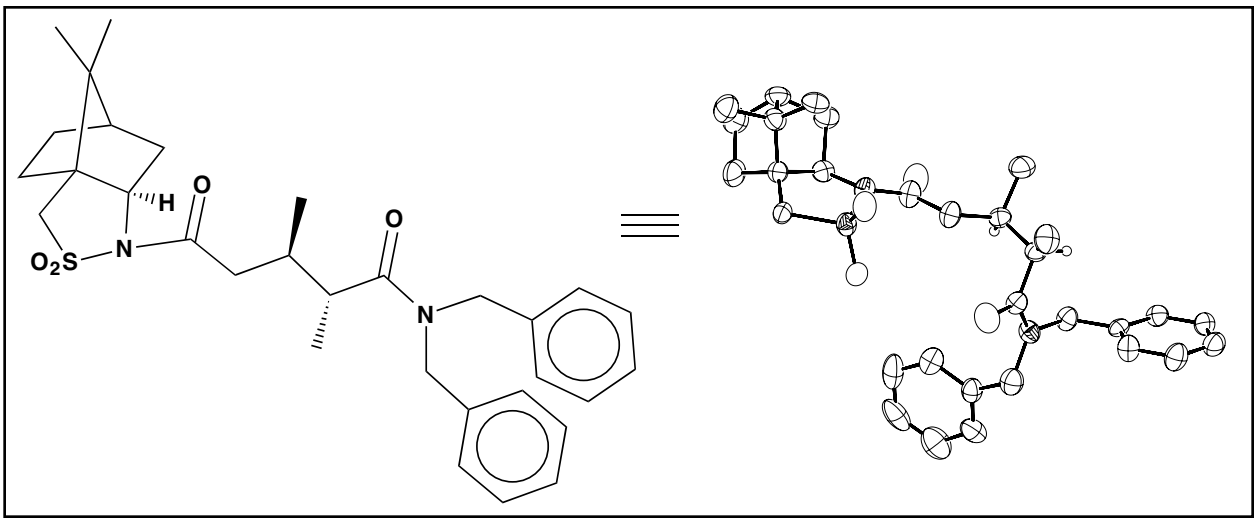
X-ray Structure Determination of Compound 772



Compound 772, $C_{31}H_{40}N_2SO_4$, 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(2)Å, b=40.328(11)Å, c=8.2705(14)Å, V=2903.4(11)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 2268 reflections were measured over the ranges $8.78 \le 2\theta \le 120.32$ °, $0 \le h \le 9$, $0 \le k \le 43$, $0 \le l \le 9$ yielding 2268 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 $w=1/[\sigma^2(F_o^2)+ 0.1501P^2 + 3.7031P]$ 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=0.0627 and wR_2=0.2330 for 1654 reflections for which $F > 4\sigma(F)$ and R_1=0.1110, wR_2=0.7450 and GOF = 1.162 for all 2223 unique, non-zero reflections and 344 variables[3]. 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 +0.278 and -0.297 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.

