Molbank **2015**, M857

Supporting Information

Unless specified, reagents were obtained from commercial sources and used without further purification. Solvents were obtained from Fisher Scientific, and H_2O was deionised before use.

NMR spectra were recorded on a Varian VNMRS-700 instrument and were calibrated to the residual solvent according to the literature [1]. Assignments are based on DEPT-135, COSY, NOESY, HSQC and HMBC spectra.

Liquid chromatography-mass spectrometry (LCMS) was performed on an Agilent HP 1100 series chromatograph (Mercury Luna 3μ C18 (2) column) attached to a Waters ZQ2000 mass spectrometer with ESCi ionisation source in ESI mode. Elution was carried out at a flow rate of 0.6 mL/min using a reverse phase gradient of MeCN–water containing 0.1% formic acid. Gradient = 0–1 min: hold MeCN 5%, 1–4 min: ramp MeCN 5–95%, 4–5 min: hold MeCN 95%, 5–7 min: ramp MeCN 95–5%, 7–8 min: hold MeCN 5%. Retention times are reported as Rt.

High resolution mass spectra (HRMS) were recorded on a Waters Micromass LCT Premier spectrometer using time of flight with positive electrospray ionisation (ESI+), an ABI/MDS Sciex Q-STAR Pulsar with ESI+ and an ASAP (atmospheric pressure solids analysis probe ionisation), or a Bruker BioApex II 4.7e FTICR utilising either ESI+ or a positive electron ionisation (EI+) source equipped with a direct insertion probe. The mass reported is that containing the most abundant isotopes (^{35}Cl and ^{79}Br). Limit: ± 5 ppm.

IR spectra were recorded neat on a Perkin-Elmer Spectrum Two FT-IR spectrometer using Universal ATR sampling accessories. Letters in parentheses refer to the relative absorbency of the peak: w—weak (<40% of the most intense peak), m—medium (40–75% of the most intense peak), s—strong (>75% of the most intense peak) and br—broad.

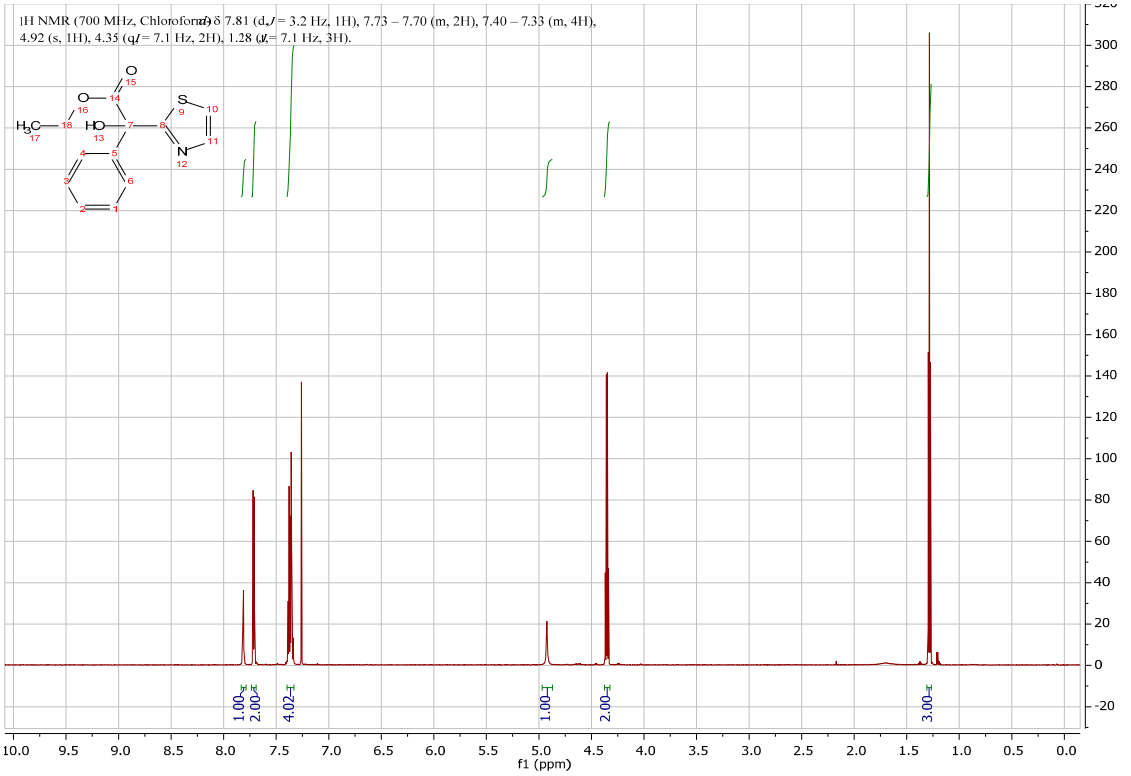
Melting points were recorded on an Optimelt automated melting point system with a heating rate of 1 °C/min (70% onset point and 10% clear point) and are uncorrected.

