

Supplementary Material for

Synthesis and Structure of N-(1-(Bromomethyl)-7,7-dimethylbicyclo[2.2.1]heptan-2-yl)benzenesulfonamide

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Experimental procedures and characterization of new compounds

Melting points were determined on a MeltEMP apparatus and are uncorrected. NMR spectra were recorded on a Bruker DPX 400 nuclear magnetic resonance spectrometer at working frequencies 400 (^1H), 100 (^{13}C). IR spectra were taken on a Bruker Vertex 70 spectrophotometer in KBr or film. High-resolution mass spectra (HRMS) were measured on an Agilent 1200 HPLC chromatograph with Agilent 6210 mass spectrometer (HR-TOF-MS, ESI + ionization in acetonitrile with 0.1% HFBA). Elemental compositions were determined by accurate mass measurement with standard deviation. Crystal data were collected on a Bruker D8 Venture diffractometer with MoK α radiation ($\lambda = 0.71073$) using the φ and ω scans.

X-ray study and refinement

Crystal data were collected on a Bruker D8 Venture diffractometer with MoK α radiation ($\lambda = 0.71073$) using the φ and ω scans. The structures were solved and refined by direct methods using the SHELX programs set [1]. Data were corrected for absorption effects using the multi-scan method (SADABS). Nonhydrogen atoms were refined anisotropically using SHELX programs set [1]. **CCDC 2174560** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

The single crystals of **3** were obtained by re-crystallization from chloroform solution. In order to investigate the molecular structure and intermolecular interactions in the solid state, X-ray structure analysis of the single crystal of compound **3** was carried out. The molecular structure is depicted in Figure S1. Crystal data, data collection and structure refinement details are summarized in Table S1. Principal bond distances, bond angles and torsion angles are presented in Table S2.

References:

1. G.M. Sheldrick, Acta Crystallogr. D64 (2008) 112.

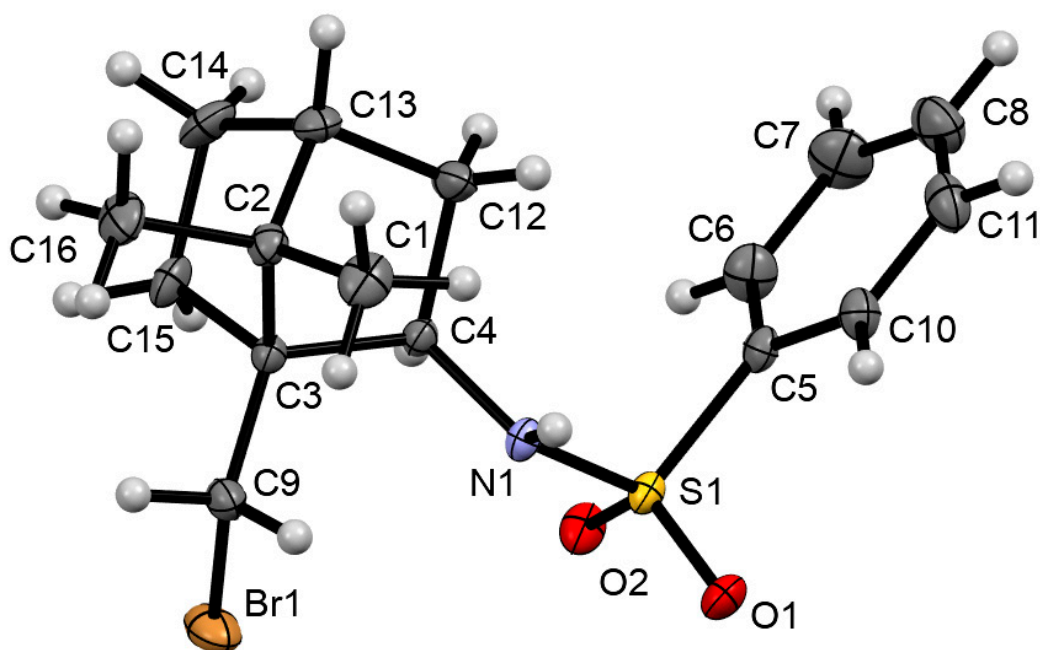


Figure S1. Molecular structure of compound **3** (ORTEP, 20% probability ellipsoids).

