Supporting information

EXPERIMENTAL INVESTIGATION OF TETRACYCLIC COMPOUNDS CONTAINING A NINE－MEMBERED SULTAM VIA COBALT ALKYNE COMPLEXES

Kyosuke Kaneda,* Misuzu Fujita, and Risa Naruse

Hokkaido Pharmaceutical University School of Pharmacy, 7-15-4-1 Maeda, Teine, Sapporo, Hokkaido 006-8590, Japan; E-mail: kaneda@hokuyakudai.ac.jp

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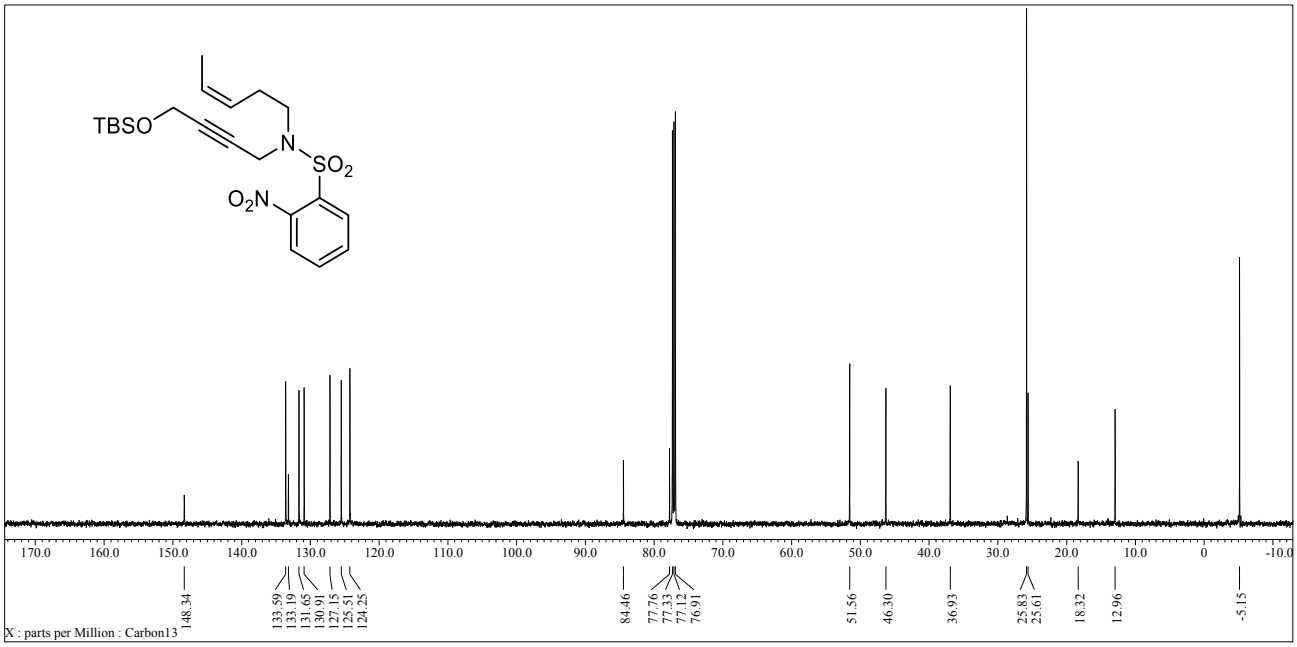
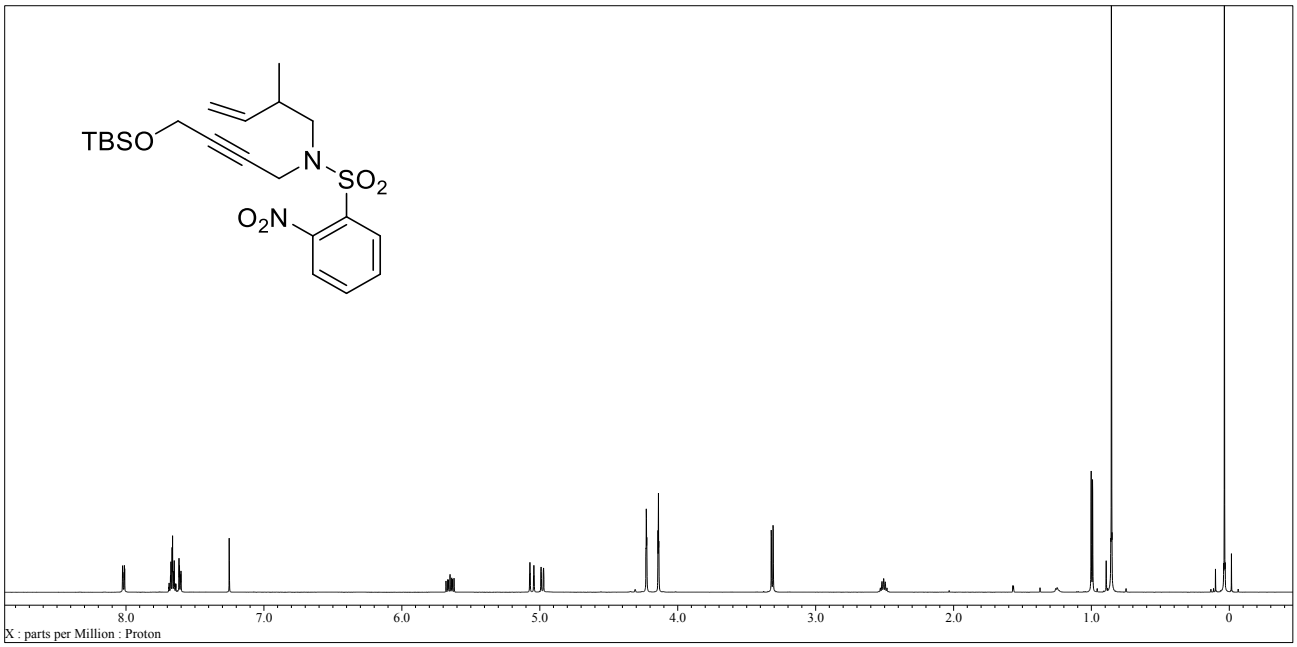
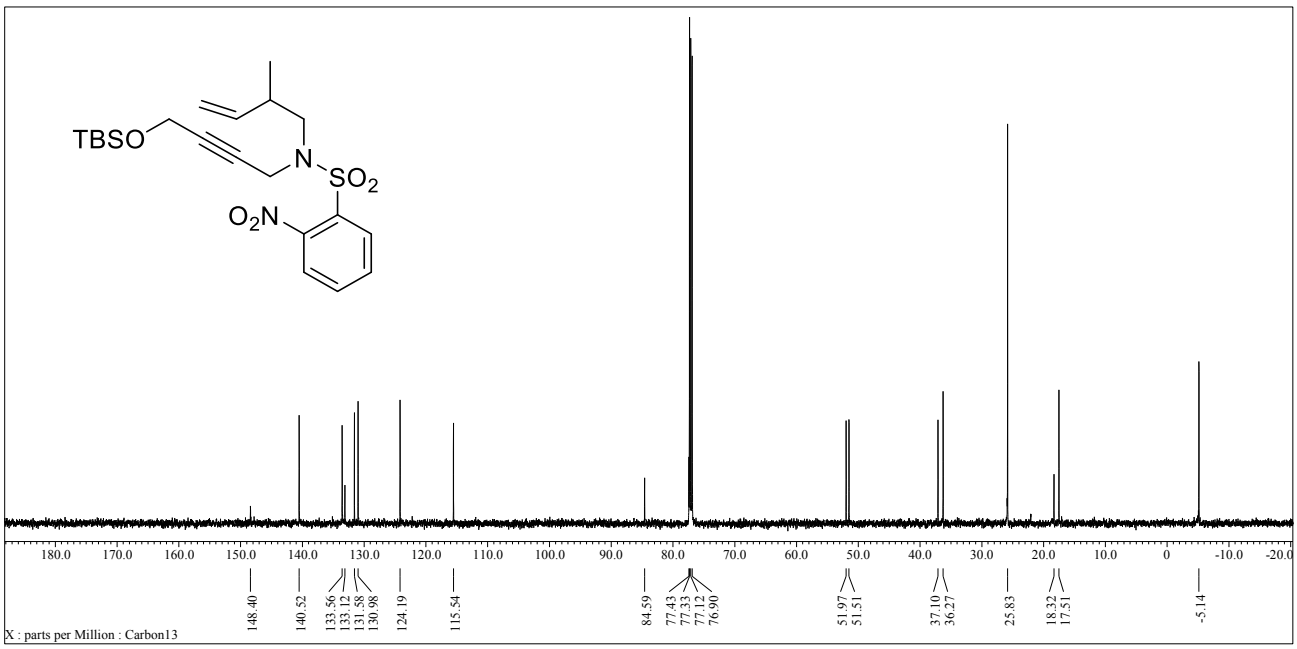
^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **2b**



^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **2b**



^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **2c**



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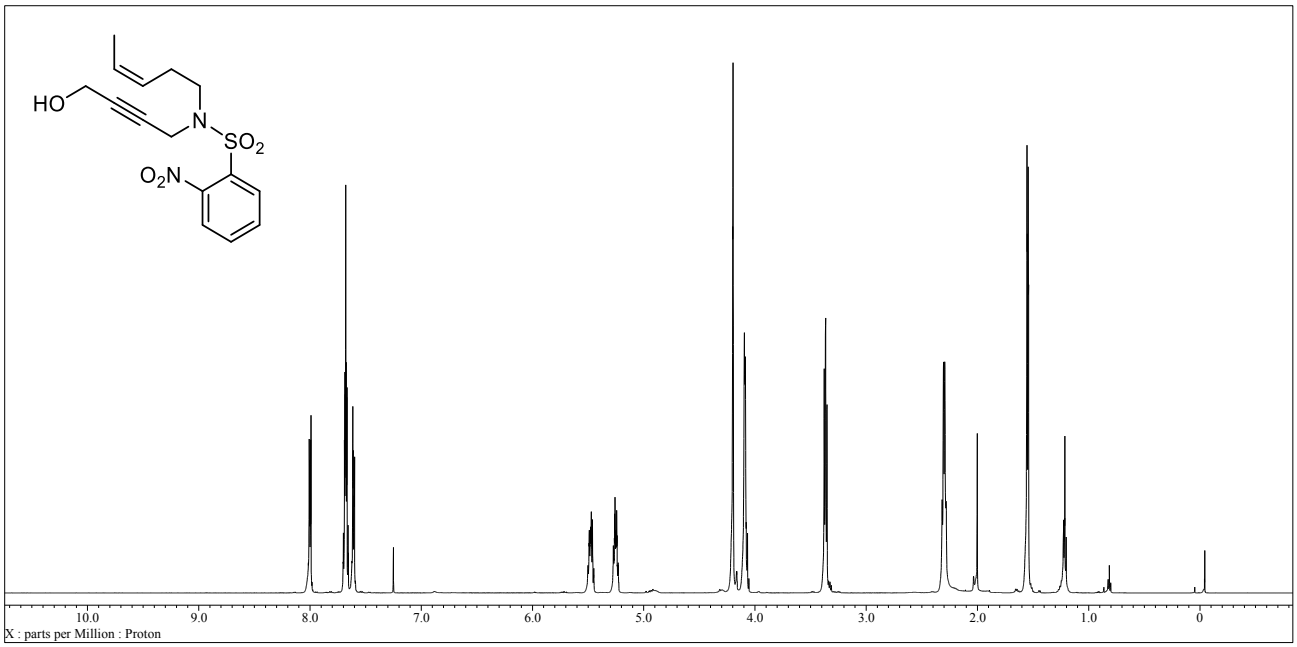

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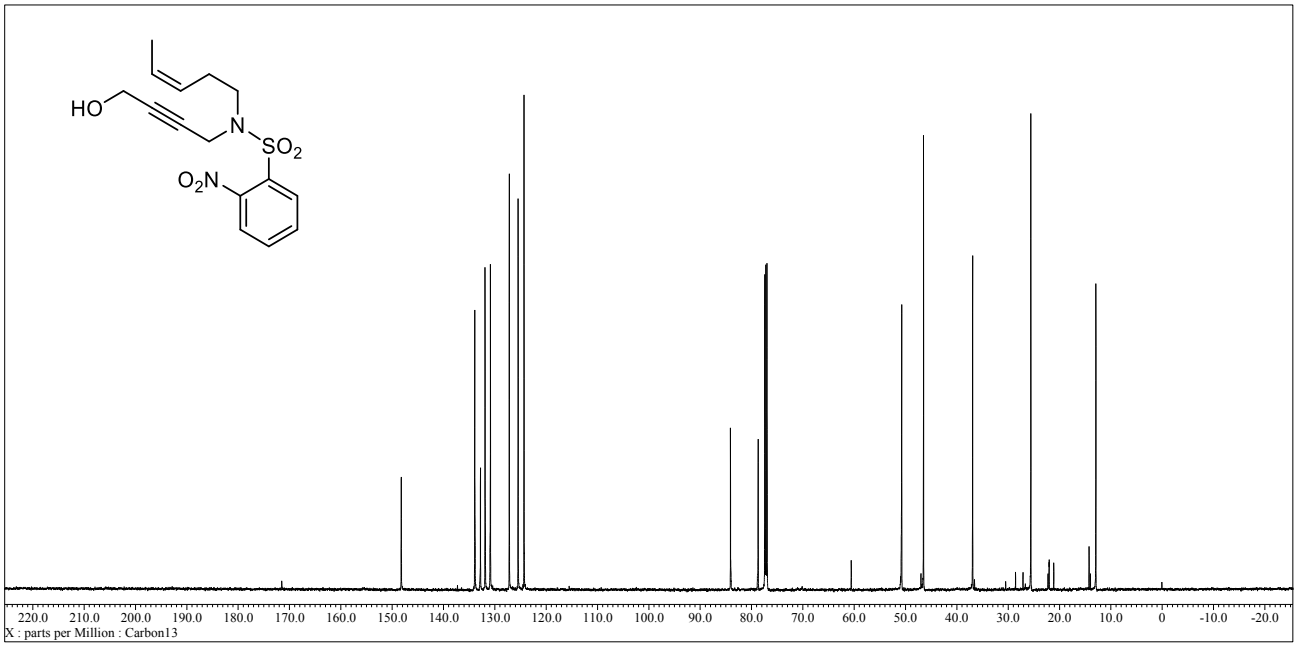

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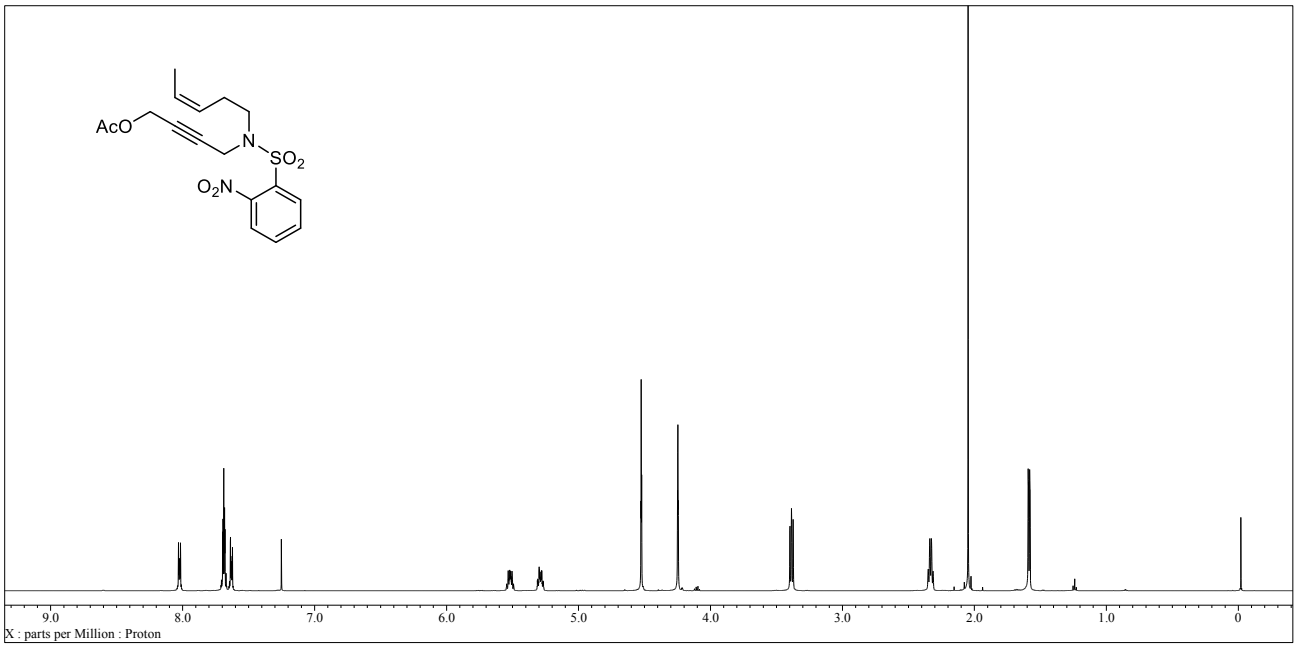

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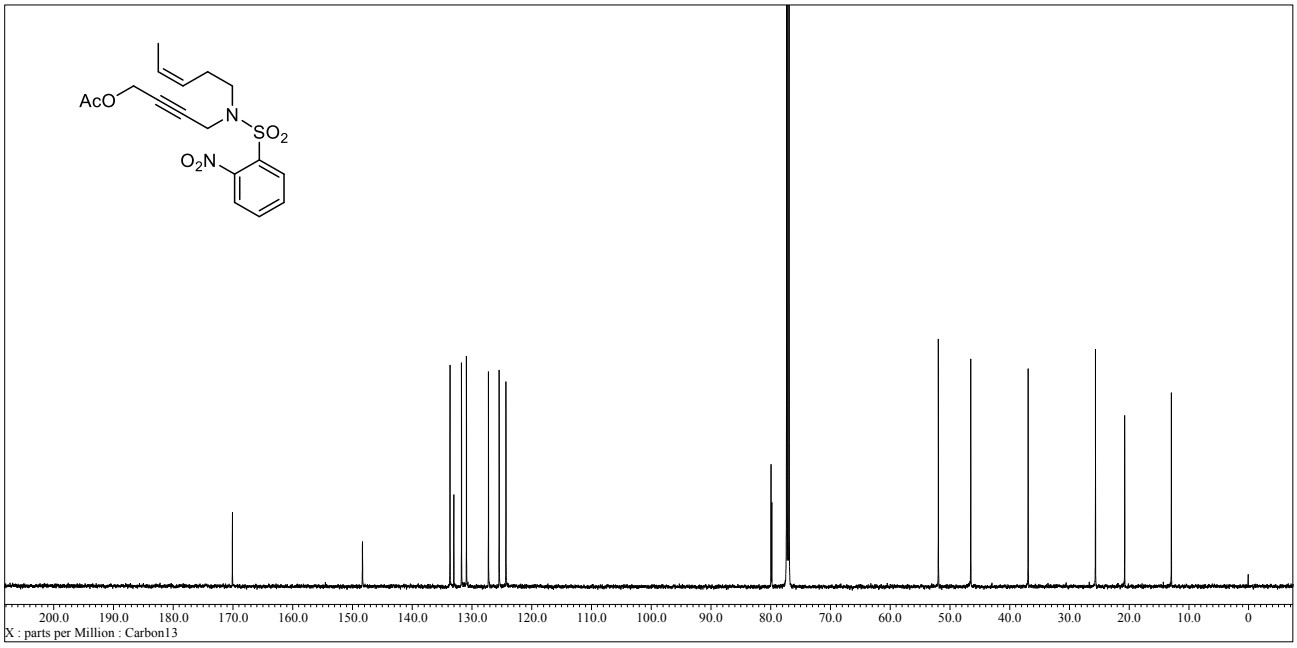

