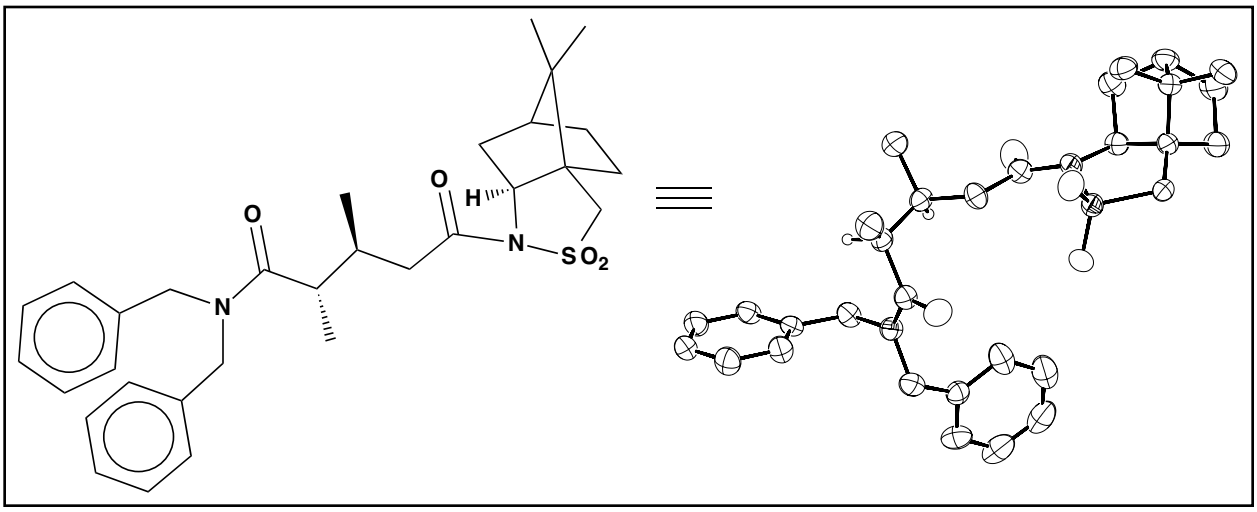
X-ray Structure Determination of Compound 773



Compound 773, $C_{31}H_{40}N_2SO_4$, crystallizes in theorthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=8.705(9)Å, b=40.336(10)Å, c=8.270(9)Å, V=2904(4)Å^3, Z=4 and d_{calc}=1.228 g/cm^3. The cell constants were determined from a least squares fit of the setting angles for 25 accurately centered reflections. X-ray intensity data were collected on a Rigaku AFC7 diffractometer employing graphite-monochromated Cu-K_α radiation (λ=1.54178Å) at a temperature of 295°K using the ω-2θ scan technique. A total of 2259 reflections were measured over the ranges 8.76 ≤ 2θ ≤ 120 °, 0 ≤ h ≤ 9, 0 ≤ k ≤ 43, 0 ≤ l ≤ 9 yielding 2259 unique reflections (R_{int} = 0.0000). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[1]). Refinement was by full-matrix least squares based on F^2 using SHELXL-93[2]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by F^2 = 0). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+0.0821P^2+0.2241P]$ where $P=(F_o^2+2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a "riding" model. Refinement converged to R_1=0.0435 and wR_2=0.1200 for 1923 reflections for which F > 4σ(F) and R_1=0.0573, wR_2=0.1299 and GOF = 1.066 for all 2259 unique, non-zero reflections and 348 variables[3]. The maximum Δ/σ in the final cycle of least squares was -0.001 and the two most prominent peaks in the final difference Fourier were +0.228 and -0.294 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[4] representation of the molecule with 30% probability thermal ellipsoids displayed.

