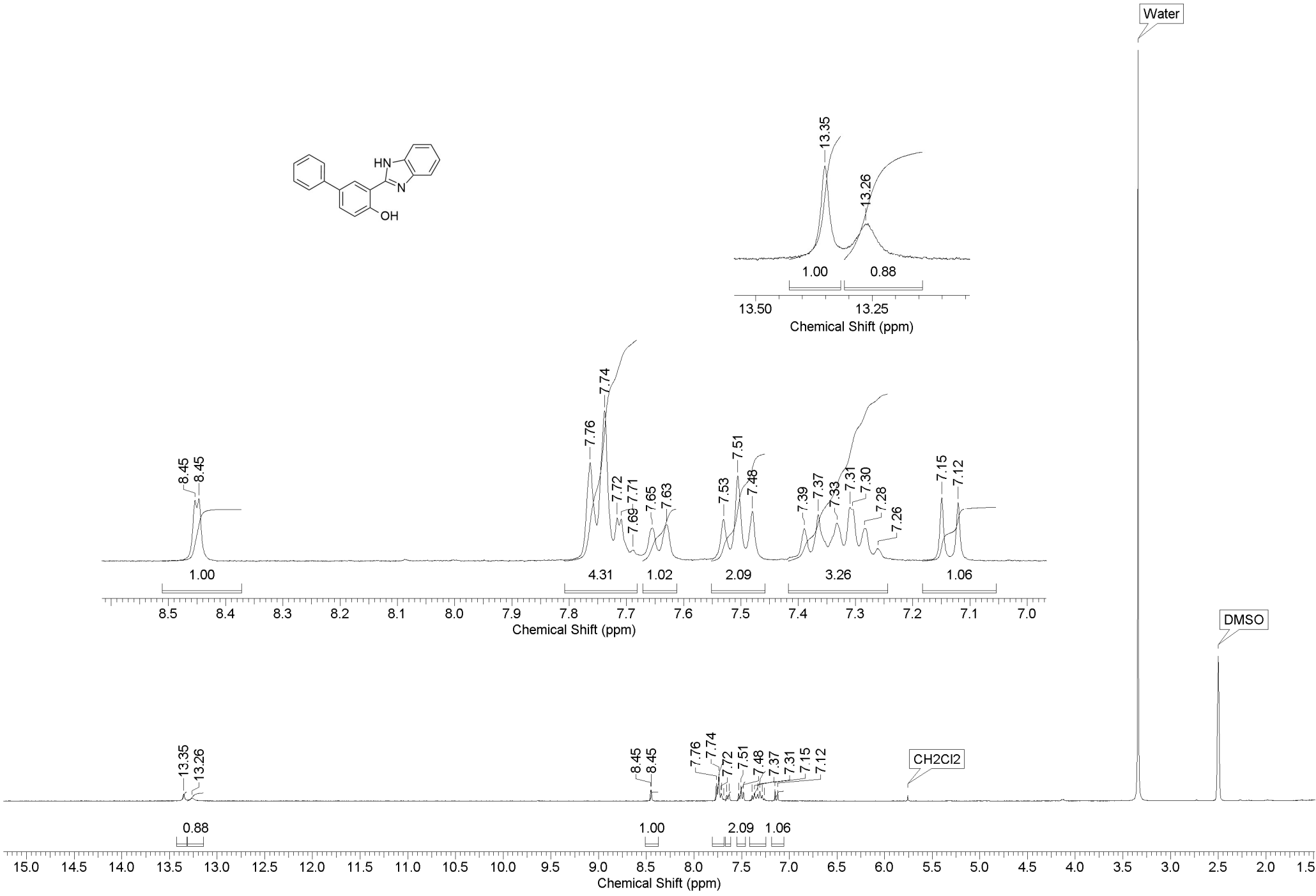

DMSO
121.02
119.93
119.37
121.0
120.5
120.0
119.5
119.0
Chemical Shift (ppm)
192.80
167.37
149.99
148.51
121.02
119.37
117.66
56.14
36.17
33.26
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

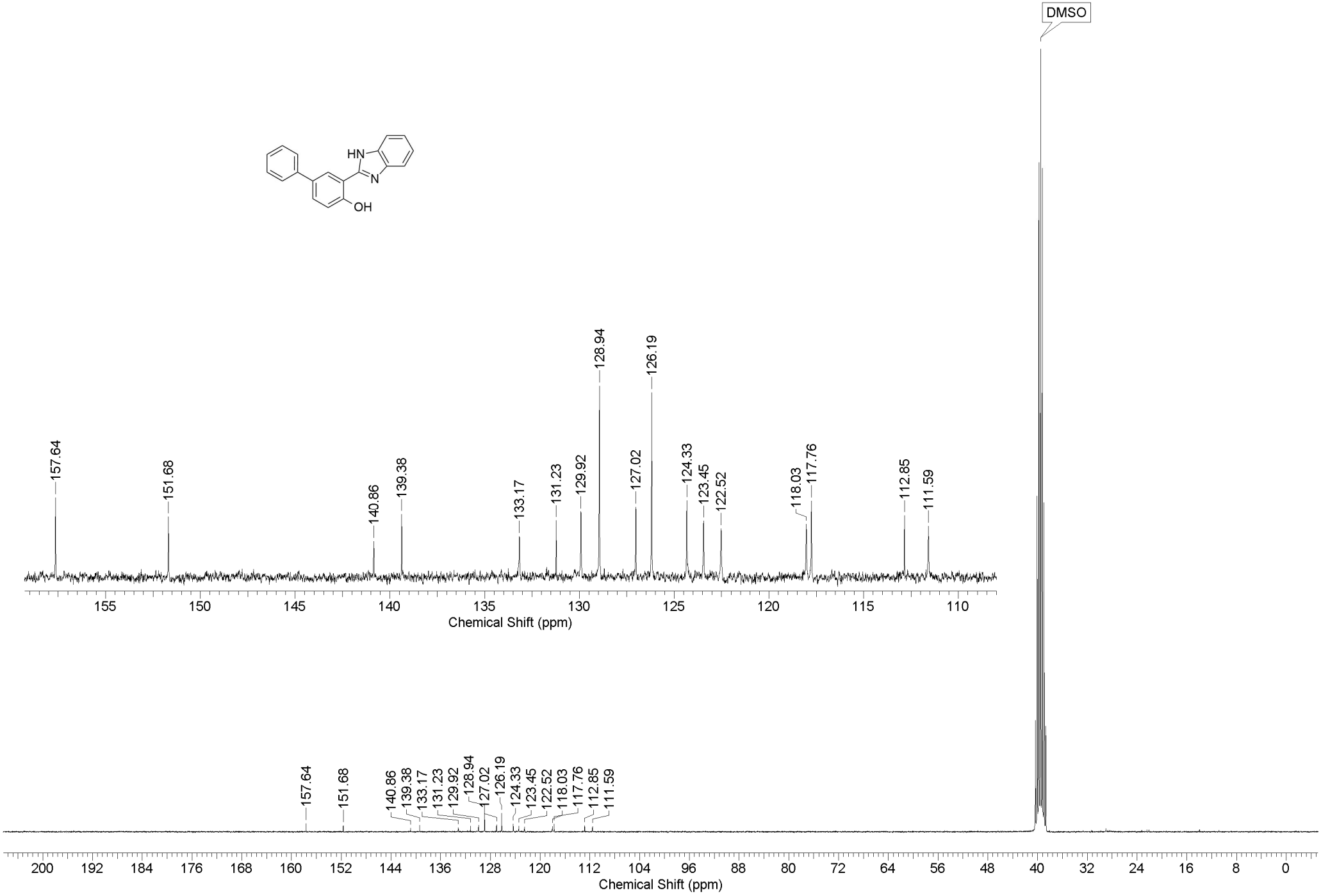

HN
N
Me
OH
Water
DMSO-d6
CH2Cl2
7.88
7.88
7.71
7.69
7.61
7.58
7.31
7.28
7.27
7.21
7.20
7.18
7.18
6.95
6.92
2.33
13.15
12.87
1.01
2.09
1.96
1.05
1.00
0.96
1.03
3.00
Chemical Shift (ppm)