Table S1. Crystal data, details of intensity measurements, and structure refinement for compound **3**

Empirical formula	C ₁₆ H ₂₂ BrNO ₂ S
Formula weight / g·mol ⁻¹	372.31
Crystal system	monoclinic
Space group	C 2/c
<i>a</i> / Å	22.4881(18)
<i>b</i> / Å	9.9283(10)
<i>c</i> / Å	16.9928(16)
α, β, γ / °	90, 118.907(3), 90
Volume / Å ³	3321.3(5)
<i>Z</i>	8
Density (calculated) / g·cm ⁻³	1.493
Absorptions coefficient / mm ⁻¹	2.605
Radiation (λ / Å)	MoK α (0.71073)
Temperature / K	293(2)
2 θ range / °	4.58 – 60.15
Crystal size / mm	0.38 × 0.40 × 0.45

Crystal habit	colorless cube
F(000)	1544
Index ranges	$-30 \leq h \leq 28, -13 \leq k \leq 13, -23 \leq l \leq 23$
Reflections collected	52719
Independent reflections	4862
Max. and min. transmission	0.4171 / 0.7460
Number of ref. parameters	196
R_1 / wR_2 [$I > 2\sigma(I)$]	0.0543 / 0.1359
R_1 / wR_2 (all data)	0.0877 / 0.1542
Goodness-of-fit on F^2	1.038
Largest diff. peak and hole / $e \cdot \text{\AA}^{-3}$	1.472 / - 1.386
Weight scheme	$w=1/[\sigma^2(F_o^2) + (0.0603 P)^2 + 9.0156 P]$ where $P=(F_o^2 + 2F_c^2)/3$

Table S2. Bond lengths, bond and torsion angles in compound **3**

Bond	<i>l</i> , Å	Angle	φ , °	Torsion angle	θ , °
Br1-C9	1.956(3)	O2-S1-O1	119.78(15)	O2-S1-N1-C4	-47.0(3)
S1-O2	1.427(2)	O2-S1-N1	108.42(14)	O1-S1-N1-C4	-176.4(2)
S1-O1	1.434(2)	O1-S1-N1	105.32(14)	C5-S1-N1-C4	68.9(2)
S1-N1	1.630(2)	O2-S1-C5	107.49(15)	C1-C2-C3-C9	60.2(4)
S1-C5	1.772(3)	O1-S1-C5	107.73(15)	C13-C2-C3-C9	-179.8(3)
N1-C4	1.471(4)	N1-S1-C5	107.55(14)	C16-C2-C3-C9	-63.1(4)
C1-C2	1.525(5)	C4-N1-S1	118.6(2)	C1-C2-C3-C15	-175.8(3)
C2-C13	1.533(5)	C1-C2-C13	115.4(3)	C13-C2-C3-C15	-55.8(3)
C2-C16	1.534(4)	C1-C2-C16	106.8(3)	C16-C2-C3-C15	60.9(4)
C2-C3	1.563(4)	C13-C2-C16	112.6(3)	C1-C2-C3-C4	-68.4(3)
C3-C9	1.492(4)	C1-C2-C3	114.4(3)	C13-C2-C3-C4	51.6(3)
C3-C15	1.555(4)	C13-C2-C3	93.5(2)	C16-C2-C3-C4	168.2(3)
C3-C4	1.572(4)	C16-C2-C3	114.0(3)	S1-N1-C4-C12	-94.1(3)
C4-C12	1.549(4)	C9-C3-C15	114.7(3)	S1-N1-C4-C3	147.1(2)
C5-C6	1.370(5)	C9-C3-C2	115.0(3)	C9-C3-C4-N1	-30.7(4)
C5-C10	1.392(5)	C15-C3-C2	100.9(2)	C15-C3-C4-N1	-158.1(3)
C6-C7	1.400(7)	C9-C3-C4	116.8(2)	C2-C3-C4-N1	96.8(3)
C7-C8	1.360(8)	C15-C3-C4	103.9(2)	C9-C3-C4-C12	-156.6(3)