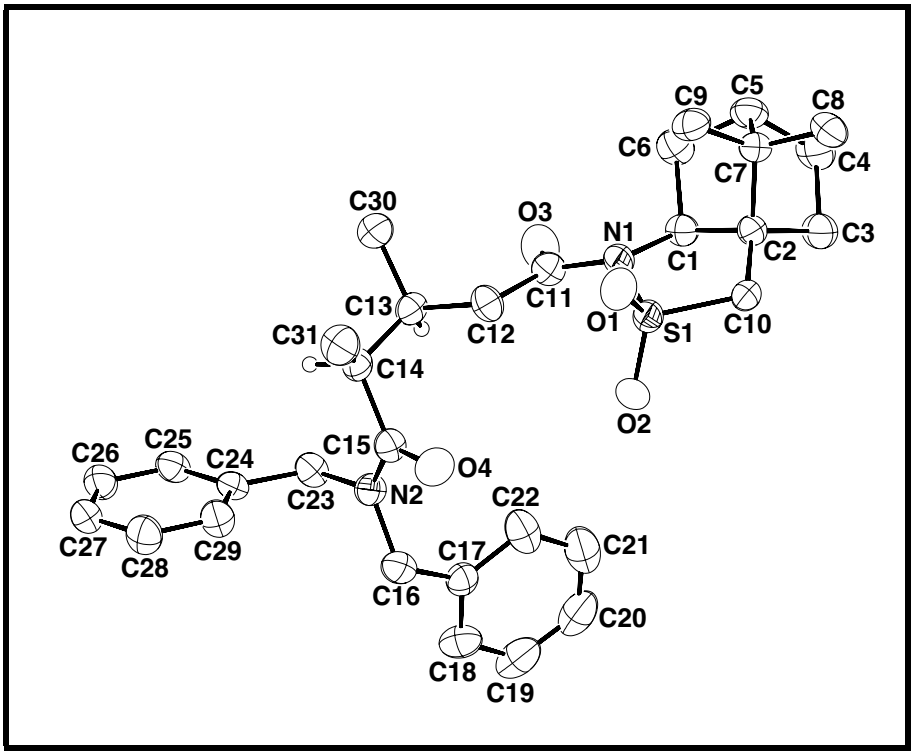


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 773

Formula:	**$C_{31}H_{40}N_2SO_4$**
Formula weight:	**536.71**
Crystal class:	**orthorhombic**
Space group:	**$P2_12_12_1$ (#19)**
Z	**4**
Cell constants:	
a	**8.705(9)Å**
b	**40.336(10)Å**
c	**8.270(9)Å**

V	**2904(4)Å^3**
μ	**12.87 cm^{-1}**
crystal size, mm	**0.44 x 0.32 x 0.01**
D_{calc}	**1.228 g/cm^3**
F(000)	**1152**
Radiation:	**Cu-K_α (λ=1.54178Å)**
2θ range	**8.76 – 120 °**
hkl collected:	**0≤ h ≤9; 0≤ k ≤43; 0≤ l ≤9**
No. reflections measured:	**2259**
No. unique reflections:	**2259 (R_{int}=0.0000)**
No. observed reflections	**1923 (F>4σ)**
No. reflections used in refinement	**2259**
No. parameters	**348**
R indices (F>4σ)	**R_1=0.0435**
	wR_2=0.1200
R indices (all data)	**R_1=0.0573**
	wR_2=0.1299
GOF:	**1.066**
Final Difference Peaks, e/Å^3	**+0.228, -0.294**