The X-ray single crystal data for compound **3** have been collected at 120.0(2)K on an Agilent XCalibur 4-circle diffractometer (Sapphire-3 CCD detector, graphite monochromator, λMoKα, λ = 0.71073Å, ω-scan, 1.0°/frame) equipped with a Cryostream (Oxford Cryosystems) open-flow nitrogen cryostat. The structures was solved by direct method and refined by full-matrix least squares on F^2 for all data using Olex2 [2] and SHELXTL [3] software. All non-hydrogen atoms were refined anisotropically, hydrogen atoms were found in the difference Fourier maps and refined isotropically. Crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC-1049429.

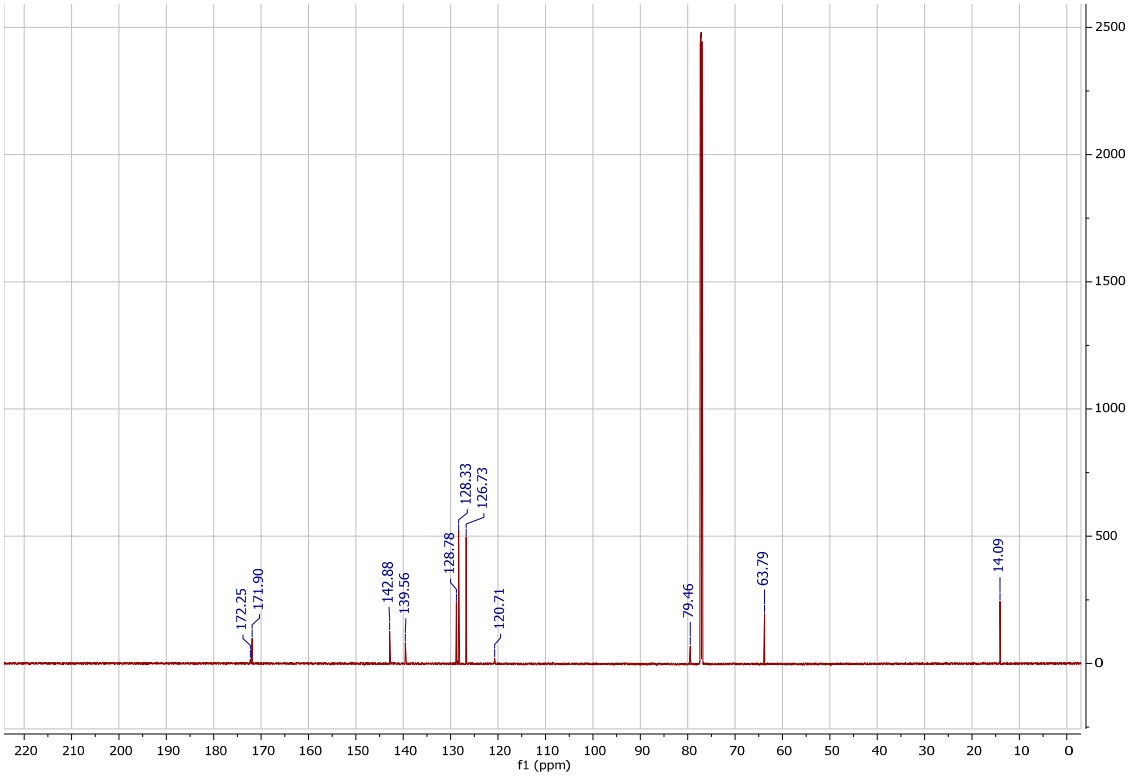
Crystal data for compound **3**: $C_{13}H_{13}NO_3S$, M = 263.30, monoclinic, space group $P2_1/n$ (no. 14), a = 7.5217(2), b = 11.1445(3), c = 14.9519(4) Å, β = 91.003(3)°, V = 1253.16(6) $Å^3$, Z = 4, T = 120.0 K, μ(MoKα) = 0.257 mm^{-1}, D_{calc} = 1.396 g/mm^3, 12888 reflections measured, 3654 unique (R_{int} = 0.0444) were used in all calculations. The final R_1 was 0.0419 (2878 refl. with I > 2σ(I)) and wR_2 was 0.0975 (all data), GOOF = 1.052.