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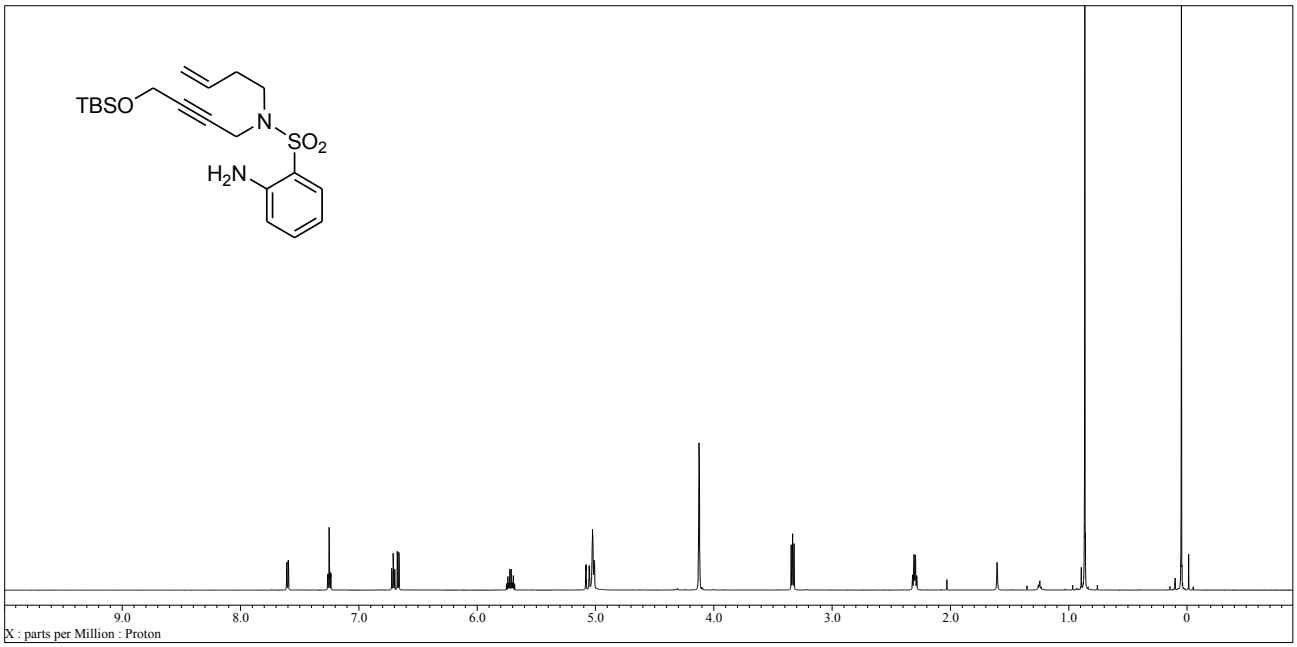

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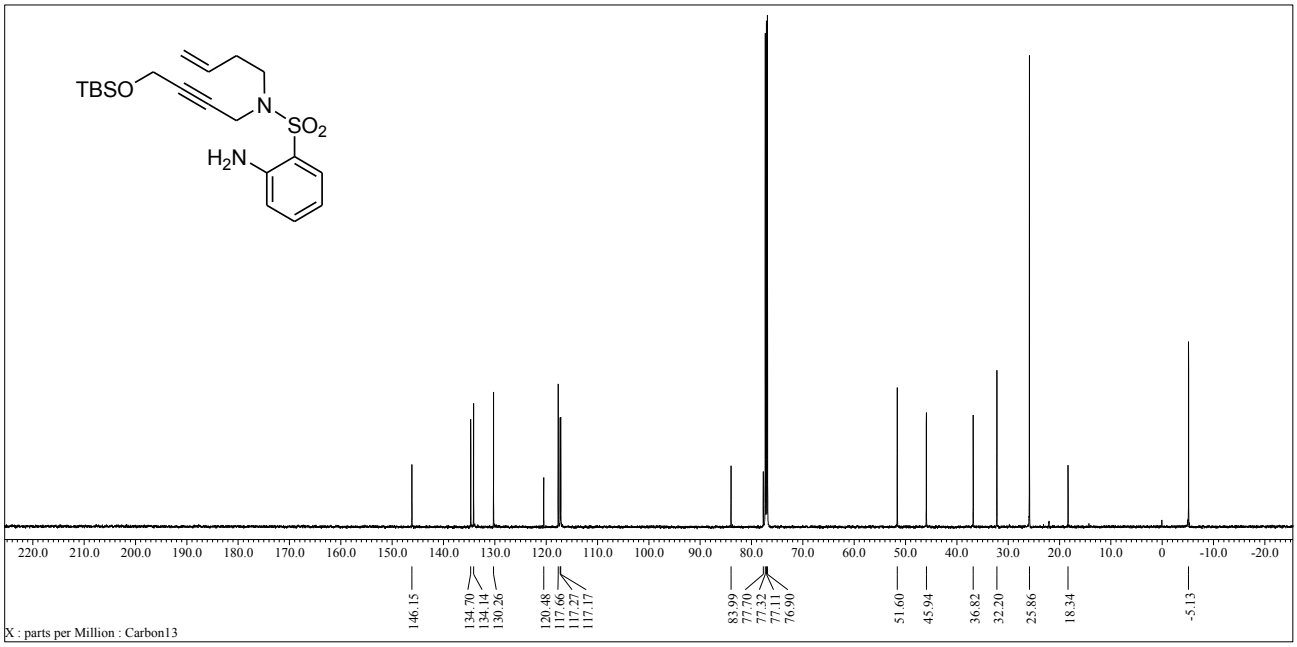

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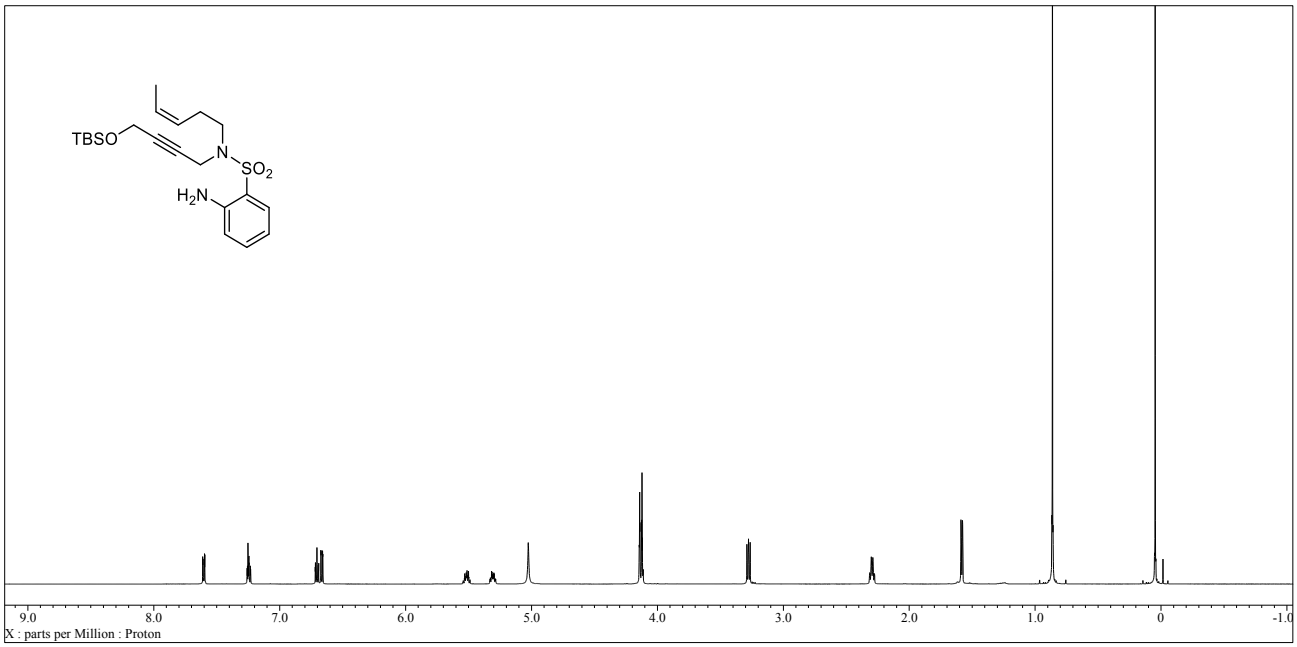

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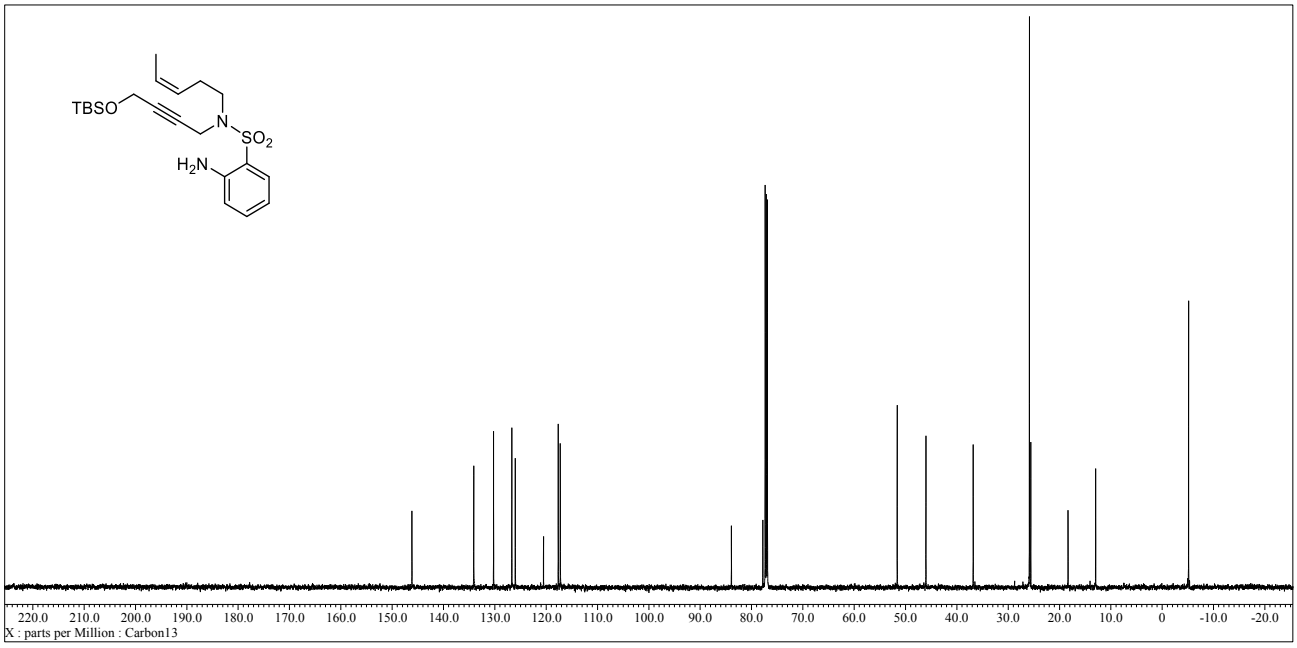


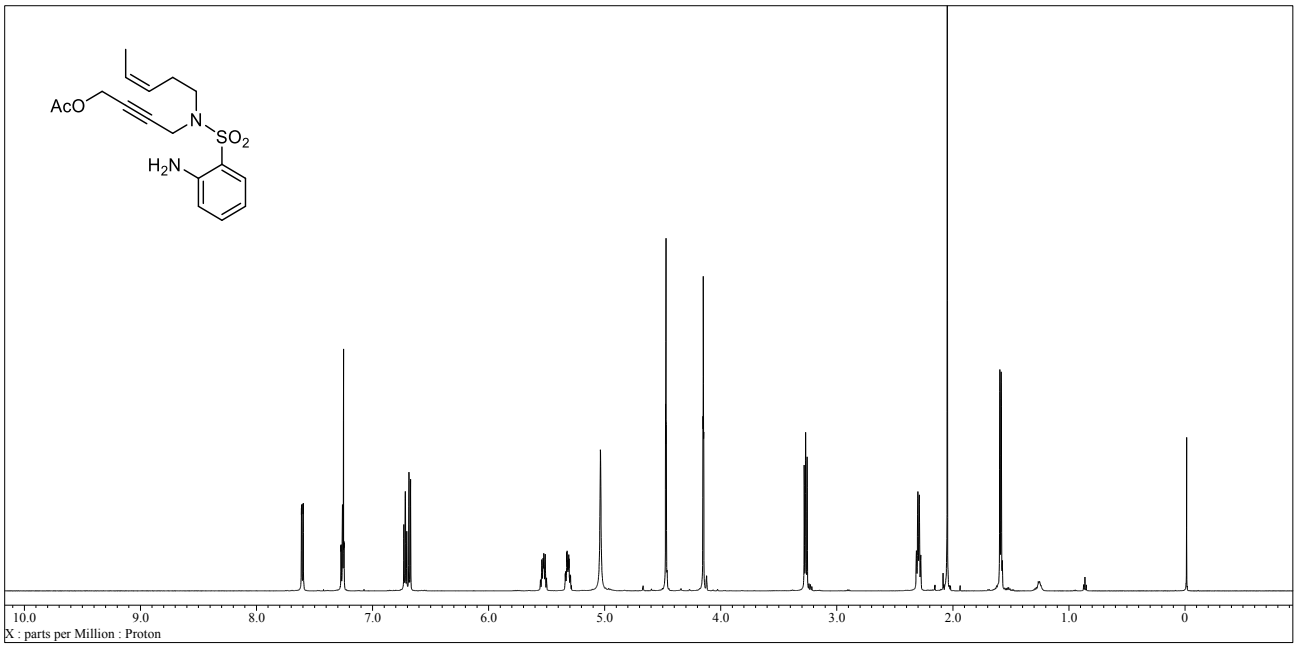
^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **5b**

^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **5c**

^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **5c**

^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **5d**

^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **5d**



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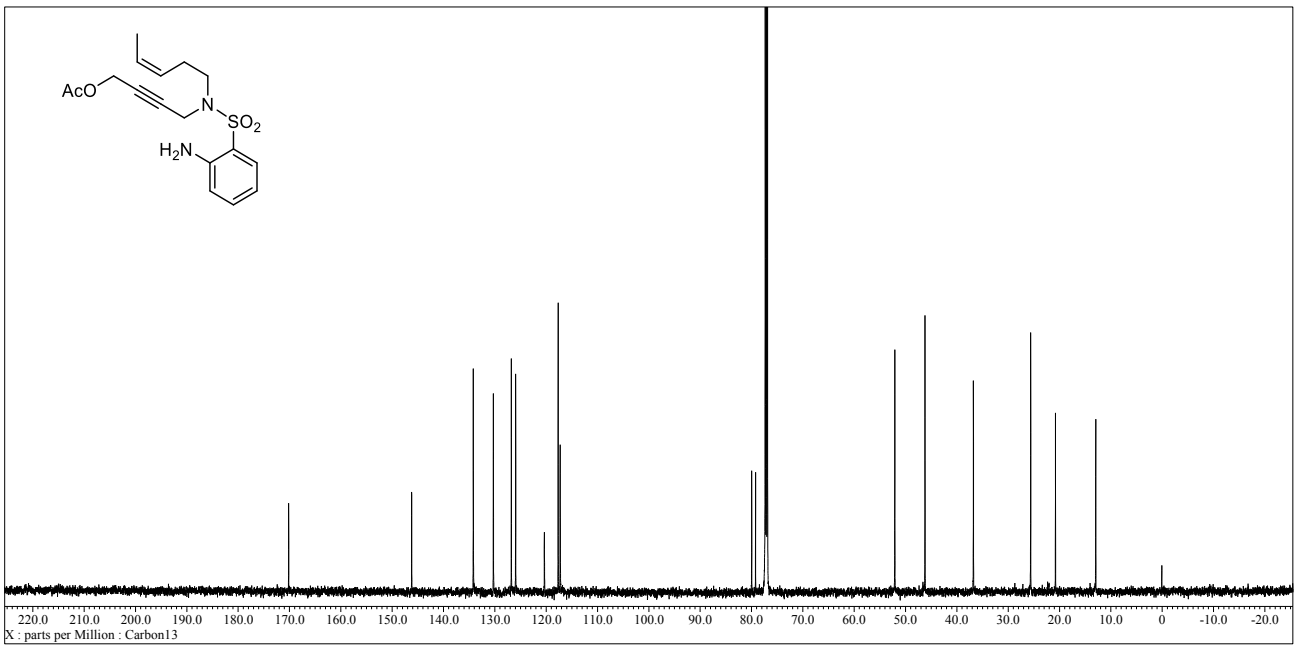

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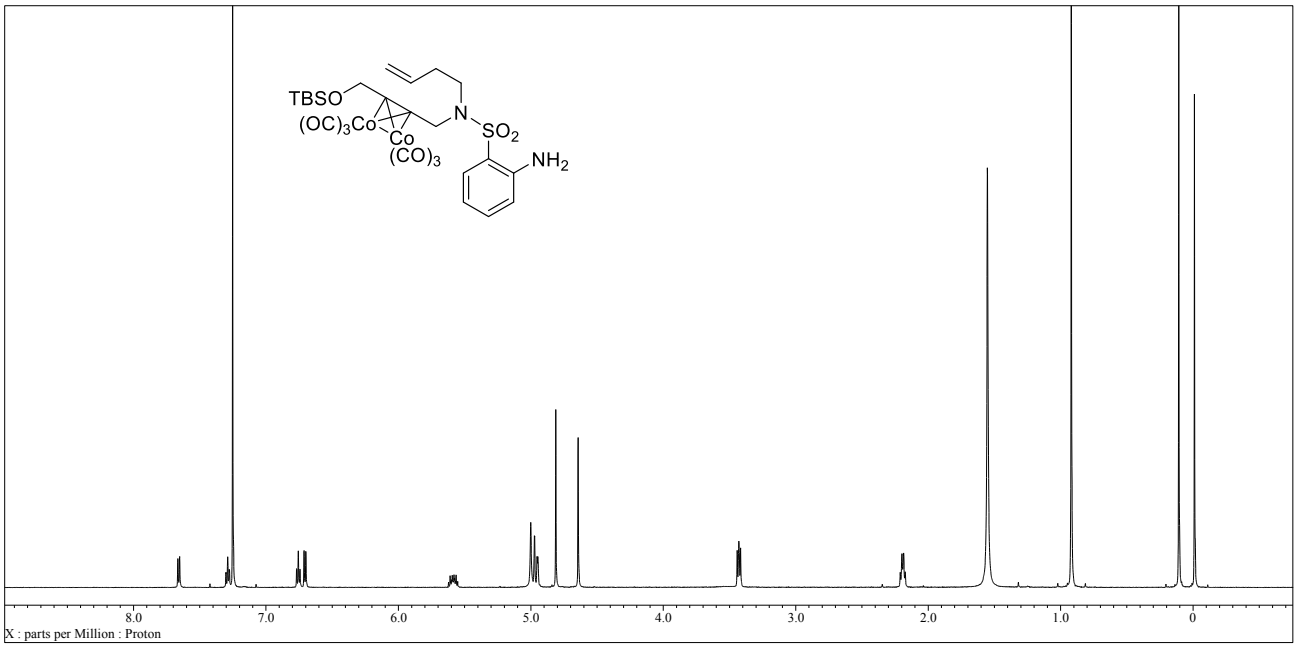