Table 2. Refined Positional Parameters for Compound 773

Atom	x	y	z	U_{eq}, Å^2
S1	0.08542(14)	0.89434(3)	0.50832(13)	0.0561(4)
C1	0.1982(6)	0.92742(11)	0.7546(5)	0.0558(12)
H1	0.2451(6)	0.91149(11)	0.8293(5)	0.074
C2	0.0234(6)	0.92756(11)	0.7779(5)	0.0533(11)
C3	-0.0027(8)	0.92416(13)	0.9610(6)	0.080(2)
H3a	0.0565(8)	0.90595(13)	1.0057(6)	0.106
H3b	-0.1105(8)	0.92087(13)	0.9859(6)	0.106
C4	0.0558(8)	0.95823(13)	1.0254(6)	0.086(2)
H4a	-0.0241(8)	0.96971(13)	1.0851(6)	0.114
H4b	0.1448(8)	0.95545(13)	1.0948(6)	0.114
C5	0.0981(7)	0.97712(12)	0.8696(6)	0.0700(14)
H5	0.0993(7)	1.00127(12)	0.8826(6)	0.093
C6	0.2475(6)	0.96295(12)	0.8009(7)	0.0692(14)
H6a	0.3285(6)	0.96277(12)	0.8815(7)	0.092
H6b	0.2817(6)	0.97534(12)	0.7071(7)	0.092
C7	-0.0228(6)	0.96465(11)	0.7475(6)	0.0594(12)
C8	-0.1905(6)	0.97207(13)	0.7934(8)	0.086(2)
H8a	-0.2574(7)	0.9573(7)	0.736(4)	0.128
H8b	-0.215(2)	0.9945(4)	0.765(5)	0.128
H8c	-0.2039(13)	0.9690(10)	0.9077(12)	0.128
C9	0.0004(7)	0.97754(12)	0.5759(6)	0.0727(14)
H9a	-0.072(3)	0.9672(7)	0.5049(10)	0.109
H9b	0.103(2)	0.9724(8)	0.540(2)	0.109
H9c	-0.014(4)	1.0011(2)	0.5742(11)	0.109
C10	-0.0465(6)	0.90113(11)	0.6712(6)	0.0610(13)
H10a	-0.1451(6)	0.90843(11)	0.6298(6)	0.081
H10b	-0.0617(6)	0.88082(11)	0.7320(6)	0.081
C11	0.3828(5)	0.91361(12)	0.5366(6)	0.0594(12)
C12	0.4068(6)	0.90130(13)	0.3663(6)	0.0628(12)
H12a	0.3543(6)	0.91605(13)	0.2919(6)	0.083
H12b	0.3608(6)	0.87949(13)	0.3558(6)	0.083
C13	0.5770(5)	0.89922(11)	0.3187(6)	0.0550(11)
H13	0.6313(5)	0.88871(11)	0.4090(6)	0.073
C14	0.6021(5)	0.87722(11)	0.1693(6)	0.0561(11)
H14	0.7120(5)	0.87739(11)	0.1434(6)	0.075
C15	0.5551(5)	0.84127(11)	0.2074(6)	0.0574(12)
C16	0.6224(7)	0.78598(12)	0.2937(6)	0.0687(14)
H16a	0.7003(7)	0.77163(12)	0.2475(6)	0.091
H16b	0.5262(7)	0.78129(12)	0.2392(6)	0.091
C17	0.6051(5)	0.77784(12)	0.4712(6)	0.0596(12)
C18	0.6697(7)	0.74928(14)	0.5306(7)	0.080(2)
H18	0.7289(7)	0.73618(14)	0.4621(7)	0.106
C19	0.6496(9)	0.7396(2)	0.6871(9)	0.102(2)
H19	0.6957(9)	0.7202(2)	0.7247(9)	0.136
C20	0.5643(9)	0.7577(2)	0.7849(9)	0.099(2)
H20	0.5479(9)	0.7505(2)	0.8903(9)	0.131
C21	0.5003(8)	0.7865(2)	0.7360(9)	0.096(2)
H21	0.4436(8)	0.7993(2)	0.8083(9)	0.128
C22	0.5200(7)	0.7974(2)	0.5733(7)	0.086(2)
H22	0.4764(7)	0.8171(2)	0.5374(7)	0.114
C23	0.8290(5)	0.82854(12)	0.2665(6)	0.0599(12)
H23a	0.8788(5)	0.81445(12)	0.3458(6)	0.080

H23b	0.8414(5)	0.85133(12)	0.3015(6)	0.080
C24	0.9088(5)	0.82418(10)	0.1053(5)	0.0496(10)
C25	1.0622(6)	0.83212(11)	0.0930(6)	0.0603(12)
H25	1.1135(6)	0.84070(11)	0.1823(6)	0.080
C26	1.1400(6)	0.82754(13)	-0.0489(7)	0.072(2)
H26	1.2438(6)	0.83287(13)	-0.0551(7)	0.095
C27	1.0658(7)	0.81512(12)	-0.1826(6)	0.0685(14)
H27	1.1195(7)	0.81196(12)	-0.2786(6)	0.091
C28	0.9137(7)	0.80745(14)	-0.1745(7)	0.077(2)
H28	0.8630(7)	0.79901(14)	-0.2645(7)	0.102
C29	0.8349(6)	0.81235(13)	-0.0305(6)	0.0662(13)
H29	0.7305(6)	0.80756(13)	-0.0255(6)	0.088
C30	0.6474(6)	0.93357(12)	0.2956(7)	0.077(2)
H30a	0.632(4)	0.9465(4)	0.391(2)	0.116
H30b	0.599(4)	0.9443(4)	0.205(3)	0.116
H30c	0.7556(11)	0.93141(13)	0.275(5)	0.116
C31	0.5130(7)	0.88865(14)	0.0177(6)	0.082(2)
H31a	0.541(4)	0.9111(4)	-0.008(3)	0.123
H31b	0.4046(7)	0.8875(10)	0.038(2)	0.123
H31c	0.538(4)	0.8745(7)	-0.0718(14)	0.123
N1	0.2293(4)	0.91730(9)	0.5864(4)	0.0537(9)
N2	0.6650(4)	0.82061(9)	0.2629(5)	0.0556(10)
O1	0.0340(4)	0.90897(9)	0.3612(4)	0.0751(11)
O2	0.1319(4)	0.86021(8)	0.5011(4)	0.0762(10)
O3	0.4846(4)	0.92107(11)	0.6285(5)	0.0831(11)
O4	0.4222(4)	0.83251(9)	0.1946(5)	0.0802(11)