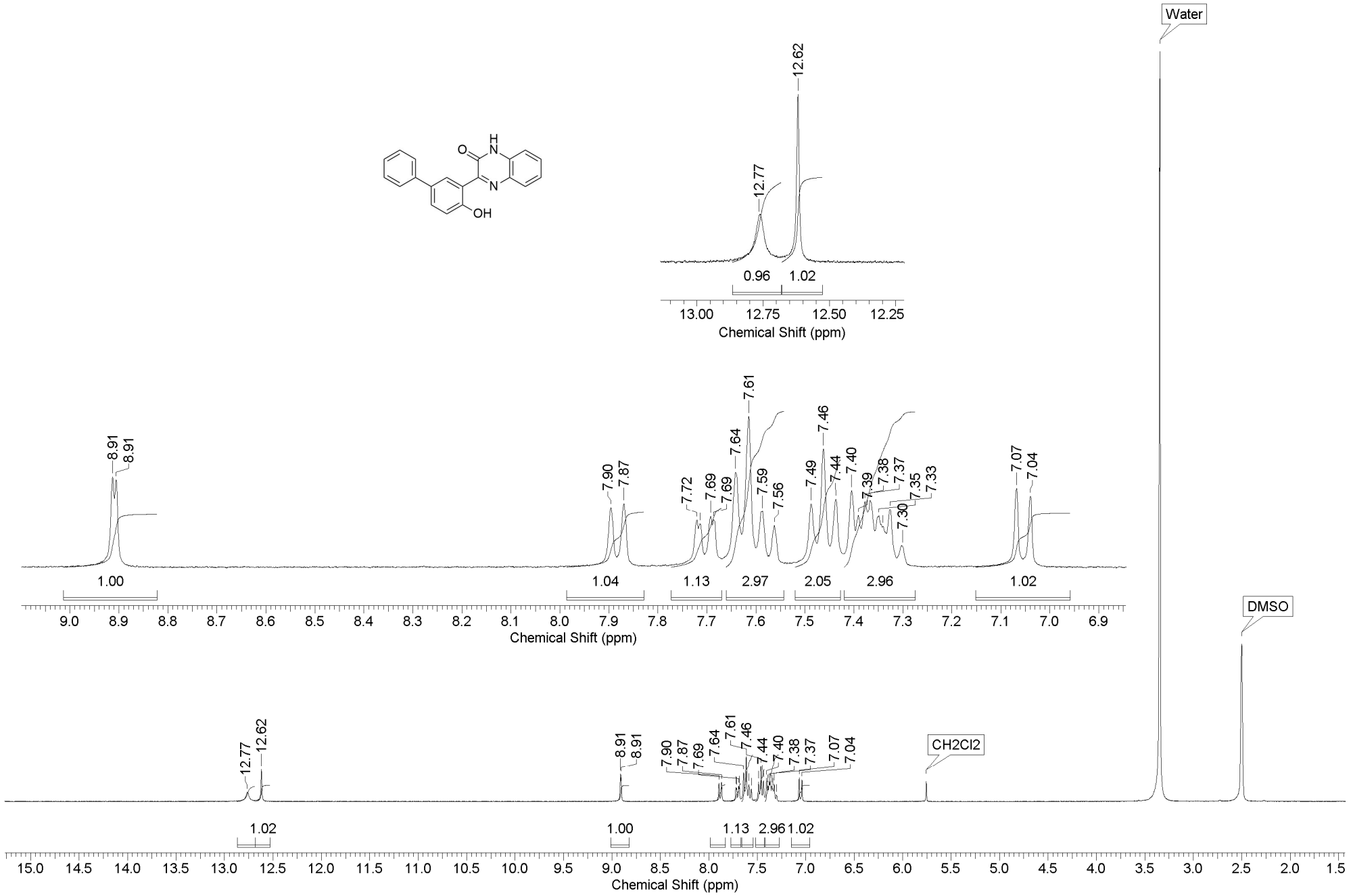

HN
Me
N
OH
DMSO
155.87
151.75
140.96
133.25
132.44
127.67
126.17
123.19
122.44
117.89
117.00
112.19
111.50
20.21
Chemical Shift (ppm)

