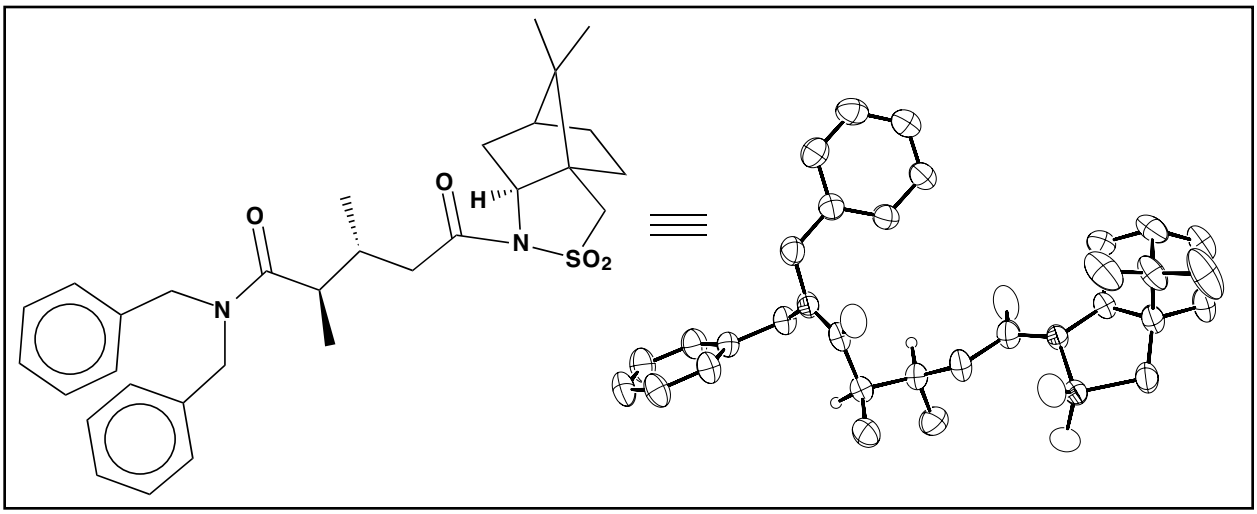
X-ray Structure Determination of Compound 774



Compound 774, $C_{31}H_{40}N_2SO_4$, crystallizes in the triclinic space group P1 with a=9.2116(8)Å, b=9.2973(7)Å, c=9.1208(7)Å, α=94.165(5)°, β=103.569(5)°, γ=103.391(5)°, V=732.00(10)Å^3, Z=1 and d_{calc}=1.218 g/cm^3. X-ray intensity data were collected on an Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 295°K. Indexing was performed from a series of 1° oscillation images with exposures of 100 seconds per frame. A hemisphere of data was collected using 8° oscillation angles with exposures of 500 seconds per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX[1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution on a Silicon Graphics Indigo R4000 computer. A total of 6067 reflections were measured over the ranges $5.64 \le 2\theta \le 50.68$ °, $-11 \le h \le 11$, $-11 \le k \le 11$, $-10 \le l \le 10$ yielding 4155 unique reflections (R_{int} = 0.0259). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[3]). Refinement was by full-matrix least squares based on F^2 using SHELXL-93[4]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by F^2 = 0). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ 0.0621P^2 + 0.1565P]$ 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=0.0470 and

wR_2=0.1194 for 3848 reflections for which F > 4σ(F) and R_1=0.0509, wR_2=0.1249 and GOF = 1.063 for all 4155 unique, non-zero reflections and 347 variables[5]. The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.141 and -0.230 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[6] representation of the molecule with 30% probability thermal ellipsoids displayed.