$$U_{eq}=1/3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$$

Table 3. Refined Thermal Parameters (U's) for Compound 773

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0608(6)	0.0600(7)	0.0476(5)	-0.0075(5)	0.0034(6)	-0.0098(6)
C1	0.067(3)	0.055(3)	0.045(2)	-0.001(2)	-0.005(2)	0.001(2)
C2	0.064(3)	0.050(3)	0.045(2)	-0.004(2)	0.005(2)	-0.002(2)
C3	0.120(5)	0.068(3)	0.053(3)	0.000(2)	0.017(3)	0.002(3)
C4	0.124(5)	0.085(4)	0.050(3)	-0.014(3)	0.004(3)	-0.001(4)
C5	0.087(4)	0.054(3)	0.070(3)	-0.013(2)	-0.002(3)	-0.007(3)
C6	0.068(3)	0.073(3)	0.067(3)	-0.017(3)	-0.011(3)	-0.007(3)
C7	0.067(3)	0.048(3)	0.063(3)	-0.002(2)	0.005(2)	0.000(2)
C8	0.082(4)	0.068(3)	0.107(5)	-0.014(3)	0.017(4)	0.012(3)
C9	0.088(4)	0.056(3)	0.074(3)	0.010(3)	-0.002(3)	-0.002(3)
C10	0.068(3)	0.053(3)	0.062(3)	-0.004(2)	0.015(2)	-0.011(2)
C11	0.056(3)	0.062(3)	0.060(3)	-0.004(2)	-0.006(2)	-0.002(2)
C12	0.055(3)	0.073(3)	0.060(3)	-0.006(2)	-0.001(2)	0.009(3)
C13	0.045(2)	0.055(3)	0.065(3)	0.009(2)	-0.003(2)	0.000(2)
C14	0.049(2)	0.059(3)	0.061(2)	0.005(2)	0.004(2)	0.002(2)
C15	0.052(3)	0.063(3)	0.057(3)	-0.003(2)	-0.002(2)	-0.001(2)
C16	0.079(4)	0.058(3)	0.069(3)	-0.009(3)	0.003(3)	-0.001(3)
C17	0.053(3)	0.062(3)	0.064(3)	-0.005(2)	0.001(2)	-0.011(2)
C18	0.096(4)	0.060(3)	0.084(4)	-0.004(3)	-0.013(3)	-0.012(3)
C19	0.122(6)	0.089(4)	0.096(5)	0.019(4)	-0.005(5)	-0.035(4)
C20	0.103(5)	0.110(5)	0.084(4)	0.012(4)	0.004(4)	-0.042(5)
C21	0.073(4)	0.125(6)	0.090(4)	-0.033(4)	0.011(4)	-0.011(4)
C22	0.073(3)	0.109(4)	0.076(3)	-0.022(3)	0.004(3)	0.006(3)
C23	0.052(3)	0.067(3)	0.061(3)	-0.010(2)	-0.006(2)	0.004(2)
C24	0.048(2)	0.044(2)	0.057(2)	-0.007(2)	-0.004(2)	0.005(2)
C25	0.057(3)	0.055(3)	0.069(3)	-0.012(2)	-0.005(2)	0.002(2)
C26	0.060(3)	0.068(3)	0.086(4)	-0.014(3)	0.011(3)	-0.003(3)
C27	0.076(3)	0.063(3)	0.066(3)	-0.006(3)	0.016(3)	0.005(3)
C28	0.082(4)	0.089(4)	0.061(3)	-0.014(3)	-0.001(3)	-0.009(3)
C29	0.056(3)	0.080(3)	0.063(3)	-0.015(3)	-0.005(2)	-0.007(3)
C30	0.073(3)	0.066(3)	0.093(4)	0.001(3)	0.011(3)	-0.005(3)
C31	0.097(4)	0.088(4)	0.061(3)	0.009(3)	-0.005(3)	0.006(3)
N1	0.060(2)	0.055(2)	0.046(2)	-0.008(2)	0.001(2)	-0.003(2)
N2	0.045(2)	0.056(2)	0.065(2)	-0.002(2)	-0.002(2)	-0.001(2)
O1	0.072(2)	0.098(3)	0.055(2)	0.007(2)	-0.009(2)	-0.009(2)
O2	0.094(2)	0.056(2)	0.079(2)	-0.018(2)	0.024(2)	-0.003(2)
O3	0.061(2)	0.118(3)	0.070(2)	-0.015(2)	-0.014(2)	-0.004(2)
O4	0.047(2)	0.080(2)	0.113(3)	-0.002(2)	-0.009(2)	-0.011(2)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$.