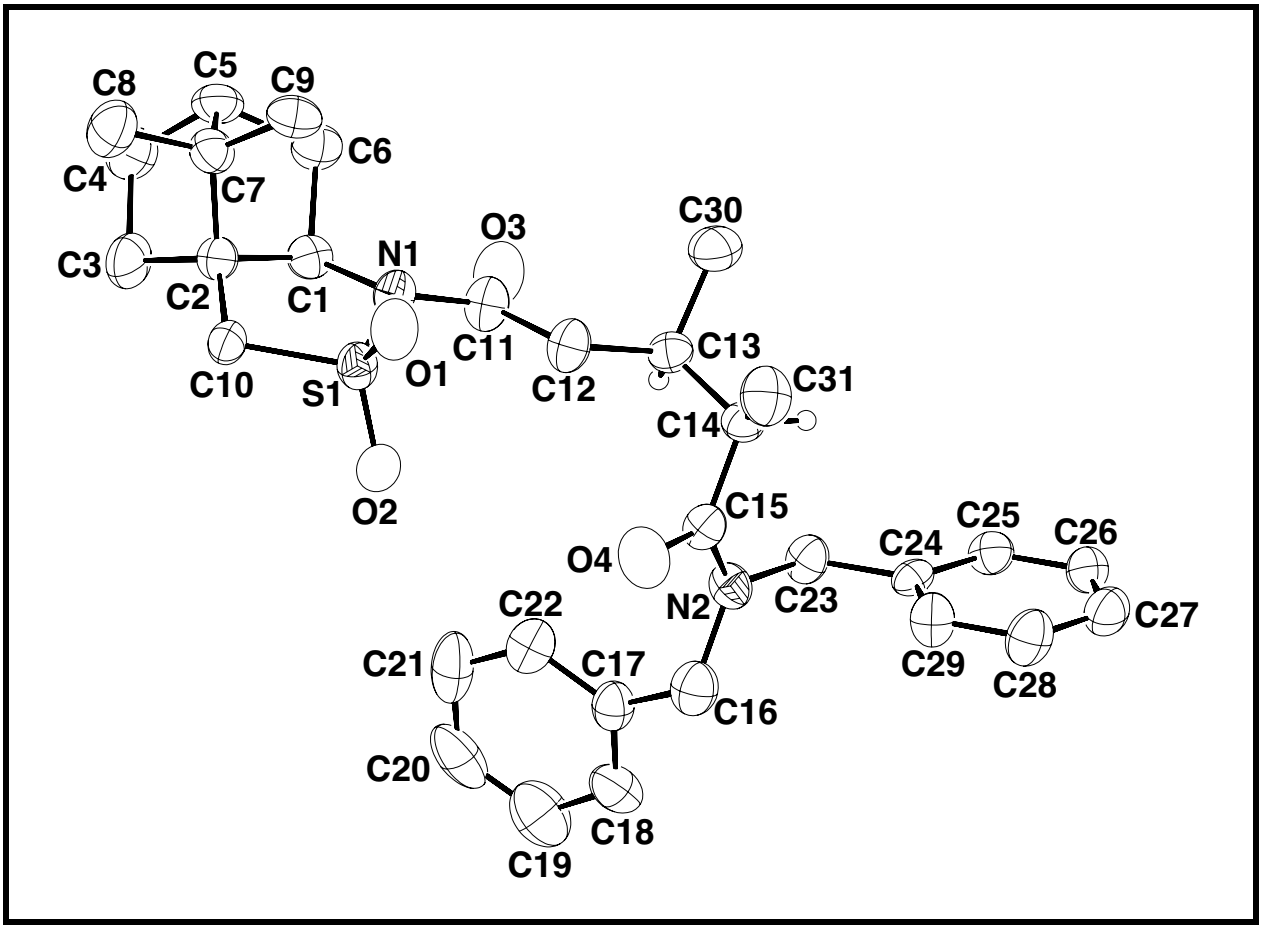


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 772

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(2)Å**
b	**40.328(11)Å**
c	**8.2705(14)Å**
V	**2903.4(11)Å^3**
μ	**12.87 cm^{-1}**
crystal size, mm	**0.38 x 0.30 x 0.03**
D_{calc}	**1.228 g/cm^3**
F(000)	**1152**
Radiation:	**Cu-K_α (λ=1.54178Å)**
2θ range	**8.78 – 120.32 °**
hkl collected:	**0≤ h ≤9; 0≤ k ≤43; 0≤ l ≤9**
No. reflections measured:	**2268**
No. unique reflections:	**2268**
No. observed reflections	**1654 (F>4σ)**
No. reflections used in refinement	**2223**
No. parameters	**344**
R indices (F>4σ)	**R_1=0.0627**
	wR_2=0.2330
R indices (all data)	**R_1=0.1110**
	wR_2=0.7450
GOF:	**1.162**
Final Difference Peaks, e/Å^3	**+0.278, -0.297**