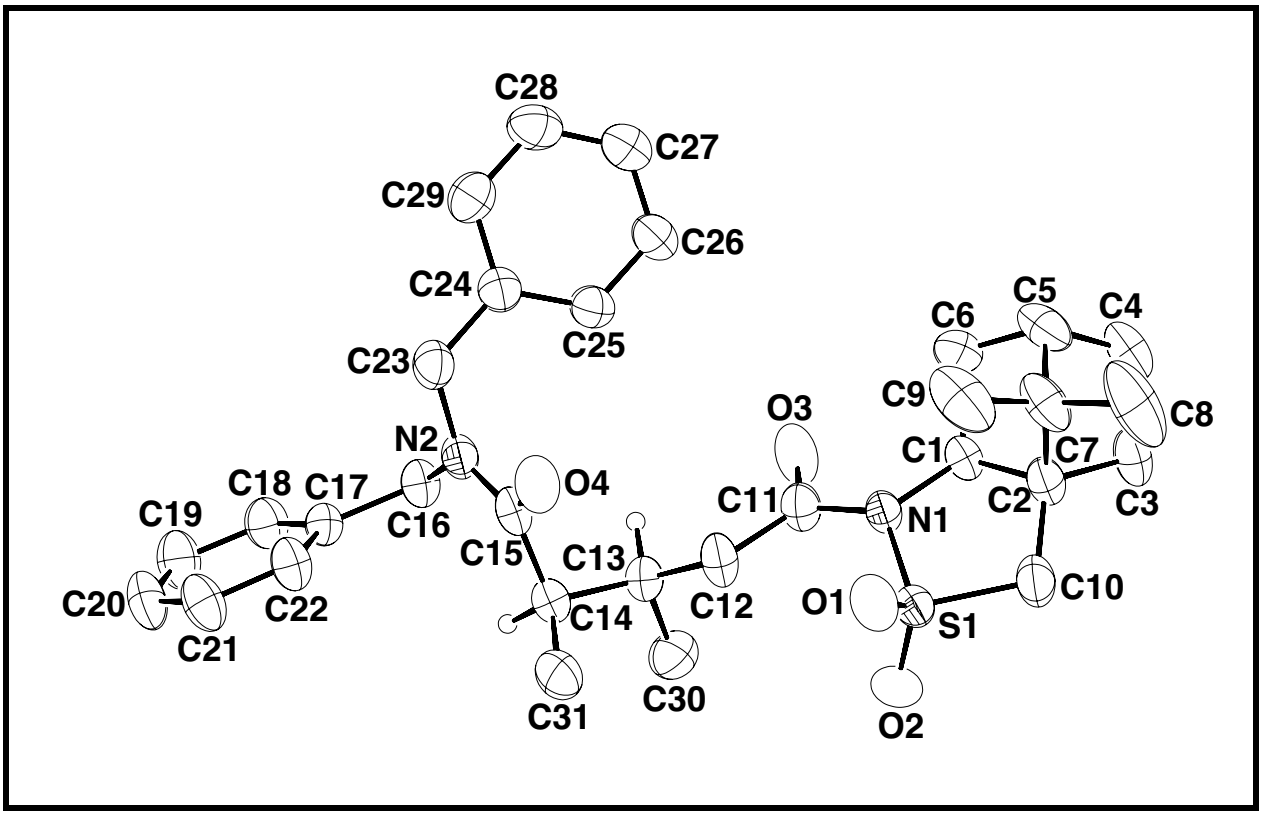


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 774

Formula:	**$C_{31}H_{40}N_2SO$**
Formula weight:	**536.71**
Crystal class:	**triclinic**

Space group:	**P1 (#1)**
Z	**1**
Cell constants:	
a	**9.2116(8)Å**
b	**9.2973(7)Å**
c	**9.1208(7)Å**
α	**94.165(5)°**
β	**103.569(5)°**
γ	**103.391(5)°**
V	**732.00(10)Å^3**
μ	**1.48 cm^{-1}**
crystal size, mm	**0.36 x 0.32 x 0.20**
D_{calc}	**1.218 g/cm^3**
F(000)	**288**
Radiation:	**Mo-K_α (λ=0.71069Å)**
2θ range	**5.64 – 50.68 °**
hkl collected:	**-11≤ h ≤11; -11≤k ≤11; -10≤l ≤10**
No. reflections measured:	**6067**
No. unique reflections:	**4155 (R_{int}=0.0259)**
No. observed reflections	**3848 (F>4σ)**
No. reflections used in refinement	**4155**
No. parameters	**347**
R indices (F>4σ)	**R_1=0.0470**
	wR_2=0.1194
R indices (all data)	**R_1=0.0509**
	wR_2=0.1249
GOF:	**1.063**
Final Difference Peaks, e/Å^3	**+0.141, -0.230**