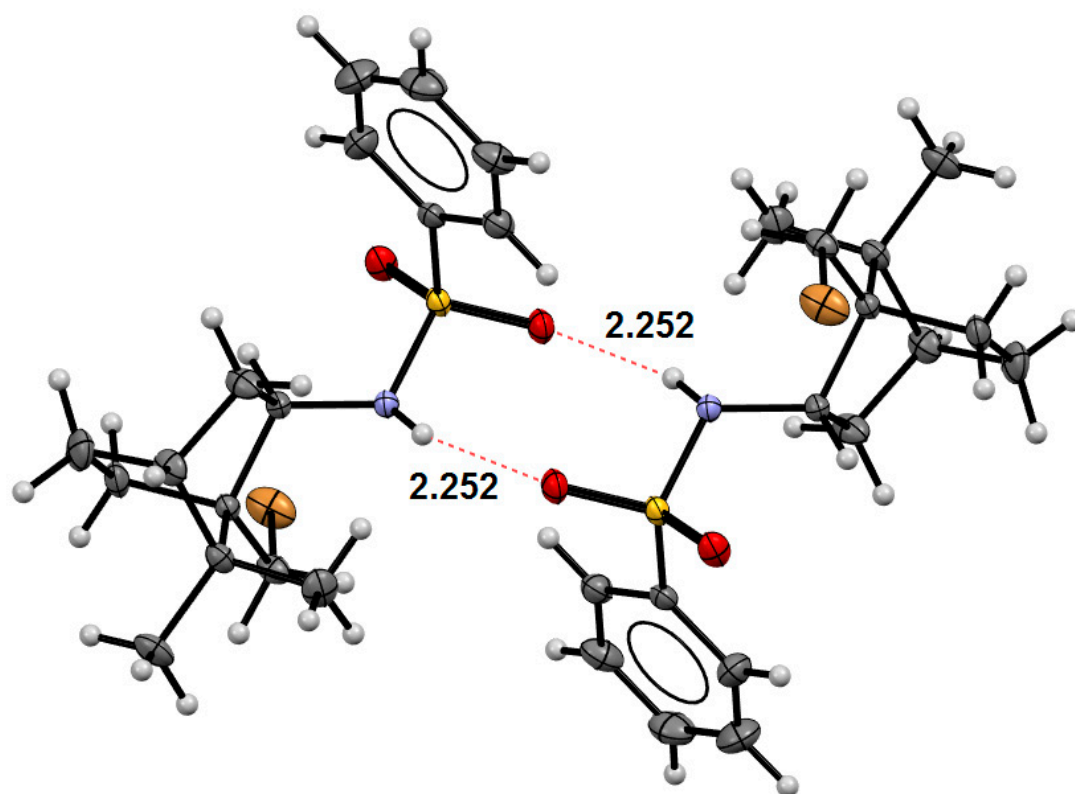