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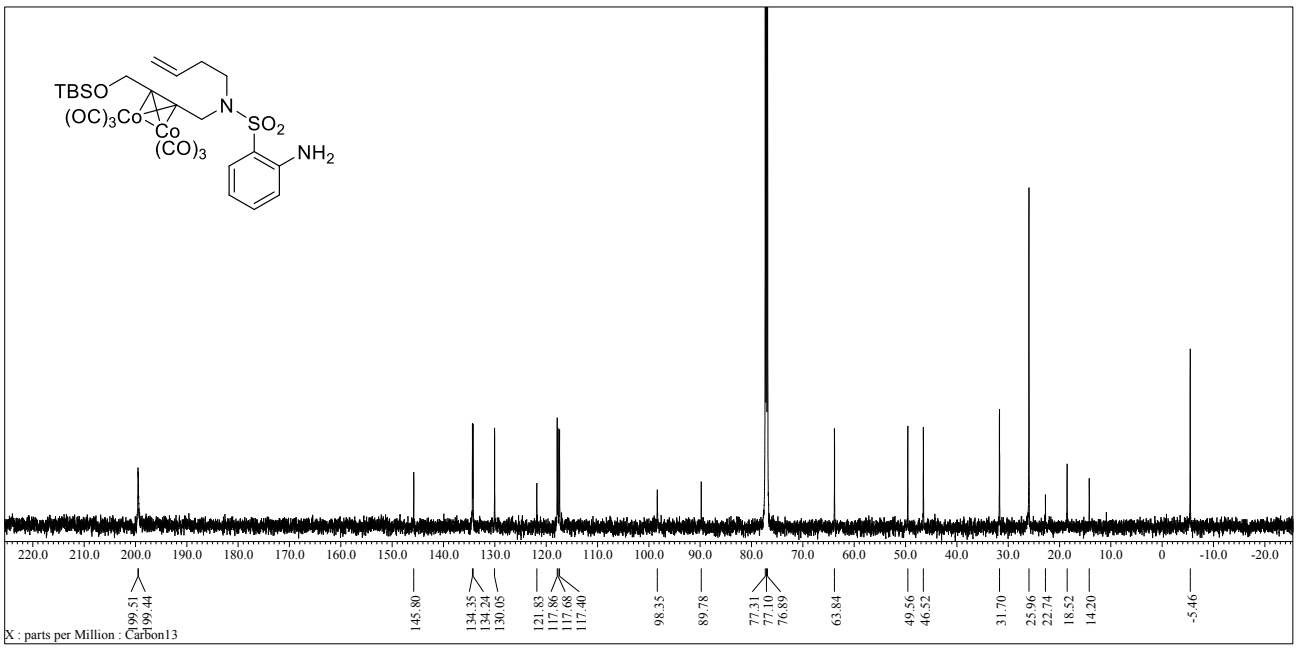
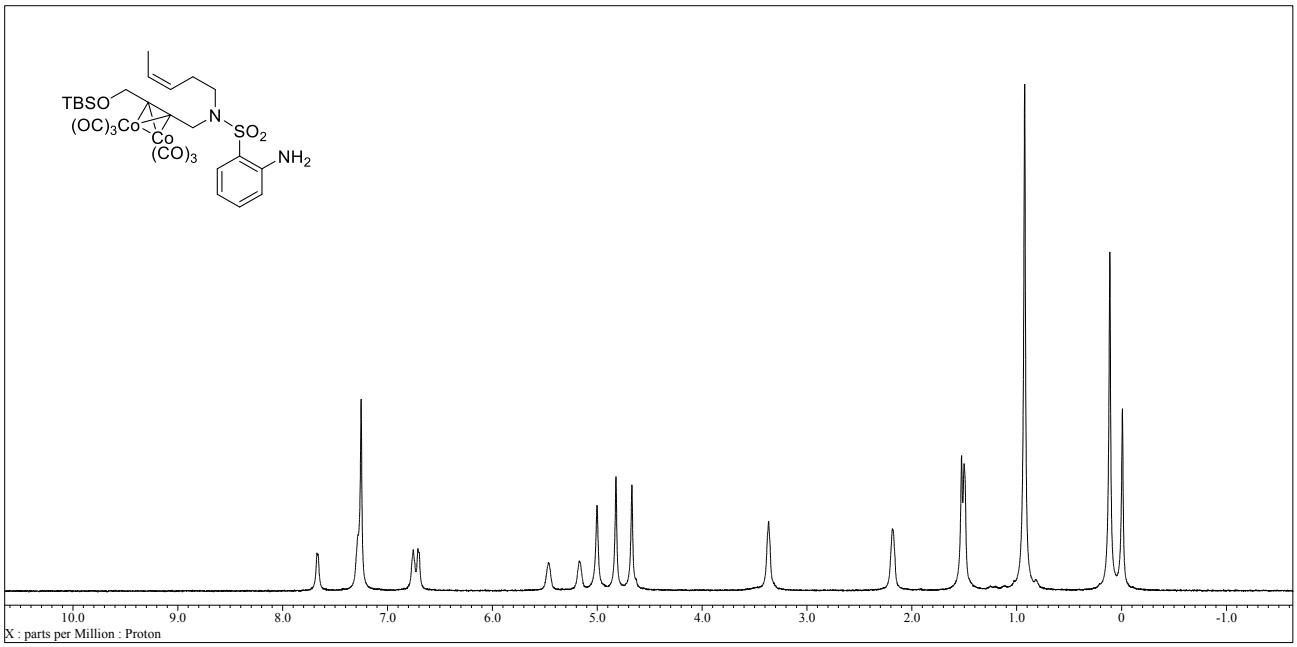


^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **6a**



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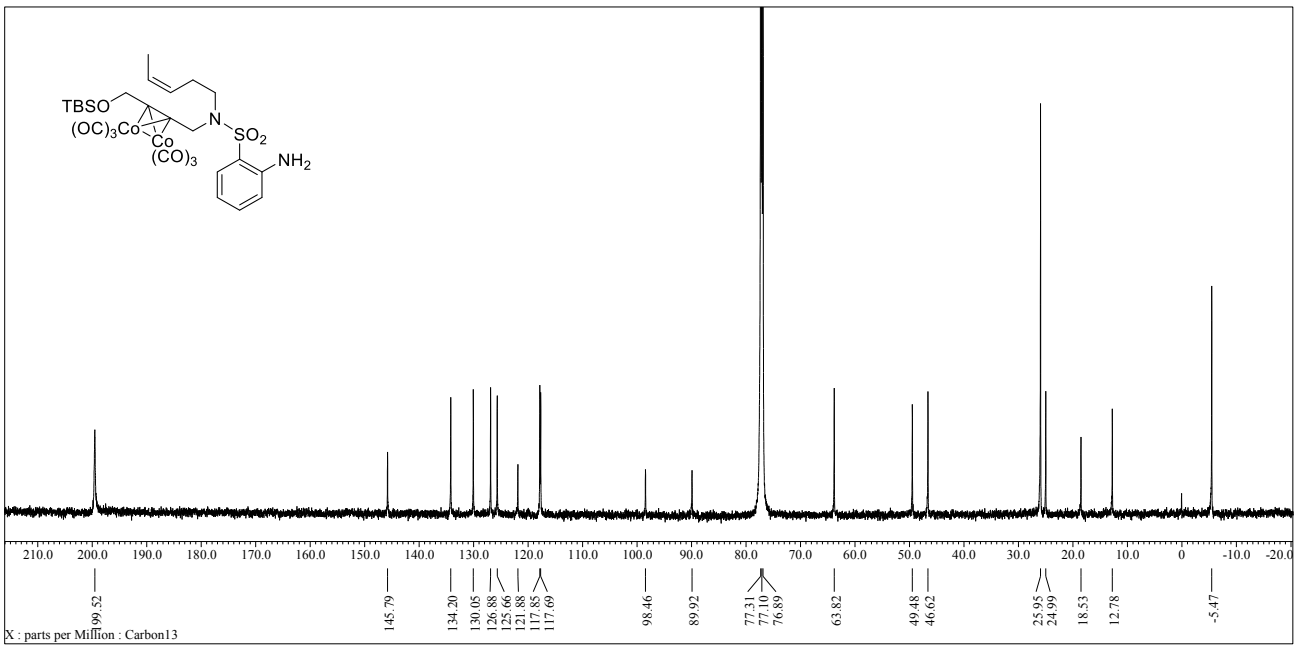

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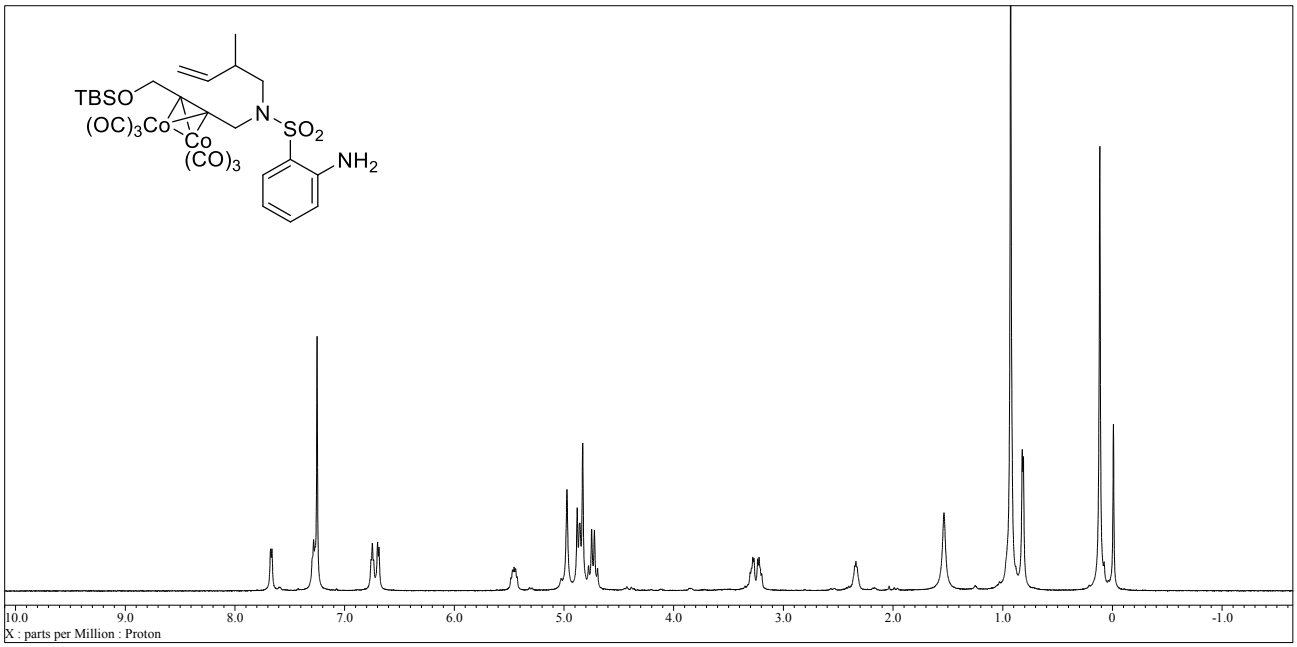
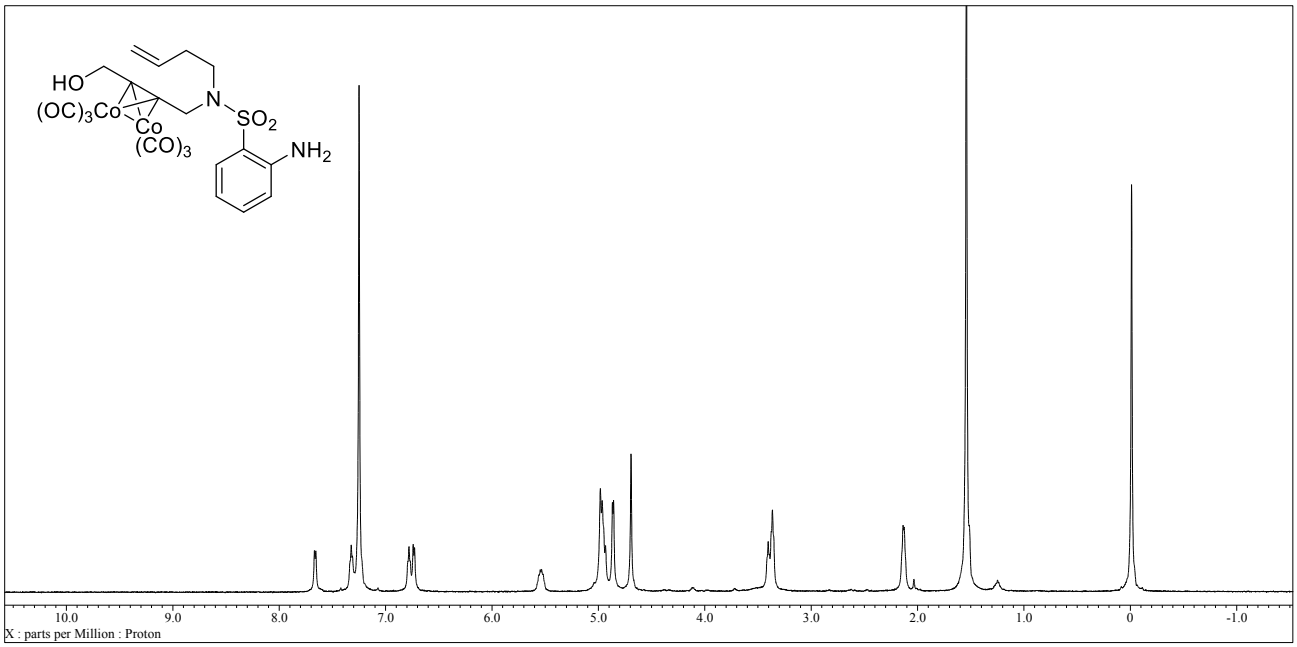


^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **6c**

^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **6c**



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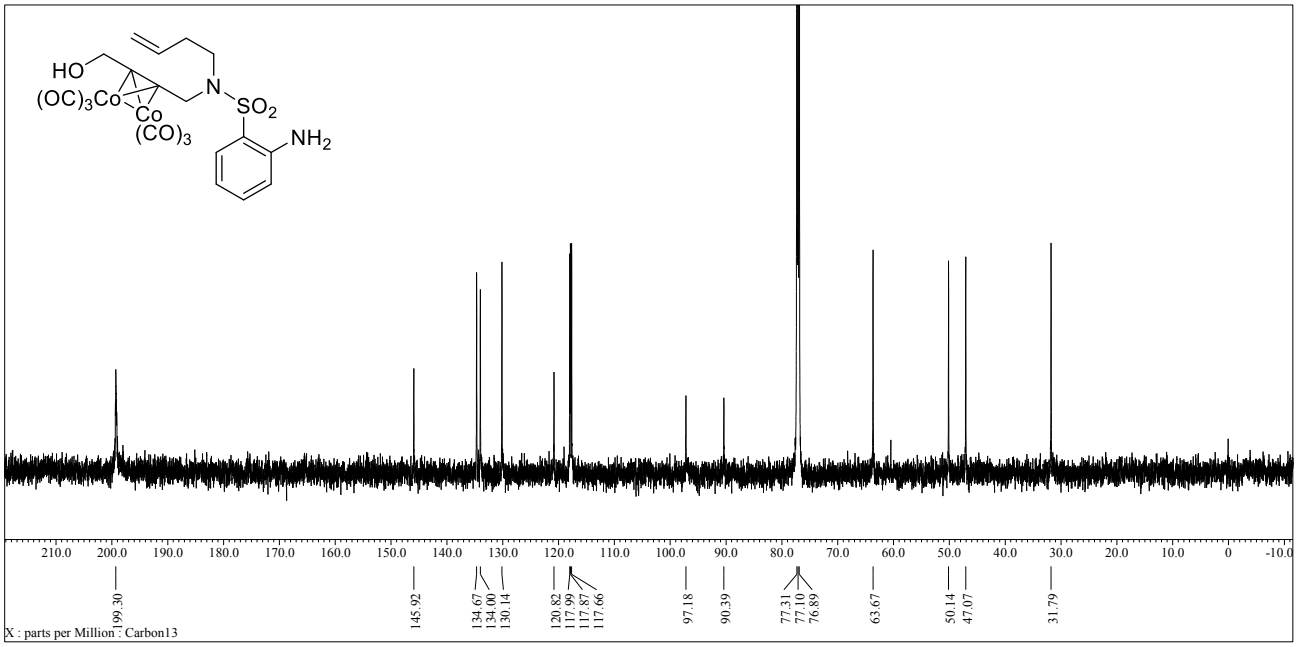
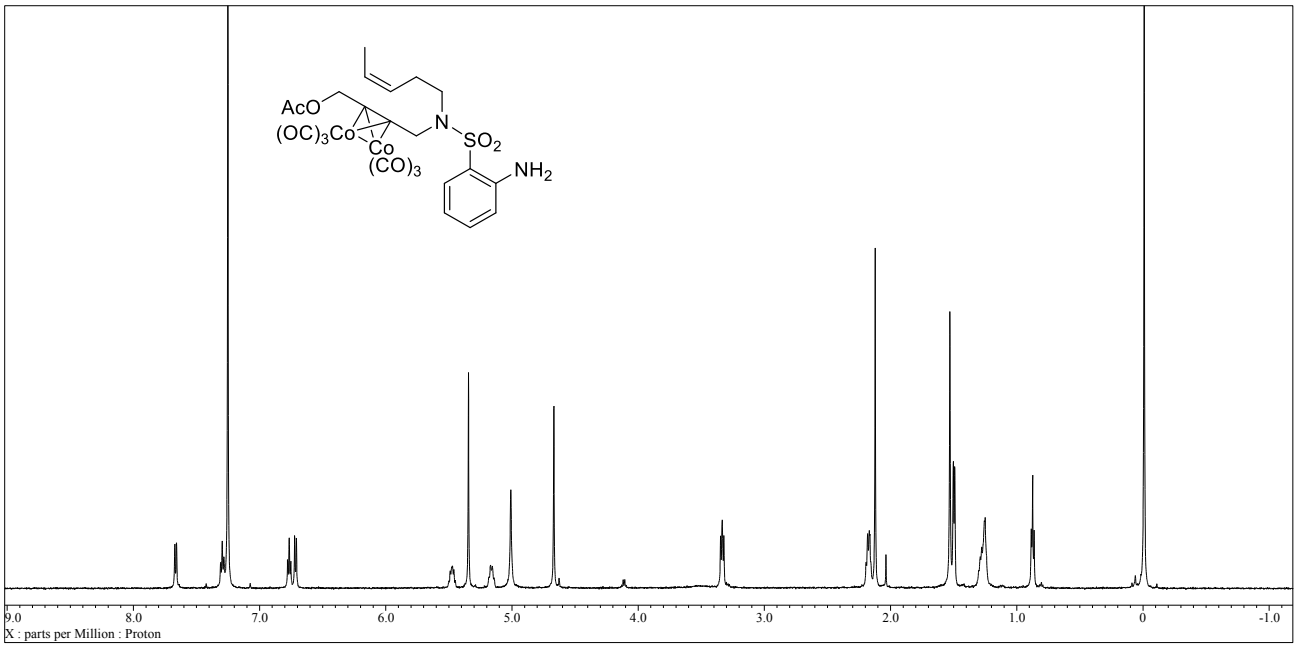
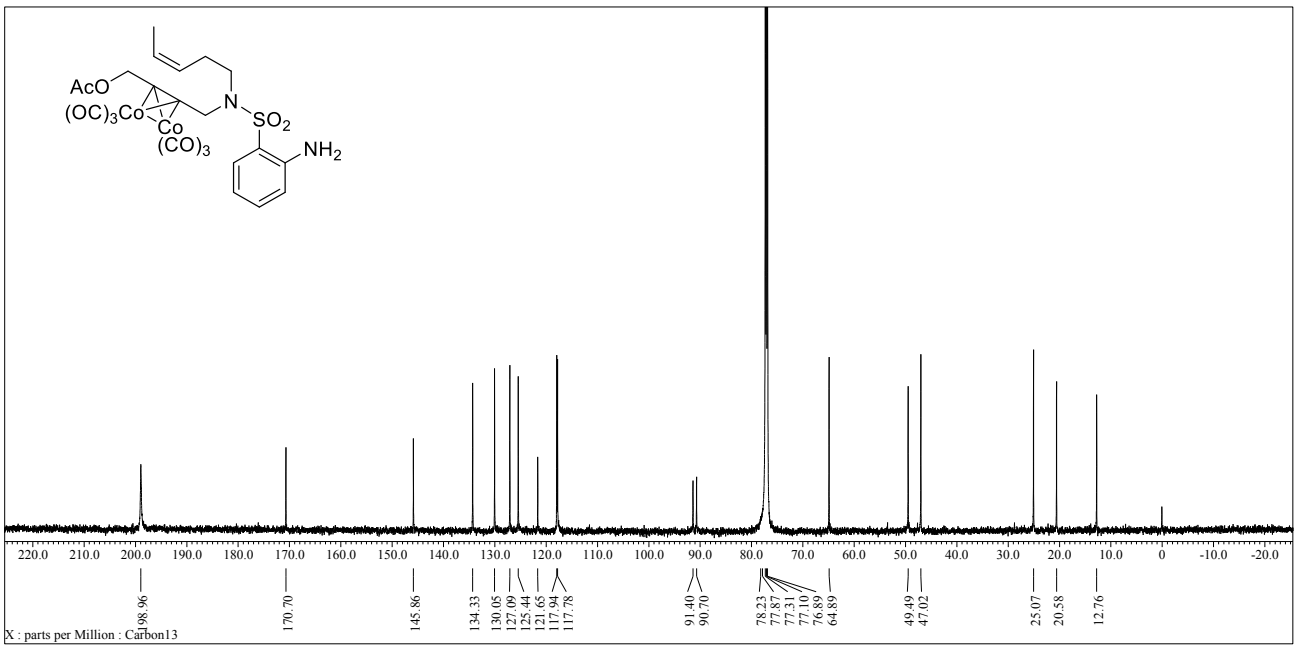