Table 2. Refined Positional Parameters for Compound 774

Atom	x	y	z	U_{eq}, Å^2
S1	0.80159(9)	0.60981(9)	0.24240(9)	0.0714(3)
C1	0.7653(4)	0.3289(3)	0.2855(4)	0.0610(7)
H1	0.8498(4)	0.3212(3)	0.3702(4)	0.081
C2	0.8221(4)	0.3423(4)	0.1417(4)	0.0716(9)
C3	0.9277(5)	0.2362(5)	0.1469(6)	0.0917(12)
H3a	1.0023(5)	0.2520(5)	0.2451(6)	0.122
H3b	0.9821(5)	0.2480(5)	0.0677(6)	0.122
C4	0.8119(6)	0.0821(5)	0.1195(6)	0.0982(13)
H4a	0.8179(6)	0.0214(5)	0.0311(6)	0.131
H4b	0.8304(6)	0.0296(5)	0.2074(6)	0.131
C5	0.6574(6)	0.1192(5)	0.0933(6)	0.0989(14)
H5	0.5700(6)	0.0370(5)	0.0369(6)	0.132
C6	0.6402(5)	0.1805(4)	0.2453(6)	0.0865(11)
H6a	0.6592(5)	0.1143(4)	0.3210(6)	0.115
H6b	0.5381(5)	0.1963(4)	0.2360(6)	0.115
C7	0.6798(6)	0.2586(5)	0.0129(5)	0.0968(14)
C8	0.7217(11)	0.2360(9)	-0.1420(5)	0.170(3)
H8a	0.755(8)	0.3314(10)	-0.174(4)	0.255
H8b	0.633(3)	0.176(7)	-0.217(2)	0.255
H8c	0.803(6)	0.186(8)	-0.130(2)	0.255
C9	0.5407(7)	0.3282(7)	-0.0188(7)	0.137(2)
H9a	0.452(2)	0.257(2)	-0.085(5)	0.206
H9b	0.565(2)	0.416(4)	-0.067(6)	0.206
H9c	0.518(4)	0.354(6)	0.0754(10)	0.206
C10	0.8959(6)	0.5051(4)	0.1419(6)	0.0978(14)
H10a	0.8834(6)	0.5283(4)	0.0384(6)	0.130
H10b	1.0054(6)	0.5287(4)	0.1917(6)	0.130
C11	0.6683(4)	0.4842(4)	0.4508(4)	0.0710(9)
C12	0.6151(5)	0.6215(4)	0.4793(4)	0.0782(10)
H12a	0.5107(5)	0.6071(4)	0.4165(4)	0.104
H12b	0.6809(5)	0.7047(4)	0.4482(4)	0.104
C13	0.6172(4)	0.6614(4)	0.6447(4)	0.0659(8)
H13	0.5752(4)	0.5687(4)	0.6822(4)	0.088
C14	0.5097(4)	0.7655(4)	0.6556(4)	0.0700(9)
H14	0.5282(4)	0.8038(4)	0.7632(4)	0.093
C15	0.3461(4)	0.6719(4)	0.6010(4)	0.0676(8)
C16	0.3370(4)	0.6247(4)	0.8652(4)	0.0681(8)
H16a	0.4489(4)	0.6555(4)	0.8901(4)	0.091
H16b	0.3076(4)	0.5311(4)	0.9044(4)	0.091
C17	0.2793(3)	0.7412(3)	0.9433(3)	0.0579(7)
C18	0.2818(4)	0.7393(5)	1.0964(4)	0.0764(9)
H18	0.3182(4)	0.6671(5)	1.1485(4)	0.102
C19	0.2308(6)	0.8430(6)	1.1707(5)	0.0932(12)
H19	0.2303(6)	0.8391(6)	1.2722(5)	0.124
C20	0.1807(5)	0.9522(6)	1.0973(5)	0.0931(13)
H20	0.1484(5)	1.0237(6)	1.1490(5)	0.124
C21	0.1784(5)	0.9554(5)	0.9463(5)	0.0899(12)
H21	0.1445(5)	1.0296(5)	0.8957(5)	0.120
C22	0.2260(4)	0.8494(4)	0.8693(4)	0.0760(9)
H22	0.2220(4)	0.8513(4)	0.7666(4)	0.101
C23	0.1253(4)	0.4960(4)	0.6416(5)	0.0779(10)
H23a	0.0706(4)	0.5279(4)	0.5505(5)	0.104