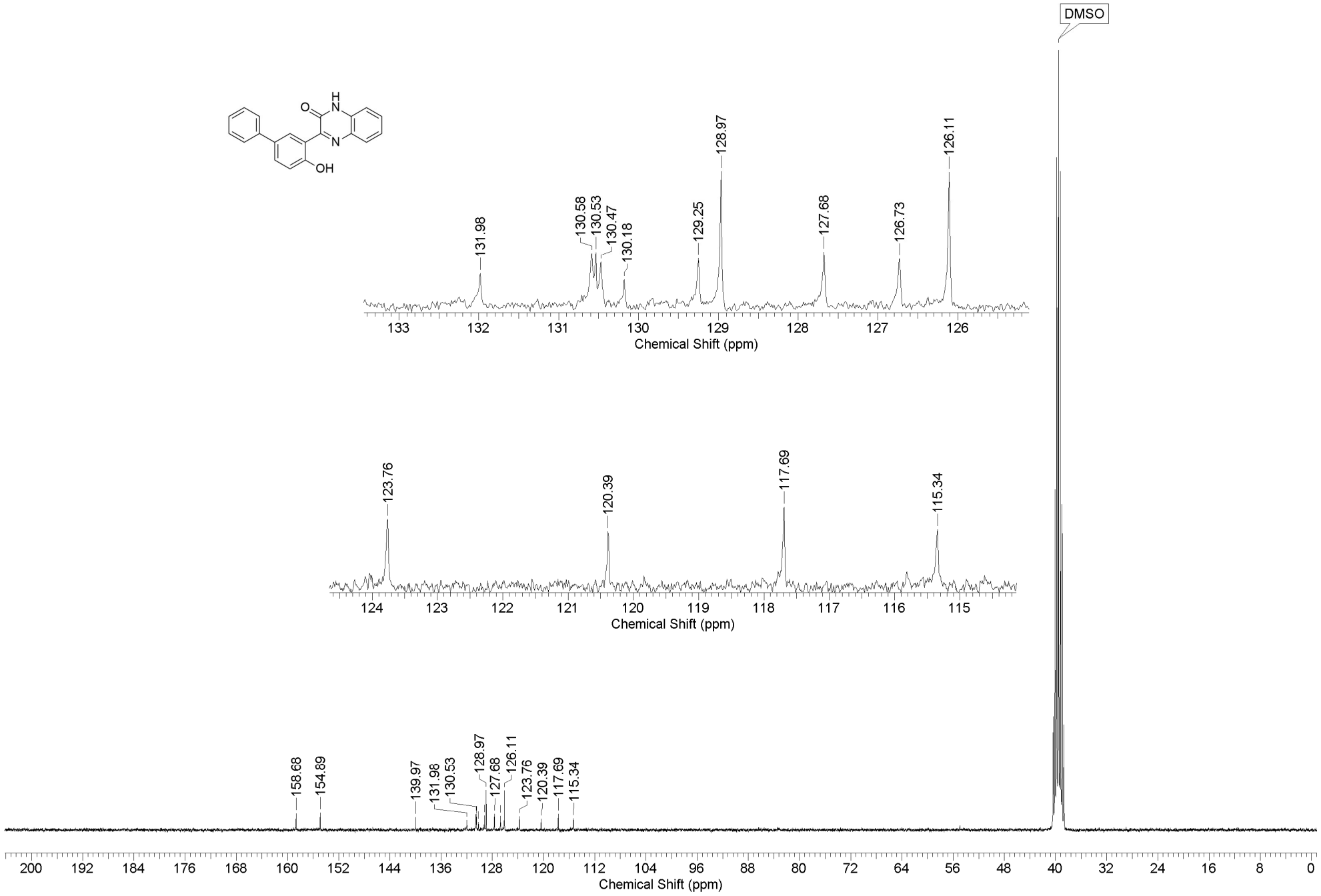

Me
OH
O
N
H
N
Water
DMSO
8.40
8.40
7.86
7.83
0.96
1.02
Chemical Shift (ppm)
7.59
7.57
7.54
7.54
7.37
7.35
7.32
7.21
7.21
7.18
7.18
6.86
6.84
1.04
2.09
1.12
1.01
Chemical Shift (ppm)
12.74
12.32
8.40
7.86
7.83
7.59
7.57
7.54
7.37
7.35
7.21
7.21
7.18
7.18
6.86
6.84
2.27
0.95
1.00
0.96
1.02
1.04
1.12
1.01
3.21
Chemical Shift (ppm)

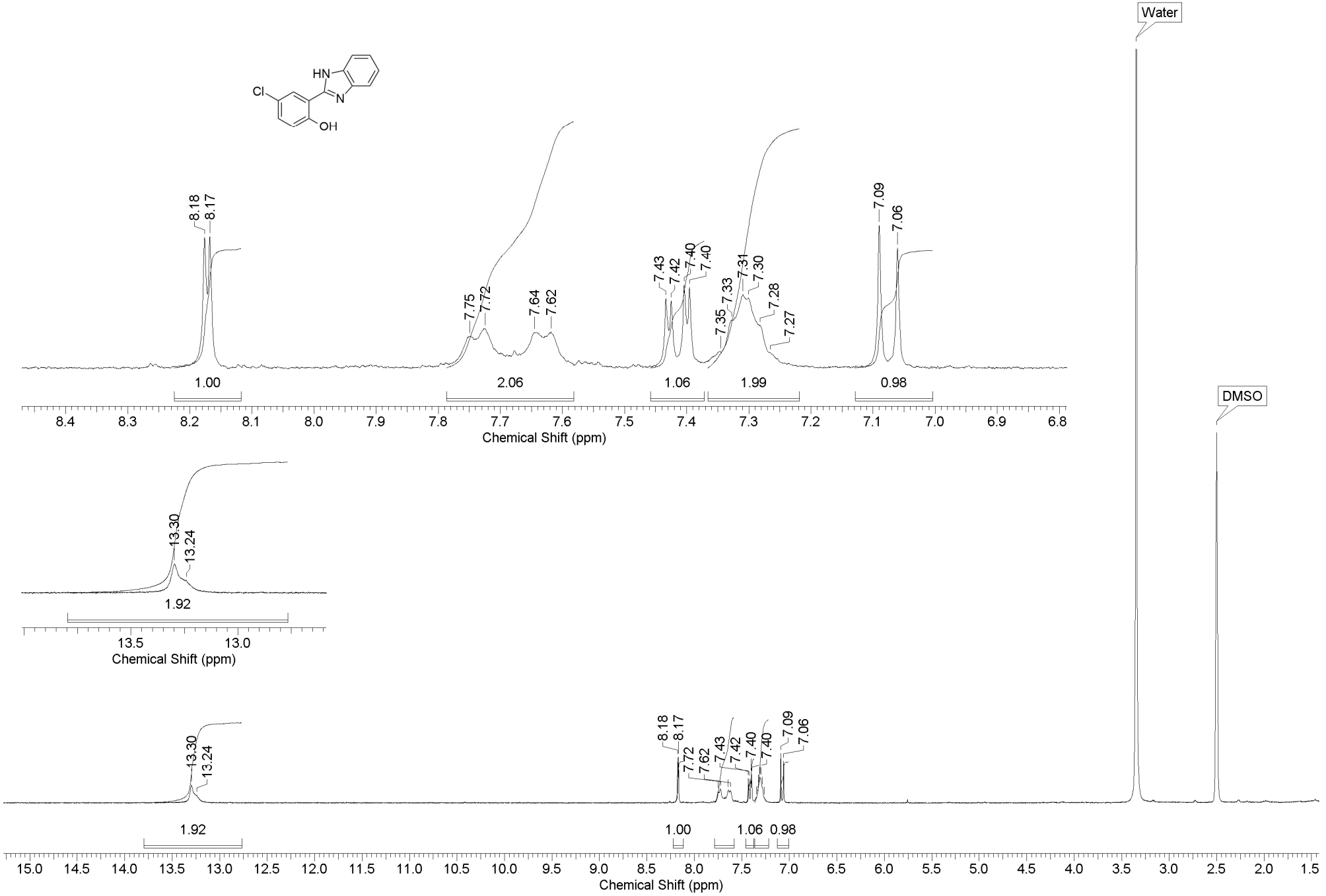

DMSO
Me
OH
156.93
154.96
154.79
157
156
155
Chemical Shift (ppm)
132.95
131.76
130.98
130.41
130.13
127.54
126.81
123.72
119.67
116.98
115.22
134
132
130
128
126
124
122
120
118
116
114
Chemical Shift (ppm)
20.35
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