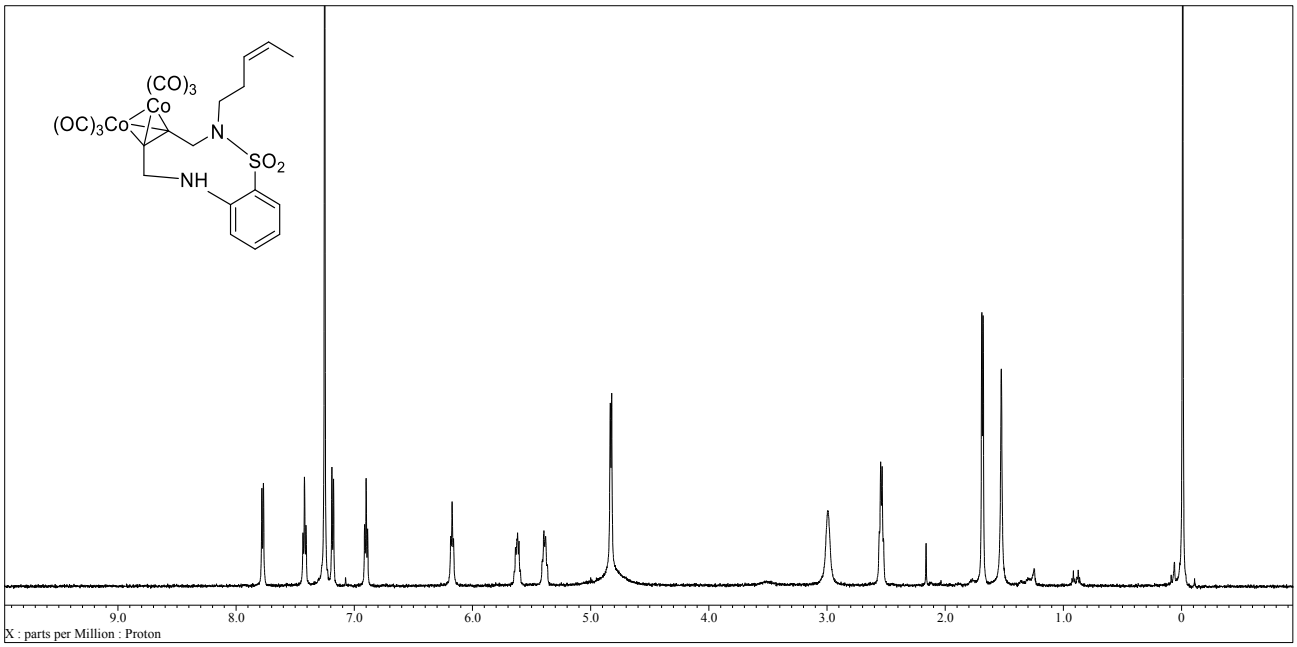
^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **6d**



^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **6f**



^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **6f**



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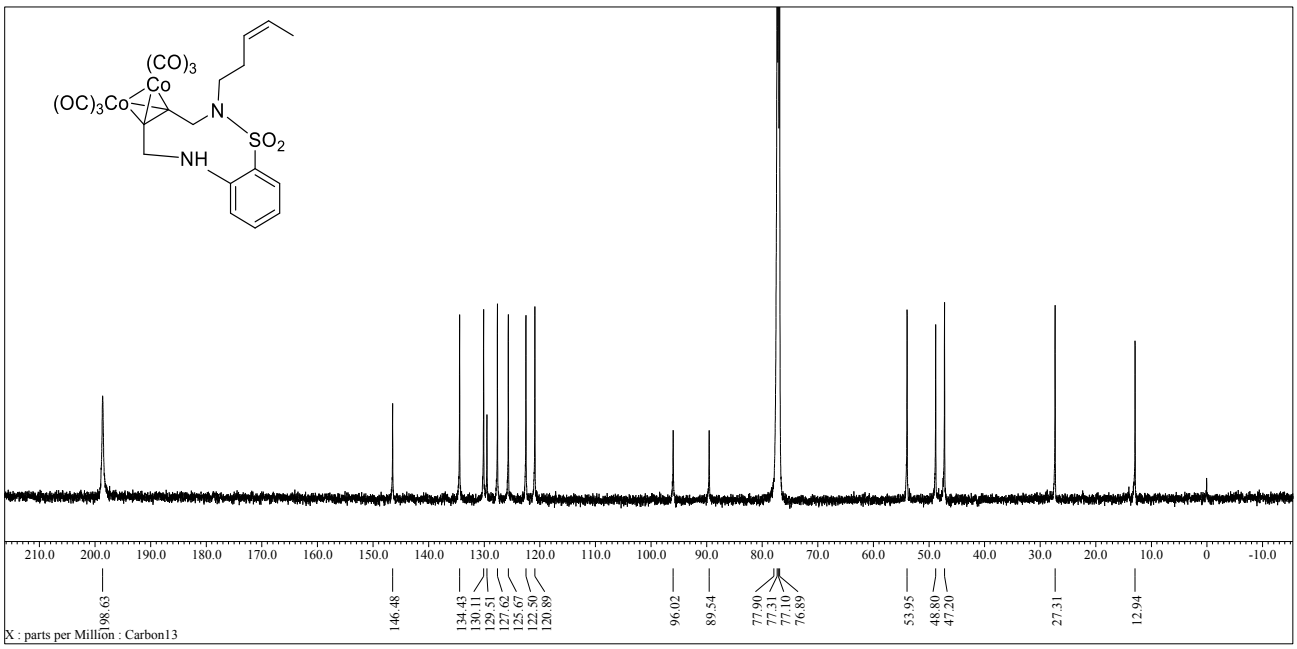
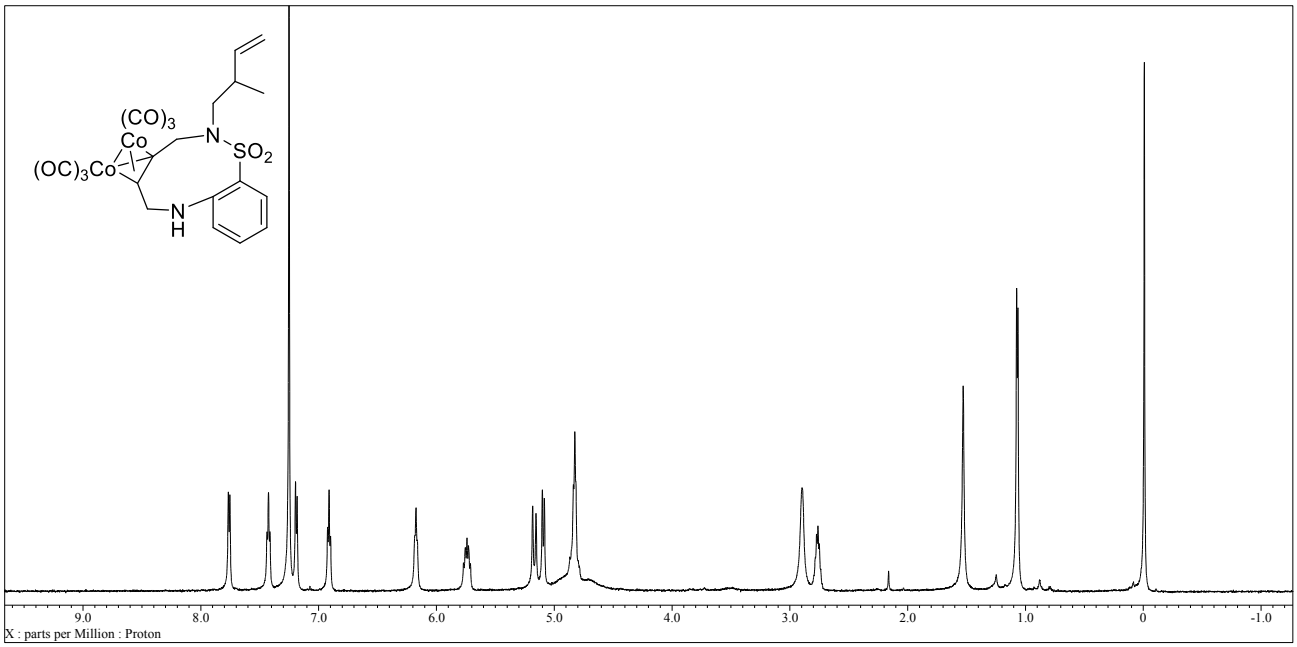


^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **7b**



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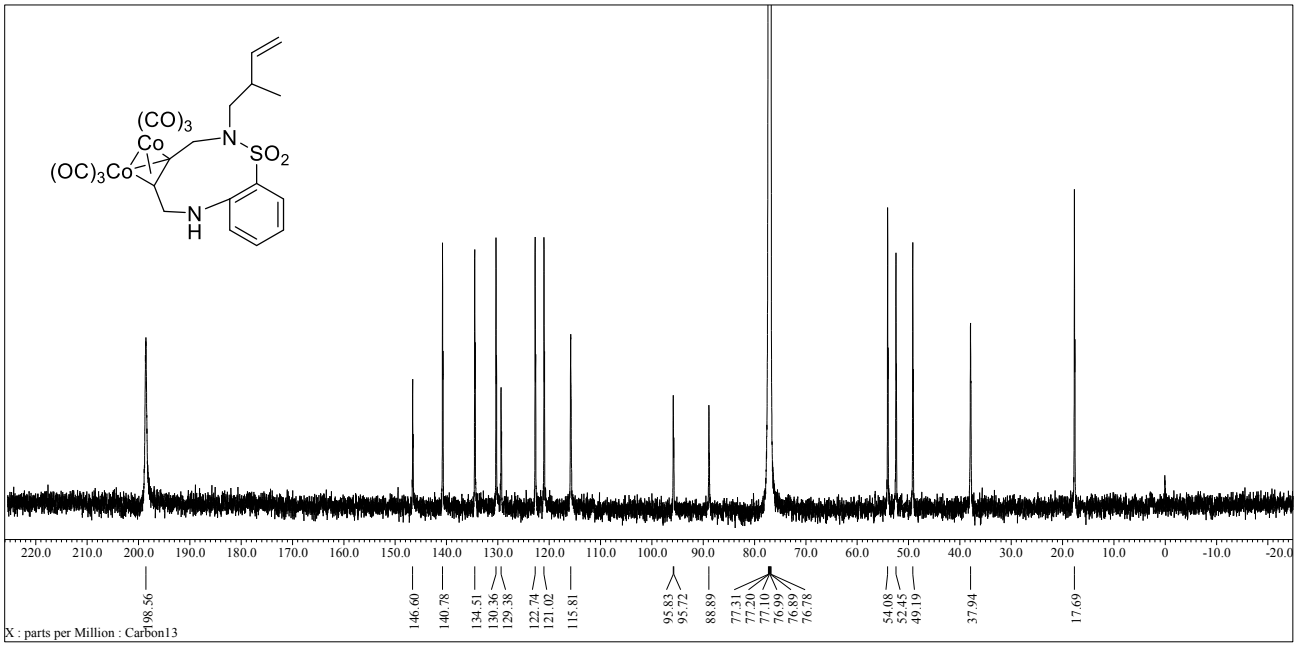
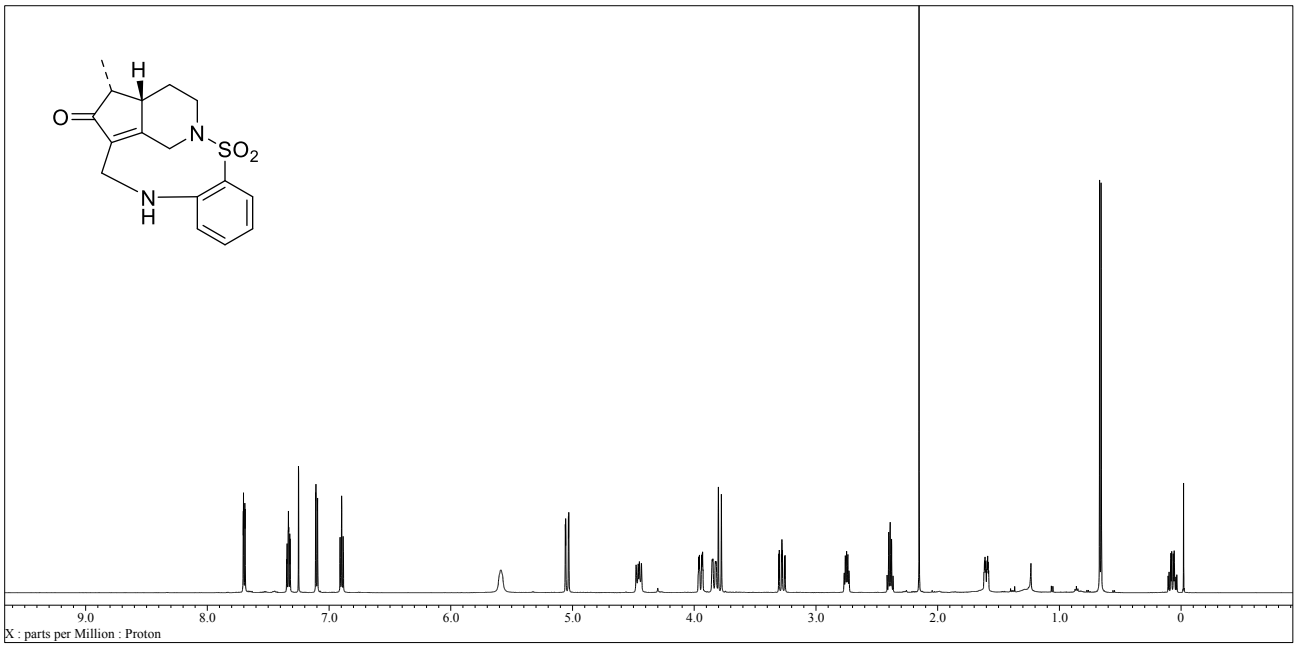


