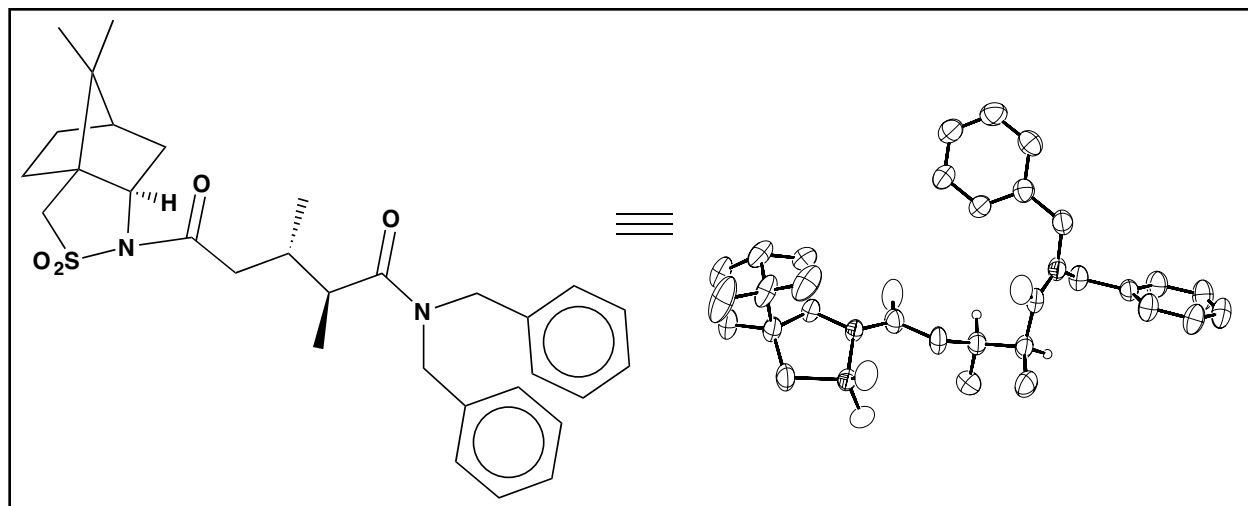


X-ray Structure Determination of Compound 770



Compound 770, $C_{31}H_{40}N_2SO_4$, crystallizes in the triclinic space group P1 with $a=9.244(4)\text{\AA}$, $b=9.313(8)\text{\AA}$, $c=9.120(3)\text{\AA}$, $\alpha=93.93(6)^\circ$, $\beta=103.63(5)^\circ$, $\gamma=103.35(5)^\circ$, $V=736.1(7)\text{\AA}^3$, $Z=1$ and $d_{\text{calc}}=1.211\text{ g/cm}^3$. 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 $\text{Cu-K}\alpha$ radiation ($\lambda=1.54178\text{\AA}$) at a temperature of 295°K using the ω - 2θ scan technique. A total of 2319 reflections were measured over the ranges $9.84 \leq 2\theta \leq 120.02^\circ$, $0 \leq h \leq 10$, $10 \leq k \leq 10$, $-10 \leq l \leq 9$ yielding 2119 unique reflections ($R_{\text{int}} = 0.7800$). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92¹). Refinement was by full-matrix least squares based on F^2 using SHELXL-93². All reflections were used during refinement (F^2 's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w=1/[\sigma^2(F_o^2) + 0.0650P^2 + 0.1718P]$ 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 $R_1=0.0323$ and $wR_2=0.0853$ for 1874 reflections for which $F > 4\sigma(F)$ and $R_1=0.0572$, $wR_2=0.1075$ and $\text{GOF} = 1.033$ for all 2119 unique, non-zero reflections and 347 variables³. 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 +0.300 and -0.246 $e/\text{\AA}^3$.

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 ORTEP⁴ representation of the molecule with 30% probability thermal ellipsoids displayed.