Microwave heating was performed using a Biotage® Initiator or Initiator$^+$.

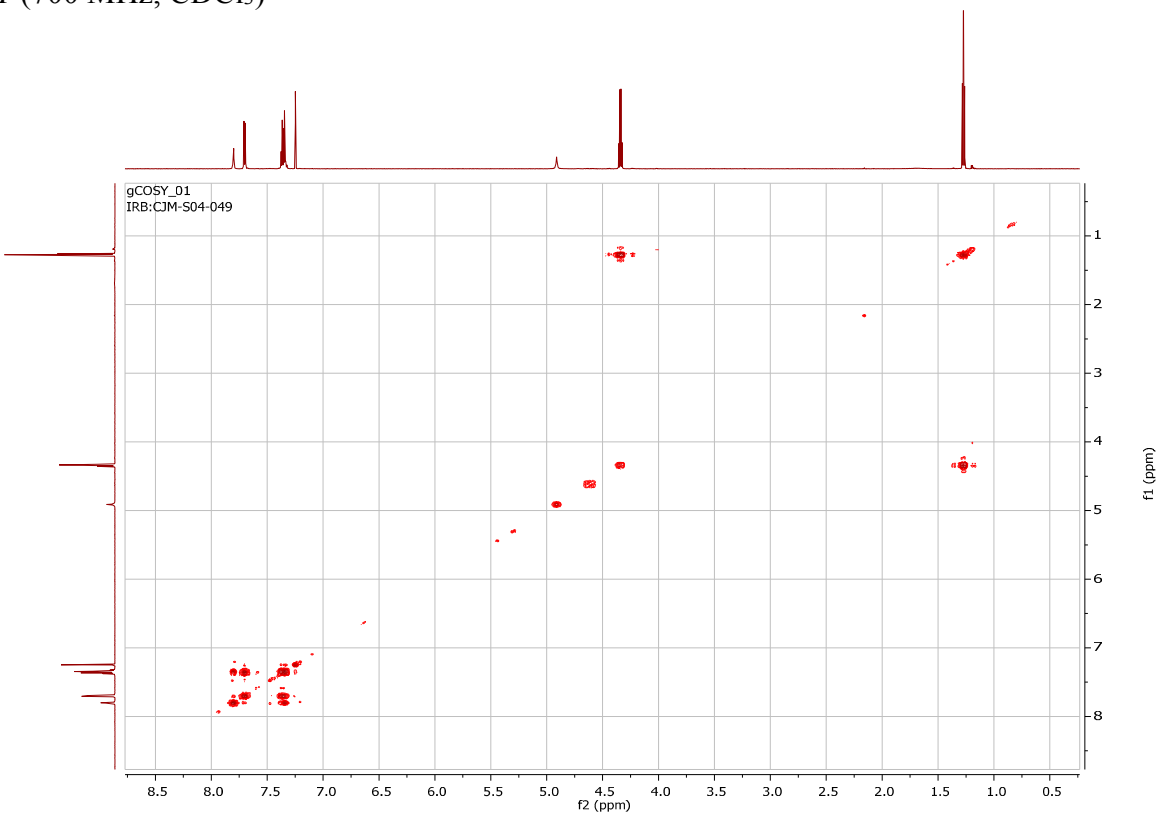
^{1}*H-NMR* (700 MHz, $CDCl_3$)