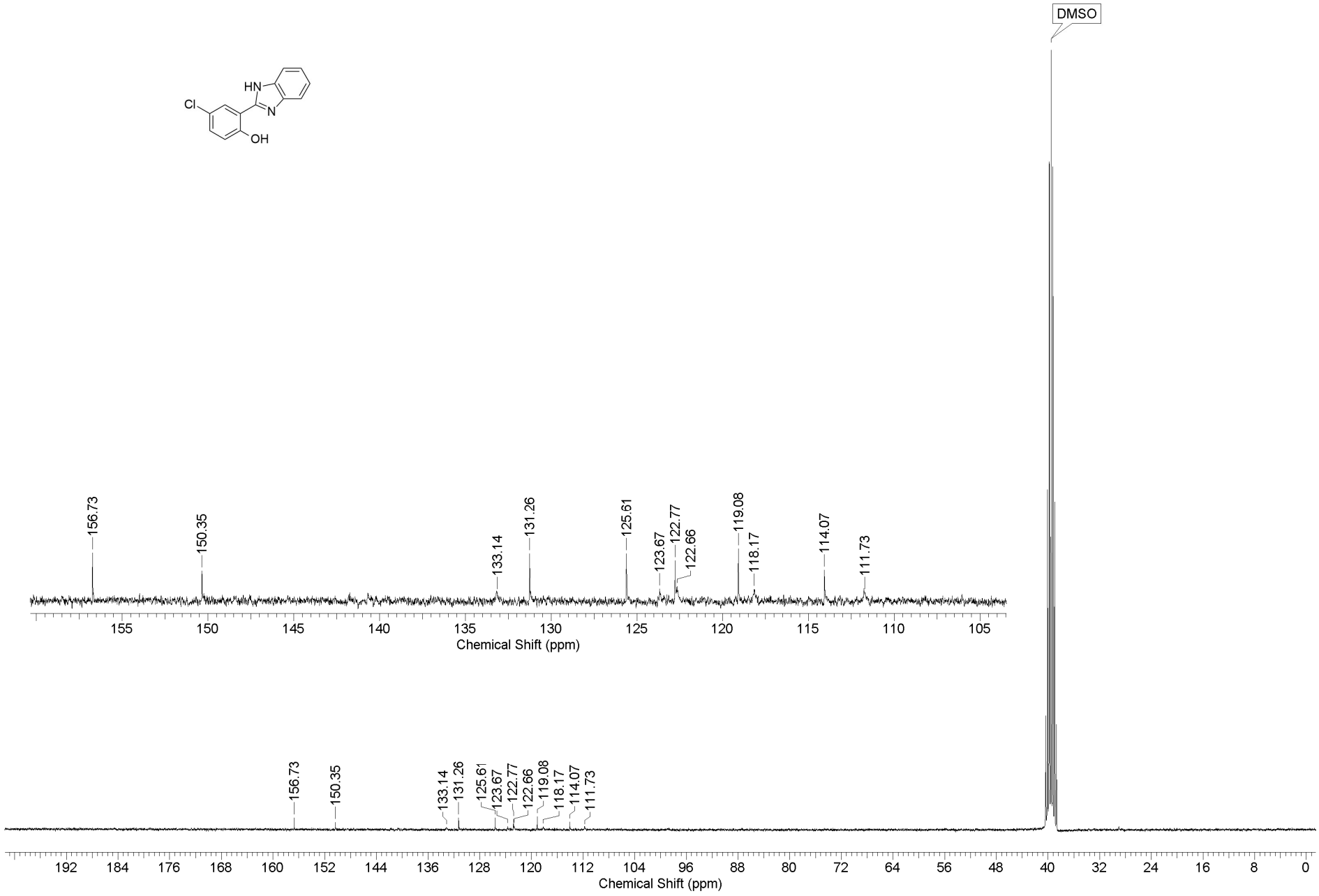

HN
N
OH
13.35
13.26
1.00
0.88
13.50
13.25
Chemical Shift (ppm)
8.45
8.45
7.76
7.74
7.72
7.71
7.69
7.65
7.63
7.53
7.51
7.48
7.39
7.37
7.33
7.31
7.30
7.28
7.26
7.15
7.12
1.00
4.31
1.02
2.09
3.26
1.06
Chemical Shift (ppm)
Water
DMSO
CH2Cl2
0.88
1.00
2.09
1.06
Chemical Shift (ppm)

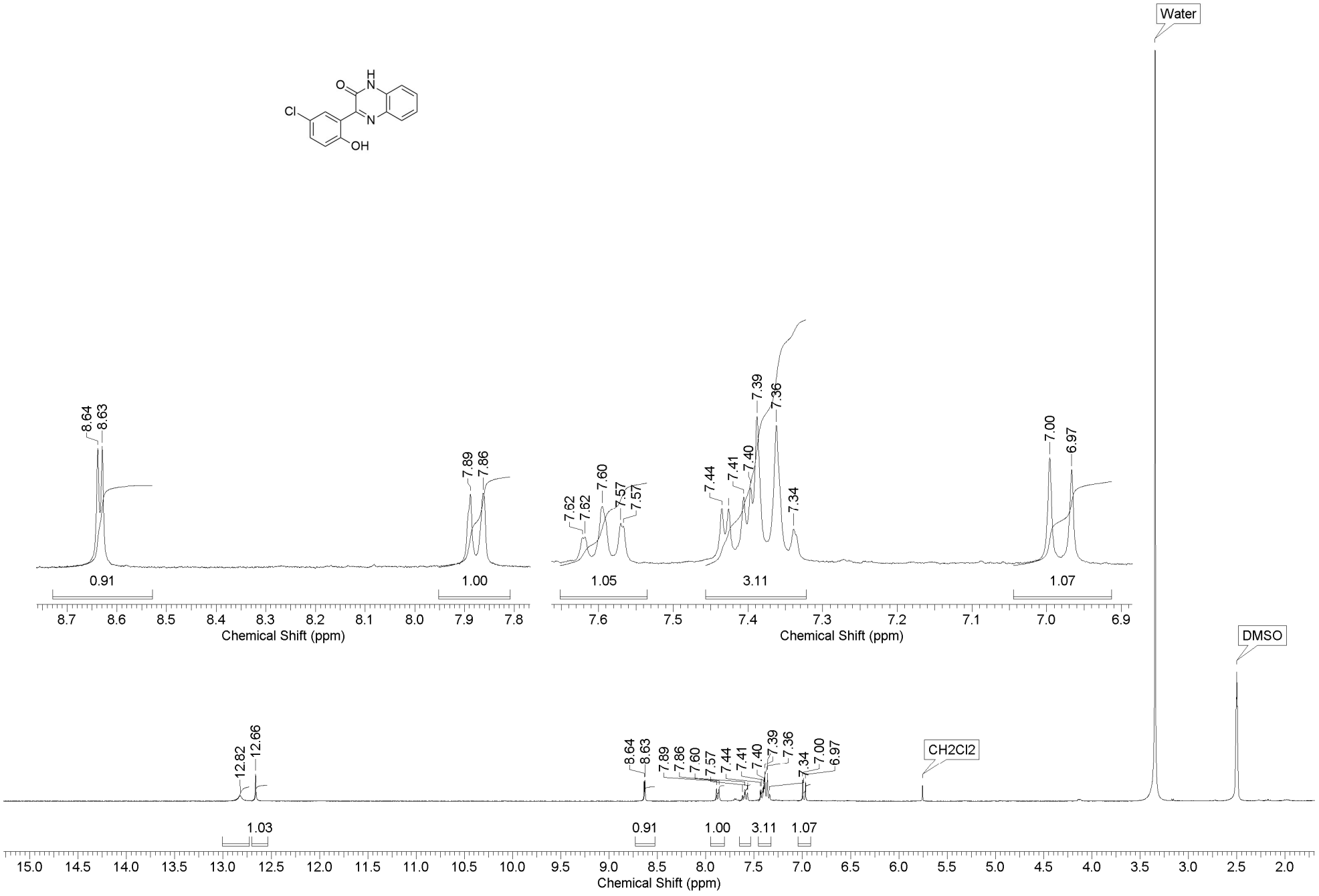

DMSO
HN
N
OH
157.64
151.68
140.86
139.38
133.17
131.23
129.92
128.94
127.02
126.19
124.33
123.45
122.52
118.03
117.76
112.85
111.59
155
150
145
140
135
130
125
120
115
110
Chemical Shift (ppm)
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