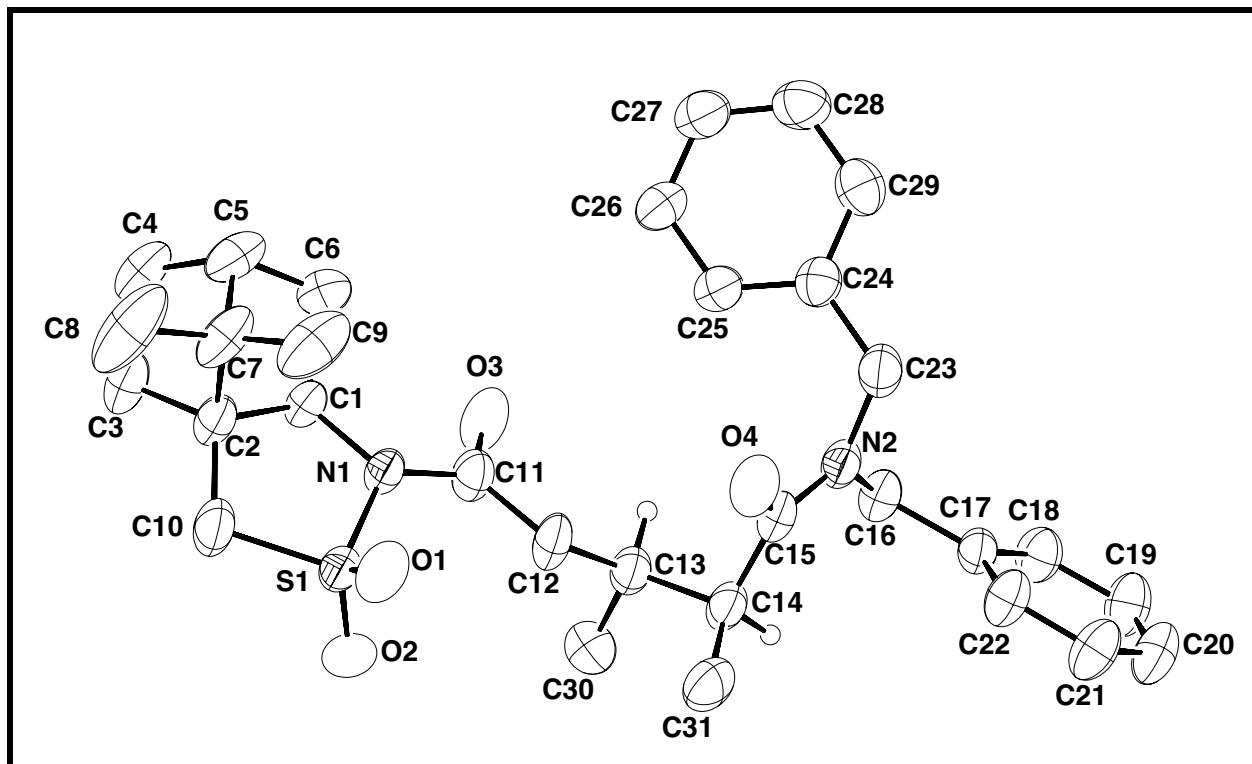


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 770

| | |
|-----------------|-----------------------|
| Formula: | $C_{31}H_{40}N_2SO_4$ |
| Formula weight: | 536.71 |
| Crystal class: | triclinic |
| Space group: | P1 (#1) |
| Z | 1 |
| Cell constants: | |
| a | 9.244(4) Å |
| b | 9.313(8) Å |
| c | 9.120(3) Å |

| | |
|--|---|
| α | 93.93(6)° |
| β | 103.63(5)° |
| γ | 103.35(5)° |
| V | 736.1(7)Å ³ |
| μ | 12.70 cm ⁻¹ |
| crystal size, mm | 0.38 x 0.12 x 0.10 |
| D _{calc} | 1.211 g/cm ³ |
| F(000) | 288 |
| Radiation: | Cu-K α (λ =1.54178Å) |
| 2 θ range | 9.84 – 120.02 ° |
| hkl collected: | 0 ≤ h ≤ 10; -10 ≤ k ≤ 10; -10 ≤ l ≤ 9 |
| No. reflections measured: | 2319 |
| No. unique reflections: | 2119 (R _{int} =0.7800) |
| No. observed reflections | 1874 (F>4 σ) |
| No. reflections used in refinement | 2119 |
| No. parameters | 347 |
| R indices (F>4 σ) | R ₁ =0.0323 wR ₂ =0.0853 |
| R indices (all data) | R ₁ =0.0572 wR ₂ =0.1075 |
| GOF: | 1.033 |
| Final Difference Peaks, e/Å ³ | +0.300, -0.246 |