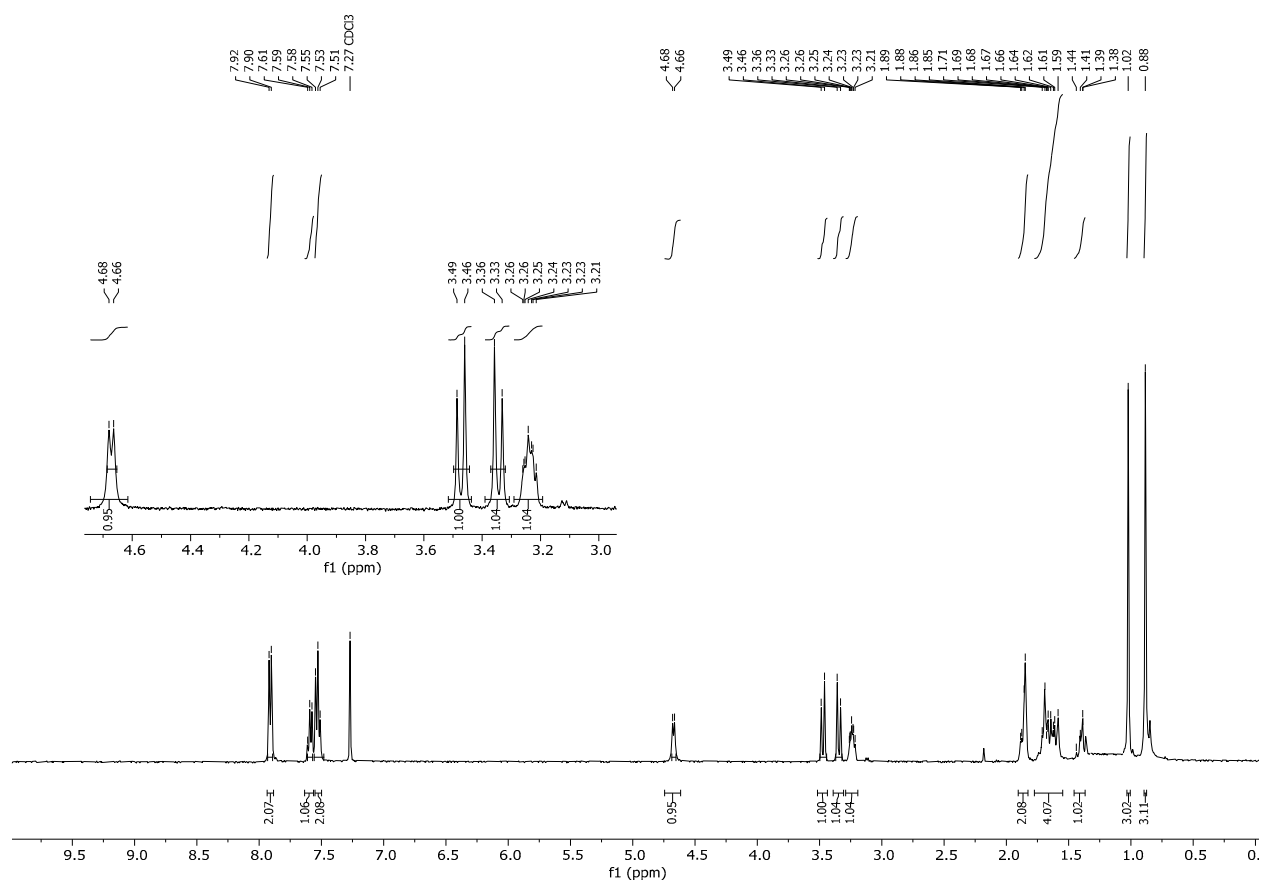