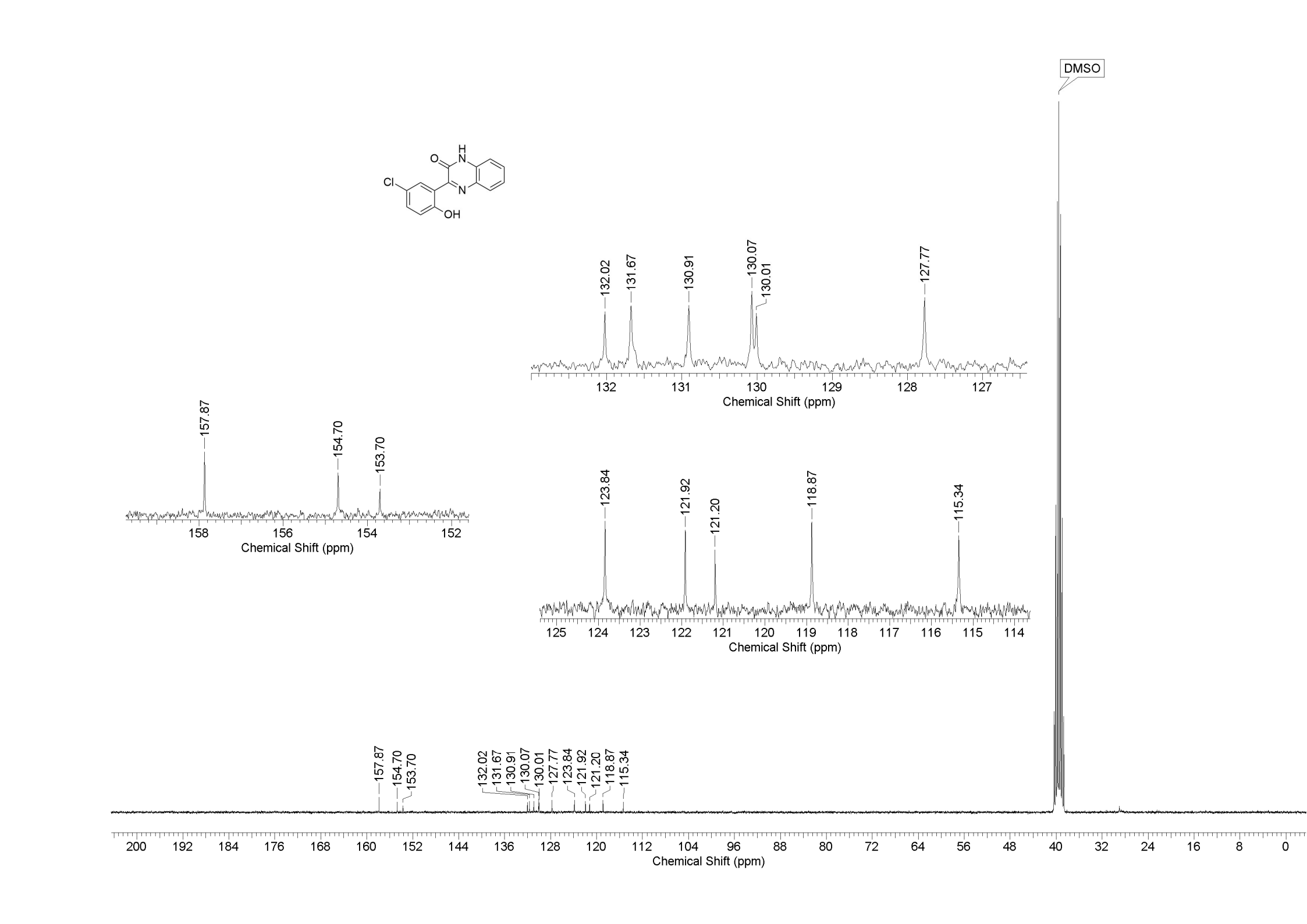

Water
DMSO
CH2Cl2
Chemical Shift (ppm)

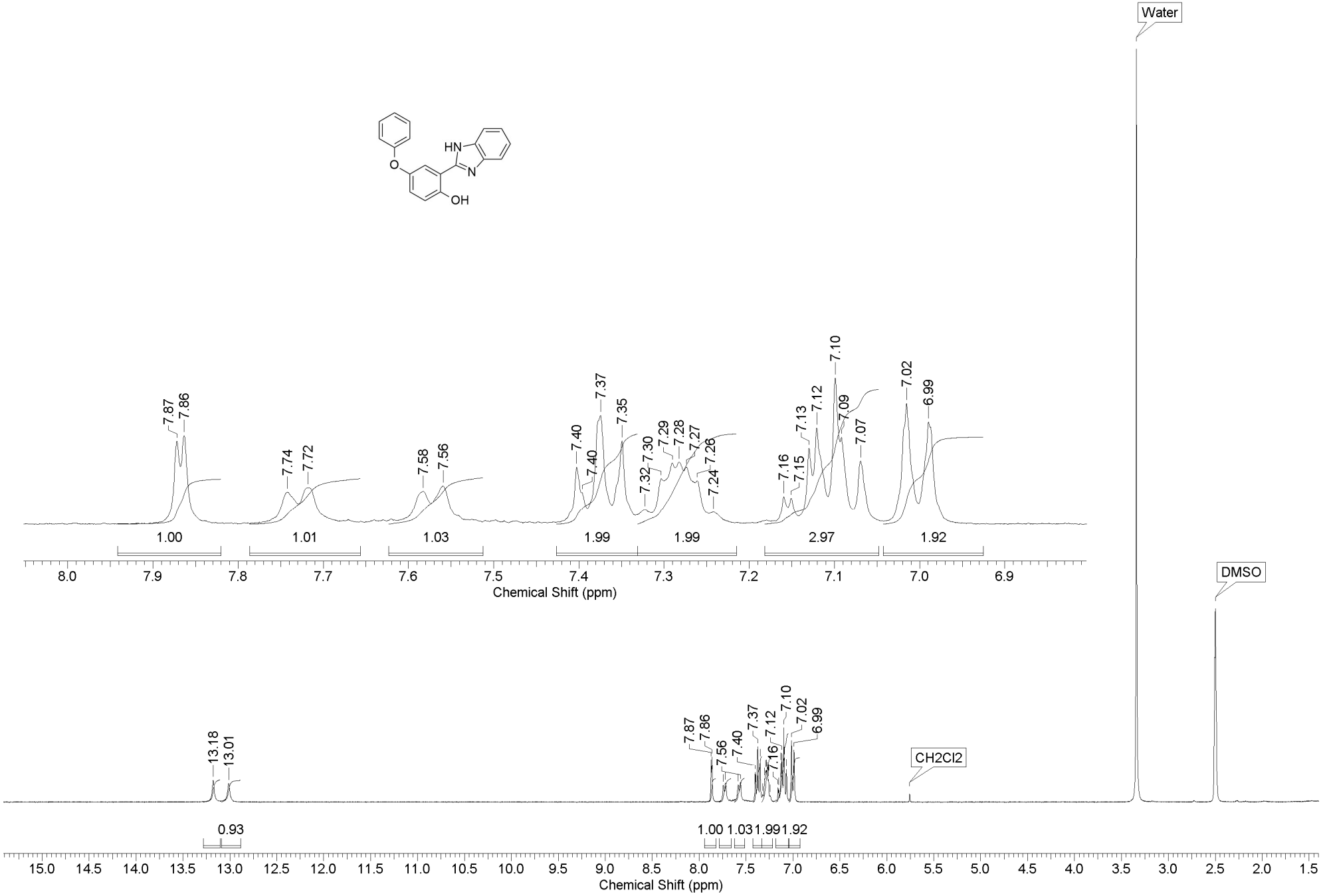

DMSO
131.98
130.58
130.53
130.47
130.18
129.25
128.97
127.68
126.73
126.11
Chemical Shift (ppm)
123.76
120.39
117.69
115.34
Chemical Shift (ppm)
158.68
154.89
139.97
131.98
130.53
128.97
127.68
126.11
123.76
120.39
117.69
115.34
Chemical Shift (ppm)