^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **7c**



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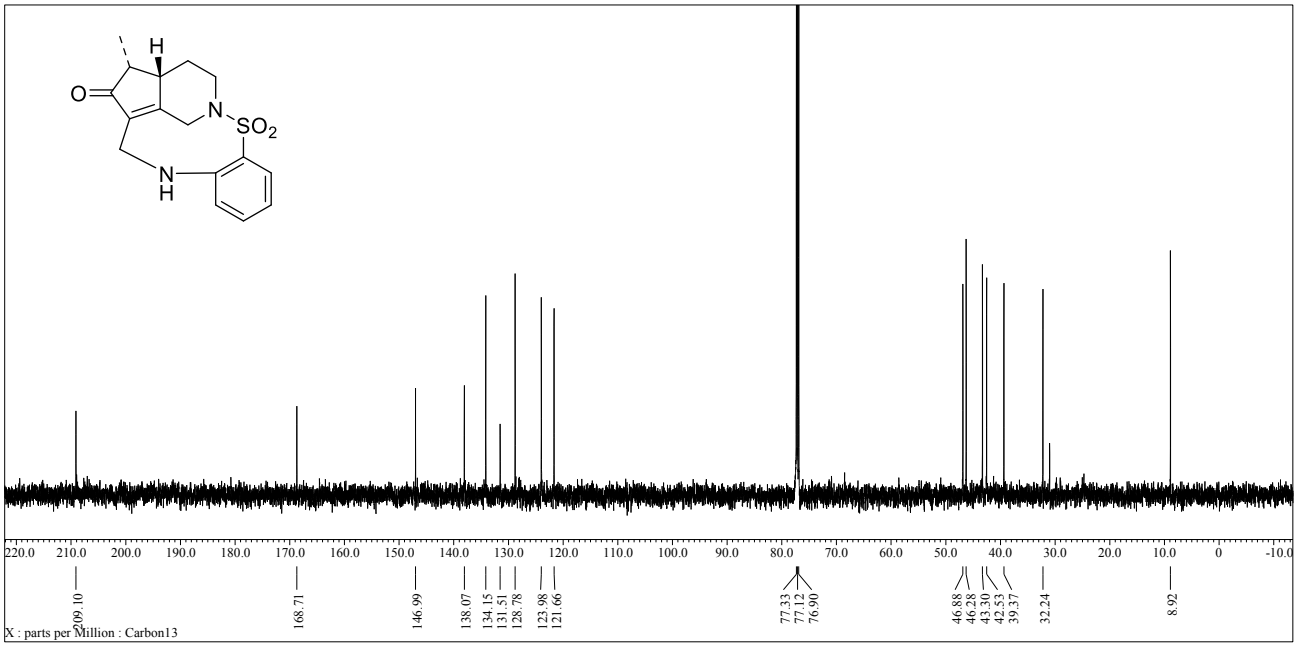
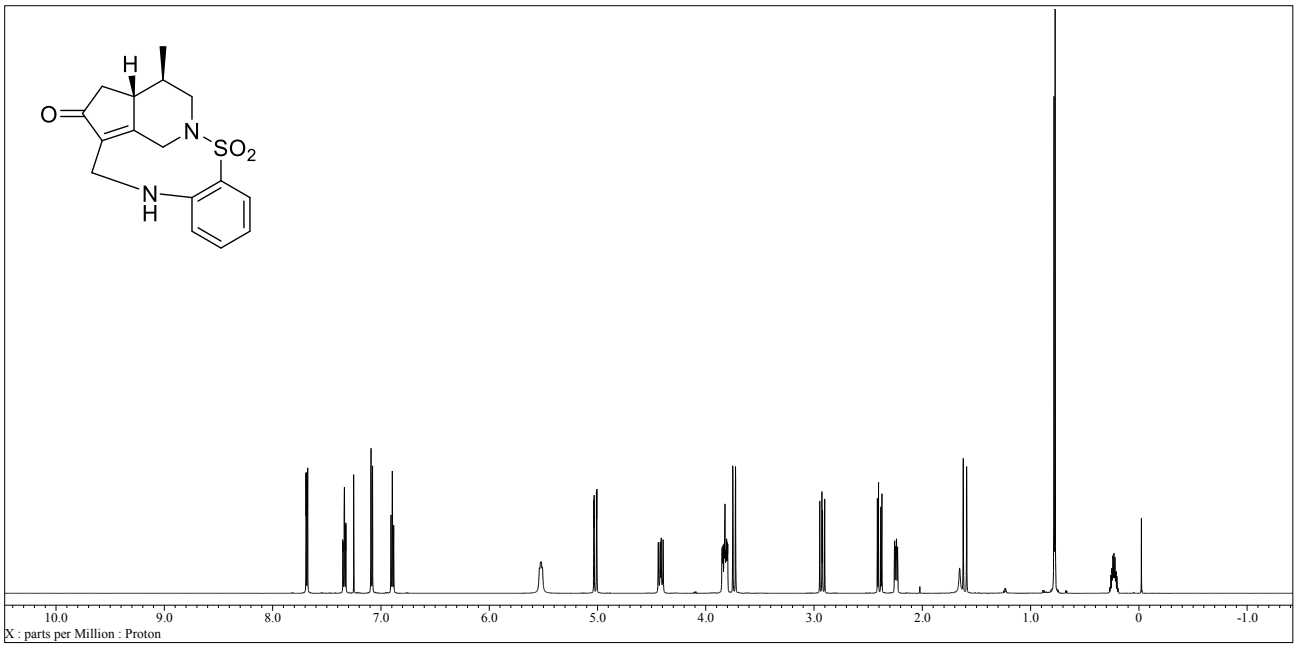
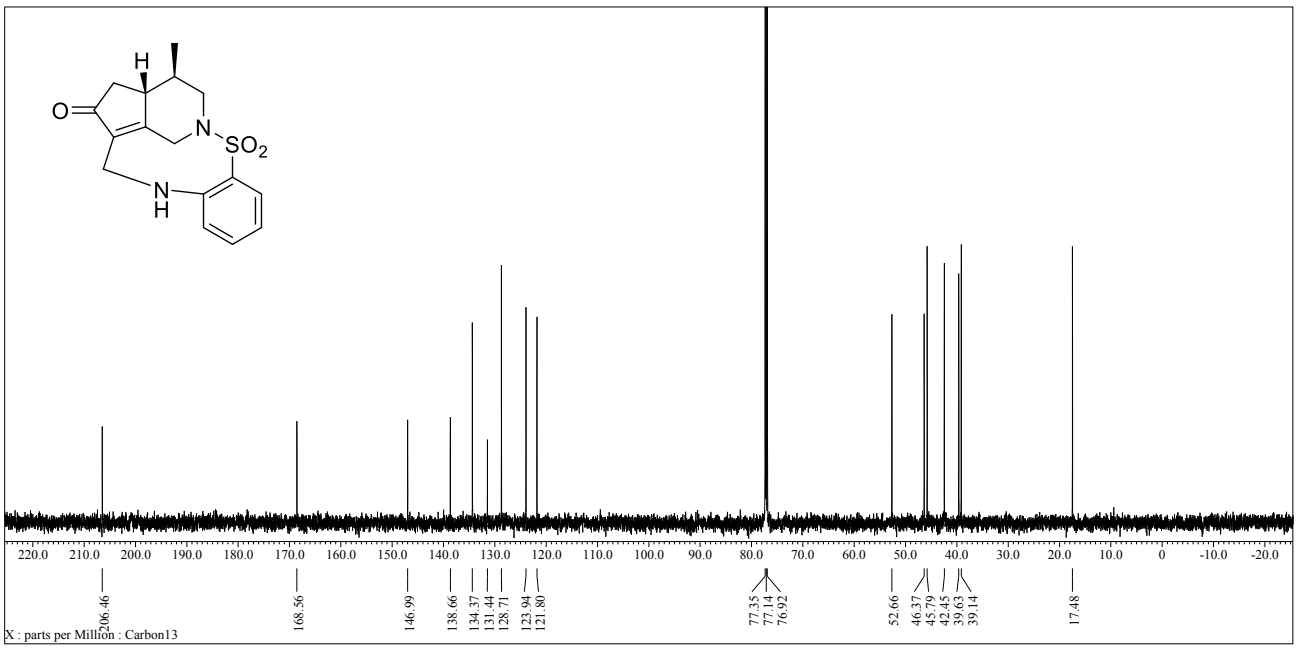


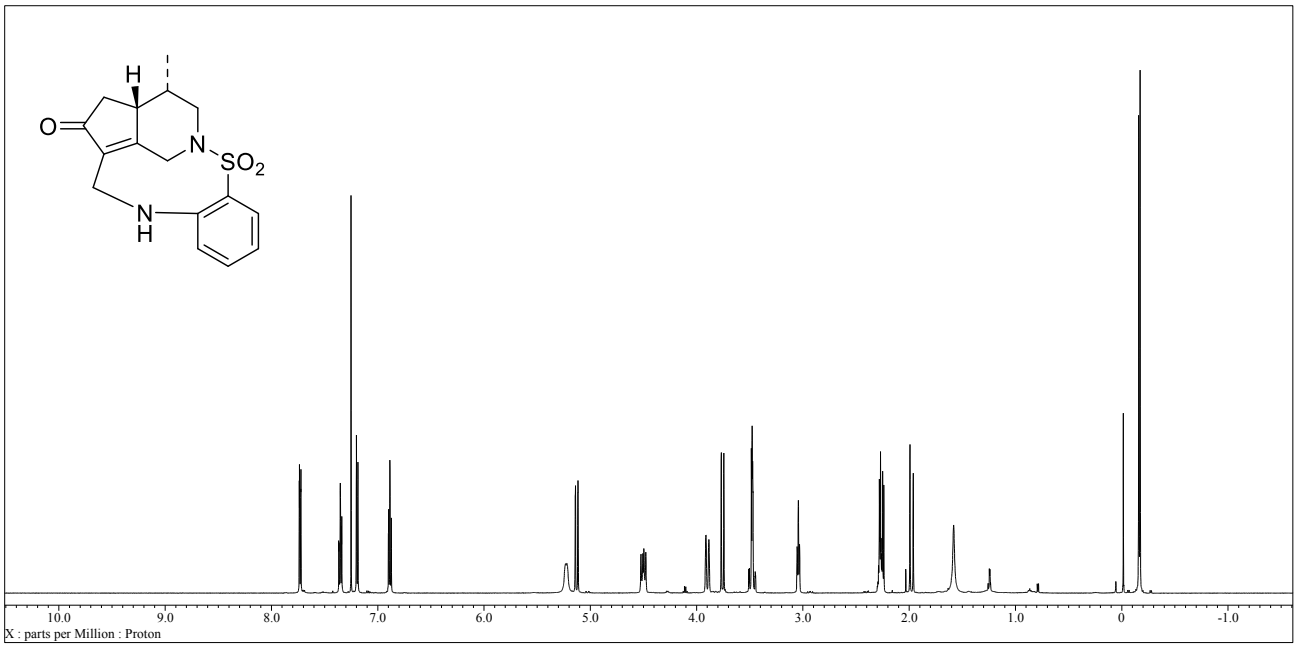
^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **8b**



^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **8c**



^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **8c**



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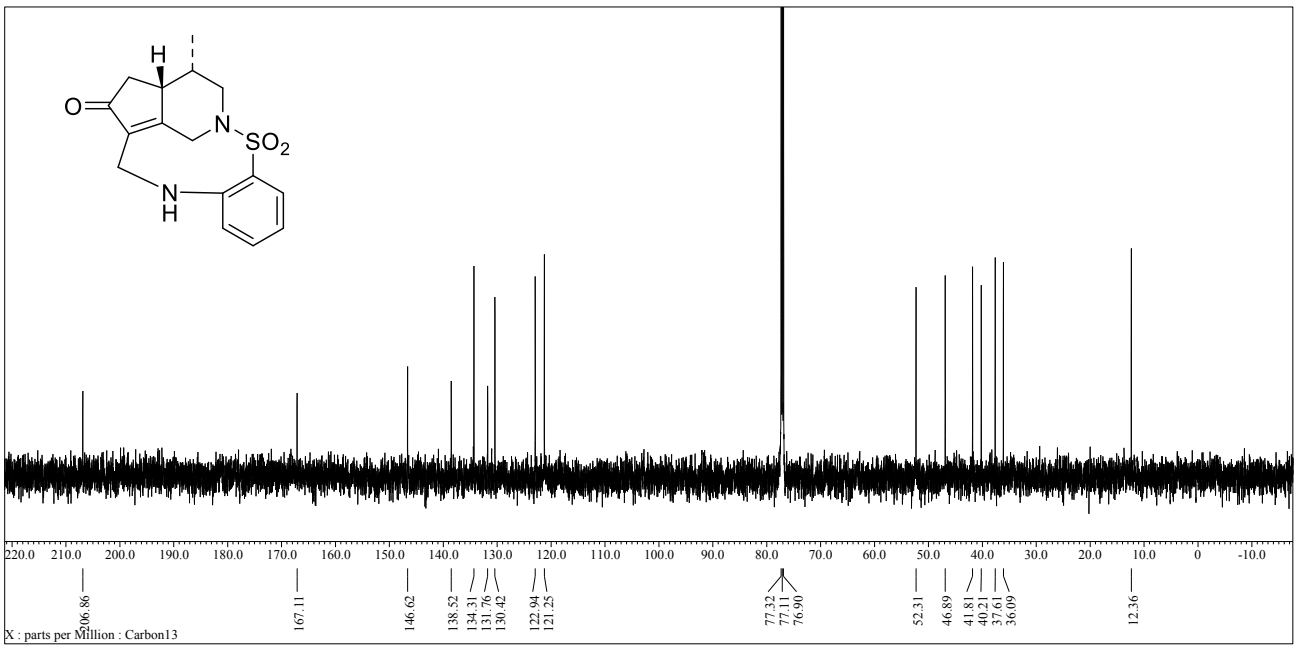
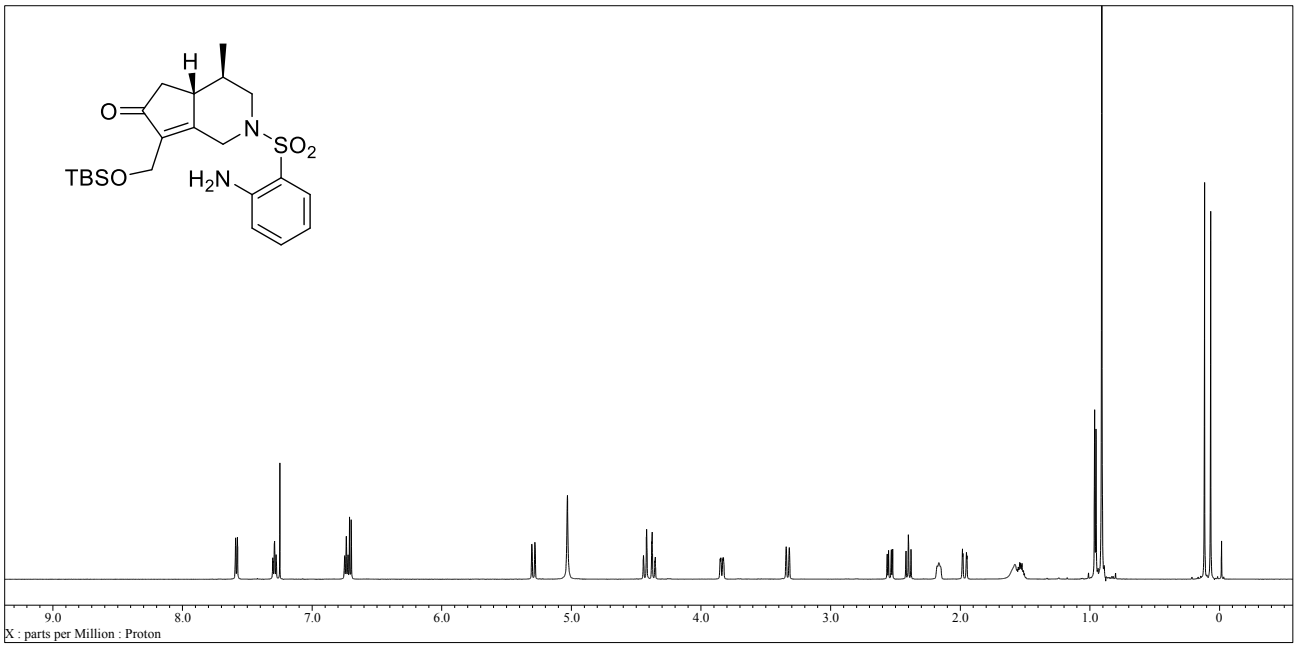
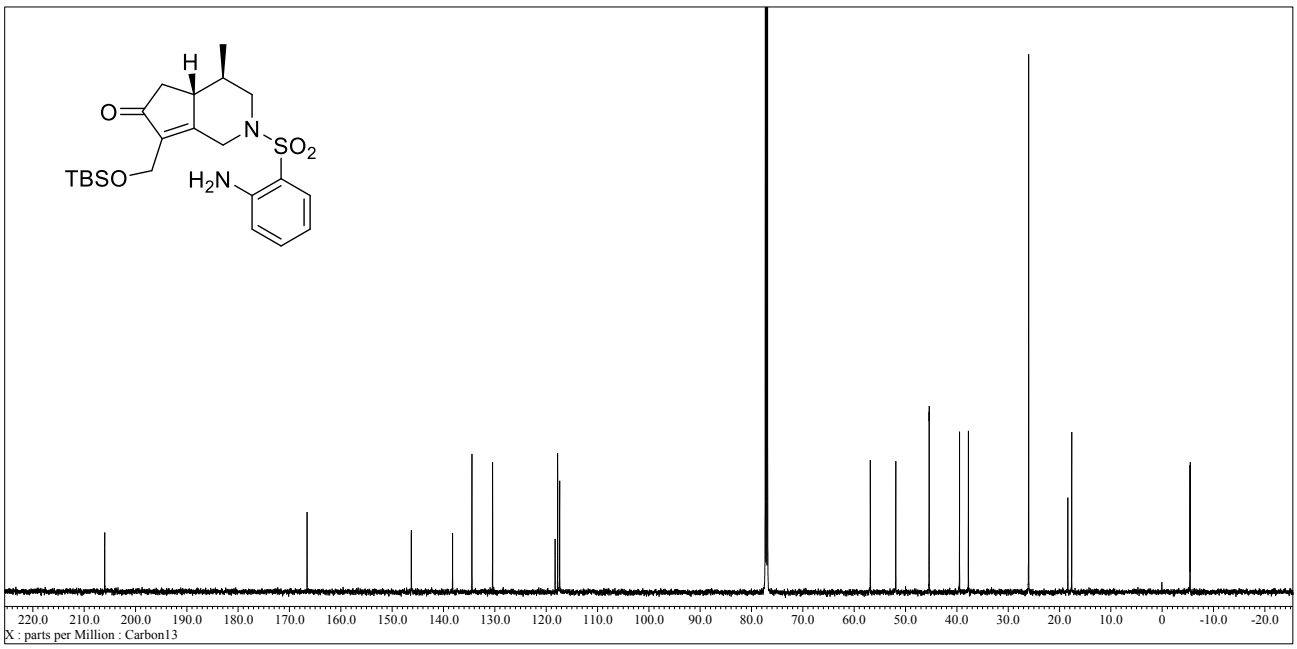


^{13}C NMR spectrum ($CDCl_3$, 150 MHz) of **8d**



^{1}H NMR spectrum ($CDCl_3$, 600 MHz) of **9**



^{13}C NMR spectrum ($CDCl_3$, 150 mHz) of **9**

Crystal data of **8b**

Experimental

Data Collection

A colorless prism crystal of C16H18N2O3S having approximate dimensions of 0.530 x 0.340 x 0.300 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn724 diffractometer using multi-layer mirror monochromated Mo-Ka radiation.

The crystal-to-detector distance was 44.85 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 11.349(9) Å
b = 11.516(9) Å b = 99.94(2)o
c = 22.31(2) Å
V = 2872(4) Å3

For Z = 8 and F.W. = 318.39, the calculated density is 1.473 g/cm3. Based on the reflection conditions of:

hkl: h+k = 2n
h0l: l = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of -169 ± 1oC to a maximum 2q value of 62.8o. A total of 720 oscillation images were collected. A sweep of data was done using w oscillations from -110.0 to 70.0o in 0.5o steps. The exposure rate was 2.0 [sec./o]. The detector swing angle was -20.07o. A second sweep was performed using w oscillations from -110.0 to 70.0o in 0.5o steps. The exposure rate was 2.0 [sec./o]. The detector swing angle was -20.07o. The crystal-to-detector distance was 44.85 mm. Readout was performed in the 0.141 mm pixel mode.