Table 2. Refined Positional Parameters for Compound 772

Atom	x	y	z	U_{eq}, Å^2
S1	0.0852(3)	0.60553(8)	0.5076(3)	0.0615(9)
C1	0.1984(13)	0.5731(3)	0.7555(13)	0.060(3)
H1	0.2451(13)	0.5889(3)	0.8309(13)	0.080
C2	0.0234(14)	0.5728(3)	0.7772(14)	0.062(3)
C3	-0.002(2)	0.5758(4)	0.9608(14)	0.089(4)
H3a	-0.109(2)	0.5793(4)	0.9861(14)	0.118
H3b	0.058(2)	0.5940(4)	1.0057(14)	0.118
C4	0.057(2)	0.5412(3)	1.026(2)	0.091(4)
H4a	0.146(2)	0.5438(3)	1.096(2)	0.121
H4b	-0.024(2)	0.5296(3)	1.085(2)	0.121
C5	0.098(2)	0.5229(3)	0.871(2)	0.075(3)
H5	0.099(2)	0.4987(3)	0.881(2)	0.100
C6	0.247(2)	0.5373(3)	0.800(2)	0.073(3)
H6a	0.280(2)	0.5250(3)	0.705(2)	0.097
H6b	0.329(2)	0.5372(3)	0.879(2)	0.097
C7	-0.0213(14)	0.5363(3)	0.746(2)	0.066(3)
C8	-0.191(2)	0.5285(4)	0.793(2)	0.094(5)
H8a	-0.211(2)	0.5364(4)	0.900(2)	0.142
H8b	-0.207(2)	0.5049(4)	0.789(2)	0.142
H8c	-0.259(2)	0.5392(4)	0.718(2)	0.142
C9	-0.001(2)	0.5227(3)	0.574(2)	0.076(4)
H9a	-0.033(2)	0.4999(3)	0.571(2)	0.114
H9b	0.105(2)	0.5243(3)	0.543(2)	0.114
H9c	-0.063(2)	0.5354(3)	0.500(2)	0.114
C10	-0.0463(13)	0.5991(3)	0.6702(14)	0.066(3)
H10a	-0.0612(13)	0.6195(3)	0.7304(14)	0.088
H10b	-0.1450(13)	0.5918(3)	0.6291(14)	0.088
C11	0.3808(13)	0.5871(3)	0.5377(14)	0.067(3)
C12	0.4066(13)	0.5989(3)	0.3683(14)	0.068(3)
H12a	0.3593(13)	0.6205(3)	0.3563(14)	0.091
H12b	0.3552(13)	0.5839(3)	0.2946(14)	0.091
C13	0.5767(12)	0.6015(3)	0.3190(13)	0.057(3)
H13	0.6319(12)	0.6121(3)	0.4084(13)	0.076
C14	0.6001(12)	0.6230(2)	0.1683(13)	0.055(3)
H14	0.7100(12)	0.6228(2)	0.1427(13)	0.073
C15	0.5564(12)	0.6582(3)	0.2050(13)	0.058(3)
C16	0.622(2)	0.7136(3)	0.295(2)	0.080(4)
H16a	0.526(2)	0.7184(3)	0.241(2)	0.107
H16b	0.700(2)	0.7280(3)	0.250(2)	0.107
C17	0.6048(13)	0.7216(3)	0.4691(14)	0.067(3)
C18	0.670(2)	0.7504(4)	0.531(2)	0.085(4)
H18	0.730(2)	0.7633(4)	0.464(2)	0.113
C19	0.649(2)	0.7607(5)	0.687(2)	0.117(6)
H19	0.693(2)	0.7803(5)	0.724(2)	0.156
C20	0.562(2)	0.7423(5)	0.786(2)	0.110(6)
H20	0.543(2)	0.7497(5)	0.891(2)	0.146
C21	0.499(2)	0.7116(6)	0.735(2)	0.105(6)
H21	0.445(2)	0.6980(6)	0.806(2)	0.140
C22	0.520(2)	0.7025(4)	0.573(2)	0.083(4)
H22	0.476(2)	0.6830(4)	0.536(2)	0.110
C23	0.8282(11)	0.6714(3)	0.2671(14)	0.063(3)
H23a	0.8409(11)	0.6487(3)	0.3022(14)	0.084

H23b	0.8776(11)	0.6856(3)	0.3464(14)	0.084
C24	0.9084(12)	0.6759(2)	0.1053(13)	0.051(2)
C25	1.0611(13)	0.6685(3)	0.093(2)	0.061(3)
H25	1.1137(13)	0.6605(3)	0.183(2)	0.081
C26	1.1378(14)	0.6727(3)	-0.051(2)	0.074(4)
H26	1.2416(14)	0.6674(3)	-0.057(2)	0.099
C27	1.065(2)	0.6844(3)	-0.182(2)	0.075(3)
H27	1.118(2)	0.6871(3)	-0.279(2)	0.100
C28	0.911(2)	0.6923(3)	-0.174(2)	0.082(4)
H28	0.861(2)	0.7007(3)	-0.264(2)	0.109
C29	0.8328(14)	0.6878(3)	-0.0304(13)	0.070(3)
H29	0.7286(14)	0.6927(3)	-0.0250(13)	0.093
C30	0.645(2)	0.5670(3)	0.296(2)	0.084(4)
H30a	0.629(2)	0.5541(3)	0.392(2)	0.126
H30b	0.754(2)	0.5689(3)	0.275(2)	0.126
H30c	0.596(2)	0.5563(3)	0.206(2)	0.126
C31	0.514(2)	0.6118(4)	0.017(2)	0.090(4)
H31a	0.541(2)	0.5893(4)	-0.007(2)	0.134
H31b	0.541(2)	0.6259(4)	-0.072(2)	0.134
H31c	0.405(2)	0.6133(4)	0.036(2)	0.134
N1	0.2292(10)	0.5834(2)	0.5864(10)	0.059(2)
N2	0.6643(9)	0.6793(2)	0.2625(11)	0.060(2)
O1	0.0340(9)	0.5912(2)	0.3625(10)	0.082(3)
O2	0.1296(9)	0.6398(2)	0.5018(10)	0.081(2)
O3	0.4840(10)	0.5789(3)	0.6282(11)	0.090(3)
O4	0.4216(9)	0.6674(2)	0.1946(13)	0.092(3)