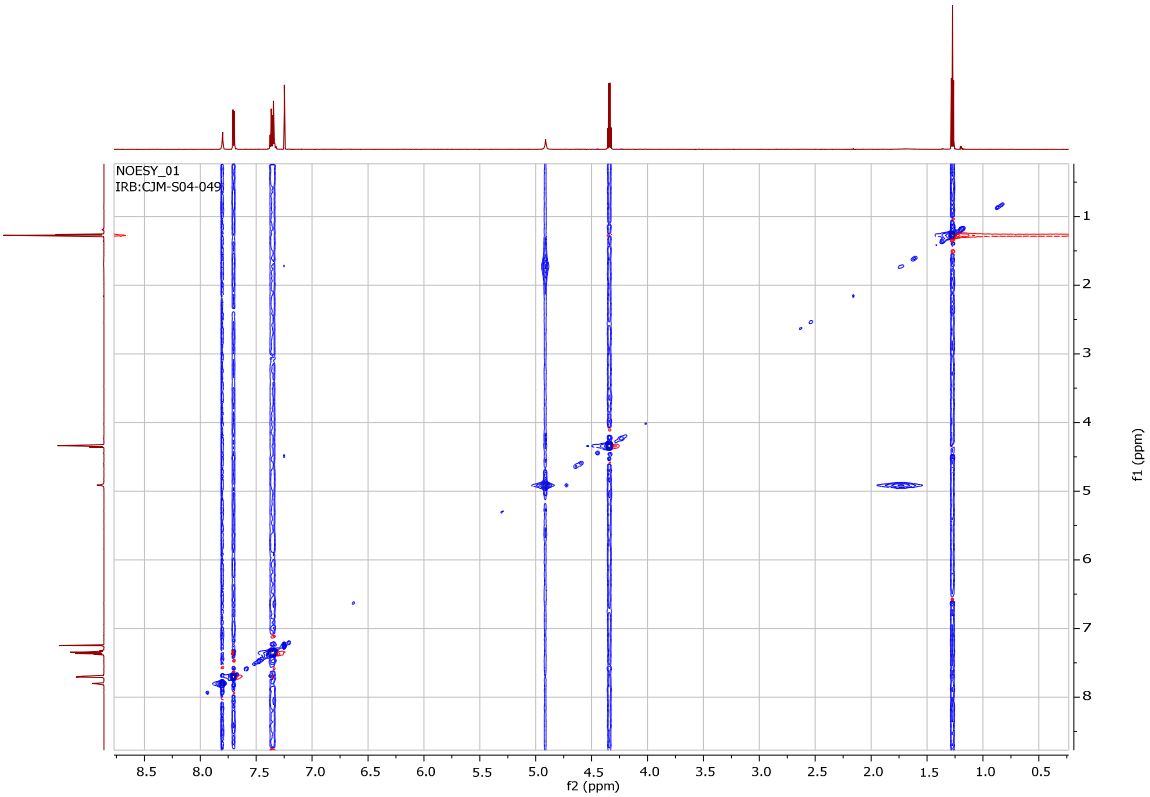
^{13}*C-NMR* (176 MHz, $CDCl_3$)