Figure S2. Hydrogen bonds $\text{NH}\cdots\text{O}=\text{S}$ in the crystal of **3**.



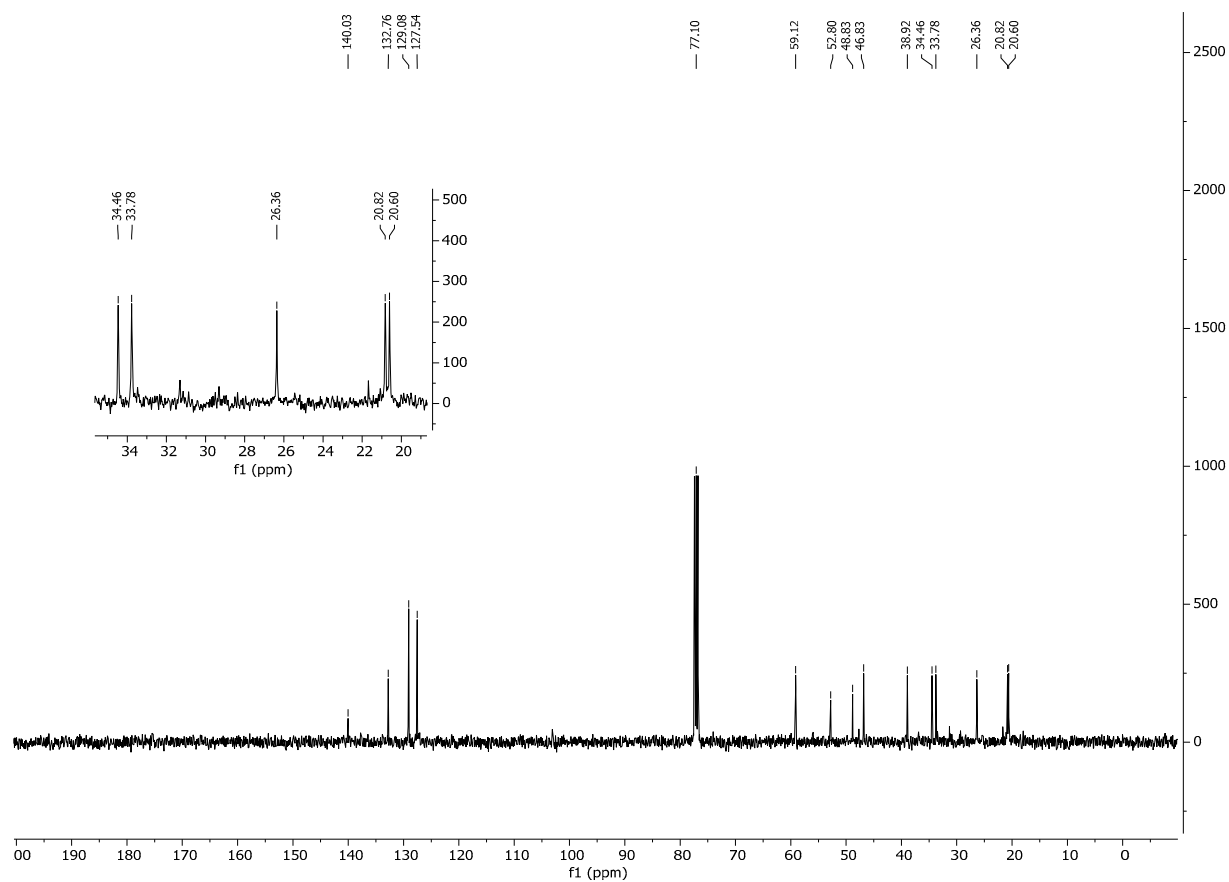


Figure S4. ^{13}C NMR spectrum of compound **3**.