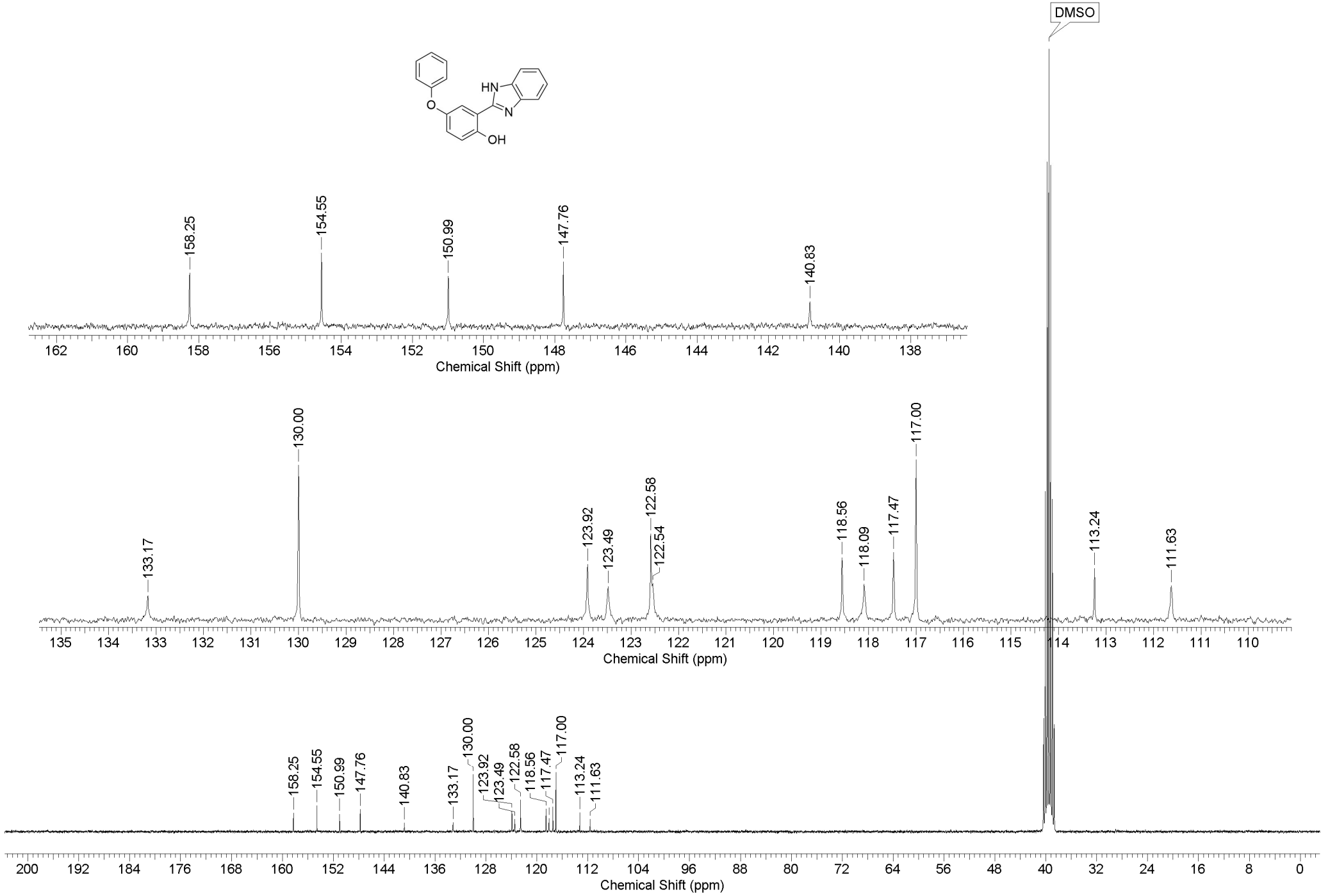

Water
DMSO
HN
Cl
N
OH
8.18
8.17
7.75
7.72
7.64
7.62
7.43
7.42
7.40
7.40
7.35
7.33
7.31
7.30
7.28
7.27
7.09
7.06
1.00
2.06
1.06
1.99
0.98
Chemical Shift (ppm)
13.30
13.24
1.92
Chemical Shift (ppm)
13.30
13.24
8.18
8.17
7.72
7.62
7.43
7.42
7.40
7.40
7.09
7.06
1.92
1.00
1.06
0.98
Chemical Shift (ppm)

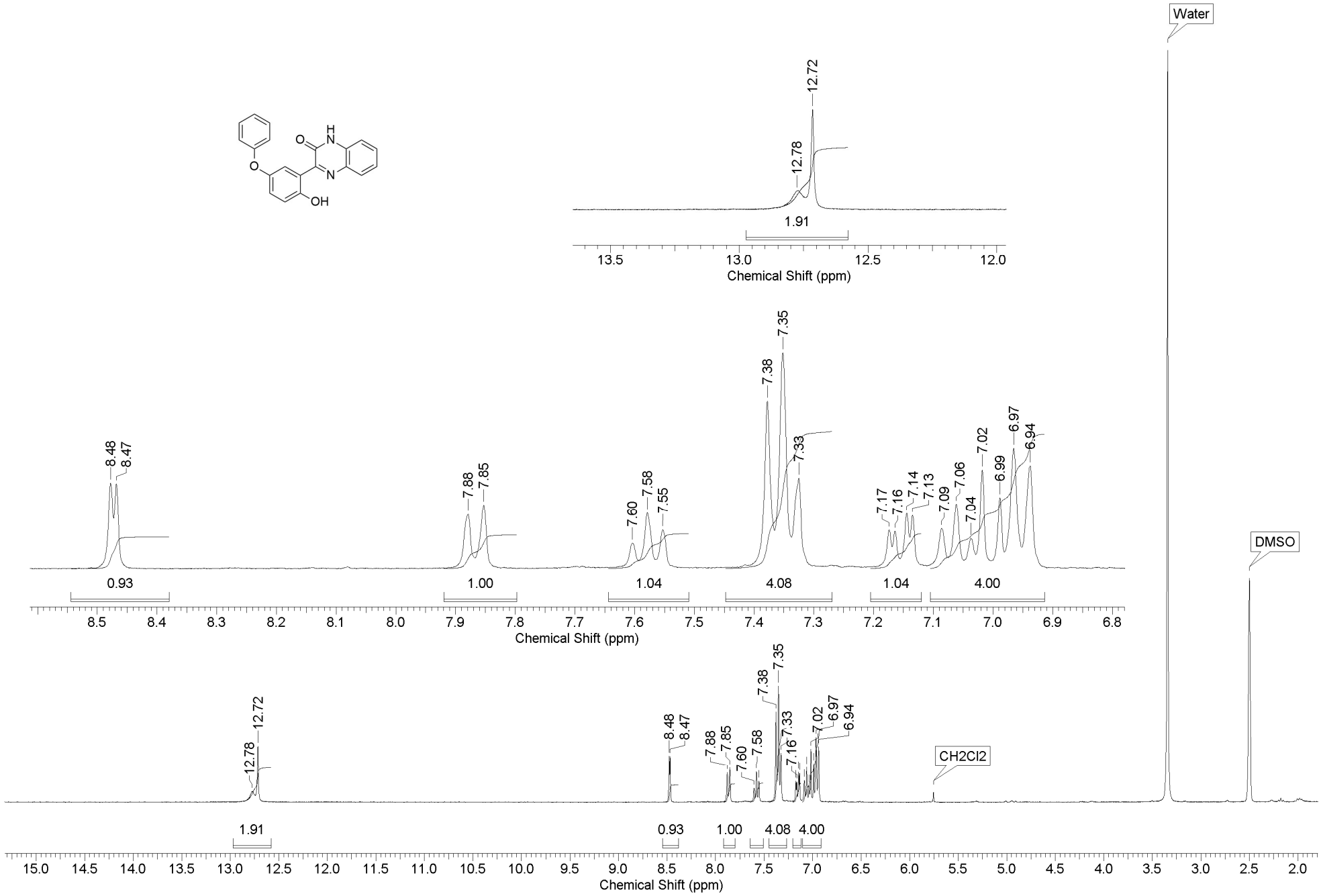

DMSO
HN
N
Cl
OH
156.73
150.35
133.14
131.26
125.61
123.67
122.77
122.66
119.08
118.17
114.07
111.73
155
150
145
140
135
130
125
120
115
110
105
Chemical Shift (ppm)
156.73
150.35
133.14
131.26
125.61
123.67
122.77
122.66
119.08
118.17
114.07
111.73
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