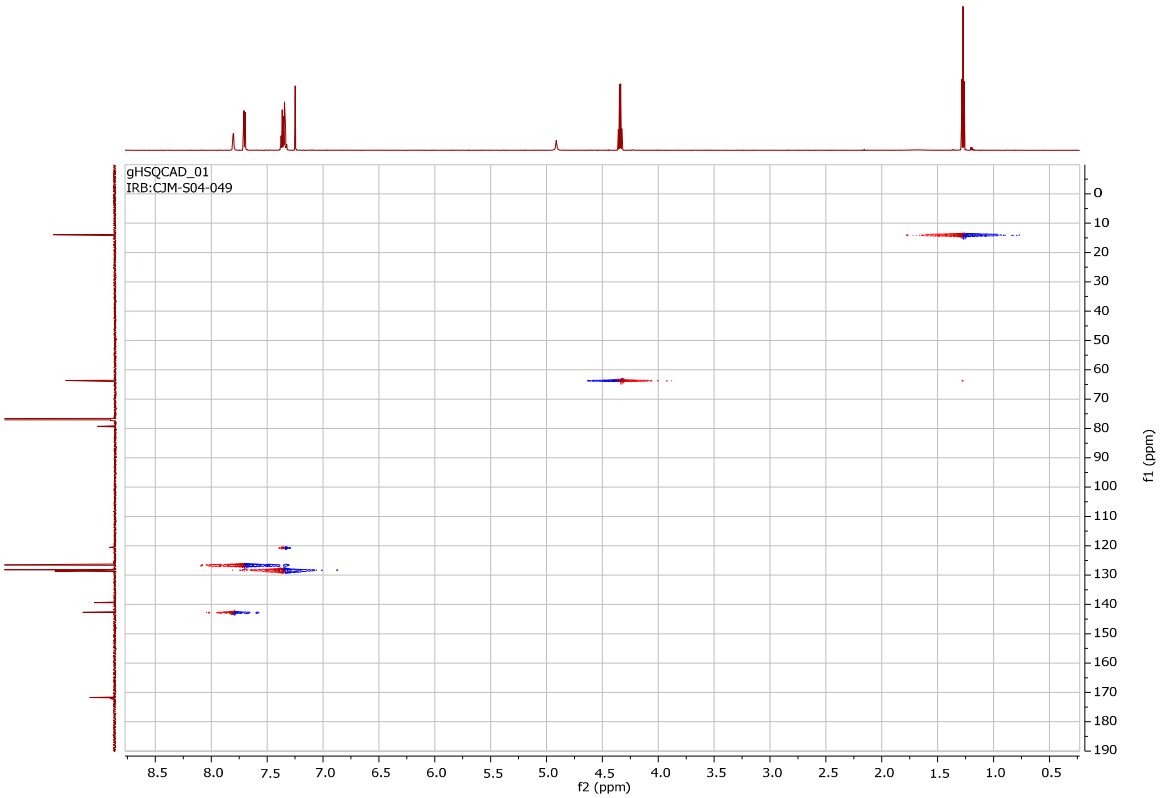
gCOSY (700 MHz, $CDCl_3$)