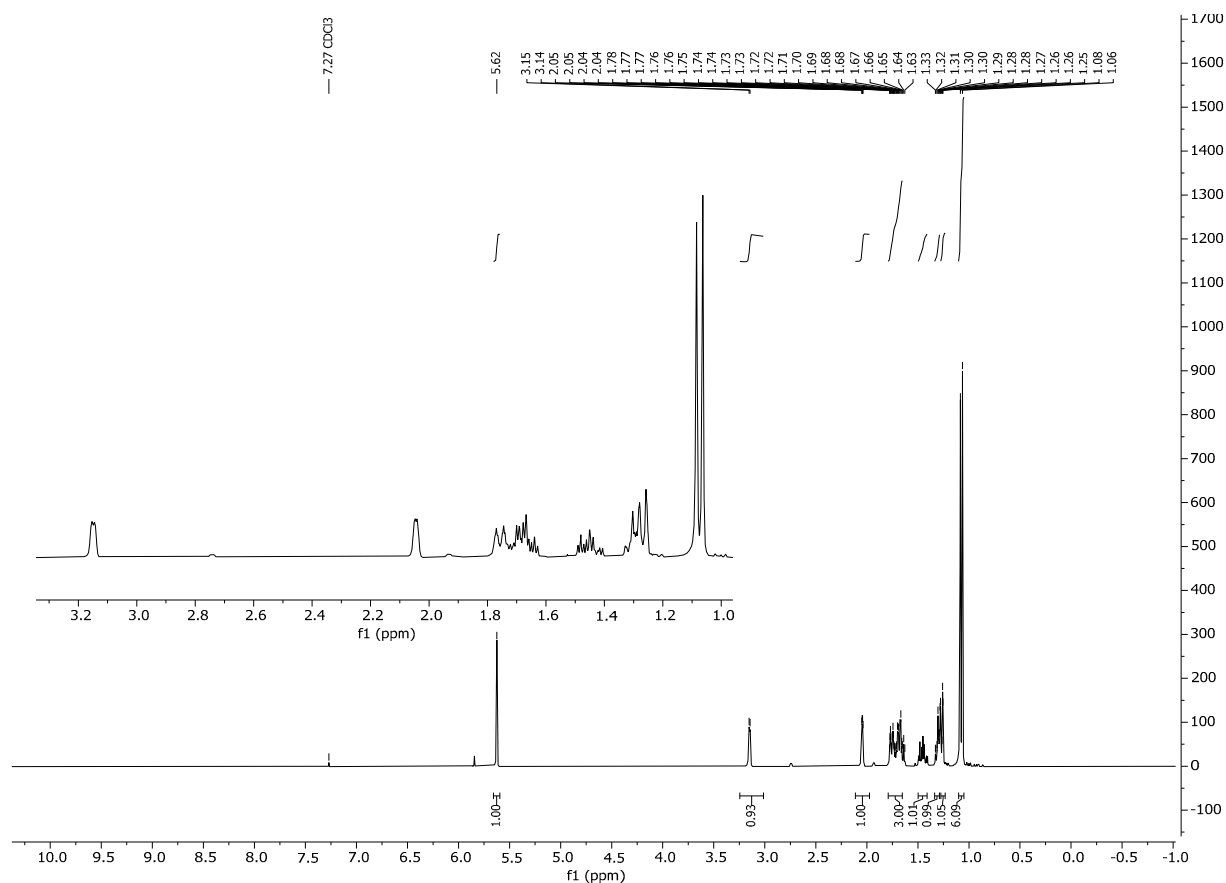


Figure S5. ^1H NMR spectrum of compound 4.