Data Reduction

Of the 8412 reflections that were collected, 2397 were unique (Rint = 0.0689); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, m, for Mo-Ka radiation is 2.405 cm-1. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.792 to 0.930. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods2 and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement3 on F2 was based on 2397 observed reflections and 203 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

R1 = S ||Fo| - |Fc|| / S |Fo| = 0.0492

wR2 = [S (w (Fo2 - Fc2)2)/ S w(Fo2)2]1/2 = 0.1364

The standard deviation of an observation of unit weight4 was 1.09. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.28 and -0.37 e-/Å3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber5. Anomalous dispersion effects were included in Fcalc6; the values for Df' and Df'' were those of Creagh and McAuley7. The values for the mass attenuation coefficients are those of Creagh and Hubbell8. All calculations were performed using the CrystalStructure9 crystallographic software package except for refinement, which was performed using SHELXL-9710.

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR97: Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G., and Spagna, R. (1999). J. Appl. Cryst., 32, 115-119.

(3) Least Squares function minimized: (SHELXL97)

S*w*(Fo2-Fc2)2 where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

[S*w*(Fo2-Fc2)2/(No-Nv)]1/2

where: No = number of observations
Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(10) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula C16H18N2O3S

Formula Weight 318.39

Crystal Color, Habit colorless, prism

Crystal Dimensions 0.530 X 0.340 X 0.300 mm

Crystal System monoclinic

Lattice Type C-centered

Lattice Parameters a = 11.349(9) Å
b = 11.516(9) Å
c = 22.31(2) Å
b = 99.94(2) o
V = 2872(4) Å3

Space Group C2/c (#15)

Z value 8

Dcalc 1.473 g/cm3

F000 1344.00

m(MoKa) 2.405 cm-1

B. Intensity Measurements

Diffractometer Saturn724

Radiation MoKa (l = 0.71075 Å)
multi-layer mirror monochromated

Voltage, Current 50kV, 24mA

Temperature -169.8oC

Detector Aperture 70 x 70 mm

Data Images 720 exposures

w oscillation Range -110.0 - 70.0o

Exposure Rate 2.0 sec./o

Detector Swing Angle -20.07o

w oscillation Range -110.0 - 70.0o

Exposure Rate 2.0 sec./o

Detector Swing Angle -20.07o

Detector Position 44.85 mm

Pixel Size 0.141 mm

2qmax 62.8o

No. of Reflections Measured Total: 8412
Unique: 2397 (Rint = 0.0689)

Corrections Lorentz-polarization
Absorption
(trans. factors: 0.792 - 0.930)

C. Structure Solution and Refinement

Structure Solution Direct Methods (SIR97)

Refinement Full-matrix least-squares on F2

Function Minimized S w (Fo2 - Fc2)2

Least Squares Weights w = 1/ [s2(Fo2) + (0.0757 . P)2
+ 4.2509 . P]
where P = (Max(Fo2,0) + 2Fc2)/3

2qmax cutoff 50.0o

Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2397
No. Variables	203
Reflection/Parameter Ratio	11.81
Residuals: R1 (I>2.00s(I))	0.0492
Residuals: R (All reflections)	0.0522
Residuals: wR2 (All reflections)	0.1364
Goodness of Fit Indicator	1.087
Max Shift/Error in Final Cycle	0.002
Maximum peak in Final Diff. Map	0.28 e-/Å3
Minimum peak in Final Diff. Map	-0.37 e-/Å3

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
S22	0.82202(4)	0.23719(4)	0.12533(2)	1.13(2)
O19	0.7217(2)	-0.25937(12)	0.06697(7)	1.75(3)
O20	0.86428(13)	0.30584(13)	0.17792(7)	1.75(3)
O21	0.76471(13)	0.29381(12)	0.07063(6)	1.53(3)
N17	0.6396(2)	0.0751(2)	0.04113(7)	1.20(3)
N18	0.9392(2)	0.1715(2)	0.10737(7)	1.26(4)
C1	0.7233(2)	0.1327(2)	0.14705(9)	1.13(4)
C2	0.6531(2)	0.0607(2)	0.10444(9)	1.12(4)
C3	0.5942(2)	-0.0324(2)	0.12765(9)	1.44(4)
C4	0.6064(2)	-0.0520(2)	0.18934(10)	1.57(4)
C5	0.6728(2)	0.0225(2)	0.23110(9)	1.53(4)

C6	0.7311(2)	0.1150(2)	0.20985(9)	1.26(4)
C7	0.6773(2)	-0.0257(2)	0.00740(9)	1.20(4)
C8	0.7944(2)	-0.0733(2)	0.04016(8)	1.17(4)
C9	0.8030(2)	-0.1894(2)	0.06908(8)	1.34(4)
C10	0.9294(2)	-0.2044(2)	0.10449(9)	1.62(4)
C11	0.9853(2)	-0.0827(2)	0.10445(9)	1.34(4)
C12	0.8970(2)	-0.0159(2)	0.05860(8)	1.09(4)
C13	0.9280(3)	-0.2635(2)	0.16576(11)	2.25(5)
C14	0.9982(2)	-0.0141(2)	0.16468(9)	1.56(4)
C15	1.0256(2)	0.1143(2)	0.15578(9)	1.67(4)
C16	0.9267(2)	0.1084(2)	0.04860(8)	1.25(4)

Beq = 8/3 p2(U11(aa*)2 + U22(bb*)2 + U33(cc*)2 + 2U12(aa*bb*)cos g + 2U13(aa*cc*)cos b + 2U23(bb*cc*)cos a)

Table 2. Atomic coordinates and Biso involving hydrogen atoms

atom	x	y	z	Biso
H3	0.5451	-0.0827	0.1002	1.72
H4	0.5687	-0.1177	0.2036	1.89
H5	0.6780	0.0100	0.2736	1.83
H6	0.7766	0.1669	0.2378	1.51
H7A	0.6861	-0.0012	-0.0341	1.44
H7B	0.6153	-0.0870	0.0038	1.44
H10	0.9735	-0.2566	0.0801	1.94
H11	1.0644	-0.0874	0.0905	1.60
H13A	0.8800	-0.2172	0.1895	2.70
H13B	1.0100	-0.2700	0.1881	2.70
H13C	0.8931	-0.3412	0.1589	2.70
H14A	0.9231	-0.0206	0.1813	1.87
H14B	1.0634	-0.0485	0.1946	1.87
H15A	1.0256	0.1558	0.1946	2.00
H15B	1.1068	0.1210	0.1456	2.00
H16A	1.0025	0.1131	0.0323	1.51
H16B	0.8625	0.1442	0.0186	1.51
H23	0.6746	0.1416	0.0310	1.46

Table 3. Anisotropic displacement parameters

atom	U11	U22	U33	U12	U13	U23
S22	0.0160(4)	0.0122(4)	0.0153(4)	-0.0028(2)	0.0041(3)	-0.0018(2)
O19	0.0285(9)	0.0165(8)	0.0229(9)	-0.0037(6)	0.0086(7)	-0.0005(6)
O20	0.0245(8)	0.0200(8)	0.0231(8)	-0.0080(6)	0.0078(7)	-0.0082(6)
O21	0.0238(8)	0.0126(7)	0.0223(8)	0.0008(6)	0.0058(6)	0.0029(6)
N17	0.0162(9)	0.0141(9)	0.0145(9)	0.0011(7)	-0.0001(7)	-0.0010(7)
N18	0.0146(9)	0.0192(9)	0.0140(9)	-0.0026(7)	0.0028(7)	-0.0023(7)
C1	0.0134(10)	0.0132(10)	0.0167(10)	0.0008(7)	0.0033(8)	-0.0001(8)
C2	0.0108(9)	0.0142(10)	0.0169(10)	0.0033(7)	0.0011(7)	-0.0012(8)
C3	0.0153(10)	0.0170(10)	0.0226(11)	-0.0027(8)	0.0041(8)	-0.0029(8)
C4	0.0202(11)	0.0163(10)	0.0251(11)	-0.0005(8)	0.0089(9)	0.0038(8)
C5	0.0207(11)	0.0209(11)	0.0175(10)	0.0037(9)	0.0060(8)	0.0035(8)
C6	0.0131(10)	0.0183(11)	0.0165(10)	-0.0002(8)	0.0027(8)	-0.0010(8)
C7	0.0186(11)	0.0134(10)	0.0129(9)	-0.0011(8)	0.0005(8)	-0.0019(8)
C8	0.0191(10)	0.0152(10)	0.0108(9)	0.0018(8)	0.0043(8)	-0.0022(8)
C9	0.0250(12)	0.0150(10)	0.0123(10)	0.0043(8)	0.0071(8)	-0.0024(8)
C10	0.0253(12)	0.0190(11)	0.0183(10)	0.0083(9)	0.0075(9)	0.0027(9)
C11	0.0141(10)	0.0207(11)	0.0168(10)	0.0047(8)	0.0046(8)	0.0013(8)
C12	0.0147(10)	0.0183(10)	0.0093(9)	0.0034(8)	0.0048(7)	-0.0014(7)
C13	0.035(2)	0.0274(12)	0.0234(12)	0.0099(10)	0.0056(10)	0.0109(9)
C14	0.0164(11)	0.0274(12)	0.0142(10)	0.0047(9)	-0.0010(8)	0.0024(9)
C15	0.0179(11)	0.0290(12)	0.0151(10)	0.0004(9)	-0.0012(8)	-0.0030(9)

C16 0.0180(10) 0.0181(10) 0.0120(9)-0.0014(8) 0.0037(8) -0.0013(8)

The general temperature factor expression: exp(-2p2(a*2U11h2 + b*2U22k2 + c*2U33l2 + 2a*b*U12hk + 2a*c*U13hl + 2b*c*U23kl))

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S22	O20	1.4274(17)		S22	O21 1.4365(16)
S22	N18	1.639(2)	S22	C1	1.768(3)
O19	C9	1.220(3)	N17	C2	1.404(3)
N17	C7	1.486(3)	N18	C15	1.482(3)
N18	C16	1.484(3)	C1	C2	1.402(3)
C1	C6	1.403(3)	C2	C3	1.408(3)
C3	C4	1.378(4)	C4	C5	1.389(3)
C5	C6	1.380(3)	C7	C8	1.505(3)
C8	C9	1.480(3)	C8	C12	1.340(3)
C9	C10	1.524(3)	C10	C11	1.539(3)
C10	C13	1.529(4)	C11	C12	1.513(3)
C11	C14	1.544(3)	C12	C16	1.497(3)
C14	C15	1.530(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
N17	H23	0.909	C3	H3	0.950
C4	H4	0.950	C5	H5	0.950
C6	H6	0.950	C7	H7A	0.990
C7	H7B	0.990	C10	H10	1.000
C11	H11	1.000	C13	H13A	0.980
C13	H13B	0.980	C13	H13C	0.980
C14	H14A	0.990	C14	H14B	0.990
C15	H15A	0.990	C15	H15B	0.990
C16	H16A	0.990	C16	H16B	0.990

Table 6. Bond angles (o)

atom	atom	atom	angle	atom	atom	atom	angle	
O20	S22	O21	119.05(10)		O20	S22	N18	106.55(10)
O20	S22	C1	106.89(11)		O21	S22	N18	105.63(10)
O21	S22	C1	109.05(10)		N18	S22	C1	109.41(11)
C2	N17	C7	115.19(16)		S22	N18	C15	119.32(15)
S22	N18	C16	118.96(13)		C15	N18	C16	112.12(17)
S22	C1	C2	121.98(17)		S22	C1	C6	116.03(14)
C2	C1	C6	121.44(19)		N17	C2	C1	124.27(19)
N17	C2	C3	118.91(17)		C1	C2	C3	116.82(19)
C2	C3	C4	121.34(18)		C3	C4	C5	121.2(2)
C4	C5	C6	118.9(2)	C1	C6	C5	120.24(18)	
N17	C7	C8	110.24(16)		C7	C8	C9	121.76(18)
C7	C8	C12	128.09(18)		C9	C8	C12	108.88(17)
O19	C9	C8	126.00(18)		O19	C9	C10	125.66(18)
C8	C9	C10	108.29(17)		C9	C10	C11	104.34(16)
C9	C10	C13	111.10(19)		C11	C10	C13	118.26(18)
C10	C11	C12	103.77(16)		C10	C11	C14	116.29(18)
C12	C11	C14	106.27(17)		C8	C12	C11	113.34(18)
C8	C12	C16	129.24(17)		C11	C12	C16	116.52(16)
C11	C14	C15	111.86(18)		N18	C15	C14	113.61(17)
N18	C16	C12	109.03(16)					

Table 7. Bond angles involving hydrogens (o)

atom	atom	atom	angle	atom	atom	atom	angle
C2	N17	H23	111.9	C7	N17	H23	110.8
C2	C3	H3	119.3	C4	C3	H3	119.3
C3	C4	H4	119.4	C5	C4	H4	119.4
C4	C5	H5	120.6	C6	C5	H5	120.6
C1	C6	H6	119.9	C5	C6	H6	119.9
N17	C7	H7A	109.6	N17	C7	H7B	109.6
C8	C7	H7A	109.6	C8	C7	H7B	109.6
H7A	C7	H7B	108.1	C9	C10	H10	107.6

C11	C10	H10	107.5	C13	C10	H10	107.6
C10	C11	H11	110.1	C12	C11	H11	110.1
C14	C11	H11	110.1	C10	C13	H13A	109.5
C10	C13	H13B	109.5	C10	C13	H13C	109.5
H13A	C13	H13B	109.5	H13A	C13	H13C	109.5
H13B	C13	H13C	109.5	C11	C14	H14A	109.2
C11	C14	H14B	109.2	C15	C14	H14A	109.2
C15	C14	H14B	109.2	H14A	C14	H14B	107.9
N18	C15	H15A	108.8	N18	C15	H15B	108.8
C14	C15	H15A	108.8	C14	C15	H15B	108.8
H15A	C15	H15B	107.7	N18	C16	H16A	109.9
N18	C16	H16B	109.9	C12	C16	H16A	109.9
C12	C16	H16B	109.9	H16A	C16	H16B	108.3

Table 8. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A		
N17	H23	S22		3.156(3)	0.91	2.69	112.93	intramol.
N17	H23	O21		2.911(3)	0.91	2.14	141.66	intramol.

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S22	N17	3.156(3)	S22	C12	3.446(3)
S22	C14	3.540(3)	O19	C3	3.381(3)
O19	C7	3.005(3)	O19	C11	3.596(3)
O19	C12	3.461(3)	O19	C13	2.926(4)
O20	C6	2.827(3)	O20	C15	2.962(3)
O21	N17	2.911(3)	O21	C2	3.116(3)
O21	C16	2.914(3)	N17	C9	3.564(4)
N17	C12	3.064(4)	N17	C16	3.257(4)
N18	C2	3.479(4)	N18	C8	3.471(3)
N18	C11	2.976(4)	C1	C4	2.758(4)
C1	C7	3.570(4)	C1	C8	3.554(4)
C1	C12	3.471(4)	C1	C14	3.510(4)
C1	C15	3.409(4)	C1	C16	3.462(4)

C2	C5	2.830(4)	C2	C8	2.793(4)
C2	C9	3.503(4)	C2	C12	3.234(4)
C2	C16	3.582(4)	C3	C6	2.770(3)
C3	C7	2.994(4)	C3	C8	3.274(4)
C3	C9	3.414(4)	C7	C16	3.217(4)
C8	C14	3.364(3)	C9	C14	3.450(3)
C12	C15	2.826(3)	C13	C14	2.982(4)
C14	C16	2.938(4)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S22	H6	2.769	S22	H14A	3.346
S22	H15A	2.715	S22	H15B	3.455
S22	H16A	3.465	S22	H16B	2.721
S22	H23	2.689	O19	H3	3.036
O19	H7B	2.607	O19	H10	2.822
O19	H13A	3.040	O19	H13C	2.739
O20	H6	2.407	O20	H15A	2.497
O21	H16B	2.448	O21	H23	2.143
N17	H3	2.583	N17	H16B	2.780
N18	H11	3.351	N18	H14A	2.785
N18	H14B	3.353	N18	H23	3.206
C1	H3	3.256	C1	H5	3.276
C1	H14A	2.871	C1	H15A	3.422
C1	H16B	3.505	C1	H23	2.555
C2	H4	3.280	C2	H6	3.298
C2	H7A	3.257	C2	H7B	2.791
C2	H14A	3.375	C2	H16B	3.436
C3	H5	3.267	C3	H7B	2.885
C3	H23	3.190	C4	H6	3.246
C5	H3	3.261	C5	H14A	3.261
C6	H4	3.241	C6	H14A	2.841
C6	H15A	3.450	C7	H3	2.835
C7	H16B	2.851	C8	H3	3.334
C8	H10	2.959	C8	H11	3.080
C8	H14A	3.292	C8	H16A	3.218
C8	H16B	2.689	C8	H23	2.814

C9	H3	3.355	C9	H7A	3.272
C9	H7B	2.641	C9	H11	3.149
C9	H13A	2.697	C9	H13B	3.359
C9	H13C	2.723	C9	H14A	3.276
C10	H14A	2.733	C10	H14B	2.920
C11	H13A	2.865	C11	H13B	2.834
C11	H13C	3.446	C11	H15A	3.389
C11	H15B	2.794	C11	H16A	2.796
C11	H16B	3.393	C12	H7A	2.886
C12	H7B	3.322	C12	H10	2.920
C12	H14A	2.704	C12	H14B	3.307

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C12	H15B	3.214	C12	H23	3.083
C13	H11	3.197	C13	H14A	2.821
C13	H14B	2.927	C14	H10	3.355
C14	H13A	2.799	C14	H13B	2.992
C14	H16A	3.304	C15	H11	2.816
C15	H16A	2.721	C15	H16B	3.309
C16	H7A	3.274	C16	H11	2.810
C16	H14A	3.320	C16	H15A	3.303
C16	H15B	2.712	C16	H23	2.847
H3	H4	2.310	H3	H7B	2.421
H3	H23	3.463	H4	H5	2.339
H5	H6	2.337	H6	H14A	3.120
H6	H15A	3.141	H7A	H16B	2.718
H7A	H23	2.213	H7B	H23	2.759
H10	H11	2.198	H10	H13A	2.859
H10	H13B	2.378	H10	H13C	2.329
H10	H14B	3.522	H11	H13B	3.163
H11	H14A	2.896	H11	H14B	2.367
H11	H15B	2.701	H11	H16A	2.683
H13A	H14A	2.330	H13A	H14B	2.835
H13B	H14A	3.031	H13B	H14B	2.620
H14A	H15A	2.335	H14A	H15B	2.866
H14B	H15A	2.392	H14B	H15B	2.331