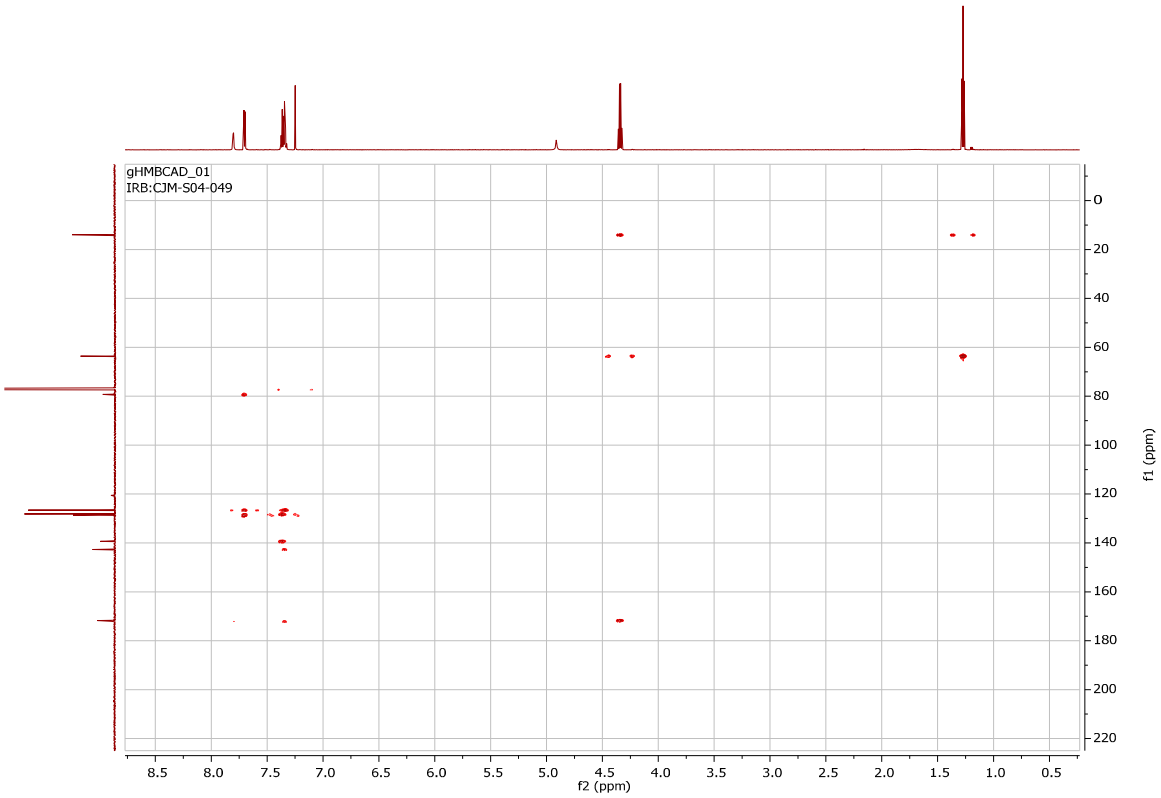
NOESY (700 MHz, $CDCl_3$)



gHSQCAD (700 MHz, $CDCl_3$)



gHMBCAD (700 MHz, $CDCl_3$)



Single crystal for X-ray for 3

14srv138 = 3 (CCDC-1049429)

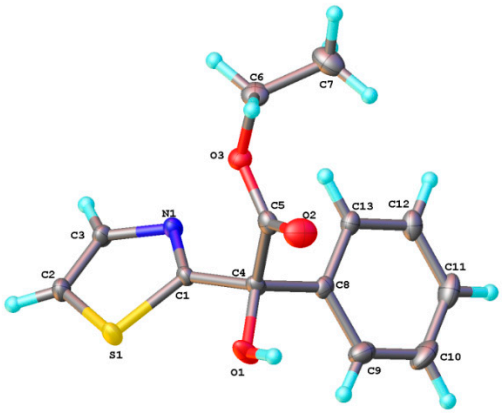


Table 1. Crystal data and structure refinement for 14srv138.	
Identification code	14srv138
Empirical formula	$C_{13}H_{13}NO_3S$
Formula weight	263.30
Temperature/K	120.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	7.5217(2)
b/Å	11.1445(3)
c/Å	14.9519(4)
α/°	90.00
β/°	91.003(3)
γ/°	90.00
Volume/Å^3	1253.16(6)
Z	4
ρ_{calc}mg/mm^3	1.396
m/mm^{-1}	0.257
F(000)	552.0
Crystal size/mm^3	0.22 × 0.2 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	4.56 to 60°
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 15$, $-20 \le l \le 21$
Reflections collected	12888
Independent reflections	3654 [R_{int} = 0.0444, R_{sigma} = 0.0470]
Data/restraints/parameters	3654/0/215
Goodness-of-fit on F^2	1.052
Final R indexes [I >= 2σ (I)]	R_1 = 0.0419, wR_2 = 0.0873
Final R indexes [all data]	R_1 = 0.0588, wR_2 = 0.0975
Largest diff. peak/hole / e Å^{-3}	0.38/−0.29