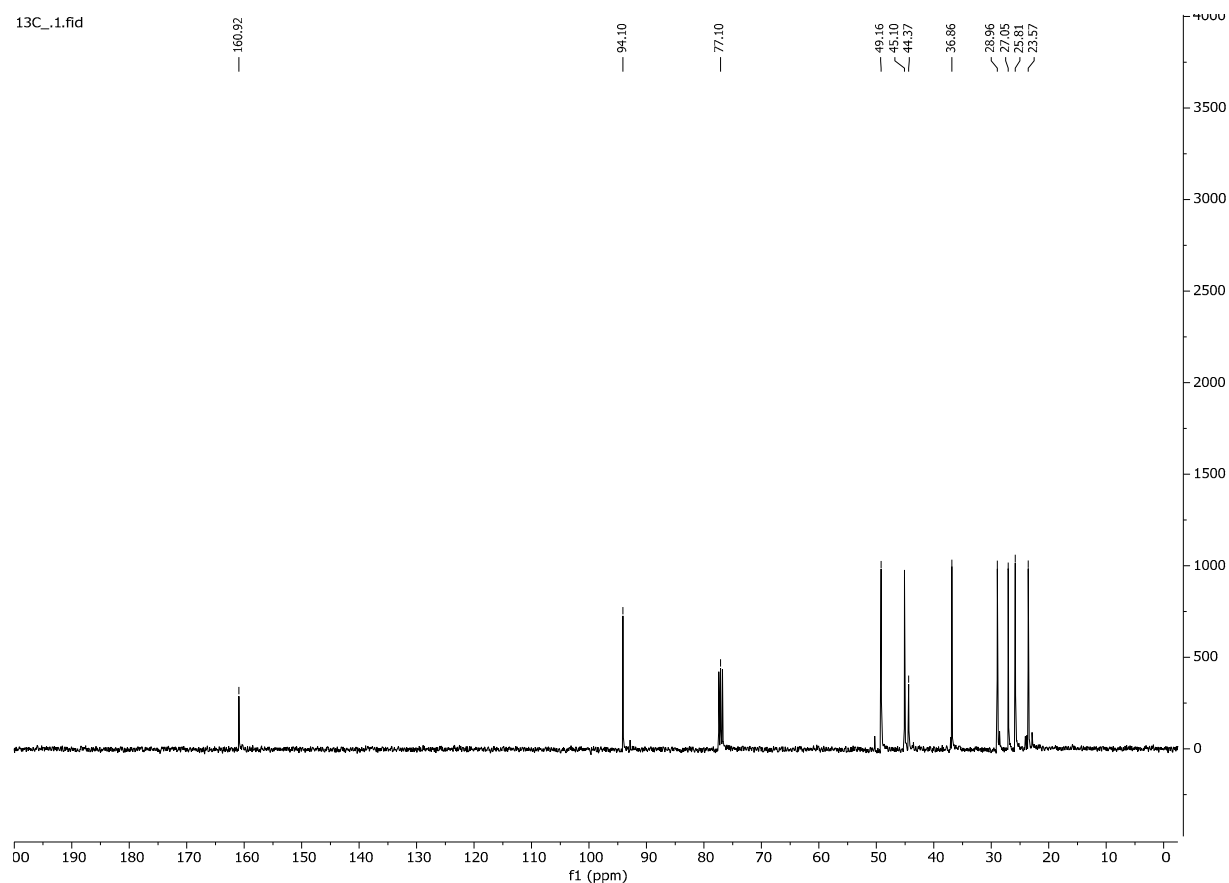


Figure S6. ^{13}C NMR spectrum of compound **4**.

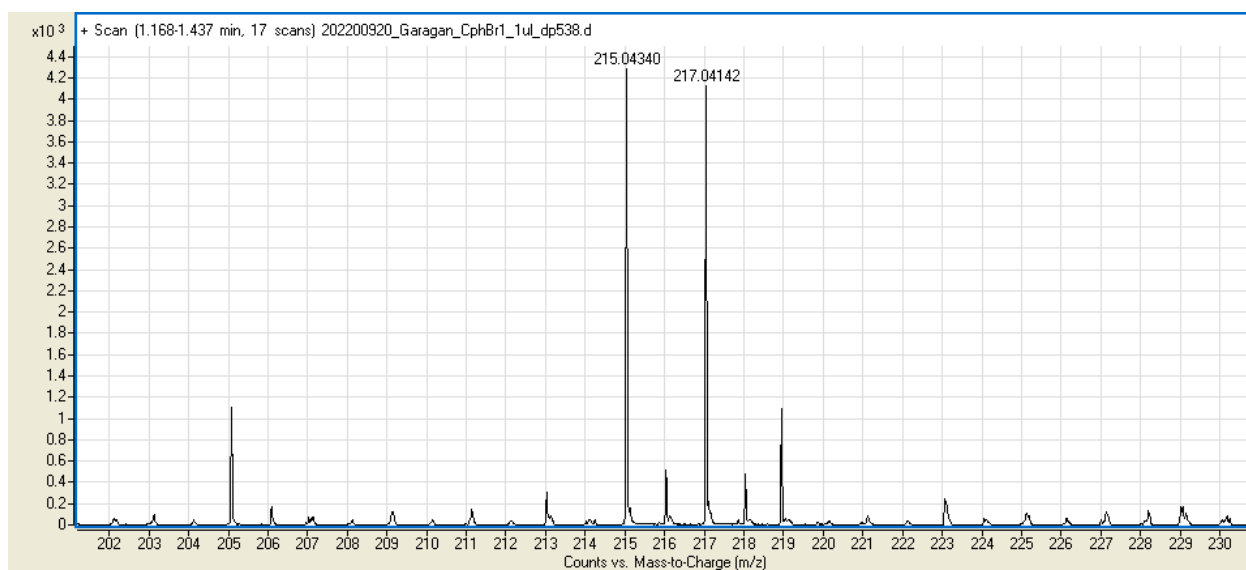


Figure S7. HRMS of compound **4**.