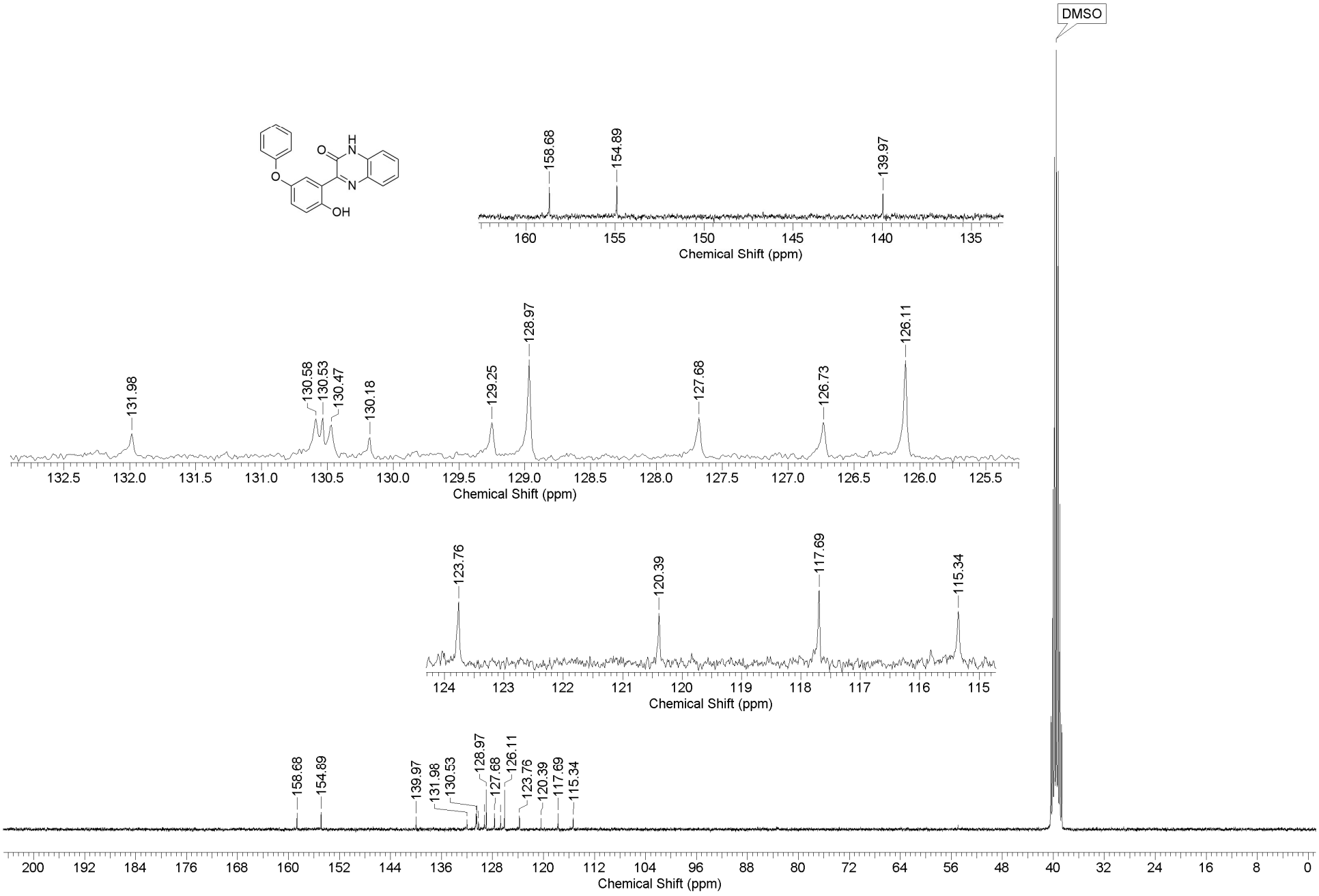

Water
DMSO
CH2Cl2
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

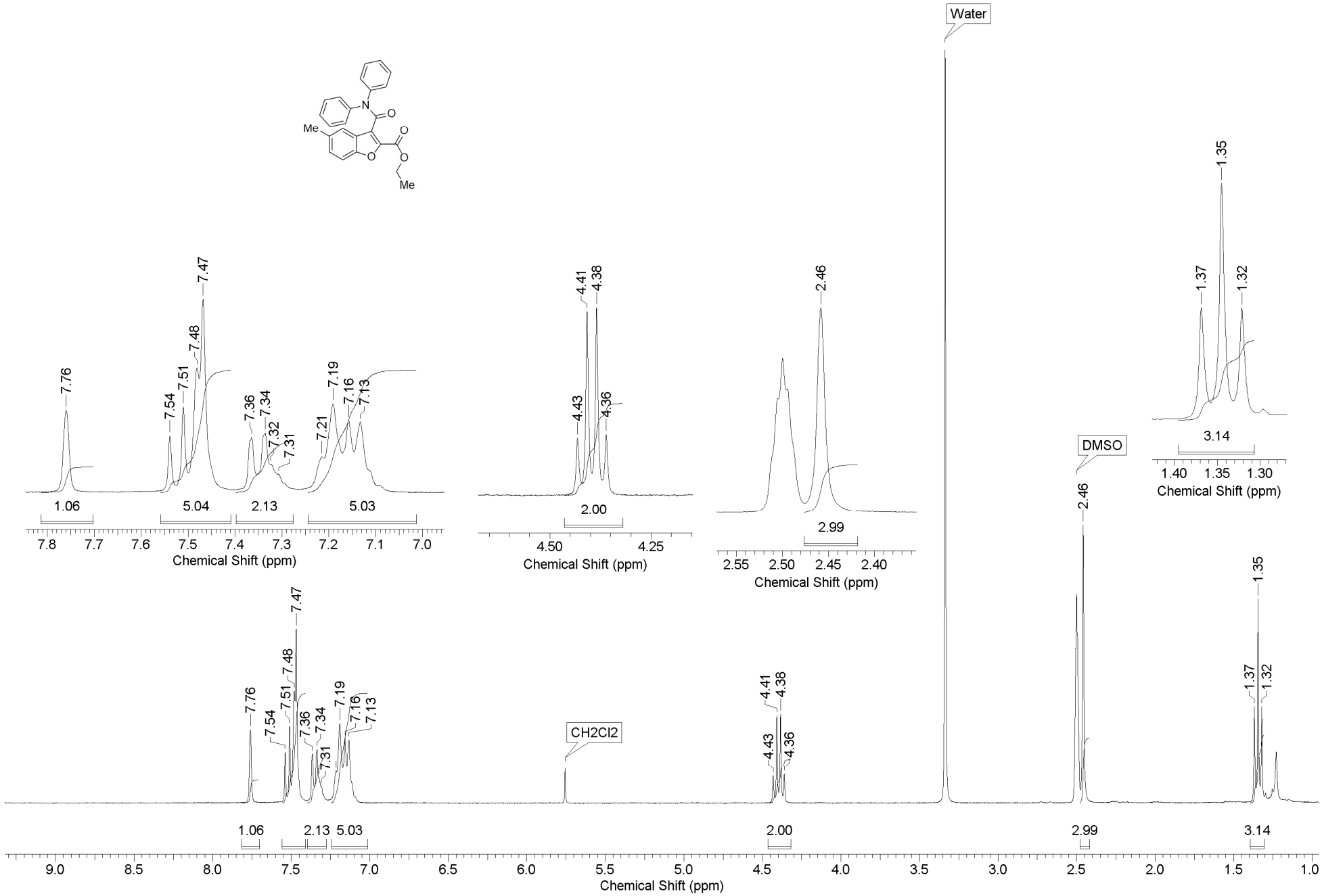

DMSO
O
H
N
N
Cl
OH
132.02
131.67
130.91
130.07
130.01
127.77
Chemical Shift (ppm)
157.87
154.70
153.70
Chemical Shift (ppm)
123.84
121.92
121.20
118.87
115.34
Chemical Shift (ppm)
157.87
154.70
153.70
132.02
131.67
130.91
130.07
130.01
127.77
123.84
121.92
121.20
118.87
115.34
Chemical Shift (ppm)

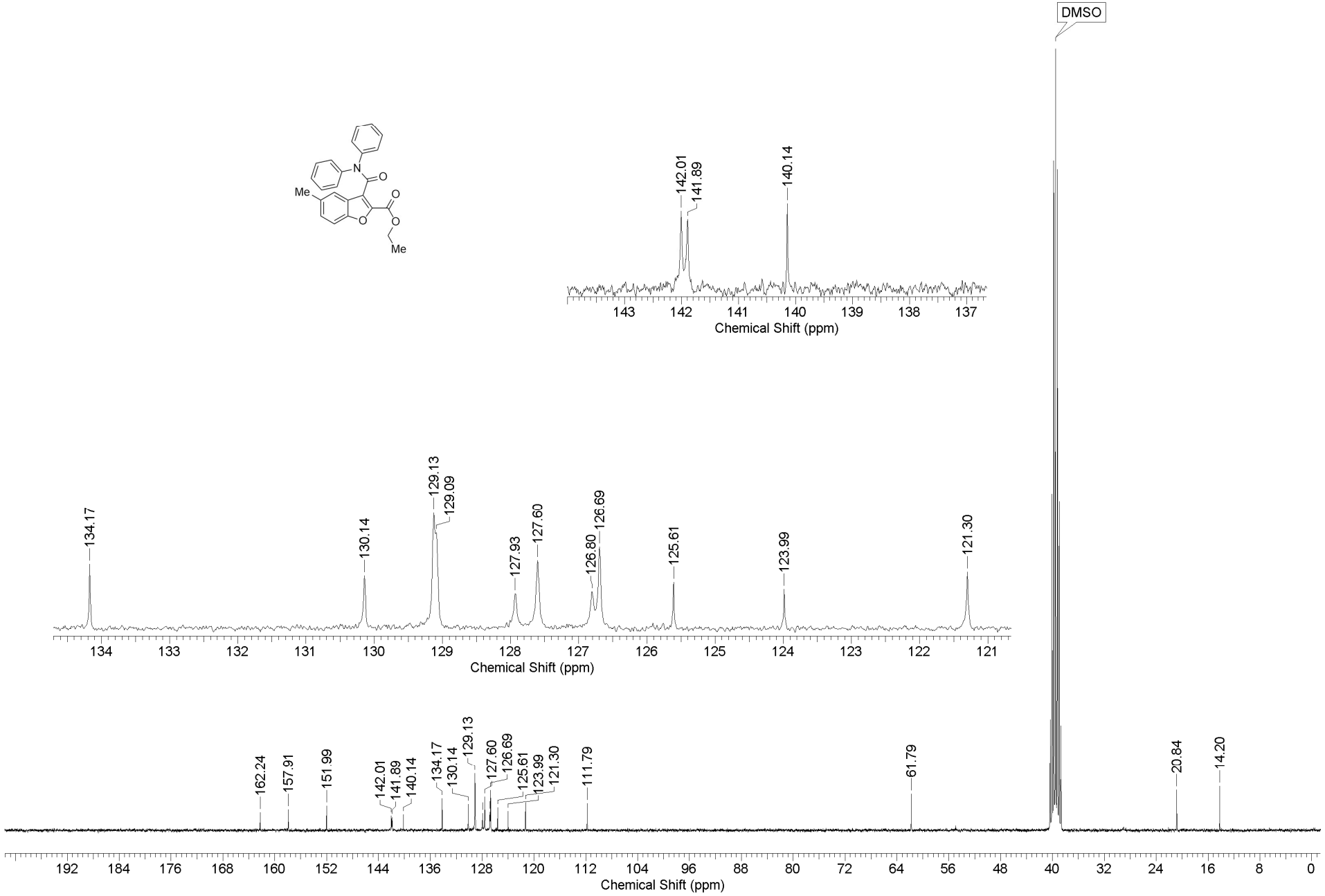

HN
O
N
OH
Water
DMSO
CH2Cl2
7.87
7.86
7.74
7.72
7.58
7.56
7.40
7.37
7.35
7.32
7.30
7.29
7.28
7.27
7.26
7.24
7.16
7.15
7.13
7.12
7.10
7.09
7.07
7.02
6.99
13.18
13.01
1.00
1.01
1.03
1.99
1.99
2.97
1.92
0.93
Chemical Shift (ppm)


DMSO
HN
O
N
OH
158.25
154.55
150.99
147.76
140.83
Chemical Shift (ppm)
133.17
130.00
123.92
123.49
122.58
122.54
118.56
118.09
117.47
117.00
113.24
111.63
Chemical Shift (ppm)
Chemical Shift (ppm)


Water
DMSO
CH2Cl2
12.72
12.78
8.48
8.47
7.88
7.85
7.60
7.58
7.55
7.38
7.35
7.33
7.17
7.16
7.14
7.13
7.09
7.06
7.04
7.02
6.99
6.97
6.94
1.91
0.93
1.00
1.04
4.08
1.04
4.00