Table 2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv138. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
S1	5673.8(5)	3140.6(3)	916.1(2)	19.66(11)
O1	7069.2(16)	4220.8(9)	2389.3(8)	24.0(3)
O2	10473.4(16)	3572.6(11)	2861.8(9)	30.5(3)
O3	9938.2(14)	1743.2(9)	2284.0(7)	19.6(2)
N1	6235.1(17)	1177.7(10)	1764.1(8)	16.5(3)
C1	6502.5(19)	2333.8(12)	1817.3(9)	14.3(3)
C2	4945(2)	1834.9(13)	435.6(10)	20.2(3)
C3	5349(2)	892.3(13)	975.9(10)	18.3(3)
C4	7432(2)	3007.7(12)	2572.3(9)	15.8(3)
C5	9468(2)	2815.3(13)	2587.6(9)	17.8(3)
C6	11832(2)	1432.8(16)	2354.0(12)	24.8(3)
C7	12292(3)	946.1(18)	3259.9(13)	32.1(4)
C8	6748.4(19)	2621.0(13)	3494.0(9)	16.2(3)
C9	5672(2)	3386.8(15)	3980.3(12)	26.1(4)
C10	5059(3)	3039.6(18)	4815.6(12)	33.2(4)
C11	5518(2)	1937.1(17)	5167.3(11)	29.6(4)
C12	6593(2)	1170.8(15)	4691.1(10)	22.7(3)
C13	7215(2)	1511.2(13)	3858.5(10)	17.5(3)

Table 3. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 14srv138. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + 2hka^*b^*U_{12} + \ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	27.2(2)	14.76(18)	16.79(18)	2.23(13)	−6.58(14)	1.44(14)
O1	32.5(7)	9.5(5)	29.3(6)	1.0(4)	−15.2(5)	−1.5(4)
O2	22.8(6)	25.5(6)	43.0(7)	−5.1(5)	−5.1(5)	−7.0(5)
O3	17.2(5)	20.7(5)	20.8(5)	−1.5(4)	−0.2(4)	0.9(4)
N1	20.2(6)	13.7(6)	15.4(6)	−0.9(4)	−4.0(5)	0.1(5)
C1	16.8(7)	13.9(6)	12.1(6)	1.7(5)	−2.1(5)	0.7(5)
C2	25.2(8)	20.2(7)	14.9(7)	−3.0(5)	−5.6(6)	1.9(6)
C3	23.3(8)	15.1(7)	16.5(7)	−3.8(5)	−4.6(6)	0.4(6)
C4	20.1(7)	10.9(6)	16.4(6)	0.7(5)	−4.3(5)	0.2(5)
C5	20.0(7)	18.3(7)	14.9(7)	3.0(5)	−1.9(6)	−2.6(6)
C6	15.8(7)	30.7(9)	27.8(9)	−0.6(7)	1.9(6)	2.1(7)
C7	22.3(9)	35.9(10)	38.1(10)	10.6(8)	−3.6(8)	1.1(8)
C8	14.8(7)	18.0(7)	15.6(6)	−4.7(5)	−2.8(5)	−2.3(5)
C9	23.9(8)	25.1(8)	29.2(9)	−7.8(7)	−1.8(7)	5.2(7)
C10	26.0(9)	44.2(11)	29.7(9)	−15.0(8)	7.1(7)	3.7(8)
C11	27.5(9)	43.6(10)	17.9(8)	−5.5(7)	5.4(7)	−9.8(8)
C12	25.0(8)	27.2(8)	16.0(7)	0.0(6)	−1.6(6)	−8.0(7)
C13	19.1(7)	19.1(7)	14.3(7)	−2.4(5)	−0.1(5)	−2.9(6)

Table 4. Bond Lengths for 14srv138.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
S1	C1	1.7272(14)		C4	C5	1.546(2)
S1	C2	1.7089(15)		C4	C8	1.541(2)
O1	C4	1.4052(16)		C6	C7	1.494(2)
O2	C5	1.2006(18)		C8	C9	1.390(2)
O3	C5	1.3282(17)		C8	C13	1.394(2)
O3	C6	1.4680(19)		C9	C10	1.394(3)
N1	C1	1.3062(17)		C10	C11	1.378(3)
N1	C3	1.3809(19)		C11	C12	1.382(2)
C1	C4	1.5165(19)		C12	C13	1.391(2)
C2	C3	1.356(2)				