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

Table 3. Refined Thermal Parameters (U's) for Compound 772

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.059(2)	0.074(2)	0.052(2)	0.007(2)	0.0030(14)	0.0096(14)
C1	0.069(7)	0.061(8)	0.051(6)	-0.004(6)	-0.006(6)	-0.003(5)
C2	0.073(7)	0.060(8)	0.053(6)	0.001(6)	0.009(6)	0.001(6)
C3	0.112(11)	0.097(11)	0.058(7)	-0.006(7)	0.021(8)	-0.014(9)
C4	0.116(11)	0.090(10)	0.067(9)	0.013(8)	0.006(9)	0.004(8)
C5	0.101(10)	0.051(7)	0.074(8)	0.010(6)	0.002(8)	0.011(7)
C6	0.081(8)	0.068(8)	0.069(8)	0.009(7)	-0.008(7)	0.006(7)
C7	0.074(7)	0.065(8)	0.060(7)	-0.005(6)	0.003(6)	0.004(6)
C8	0.074(8)	0.107(11)	0.103(11)	0.016(9)	0.008(9)	-0.007(8)
C9	0.091(9)	0.056(8)	0.082(8)	-0.012(6)	-0.012(8)	0.000(7)
C10	0.064(7)	0.065(8)	0.070(7)	0.018(6)	0.020(6)	0.009(6)
C11	0.048(6)	0.096(9)	0.058(7)	0.003(7)	-0.010(5)	-0.006(6)
C12	0.050(6)	0.093(9)	0.062(7)	0.005(7)	-0.005(6)	-0.007(6)
C13	0.056(6)	0.053(6)	0.062(6)	-0.002(6)	-0.006(6)	-0.007(5)
C14	0.054(6)	0.050(6)	0.062(6)	-0.007(5)	-0.003(5)	-0.005(5)
C15	0.048(6)	0.067(8)	0.060(7)	0.010(6)	-0.007(5)	-0.002(5)
C16	0.071(8)	0.095(11)	0.074(8)	0.007(8)	0.000(7)	0.001(7)
C17	0.058(7)	0.081(8)	0.061(7)	0.008(7)	0.003(6)	0.007(6)
C18	0.096(10)	0.076(9)	0.083(11)	-0.007(8)	-0.002(8)	0.023(8)
C19	0.119(14)	0.14(2)	0.097(12)	-0.026(12)	-0.012(11)	0.024(13)
C20	0.107(13)	0.13(2)	0.090(11)	-0.014(12)	-0.007(11)	0.072(12)
C21	0.063(8)	0.17(2)	0.084(10)	0.032(11)	0.016(8)	0.021(10)
C22	0.076(8)	0.096(10)	0.076(8)	0.010(8)	0.000(8)	-0.008(7)
C23	0.044(6)	0.077(8)	0.068(7)	0.009(6)	-0.005(5)	-0.002(5)
C24	0.053(6)	0.042(6)	0.057(6)	0.000(5)	-0.002(6)	-0.014(5)
C25	0.052(6)	0.060(7)	0.071(7)	0.003(6)	-0.005(6)	-0.003(5)
C26	0.057(7)	0.069(8)	0.097(10)	0.008(7)	0.019(7)	0.004(6)
C27	0.086(9)	0.077(9)	0.063(7)	0.006(7)	0.010(7)	-0.007(7)
C28	0.079(9)	0.106(11)	0.060(7)	0.019(7)	-0.003(7)	-0.002(8)
C29	0.056(6)	0.096(10)	0.056(7)	0.007(7)	-0.006(6)	0.007(6)
C30	0.085(9)	0.064(8)	0.103(10)	-0.004(8)	0.001(8)	-0.009(7)
C31	0.090(9)	0.113(12)	0.066(7)	-0.009(8)	-0.004(8)	-0.006(8)
N1	0.057(5)	0.073(6)	0.047(5)	0.006(5)	-0.005(4)	-0.002(4)
N2	0.045(5)	0.068(6)	0.067(6)	-0.006(5)	-0.005(4)	0.003(4)
O1	0.067(5)	0.121(8)	0.057(5)	0.003(5)	-0.009(4)	0.001(5)
O2	0.092(6)	0.074(6)	0.077(5)	0.016(5)	0.023(5)	0.006(4)
O3	0.063(5)	0.138(8)	0.068(5)	0.020(6)	-0.016(5)	0.005(5)
O4	0.043(4)	0.121(7)	0.111(7)	0.000(6)	-0.011(5)	0.014(5)