H15B	H16A	2.600	H16B	H23	2.198

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O19	O19[1]	3.171(4)	O19	N18[2]	3.566(4)
O19	C7[1]	3.294(3)	O19	C8[1]	3.051(3)
O19	C9[1]	3.057(4)	O19	C15[2]	3.538(4)
O20	C3[3]	3.543(4)	O20	C4[3]	3.171(4)
O20	C4[4]	3.350(4)	O20	C5[4]	3.289(3)
O21	O21[5]	3.271(3)	O21	N17[5]	3.257(3)
O21	C7[5]	3.315(3)	O21	C16[5]	3.327(3)
N17	O21[5]	3.257(3)	N18	O19[3]	3.566(4)
C3	O20[2]	3.543(4)	C4	O20[2]	3.171(4)
C4	O20[6]	3.350(4)	C5	O20[6]	3.289(3)
C7	O19[1]	3.294(3)	C7	O21[5]	3.315(3)
C8	O19[1]	3.051(3)	C9	O19[1]	3.057(4)
C9	C9[1]	3.408(4)	C12	C16[7]	3.537(4)
C15	O19[3]	3.538(4)	C16	O21[5]	3.327(3)
C16	C12[7]	3.537(4)			

Symmetry Operators:

(1) -X+1/2+1,-Y+1/2-1,-Z (2) X+1/2-1,Y+1/2-1,Z
(3) X+1/2,Y+1/2,Z (4) -X+1/2+1,Y+1/2,-Z+1/2
(5) -X+1/2+1,-Y+1/2,-Z (6) -X+1/2+1,Y+1/2-1,-Z+1/2
(7) -X+2,-Y,-Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S22	H3[1]	3.394	S22	H4[1]	3.463
S22	H11[2]	3.528	O19	H7A[3]	3.082
O19	H7B[3]	3.170	O19	H15B[4]	2.732
O19	H16A[4]	2.877	O20	H3[1]	3.177

O20	H4[1]	2.455	O20	H4[5]	2.766
O20	H5[5]	2.666	O20	H15A[6]	3.377
O21	H3[1]	3.444	O21	H7A[7]	2.615
O21	H10[2]	3.398	O21	H11[2]	2.754
O21	H16B[7]	2.360	O21	H23[7]	2.590
N17	H3[8]	3.473	N17	H7B[8]	2.896
N17	H10[2]	2.938	N17	H16B[7]	3.495
N18	H3[1]	3.091	N18	H4[1]	3.402
C1	H10[2]	3.229	C1	H13B[2]	2.953
C2	H7B[8]	3.559	C2	H10[2]	2.915
C2	H13B[2]	3.313	C2	H13C[2]	3.564
C3	H7A[8]	3.512	C3	H10[2]	3.548
C3	H13B[2]	3.509	C3	H13C[2]	3.331
C4	H4[9]	3.445	C4	H5[9]	3.540
C4	H13B[2]	3.426	C4	H13C[2]	3.413
C4	H15A[4]	3.495	C5	H13A[5]	3.584
C5	H13B[2]	3.071	C5	H13C[5]	3.110
C5	H14B[6]	3.266	C5	H15B[6]	3.569
C6	H13A[5]	3.368	C6	H13B[2]	2.804
C6	H13C[5]	3.498	C6	H14B[6]	3.437
C6	H15A[6]	3.219	C6	H15B[6]	3.427
C7	H3[8]	3.406	C7	H7B[8]	3.534
C7	H10[3]	3.446	C8	H16A[10]	3.071
C9	H7B[3]	3.264	C9	H16A[10]	3.534
C10	H7B[3]	3.381	C10	H16A[10]	3.441
C11	H16A[10]	3.099	C11	H16B[10]	3.558
C12	H16A[10]	2.734	C13	H5[11]	3.262
C13	H6[11]	3.519	C13	H13B[6]	3.215
C14	H14A[6]	3.398	C14	H14B[6]	3.355
C15	H4[1]	3.275	C15	H6[6]	3.034
C15	H15A[6]	3.518	C16	H11[10]	3.132
C16	H16A[10]	3.304	C16	H23[7]	3.473
H3	S22[4]	3.394	H3	O20[4]	3.177

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance

H3	O21[4]	3.444	H3	N17[8]	3.473
H3	N18[4]	3.091	H3	C7[8]	3.406
H3	H7A[8]	2.942	H3	H7B[8]	3.325
H3	H15B[4]	3.595	H3	H23[8]	3.567
H4	S22[4]	3.463	H4	O20[4]	2.455
H4	O20[11]	2.766	H4	N18[4]	3.402
H4	C4[9]	3.445	H4	C15[4]	3.275
H4	H4[9]	2.800	H4	H5[9]	3.279
H4	H6[11]	3.186	H4	H15A[4]	2.655
H4	H15B[4]	3.334	H5	O20[11]	2.666
H5	C4[9]	3.540	H5	C13[5]	3.262
H5	H4[9]	3.279	H5	H13A[5]	3.342
H5	H13B[2]	3.526	H5	H13B[5]	3.512
H5	H13C[5]	2.504	H5	H14B[6]	2.975
H5	H15B[6]	3.054	H6	C13[5]	3.519
H6	C15[6]	3.034	H6	H4[5]	3.186
H6	H13A[5]	2.926	H6	H13B[2]	3.123
H6	H13C[5]	3.248	H6	H14B[6]	3.283
H6	H15A[6]	2.482	H6	H15B[6]	2.758
H7A	O19[3]	3.082	H7A	O21[7]	2.615
H7A	C3[8]	3.512	H7A	H3[8]	2.942
H7A	H10[3]	3.387	H7A	H11[10]	3.446
H7A	H13C[3]	3.315	H7B	O19[3]	3.170
H7B	N17[8]	2.896	H7B	C2[8]	3.559
H7B	C7[8]	3.534	H7B	C9[3]	3.264
H7B	C10[3]	3.381	H7B	H3[8]	3.325
H7B	H7B[8]	3.278	H7B	H10[3]	2.667
H7B	H23[8]	3.307	H10	O21[12]	3.398
H10	N17[12]	2.938	H10	C1[12]	3.229
H10	C2[12]	2.915	H10	C3[12]	3.548
H10	C7[3]	3.446	H10	H7A[3]	3.387
H10	H7B[3]	2.667	H10	H16A[10]	3.056
H10	H16B[10]	3.378	H10	H23[12]	2.941
H11	S22[12]	3.528	H11	O21[12]	2.754
H11	C16[10]	3.132	H11	H7A[10]	3.446
H11	H16A[10]	2.732	H11	H16B[10]	2.783
H13A	C5[11]	3.584	H13A	C6[11]	3.368

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H13A	H5[11]	3.342	H13A	H6[11]	2.926
H13A	H13A[6]	3.488	H13A	H13B[6]	2.865
H13A	H14B[6]	3.209	H13B	C1[12]	2.953
H13B	C2[12]	3.313	H13B	C3[12]	3.509
H13B	C4[12]	3.426	H13B	C5[12]	3.071
H13B	C6[12]	2.804	H13B	C13[6]	3.215
H13B	H5[12]	3.526	H13B	H5[11]	3.512
H13B	H6[12]	3.123	H13B	H13A[6]	2.865
H13B	H13B[6]	2.809	H13B	H13C[6]	3.498
H13C	C2[12]	3.564	H13C	C3[12]	3.331
H13C	C4[12]	3.413	H13C	C5[11]	3.110
H13C	C6[11]	3.498	H13C	H5[11]	2.504
H13C	H6[11]	3.248	H13C	H7A[3]	3.315
H13C	H13B[6]	3.498	H13C	H15B[4]	3.240
H14A	C14[6]	3.398	H14A	H14A[6]	3.254
H14A	H14B[6]	2.764	H14A	H15A[6]	3.402
H14B	C5[6]	3.266	H14B	C6[6]	3.437
H14B	C14[6]	3.355	H14B	H5[6]	2.975
H14B	H6[6]	3.283	H14B	H13A[6]	3.209
H14B	H14A[6]	2.764	H14B	H14B[6]	3.066
H15A	O20[6]	3.377	H15A	C4[1]	3.495
H15A	C6[6]	3.219	H15A	C15[6]	3.518
H15A	H4[1]	2.655	H15A	H6[6]	2.482
H15A	H14A[6]	3.402	H15A	H15A[6]	2.634
H15B	O19[1]	2.732	H15B	C5[6]	3.569
H15B	C6[6]	3.427	H15B	H3[1]	3.595
H15B	H4[1]	3.334	H15B	H5[6]	3.054
H15B	H6[6]	2.758	H15B	H13C[1]	3.240
H16A	O19[1]	2.877	H16A	C8[10]	3.071
H16A	C9[10]	3.534	H16A	C10[10]	3.441
H16A	C11[10]	3.099	H16A	C12[10]	2.734
H16A	C16[10]	3.304	H16A	H10[10]	3.056
H16A	H11[10]	2.732	H16A	H16A[10]	2.975
H16B	O21[7]	2.360	H16B	N17[7]	3.495

H16B	C11[10]	3.558	H16B	H10[10]	3.378
H16B	H11[10]	2.783	H16B	H16B[7]	3.525
H16B	H23[7]	2.707	H23	O21[7]	2.590

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H23	C16[7]	3.473	H23	H3[8]	3.567
H23	H7B[8]	3.307	H23	H10[2]	2.941
H23	H16B[7]	2.707	H23	H23[7]	3.449

Symmetry Operators:

(1) X+1/2,Y+1/2,Z (2) X+1/2-1,Y+1/2,Z
(3) -X+1/2+1,-Y+1/2-1,-Z (4) X+1/2-1,Y+1/2-1,Z
(5) -X+1/2+1,Y+1/2,-Z+1/2 (6) -X+2,Y,-Z+1/2
(7) -X+1/2+1,-Y+1/2,-Z (8) -X+1,-Y,-Z
(9) -X+1,Y,-Z+1/2 (10) -X+2,-Y,-Z
(11) -X+1/2+1,Y+1/2-1,-Z+1/2 (12) X+1/2,Y+1/2-1,Z

Crystal data of **8c**

Experimental

Data Collection

A colorless prism crystal of C16H18N2O3S having approximate dimensions of 0.230 x 0.190 x 0.180 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn724 diffractometer using multi-layer mirror monochromated Mo-Ka radiation.

The crystal-to-detector distance was 44.92 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 8.122(7) Å a = 106.57(2)o
b = 8.129(8) Å b = 97.642(10)o
c = 12.103(12) Å g = 90.74(2)o
V = 758.1(13) Å3

For Z = 2 and F.W. = 318.39, the calculated density is 1.395 g/cm3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -169 ± 1oC to a maximum 2q value of 62.8o. A total of 720 oscillation images were collected. A sweep of data was done using w oscillations from -110.0 to 70.0o in 0.5o steps. The exposure rate was 2.0 [sec./o]. The detector swing angle was -20.01o. A second sweep was performed using w oscillations from -110.0 to 70.0o in 0.5o steps. The exposure rate was 2.0 [sec./o]. The detector swing angle was -20.01o. The crystal-to-detector distance was 44.92 mm. Readout was performed in the 0.141 mm pixel mode.

Data Reduction

Of the 4995 reflections that were collected, 2605 were unique (Rint = 0.0488); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, m, for Mo-Ka radiation is 2.278 cm-1. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.216 to 0.960. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods2 and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement3 on F2 was based on 2605 observed reflections and 203 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

R1 = S ||Fo| - |Fc|| / S |Fo| = 0.0504

wR2 = [S (w (Fo2 - Fc2)2)/ S w(Fo2)2]1/2 = 0.1485

The standard deviation of an observation of unit weight4 was 1.11. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.44 and -0.42 e-/Å3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber5. Anomalous dispersion effects were included in Fcalc6; the values for Df' and Df'' were those of Creagh and McAuley7. The values for the mass attenuation coefficients are those of Creagh and Hubbell8. All calculations were performed using the CrystalStructure9 crystallographic software package except for refinement, which was performed using SHELXL-9710.

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR97: Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G., and Spagna, R. (1999). J. Appl. Cryst., 32, 115-119.

(3) Least Squares function minimized: (SHELXL97)

S*w*(Fo2-Fc2)2 where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$[S w(Fo2-Fc2)2/(No-Nv)]1/2$$

where: No = number of observations
Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(10) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula C16H18N2O3S

Formula Weight 318.39

Crystal Color, Habit colorless, prism

Crystal Dimensions 0.230 X 0.190 X 0.180 mm

Crystal System triclinic

Lattice Type Primitive

Lattice Parameters a = 8.122(7) Å
b = 8.129(8) Å
c = 12.103(12) Å
a = 106.57(2) o
b = 97.642(10) o
g = 90.74(2) o
V = 758.1(13) Å3

Space Group P-1 (#2)

Z value 2

Dcalc 1.395 g/cm3

F000 336.00

m(MoKa)2.278 cm-1

B. Intensity Measurements

Diffractometer Saturn724

Radiation MoKa (l = 0.71075 Å)
multi-layer mirror monochromated

Voltage, Current 50kV, 24mA

Temperature -169.8oC

Detector Aperture 70 x 70 mm

Data Images 720 exposures

w oscillation Range -110.0 - 70.0o

Exposure Rate 2.0 sec./o

Detector Swing Angle -20.01o

w oscillation Range -110.0 - 70.0o

Exposure Rate 2.0 sec./o

Detector Swing Angle -20.01o

Detector Position 44.92 mm

Pixel Size 0.141 mm

2qmax 62.8o

No. of Reflections Measured Total: 4995
Unique: 2605 (Rint = 0.0488)

Corrections Lorentz-polarization
Absorption
(trans. factors: 0.216 - 0.960)

C. Structure Solution and Refinement

Structure Solution Direct Methods (SIR97)

Refinement Full-matrix least-squares on F2

Function Minimized S w (Fo2 - Fc2)2

Least Squares Weights w = 1/ [s2(Fo2) + (0.0851 . P)2
+ 0.4060 . P]
where P = (Max(Fo2,0) + 2Fc2)/3

2qmax cutoff 50.0o

Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2605
No. Variables	203
Reflection/Parameter Ratio	12.83
Residuals: R1 (I>2.00s(I))	0.0504
Residuals: R (All reflections)	0.0570
Residuals: wR2 (All reflections)	0.1485
Goodness of Fit Indicator	1.111
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.44 e-/Å3
Minimum peak in Final Diff. Map	-0.42 e-/Å3

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
S22	0.01726(6)	0.05145(7)	0.23474(5)	1.35(2)
O19	0.5917(2)	-0.4049(3)	0.1355(2)	2.10(4)
O20	-0.0220(2)	0.0996(3)	0.1289(2)	1.83(4)
O21	-0.0638(2)	0.1323(2)	0.3328(2)	1.79(4)
N17	0.3103(3)	-0.0162(3)	0.0827(2)	1.50(4)
N18	-0.0327(3)	-0.1538(3)	0.2014(2)	1.45(4)
C1	0.2332(3)	0.0889(3)	0.2822(2)	1.28(4)
C2	0.3506(3)	0.0406(3)	0.2035(2)	1.34(4)
C3	0.5185(3)	0.0547(4)	0.2535(3)	1.90(5)
C4	0.5643(4)	0.1175(4)	0.3719(3)	2.52(6)
C5	0.4476(3)	0.1707(4)	0.4467(3)	2.40(5)

C6	0.2815(3)	0.1557(3)	0.4014(2)	1.66(5)
C7	0.3635(3)	-0.1885(3)	0.0235(2)	1.59(5)
C8	0.3164(3)	-0.3176(3)	0.0835(2)	1.35(4)
C9	0.4420(3)	-0.4098(3)	0.1395(2)	1.59(5)
C10	0.3532(4)	-0.5114(4)	0.2042(3)	2.01(5)
C11	0.1749(3)	-0.4507(3)	0.1990(2)	1.60(5)
C12	0.1330(3)	-0.3301(3)	0.3150(2)	1.61(5)
C13	-0.0286(3)	-0.2391(3)	0.2941(2)	1.71(5)
C14	0.0059(3)	-0.2694(3)	0.0899(2)	1.53(4)
C15	0.1680(3)	-0.3479(3)	0.1128(2)	1.38(4)
C16	0.1143(4)	-0.4271(4)	0.4041(3)	2.38(5)

Beq = 8/3 p2(U11(aa*)2 + U22(bb*)2 + U33(cc*)2 + 2U12(aa*bb*)cos g + 2U13(aa*cc*)cos b + 2U23(bb*cc*)cos a)

Table 2. Atomic coordinates and Biso involving hydrogen atoms

atom	x	y	z	Biso
H3	0.6017	0.0201	0.2046	2.28
H4	0.6787	0.1247	0.4032	3.03
H5	0.4818	0.2168	0.5282	2.88
H6	0.2000	0.1911	0.4516	1.99
H7A	0.4855	-0.1837	0.0243	1.90
H7B	0.3099	-0.2248	-0.0587	1.90
H10A	0.4073	-0.4867	0.2859	2.42
H10B	0.3537	-0.6364	0.1657	2.42
H11	0.0928	-0.5516	0.1695	1.92
H12	0.2260	-0.2405	0.3482	1.93
H13A	-0.1236	-0.3249	0.2739	2.05
H13B	-0.0438	-0.1521	0.3677	2.05
H14A	0.0137	-0.2034	0.0333	1.83
H14B	-0.0837	-0.3607	0.0566	1.83
H16A	0.0924	-0.3461	0.4771	2.85
H16B	0.2171	-0.4844	0.4183	2.85
H16C	0.0214	-0.5133	0.3739	2.85
H23	0.2039	-0.0069	0.0550	2.42

Table 3. Anisotropic displacement parameters

atom	U11	U22	U33	U12	U13	U23
S22	0.0112(4)	0.0198(4)	0.0188(4)	0.0013(3)	0.0029(3)	0.0030(3)
O19	0.0210(10)	0.0299(10)	0.0288(11)	0.0057(8)	0.0042(8)	0.0076(8)
O20	0.0186(9)	0.0265(10)	0.0238(10)	0.0057(7)	-0.0006(7)	0.0077(8)
O21	0.0155(9)	0.0250(9)	0.0228(10)	0.0018(7)	0.0057(7)	-0.0019(8)
N17	0.0175(11)	0.0225(11)	0.0200(11)	0.0019(8)	0.0064(9)	0.0092(9)
N18	0.0146(10)	0.0222(11)	0.0172(11)	-0.0029(8)	0.0054(8)	0.0026(8)
C1	0.0112(11)	0.0165(11)	0.0224(13)	-0.0008(9)	0.0031(9)	0.0078(10)
C2	0.0166(12)	0.0146(11)	0.0223(13)	-0.0005(9)	0.0051(10)	0.0087(10)
C3	0.0132(12)	0.029(2)	0.031(2)	-0.0007(10)	0.0084(10)	0.0072(11)
C4	0.0143(12)	0.043(2)	0.034(2)	-0.0030(11)	-0.0004(11)	0.0050(13)
C5	0.022(2)	0.040(2)	0.024(2)	-0.0018(12)	-0.0007(11)	0.0032(12)
C6	0.0168(12)	0.0223(12)	0.0228(13)	0.0008(10)	0.0046(10)	0.0041(10)
C7	0.0193(12)	0.0230(12)	0.0213(13)	0.0007(10)	0.0101(10)	0.0084(10)
C8	0.0216(12)	0.0162(11)	0.0129(12)	-0.0002(9)	0.0050(9)	0.0023(9)
C9	0.0256(13)	0.0176(12)	0.0160(12)	0.0032(10)	0.0050(10)	0.0015(10)
C10	0.034(2)	0.0239(13)	0.0229(13)	0.0068(11)	0.0096(11)	0.0102(11)
C11	0.0257(13)	0.0168(12)	0.0188(13)	-0.0027(10)	0.0072(10)	0.0043(10)
C12	0.0267(13)	0.0177(12)	0.0179(13)	-0.0031(10)	0.0063(10)	0.0056(10)