Table 5. Bond Angles for 14srv138.								
Atom	**Atom**	**Atom**	**Angle/°**		**Atom**	**Atom**	**Atom**	**Angle/°**
C2	S1	C1	89.62(7)		C8	C4	C5	107.10(11)
C5	O3	C6	116.83(12)		O2	C5	O3	125.41(15)
C1	N1	C3	110.53(12)		O2	C5	C4	121.68(14)
N1	C1	S1	114.39(10)		O3	C5	C4	112.90(12)
N1	C1	C4	127.03(12)		O3	C6	C7	111.03(13)
C4	C1	S1	118.58(10)		C9	C8	C4	120.17(14)
C3	C2	S1	110.02(12)		C9	C8	C13	119.03(14)
C2	C3	N1	115.44(13)		C13	C8	C4	120.80(12)
O1	C4	C1	104.24(11)		C8	C9	C10	120.20(16)
O1	C4	C5	108.98(12)		C11	C10	C9	120.32(16)
O1	C4	C8	112.12(12)		C10	C11	C12	119.96(16)
C1	C4	C5	112.70(11)		C11	C12	C13	120.13(16)
C1	C4	C8	111.75(11)		C12	C13	C8	120.36(14)

Table 6. Hydrogen Bonds for 14srv138.						
D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
O1	H1	N1[1]	0.83(2)	2.02(2)	2.8159(16)	161(2)

[1]3/2−X,1/2+Y,1/2−Z.

Table 7. Selected Torsion Angles for 14srv138.										
A	**B**	**C**	**D**	**Angle/°**		**A**	**B**	**C**	**D**	**Angle/°**
O1	C4	C1	S1	10.58(15)		C5	C4	C8	C9	128.58(15)
O1	C4	C5	O2	34.68(19)		C5	C4	C8	C13	−50.51(17)
O1	C4	C5	O3	−146.25(12)		C6	O3	C5	O2	4.8(2)
N1	C1	C4	O1	−169.10(14)		C7	C6	O3	C5	84.04(17)
C1	C4	C5	O2	149.89(14)		C8	C4	C1	S1	131.88(11)
C1	C4	C5	O3	−31.04(16)		C8	C4	C1	N1	−47.80(19)
C1	C4	C8	C9	−107.54(16)		C8	C4	C5	O2	−86.82(16)
C1	C4	C8	C13	73.37(17)		C8	C4	C5	O3	92.25(13)
C4	C5	O3	C6	−174.23(12)		C9	C8	C4	O1	9.1(2)
C5	C4	C1	S1	−107.45(12)		C13	C8	C4	O1	−170.01(13)
C5	C4	C1	N1	72.87(18)						

Table 8. Hydrogen Atom Coordinates (Å × 10^4) and Isotropic Displacement Parameters (Å^2 × 10^3) for 14srv138.				
Atom	***x***	***y***	***z***	**U(eq)**
H1	7760(30)	4680(20)	2649(15)	47(7)
H2	4330(30)	1828(16)	−118(14)	34(5)
H3	5030(20)	70(17)	856(11)	26(5)
H6A	12520(20)	2147(16)	2213(12)	24(5)
H6B	11980(30)	826(18)	1882(13)	37(5)
H7A	13550(30)	644(17)	3265(13)	35(5)
H7B	12120(30)	1541(18)	3737(14)	38(6)
H7C	11570(30)	250(20)	3380(15)	51(7)
H9	5400(20)	4119(16)	3725(12)	23(5)
H10	4290(30)	3571(19)	5128(14)	47(6)
H11	5090(30)	1696(17)	5723(14)	36(6)
H12	6900(20)	373(16)	4938(12)	28(5)

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2. Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H. *OLEX2*: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr*. **2009**, *42*, 339–341.
3. Sheldrick, G.M. A short history of *SHELX*. *Acta Crystallogr*. **2008**, *A64*, 112–122.