Chemical Shift (ppm)


DMSO
158.68
154.89
139.97
160
155
150
145
140
135
Chemical Shift (ppm)
131.98
130.58
130.53
130.47
130.18
129.25
128.97
127.68
126.73
126.11
132.5
132.0
131.5
131.0
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
125.5
Chemical Shift (ppm)
123.76
120.39
117.69
115.34
124
123
122
121
120
119
118
117
116
115
Chemical Shift (ppm)
158.68
154.89
139.97
131.98
130.53
128.97
127.68
126.11
123.76
120.39
117.69
115.34
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)


Water
DMSO
CH2Cl2
Me
Me
7.76
7.54
7.51
7.48
7.47
7.36
7.34
7.32
7.31
7.21
7.19
7.16
7.13
4.41
4.38
4.43
4.36
2.46
1.37
1.35
1.32
1.06
5.04
2.13
5.03
2.00
2.99
3.14
Chemical Shift (ppm)


DMSO
142.01
141.89
140.14
143
142
141
140
139
138
137
Chemical Shift (ppm)
134.17
130.14
129.13
129.09
127.93
127.60
126.80
126.69
125.61
123.99
121.30
134
133
132
131
130
129
128
127
126
125
124
123
122
121
Chemical Shift (ppm)
162.24
157.91
151.99
142.01
141.89
140.14
134.17
130.14
129.13
127.60
126.69
125.61
123.99
121.30
111.79
61.79
20.84
14.20
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)