C13	0.0208(13)	0.0237(13)	0.0198(13)	-0.0057(10)	0.0081(10) 0.0031(10)
C14	0.0187(12)	0.0224(12)	0.0147(12)	-0.0031(10)	0.0032(9) 0.0018(10)
C15	0.0210(12)	0.0154(11)	0.0134(12)	-0.0028(9)	0.0042(9) -0.0006(9)
C16	0.046(2)	0.0243(13)	0.024(2)	-0.0004(12)	0.0152(12) 0.0090(11)

The general temperature factor expression: exp(-2p2(a*2U11h2 + b*2U22k2 + c*2U33l2 + 2a*b*U12hk + 2a*c*U13hl + 2b*c*U23kl))

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S22	O20	1.441(3)	S22	O21	1.432(2)
S22	N18	1.633(3)	S22	C1	1.766(3)
O19	C9	1.224(4)	N17	C2	1.394(4)
N17	C7	1.479(4)	N18	C13	1.476(4)
N18	C14	1.483(3)	C1	C2	1.417(4)
C1	C6	1.388(4)	C2	C3	1.405(4)
C3	C4	1.375(4)	C4	C5	1.386(4)
C5	C6	1.378(4)	C7	C8	1.508(4)
C8	C9	1.478(4)	C8	C15	1.340(4)
C9	C10	1.523(5)	C10	C11	1.535(4)
C11	C12	1.549(4)	C11	C15	1.508(4)
C12	C13	1.542(4)	C12	C16	1.527(5)
C14	C15	1.498(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
N17	H23	0.897	C3	H3	0.950
C4	H4	0.950	C5	H5	0.950
C6	H6	0.950	C7	H7A	0.990

C7	H7B	0.990	C10	H10A	0.990
C10	H10B	0.990	C11	H11	1.000
C12	H12	1.000	C13	H13A	0.990
C13	H13B	0.990	C14	H14A	0.990
C14	H14B	0.990	C16	H16A	0.980
C16	H16B	0.980	C16	H16C	0.980

Table 6. Bond angles (o)

atom	atom	atom	angle	atom	atom	atom	angle	
O20	S22	O21	119.06(12)		O20	S22	N18	106.32(11)
O20	S22	C1	108.62(12)		O21	S22	N18	106.18(12)
O21	S22	C1	106.62(11)		N18	S22	C1	109.85(11)
C2	N17	C7	116.6(2)	S22	N18	C13	119.97(15)	
S22	N18	C14	119.22(19)		C13	N18	C14	113.0(2)
S22	C1	C2	121.52(17)		S22	C1	C6	116.30(18)
C2	C1	C6	122.0(2)	N17	C2	C1	124.6(2)	
N17	C2	C3	119.2(3)	C1	C2	C3	116.2(3)	
C2	C3	C4	121.1(3)	C3	C4	C5	121.6(3)	
C4	C5	C6	119.1(3)	C1	C6	C5	119.9(3)	
N17	C7	C8	110.5(3)	C7	C8	C9	122.3(3)	
C7	C8	C15	128.2(3)	C9	C8	C15	108.6(3)	
O19	C9	C8	126.2(3)	O19	C9	C10	125.6(3)	
C8	C9	C10	108.2(3)	C9	C10	C11	104.8(3)	
C10	C11	C12	114.76(19)		C10	C11	C15	103.4(3)
C12	C11	C15	107.5(2)	C11	C12	C13	110.7(2)	
C11	C12	C16	111.9(3)	C13	C12	C16	109.1(3)	
N18	C13	C12	114.5(2)	N18	C14	C15	108.68(18)	
C8	C15	C11	113.9(3)	C8	C15	C14	129.3(3)	
C11	C15	C14	115.8(3)					

Table 7. Bond angles involving hydrogens (o)

atom	atom	atom	angle	atom	atom	atom	angle
C2	N17	H23	115.4	C7	N17	H23	109.3
C2	C3	H3	119.4	C4	C3	H3	119.4

C3	C4	H4	119.2	C5	C4	H4	119.2
C4	C5	H5	120.5	C6	C5	H5	120.5
C1	C6	H6	120.0	C5	C6	H6	120.0
N17	C7	H7A	109.5	N17	C7	H7B	109.5
C8	C7	H7A	109.6	C8	C7	H7B	109.5
H7A	C7	H7B	108.1	C9	C10	H10A	110.8
C9	C10	H10B	110.8	C11	C10	H10A	110.8
C11	C10	H10B	110.8	H10A	C10	H10B	108.9
C10	C11	H11	110.3	C12	C11	H11	110.3
C15	C11	H11	110.3	C11	C12	H12	108.4
C13	C12	H12	108.4	C16	C12	H12	108.4
N18	C13	H13A	108.6	N18	C13	H13B	108.6
C12	C13	H13A	108.6	C12	C13	H13B	108.6
H13A	C13	H13B	107.6	N18	C14	H14A	110.0
N18	C14	H14B	110.0	C15	C14	H14A	110.0
C15	C14	H14B	110.0	H14A	C14	H14B	108.3
C12	C16	H16A	109.5	C12	C16	H16B	109.5
C12	C16	H16C	109.5	H16A	C16	H16B	109.5
H16A	C16	H16C	109.5	H16B	C16	H16C	109.5

Table 8. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A		
N17	H23	S22		3.159(3)	0.90	2.75	109.10	intramol.
N17	H23	O20		2.941(4)	0.90	2.22	136.56	intramol.

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S22	N17	3.159(3)	S22	C15	3.474(4)
O19	C7	3.024(4)	O19	C15	3.463(5)
O20	N17	2.941(4)	O20	C2	3.125(4)
O20	C14	2.917(5)	O21	C6	2.805(4)
O21	C13	2.945(5)	N17	C14	3.222(4)
N17	C15	3.055(4)	N18	C2	3.467(4)
N18	C8	3.455(4)	N18	C11	2.957(4)

C1	C4	2.744(4)	C1	C7	3.595(4)
C1	C13	3.437(5)	C1	C14	3.487(4)
C1	C15	3.551(4)	C2	C5	2.827(5)
C2	C8	2.850(4)	C2	C14	3.591(4)
C2	C15	3.293(4)	C3	C6	2.775(4)
C3	C7	3.018(4)	C3	C8	3.392(4)
C7	C14	3.222(5)	C8	C12	3.369(5)
C9	C12	3.450(5)	C10	C16	3.229(5)
C12	C14	2.961(5)	C13	C15	2.831(4)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S22	H6	2.757	S22	H12	3.426
S22	H13A	3.431	S22	H13B	2.698
S22	H14A	2.708	S22	H14B	3.448
S22	H23	2.749	O19	H3	3.312
O19	H7A	2.627	O19	H10A	2.718
O19	H10B	2.811	O20	H14A	2.444
O20	H23	2.225	O21	H6	2.377
O21	H13A	3.593	O21	H13B	2.469
N17	H3	2.583	N17	H14A	2.735
N18	H11	3.335	N18	H12	2.798
N18	H23	3.210	C1	H3	3.262
C1	H5	3.256	C1	H12	3.008
C1	H13B	3.411	C1	H14A	3.504
C1	H23	2.611	C2	H4	3.272
C2	H6	3.307	C2	H7A	2.767
C2	H7B	3.261	C2	H12	3.465
C2	H14A	3.420	C3	H5	3.264
C3	H7A	2.872	C3	H23	3.194
C4	H6	3.243	C5	H3	3.259
C5	H12	3.583	C6	H4	3.237
C6	H12	3.114	C6	H13B	3.517
C7	H3	2.845	C7	H14A	2.861
C8	H3	3.419	C8	H10A	3.146
C8	H10B	3.035	C8	H11	3.089

C8	H12	3.269	C8	H14A	2.694
C8	H14B	3.227	C8	H23	2.794
C9	H3	3.544	C9	H7A	2.650
C9	H7B	3.252	C9	H11	3.152
C9	H12	3.257	C10	H12	2.703
C10	H16B	2.904	C11	H13A	2.805
C11	H13B	3.386	C11	H14A	3.382
C11	H14B	2.777	C11	H16A	3.389
C11	H16B	2.723	C11	H16C	2.756
C12	H10A	2.595	C12	H10B	3.330
C12	H14B	3.321	C13	H11	2.824
C13	H14A	3.313	C13	H14B	2.733
C13	H16A	2.677	C13	H16B	3.351

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C13	H16C	2.686	C14	H7B	3.315
C14	H11	2.795	C14	H12	3.333
C14	H13A	2.730	C14	H13B	3.304
C14	H23	2.815	C15	H7A	3.322
C15	H7B	2.903	C15	H10A	3.125
C15	H10B	2.980	C15	H12	2.705
C15	H13A	3.237	C15	H23	3.065
C16	H10A	2.913	C16	H11	2.709
C16	H13A	2.625	C16	H13B	2.707
H3	H4	2.305	H3	H7A	2.398
H3	H23	3.459	H4	H5	2.331
H5	H6	2.335	H6	H12	3.392
H6	H13B	3.231	H7A	H23	2.728
H7B	H14A	2.770	H7B	H23	2.177
H10A	H11	2.715	H10A	H12	2.501
H10A	H16B	2.366	H10A	H16C	3.465
H10B	H11	2.239	H10B	H12	3.599
H10B	H16B	3.300	H11	H12	2.903
H11	H13A	2.726	H11	H14B	2.660
H11	H16B	2.937	H11	H16C	2.550

H12	H13A	2.874	H12	H13B	2.336
H12	H16A	2.353	H12	H16B	2.372
H12	H16C	2.865	H13A	H14B	2.627
H13A	H16A	2.874	H13A	H16B	3.535
H13A	H16C	2.434	H13B	H16A	2.512
H13B	H16B	3.593	H13B	H16C	3.011
H14A	H23	2.144			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O19	$O19^{1}$	3.335(4)	O19	$N18^{2}$	3.518(4)
O19	$C7^{1}$	3.369(4)	O19	$C8^{1}$	3.136(4)
O19	$C9^{1}$	3.211(4)	O19	$C13^{2}$	3.443(4)
O20	$O20^{3}$	3.149(4)	O20	$N17^{3}$	3.142(4)
O20	$C7^{3}$	3.327(4)	O20	$C14^{3}$	3.333(5)
O21	$C3^{4}$	3.400(4)	O21	$C4^{4}$	3.125(5)
O21	$C16^{5}$	3.474(4)	N17	$O20^{3}$	3.142(4)
N18	$O19^{4}$	3.518(4)	C3	$O21^{2}$	3.400(4)
C4	$O21^{2}$	3.125(5)	C5	$C5^{6}$	3.449(6)
C7	$O19^{1}$	3.369(4)	C7	$O20^{3}$	3.327(4)
C8	$O19^{1}$	3.136(4)	C9	$O19^{1}$	3.211(4)
C9	$C9^{1}$	3.536(5)	C13	$O19^{4}$	3.443(4)
C14	$O20^{3}$	3.333(5)	C14	$C15^{7}$	3.507(4)
C15	$C14^{7}$	3.507(4)	C16	$O21^{5}$	3.474(4)
C16	$C16^{8}$	3.592(5)			

Symmetry Operators:

(1) -X+1,-Y-1,-Z	(2) X+1,Y,Z
(3) -X,-Y,-Z	(4) X-1,Y,Z
(5) -X,-Y,-Z+1	(6) -X+1,-Y,-Z+1
(7) -X,-Y-1,-Z	(8) -X,-Y-1,-Z+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S22	H3[1]	3.345	S22	H4[1]	3.594
S22	H11[2]	3.598	S22	H16C[2]	3.455
O19	H7A[3]	3.354	O19	H7B[3]	3.037
O19	H13A[4]	2.624	O19	H14B[4]	2.973
O20	H3[1]	3.410	O20	H7B[5]	2.693
O20	H11[2]	2.858	O20	H14A[5]	2.356
O20	H23[5]	2.418	O21	H3[1]	2.937
O21	H4[1]	2.367	O21	H13B[6]	3.571
O21	H16A[6]	2.497	O21	H16C[2]	2.840
N17	H7A[7]	2.962	N17	H10B[2]	2.962
N17	H14A[5]	3.557	N18	H3[1]	3.306
C1	H10B[2]	3.167	C1	H16B[2]	3.402
C2	H10B[2]	2.791	C3	H7B[7]	3.479
C3	H10B[2]	3.234	C4	H5[8]	3.310
C4	H12[8]	3.445	C4	H16A[8]	3.338
C4	H16B[8]	3.583	C5	H5[8]	3.300
C5	H10A[8]	3.554	C5	H12[8]	3.304
C5	H16B[2]	3.465	C5	H16B[8]	3.584
C6	H16B[2]	2.933	C6	H16C[2]	3.504
C7	H3[7]	3.454	C7	H7A[7]	3.477
C7	H10B[3]	3.489	C8	H14B[9]	3.127
C9	H7A[3]	3.438	C9	H7B[3]	3.586
C10	H5[8]	3.516	C10	H7A[3]	3.556
C10	H14B[9]	3.473	C11	H14A[9]	3.521
C11	H14B[9]	3.021	C12	H4[8]	3.472
C12	H5[8]	3.404	C13	H6[6]	3.468
C14	H11[9]	3.052	C14	H14B[9]	3.145
C14	H23[5]	3.546	C15	H14B[9]	2.664
C16	H4[8]	3.145	C16	H5[8]	3.563
C16	H6[10]	3.379	C16	H6[6]	3.557
C16	H16A[11]	3.218	C16	H16C[11]	3.191
H3	S22[4]	3.345	H3	O20[4]	3.410
H3	O21[4]	2.937	H3	N18[4]	3.306
H3	C7[7]	3.454	H3	H7A[7]	3.399
H3	H7B[7]	2.896	H3	H10B[2]	3.566
H4	S22[4]	3.594	H4	O21[4]	2.367

H4	C12[8]	3.472	H4	C16[8]	3.145

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4	H5[8]	3.407	H4	H6[8]	3.581
H4	H12[8]	2.878	H4	H13B[4]	3.189
H4	H16A[8]	2.550	H4	H16B[8]	3.127
H5	C4[8]	3.310	H5	C5[8]	3.300
H5	C10[8]	3.516	H5	C12[8]	3.404
H5	C16[8]	3.563	H5	H4[8]	3.407
H5	H5[8]	3.419	H5	H10A[8]	2.699
H5	H12[8]	2.605	H5	H16B[8]	3.126
H6	C13[6]	3.468	H6	C16[2]	3.379
H6	C16[6]	3.557	H6	H4[8]	3.581
H6	H13A[6]	3.333	H6	H13B[6]	2.760
H6	H16A[6]	2.824	H6	H16B[2]	2.785
H6	H16C[2]	3.127	H6	H16C[6]	3.540
H7A	O19[3]	3.354	H7A	N17[7]	2.962
H7A	C7[7]	3.477	H7A	C9[3]	3.438
H7A	C10[3]	3.556	H7A	H3[7]	3.399
H7A	H7A[7]	3.217	H7A	H7B[7]	3.585
H7A	H10B[3]	2.840	H7A	H23[7]	3.329
H7B	O19[3]	3.037	H7B	O20[5]	2.693
H7B	C3[7]	3.479	H7B	C9[3]	3.586
H7B	H3[7]	2.896	H7B	H7A[7]	3.585
H7B	H10B[3]	3.257	H10A	C5[8]	3.554
H10A	H5[8]	2.699	H10B	N17[10]	2.962
H10B	C1[10]	3.167	H10B	C2[10]	2.791
H10B	C3[10]	3.234	H10B	C7[3]	3.489
H10B	H3[10]	3.566	H10B	H7A[3]	2.840
H10B	H7B[3]	3.257	H10B	H14A[9]	3.550
H10B	H14B[9]	3.231	H10B	H23[10]	3.085
H11	S22[10]	3.598	H11	O20[10]	2.858
H11	C14[9]	3.052	H11	H14A[9]	2.710
H11	H14B[9]	2.614	H12	C4[8]	3.445
H12	C5[8]	3.304	H12	H4[8]	2.878

H12	H5[8]	2.605	H13A	O19[1]	2.624
H13A	H6[6]	3.333	H13B	O21[6]	3.571
H13B	H4[1]	3.189	H13B	H6[6]	2.760
H13B	H13B[6]	3.423	H14A	O20[5]	2.356
H14A	N17[5]	3.557	H14A	C11[9]	3.521

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14A	H10B[9]	3.550	H14A	H11[9]	2.710
H14A	H14B[9]	3.475	H14A	H23[5]	2.794
H14B	O19[1]	2.973	H14B	C8[9]	3.127
H14B	C10[9]	3.473	H14B	C11[9]	3.021
H14B	C14[9]	3.145	H14B	C15[9]	2.664
H14B	H10B[9]	3.231	H14B	H11[9]	2.614
H14B	H14A[9]	3.475	H14B	H14B[9]	2.767
H16A	O21[6]	2.497	H16A	C4[8]	3.338
H16A	C16[11]	3.218	H16A	H4[8]	2.550
H16A	H6[6]	2.824	H16A	H16A[11]	3.121
H16A	H16B[11]	3.402	H16A	H16C[11]	2.646
H16B	C1[10]	3.402	H16B	C4[8]	3.583
H16B	C5[10]	3.465	H16B	C5[8]	3.584
H16B	C6[10]	2.933	H16B	H4[8]	3.127
H16B	H5[8]	3.126	H16B	H6[10]	2.785
H16B	H16A[11]	3.402	H16B	H16C[11]	3.376
H16C	S22[10]	3.455	H16C	O21[10]	2.840
H16C	C6[10]	3.504	H16C	C16[11]	3.191
H16C	H6[10]	3.127	H16C	H6[6]	3.540
H16C	H16A[11]	2.646	H16C	H16B[11]	3.376
H16C	H16C[11]	3.064	H23	O20[5]	2.418
H23	C14[5]	3.546	H23	H7A[7]	3.329
H23	H10B[2]	3.085	H23	H14A[5]	2.794
H23	H23[5]	3.417			

Symmetry Operators:

(1) X-1,Y,Z
(2) X,Y+1,Z
(3) -X+1,-Y-1,-Z
(4) X+1,Y,Z
(5) -X,-Y,-Z
(6) -X,-Y,-Z+1
(7) -X+1,-Y,-Z
(8) -X+1,-Y,-Z+1
(9) -X,-Y-1,-Z
(10) X,Y-1,Z
(11) -X,-Y-1,-Z+1