H23b	0.0666(4)	0.4989(4)	0.7167(5)	0.104
C24	0.1325(4)	0.3385(4)	0.6045(4)	0.0687(9)
C25	0.2542(4)	0.3062(4)	0.5588(5)	0.0797(10)
H25	0.3380(4)	0.3841(4)	0.5581(5)	0.106
C26	0.2566(5)	0.1621(4)	0.5139(5)	0.0851(11)
H26	0.3414(5)	0.1447(4)	0.4838(5)	0.113
C27	0.1365(5)	0.0457(5)	0.5132(5)	0.0860(11)
H27	0.1383(5)	-0.0515(5)	0.4833(5)	0.114
C28	0.0128(6)	0.0736(5)	0.5572(5)	0.0933(13)
H28	-0.0707(6)	-0.0053(5)	0.5563(5)	0.124
C29	0.0102(5)	0.2181(5)	0.6031(5)	0.0837(11)
H29	-0.0747(5)	0.2347(5)	0.6336(5)	0.111
C30	0.7813(5)	0.7278(5)	0.7436(6)	0.0926(13)
H30a	0.7787(6)	0.749(4)	0.8474(9)	0.139
H30b	0.826(2)	0.818(2)	0.709(3)	0.139
H30c	0.8423(12)	0.658(2)	0.737(3)	0.139
C31	0.5368(6)	0.8994(5)	0.5677(6)	0.0981(13)
H31a	0.6443(10)	0.952(3)	0.598(3)	0.147
H31b	0.476(3)	0.965(2)	0.590(3)	0.147
H31c	0.507(4)	0.8650(6)	0.4605(7)	0.147
N1	0.7171(3)	0.4653(3)	0.3192(3)	0.0592(6)
N2	0.2768(3)	0.6006(3)	0.6999(3)	0.0650(7)
O1	0.6860(4)	0.6646(3)	0.1444(3)	0.0939(8)
O2	0.9130(3)	0.7141(3)	0.3599(4)	0.1044(10)
O3	0.6600(4)	0.3835(3)	0.5301(4)	0.1025(10)
O4	0.2794(3)	0.6517(3)	0.4634(3)	0.0889(8)