Table 4. Bond Distances in Compound 773, Å

S1-O1	1.424(4)	S1-O2	1.436(3)	S1-N1	1.686(4)
S1-C10	1.791(5)	C1-N1	1.475(6)	C1-C2	1.534(7)
C1-C6	1.544(6)	C2-C10	1.512(6)	C2-C3	1.538(6)
C2-C7	1.569(6)	C3-C4	1.559(8)	C4-C5	1.541(8)
C5-C6	1.530(7)	C5-C7	1.543(7)	C7-C9	1.525(7)
C7-C8	1.537(8)	C11-O3	1.206(5)	C11-N1	1.406(6)
C11-C12	1.508(7)	C12-C13	1.535(7)	C13-C30	1.527(6)
C13-C14	1.537(6)	C14-C15	1.539(6)	C14-C31	1.545(7)
C15-O4	1.215(6)	C15-N2	1.349(6)	C16-N2	1.467(6)
C16-C17	1.512(7)	C17-C22	1.372(7)	C17-C18	1.373(7)
C18-C19	1.363(9)	C19-C20	1.320(10)	C20-C21	1.351(9)
C21-C22	1.425(10)	C23-N2	1.463(6)	C23-C24	1.513(6)
C24-C25	1.377(7)	C24-C29	1.379(6)	C25-C26	1.368(7)
C26-C27	1.375(7)	C27-C28	1.361(8)	C28-C29	1.388(7)

Table 5. Bond Angles in Compound 773, °

O1-S1-O2	116.8(2)	O1-S1-N1	109.4(2)	O2-S1-N1	109.5(2)
O1-S1-C10	112.2(2)	O2-S1-C10	111.0(2)	N1-S1-C10	96.0(2)
N1-C1-C2	107.5(4)	N1-C1-C6	116.1(4)	C2-C1-C6	103.9(4)
C10-C2-C1	108.9(4)	C10-C2-C3	116.9(4)	C1-C2-C3	105.7(5)
C10-C2-C7	118.4(4)	C1-C2-C7	103.8(4)	C3-C2-C7	101.8(4)
C2-C3-C4	102.1(4)	C5-C4-C3	103.2(4)	C6-C5-C4	109.2(5)
C6-C5-C7	102.4(4)	C4-C5-C7	102.9(4)	C5-C6-C1	101.7(4)
C9-C7-C8	106.8(5)	C9-C7-C5	114.1(4)	C8-C7-C5	115.0(5)
C9-C7-C2	116.1(4)	C8-C7-C2	112.9(4)	C5-C7-C2	91.8(4)
C2-C10-S1	106.8(3)	O3-C11-N1	119.2(4)	O3-C11-C12	124.7(5)
N1-C11-C12	116.1(4)	C11-C12-C13	113.0(4)	C30-C13-C12	111.7(4)
C30-C13-C14	111.5(4)	C12-C13-C14	112.0(4)	C13-C14-C15	110.0(4)
C13-C14-C31	114.1(4)	C15-C14-C31	108.3(4)	O4-C15-N2	121.7(4)
O4-C15-C14	120.6(4)	N2-C15-C14	117.6(4)	N2-C16-C17	113.6(4)
C22-C17-C18	118.9(5)	C22-C17-C16	121.8(5)	C18-C17-C16	119.2(5)
C19-C18-C17	121.9(7)	C20-C19-C18	119.7(7)	C19-C20-C21	121.7(7)
C20-C21-C22	119.8(7)	C17-C22-C21	118.1(6)	N2-C23-C24	113.9(4)
C25-C24-C29	118.2(5)	C25-C24-C23	118.9(4)	C29-C24-C23	122.9(4)
C26-C25-C24	120.8(5)	C25-C26-C27	120.4(5)	C28-C27-C26	120.0(5)
C27-C28-C29	119.4(5)	C24-C29-C28	121.2(5)	C11-N1-C1	118.7(4)
C11-N1-S1	122.4(3)	C1-N1-S1	112.1(3)	C15-N2-C23	124.3(4)
C15-N2-C16	117.9(4)	C23-N2-C16	116.8(4)		

References

1. SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. (1994). J. Appl. Cryst., 27, 435.

2. SHELXL-93: Program for the Refinement of Crystal Structures, Sheldrick, G.M. (1993), University of Göttingen, Germany.

3. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

$wR_2 = \{ \sum w (F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2 \}^{1/2}$

$GOF = \{ \sum w (F_o^2 - F_c^2)^2 / (n - p) \}^{1/2}$
where n = the number of reflections and p = the number of parameters refined.

4. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.