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 772, Å

S1-O1	1.405(9)	S1-O2	1.437(9)	S1-N1	1.671(9)
S1-C10	1.785(11)	C1-N1	1.484(14)	C1-C2	1.53(2)
C1-C6	1.55(2)	C2-C10	1.51(2)	C2-C3	1.54(2)
C2-C7	1.54(2)	C3-C4	1.58(2)	C4-C5	1.53(2)
C5-C6	1.53(2)	C5-C7	1.56(2)	C7-C9	1.54(2)
C7-C8	1.56(2)	C11-O3	1.215(13)	C11-N1	1.387(14)
C11-C12	1.50(2)	C12-C13	1.54(2)	C13-C30	1.53(2)
C13-C14	1.53(2)	C14-C15	1.50(2)	C14-C31	1.53(2)
C15-O4	1.234(13)	C15-N2	1.353(13)	C16-N2	1.46(2)
C16-C17	1.48(2)	C17-C22	1.37(2)	C17-C18	1.39(2)
C18-C19	1.37(2)	C19-C20	1.34(2)	C20-C21	1.42(2)
C21-C22	1.40(2)	C23-N2	1.462(13)	C23-C24	1.52(2)
C24-C25	1.37(2)	C24-C29	1.39(2)	C25-C26	1.37(2)
C26-C27	1.35(2)	C27-C28	1.38(2)	C28-C29	1.38(2)

Table 5. Bond Angles in Compound 772, °

O1-S1-O2	117.0(6)	O1-S1-N1	110.5(5)	O2-S1-N1	108.9(5)
O1-S1-C10	112.4(6)	O2-S1-C10	109.8(5)	N1-S1-C10	96.3(5)
N1-C1-C2	107.0(10)	N1-C1-C6	115.9(10)	C2-C1-C6	103.6(10)
C10-C2-C1	109.0(10)	C10-C2-C3	117.8(11)	C1-C2-C3	104.8(12)
C10-C2-C7	118.0(10)	C1-C2-C7	103.8(10)	C3-C2-C7	101.8(10)
C2-C3-C4	102.9(10)	C5-C4-C3	102.3(9)	C4-C5-C6	109.8(12)
C4-C5-C7	103.4(10)	C6-C5-C7	100.3(9)	C5-C6-C1	102.4(10)
C9-C7-C2	117.8(10)	C9-C7-C8	105.4(12)	C2-C7-C8	113.0(10)
C9-C7-C5	114.3(10)	C2-C7-C5	93.0(9)	C8-C7-C5	113.4(11)
C2-C10-S1	106.6(8)	O3-C11-N1	119.7(10)	O3-C11-C12	123.5(11)
N1-C11-C12	116.6(10)	C11-C12-C13	114.5(10)	C30-C13-C14	111.1(10)
C30-C13-C12	110.4(9)	C14-C13-C12	112.5(9)	C15-C14-C31	108.7(10)
C15-C14-C13	109.7(8)	C31-C14-C13	115.8(9)	O4-C15-N2	119.6(11)
O4-C15-C14	120.9(10)	N2-C15-C14	119.3(9)	N2-C16-C17	114.5(11)
C22-C17-C18	117.0(12)	C22-C17-C16	122.8(13)	C18-C17-C16	120.1(12)
C19-C18-C17	123(2)	C20-C19-C18	119(2)	C19-C20-C21	121(2)
C22-C21-C20	117(2)	C17-C22-C21	122(2)	N2-C23-C24	113.6(9)
C25-C24-C29	118.4(11)	C25-C24-C23	119.2(10)	C29-C24-C23	122.4(9)
C24-C25-C26	120.9(12)	C27-C26-C25	120.9(11)	C26-C27-C28	119.7(12)
C27-C28-C29	119.8(12)	C28-C29-C24	120.3(10)	C11-N1-C1	118.4(9)
C11-N1-S1	123.0(8)	C1-N1-S1	112.4(8)	C15-N2-C16	119.1(9)
C15-N2-C23	123.4(10)	C16-N2-C23	116.5(9)		

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.