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

Table 3. Refined Thermal Parameters (U's) for Compound 774

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0820(5)	0.0578(4)	0.0832(6)	0.0071(4)	0.0372(5)	0.0195(4)
C1	0.072(2)	0.059(2)	0.057(2)	0.0044(14)	0.0163(14)	0.029(2)
C2	0.090(2)	0.069(2)	0.067(2)	0.007(2)	0.031(2)	0.033(2)
C3	0.110(3)	0.092(3)	0.098(3)	0.011(2)	0.041(2)	0.061(3)
C4	0.136(4)	0.083(3)	0.084(3)	-0.005(2)	0.020(3)	0.057(3)
C5	0.110(3)	0.067(2)	0.103(3)	-0.018(2)	0.000(2)	0.026(2)
C6	0.092(2)	0.060(2)	0.108(3)	0.010(2)	0.031(2)	0.016(2)
C7	0.133(4)	0.093(3)	0.063(2)	-0.011(2)	0.001(2)	0.057(3)
C8	0.310(10)	0.176(6)	0.058(3)	-0.001(3)	0.040(4)	0.141(7)
C9	0.139(4)	0.131(4)	0.112(4)	-0.032(4)	-0.046(3)	0.070(4)
C10	0.126(3)	0.077(2)	0.123(4)	0.018(2)	0.080(3)	0.038(2)
C11	0.086(2)	0.073(2)	0.069(2)	0.019(2)	0.032(2)	0.034(2)
C12	0.099(2)	0.086(2)	0.072(2)	0.015(2)	0.038(2)	0.050(2)
C13	0.069(2)	0.071(2)	0.063(2)	0.006(2)	0.025(2)	0.023(2)
C14	0.076(2)	0.066(2)	0.072(2)	-0.001(2)	0.026(2)	0.023(2)
C15	0.081(2)	0.068(2)	0.065(2)	0.004(2)	0.024(2)	0.039(2)
C16	0.073(2)	0.075(2)	0.064(2)	0.009(2)	0.020(2)	0.033(2)
C17	0.056(2)	0.064(2)	0.055(2)	0.0068(14)	0.0147(13)	0.0210(14)
C18	0.090(2)	0.086(2)	0.060(2)	0.014(2)	0.024(2)	0.033(2)
C19	0.113(3)	0.116(3)	0.062(2)	0.003(2)	0.032(2)	0.044(3)
C20	0.102(3)	0.111(3)	0.074(3)	-0.014(2)	0.022(2)	0.051(3)
C21	0.106(3)	0.091(3)	0.085(3)	0.007(2)	0.016(2)	0.060(2)
C22	0.092(2)	0.085(2)	0.064(2)	0.012(2)	0.022(2)	0.046(2)
C23	0.066(2)	0.089(2)	0.081(2)	0.000(2)	0.018(2)	0.028(2)
C24	0.065(2)	0.075(2)	0.063(2)	0.004(2)	0.009(2)	0.020(2)
C25	0.067(2)	0.068(2)	0.098(3)	-0.005(2)	0.017(2)	0.013(2)
C26	0.093(3)	0.067(2)	0.095(3)	-0.002(2)	0.025(2)	0.024(2)
C27	0.103(3)	0.070(2)	0.082(3)	0.007(2)	0.019(2)	0.022(2)
C28	0.112(3)	0.073(2)	0.090(3)	0.019(2)	0.027(3)	0.010(2)
C29	0.082(2)	0.100(3)	0.074(2)	0.023(2)	0.025(2)	0.024(2)
C30	0.074(2)	0.094(3)	0.108(3)	0.012(3)	0.023(2)	0.019(2)
C31	0.113(3)	0.068(2)	0.130(4)	0.018(2)	0.049(3)	0.037(2)
N1	0.068(2)	0.0572(14)	0.0596(14)	0.0062(12)	0.0238(12)	0.0238(12)
N2	0.064(2)	0.069(2)	0.066(2)	-0.0023(14)	0.0187(13)	0.0247(13)
O1	0.120(2)	0.090(2)	0.091(2)	0.026(2)	0.033(2)	0.054(2)
O2	0.090(2)	0.069(2)	0.140(3)	-0.017(2)	0.031(2)	0.001(2)
O3	0.161(3)	0.095(2)	0.094(2)	0.035(2)	0.073(2)	0.068(2)
O4	0.096(2)	0.113(2)	0.065(2)	0.006(2)	0.0181(13)	0.045(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 774, Å

S1-O2	1.419(3)	S1-O1	1.436(3)	S1-N1	1.676(3)
S1-C10	1.783(4)	C1-N1	1.472(4)	C1-C2	1.526(5)
C1-C6	1.536(5)	C2-C10	1.508(5)	C2-C3	1.534(5)
C2-C7	1.542(6)	C3-C4	1.539(7)	C4-C5	1.511(6)
C5-C6	1.517(7)	C5-C7	1.532(7)	C7-C9	1.543(7)
C7-C8	1.561(7)	C11-O3	1.223(5)	C11-N1	1.390(4)
C11-C12	1.496(5)	C12-C13	1.521(5)	C13-C30	1.526(5)
C13-C14	1.551(5)	C14-C15	1.501(5)	C14-C31	1.533(6)
C15-O4	1.240(4)	C15-N2	1.350(5)	C16-N2	1.460(5)
C16-C17	1.514(4)	C17-C22	1.372(5)	C17-C18	1.393(5)
C18-C19	1.369(6)	C19-C20	1.364(6)	C20-C21	1.376(6)
C21-C22	1.379(5)	C23-N2	1.457(4)	C23-C24	1.499(5)
C24-C25	1.370(5)	C24-C29	1.389(5)	C25-C26	1.379(5)
C26-C27	1.356(6)	C27-C28	1.365(6)	C28-C29	1.385(6)

Table 5. Bond Angles in Compound 774, °

O2-S1-O1	117.2(2)	O2-S1-N1	109.0(2)	O1-S1-N1	110.0(2)
O2-S1-C10	109.8(2)	O1-S1-C10	113.5(2)	N1-S1-C10	95.1(2)
N1-C1-C2	107.5(3)	N1-C1-C6	117.5(3)	C2-C1-C6	103.1(3)
C10-C2-C1	107.5(3)	C10-C2-C3	116.5(3)	C1-C2-C3	105.2(3)
C10-C2-C7	120.3(4)	C1-C2-C7	103.9(3)	C3-C2-C7	101.9(3)
C2-C3-C4	102.2(3)	C5-C4-C3	103.3(3)	C4-C5-C6	108.3(4)
C4-C5-C7	103.7(4)	C6-C5-C7	102.1(3)	C5-C6-C1	102.3(3)
C5-C7-C2	92.1(3)	C5-C7-C9	114.9(5)	C2-C7-C9	116.9(3)
C5-C7-C8	114.6(4)	C2-C7-C8	111.9(5)	C9-C7-C8	106.4(5)
C2-C10-S1	107.1(3)	O3-C11-N1	118.6(3)	O3-C11-C12	123.7(3)
N1-C11-C12	117.5(3)	C11-C12-C13	113.8(3)	C12-C13-C30	111.6(3)
C12-C13-C14	110.1(3)	C30-C13-C14	112.3(3)	C15-C14-C31	111.3(3)
C15-C14-C13	107.4(3)	C31-C14-C13	113.9(3)	O4-C15-N2	120.3(3)
O4-C15-C14	119.8(3)	N2-C15-C14	119.8(3)	N2-C16-C17	113.4(3)
C22-C17-C18	118.7(3)	C22-C17-C16	122.4(3)	C18-C17-C16	118.8(3)
C19-C18-C17	120.4(4)	C20-C19-C18	120.6(4)	C19-C20-C21	119.4(4)
C20-C21-C22	120.5(4)	C17-C22-C21	120.3(4)	N2-C23-C24	113.5(3)
C25-C24-C29	116.5(4)	C25-C24-C23	121.8(3)	C29-C24-C23	121.4(3)
C24-C25-C26	122.3(4)	C27-C26-C25	120.6(4)	C26-C27-C28	118.9(4)
C27-C28-C29	120.7(4)	C28-C29-C24	121.0(4)	C11-N1-C1	118.7(3)
C11-N1-S1	122.4(2)	C1-N1-S1	112.8(2)	C15-N2-C23	118.9(3)
C15-N2-C16	125.4(3)	C23-N2-C16	115.6(3)		

References

1. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Molecular Structure Corporation (1995).

2. teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

3. SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A.,

Polidoro, G. (1994). J. Appl. Cryst., 27, 435.

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

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

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

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

where n = the number of reflections and p = the number of parameters refined.

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