Table 2. Refined Positional Parameters for Compound 770

| Atom | x | y | z | U _{eq} , Å ² |
|------|-------------|-------------|-------------|----------------------------------|
| S1 | 0.19825(13) | 0.39023(11) | 0.75770(12) | 0.0744(4) |
| O1 | 0.3126(5) | 0.3354(4) | 0.8550(4) | 0.0988(12) |
| O2 | 0.0870(4) | 0.2864(4) | 0.6402(5) | 0.1073(13) |
| O3 | 0.3392(6) | 0.6163(4) | 0.4697(4) | 0.1054(13) |
| O4 | 0.7196(5) | 0.3482(4) | 0.5362(4) | 0.0921(10) |
| N1 | 0.2823(4) | 0.5354(3) | 0.6800(4) | 0.0612(8) |
| N2 | 0.7228(4) | 0.3991(4) | 0.3007(4) | 0.0691(9) |
| C1 | 0.2346(5) | 0.6716(4) | 0.7141(5) | 0.0627(10) |
| H1 | 0.1504(5) | 0.6798(4) | 0.6294(5) | 0.083 |
| C2 | 0.1771(6) | 0.6572(5) | 0.8588(5) | 0.0730(12) |
| C3 | 0.0724(8) | 0.7640(6) | 0.8541(7) | 0.097(2) |
| H3a | -0.0020(8) | 0.7486(6) | 0.7560(7) | 0.129 |
| H3b | 0.0183(8) | 0.7517(6) | 0.9333(7) | 0.129 |
| C4 | 0.1862(8) | 0.9176(6) | 0.8813(6) | 0.100(2) |
| H4a | 0.1671(8) | 0.9703(6) | 0.7935(6) | 0.133 |
| H4b | 0.1800(8) | 0.9775(6) | 0.9698(6) | 0.133 |
| C5 | 0.3416(8) | 0.8809(6) | 0.9071(7) | 0.101(2) |

| | | | | |
|------|------------|------------|------------|------------|
| H5 | 0.4286(8) | 0.9630(6) | 0.9630(7) | 0.135 |
| C6 | 0.3582(7) | 0.8196(5) | 0.7547(7) | 0.093(2) |
| H6a | 0.4601(7) | 0.8040(5) | 0.7640(7) | 0.123 |
| H6b | 0.3391(7) | 0.8859(5) | 0.6790(7) | 0.123 |
| C7 | 0.3199(8) | 0.7425(6) | 0.9876(6) | 0.101(2) |
| C8 | 0.278(2) | 0.7650(11) | 1.1410(7) | 0.180(5) |
| H8a | 0.367(3) | 0.822(10) | 1.217(3) | 0.270 |
| H8b | 0.241(11) | 0.6698(11) | 1.172(6) | 0.270 |
| H8c | 0.199(9) | 0.818(10) | 1.129(3) | 0.270 |
| C9 | 0.4589(9) | 0.6728(8) | 1.0176(8) | 0.137(3) |
| H9a | 0.433(3) | 0.583(4) | 1.062(7) | 0.205 |
| H9b | 0.546(2) | 0.742(3) | 1.086(6) | 0.205 |
| H9c | 0.483(5) | 0.651(7) | 0.9234(14) | 0.205 |
| C10 | 0.1042(8) | 0.4950(6) | 0.8593(8) | 0.104(2) |
| H10a | -0.0051(8) | 0.4714(6) | 0.8100(8) | 0.139 |
| H10b | 0.1174(8) | 0.4716(6) | 0.9629(8) | 0.139 |
| C11 | 0.3315(6) | 0.5168(5) | 0.5492(5) | 0.0738(12) |
| C12 | 0.3850(7) | 0.3797(6) | 0.5217(5) | 0.0808(13) |
| H12a | 0.3202(7) | 0.2969(6) | 0.5540(5) | 0.108 |
| H12b | 0.4893(7) | 0.3946(6) | 0.5842(5) | 0.108 |
| C13 | 0.3823(5) | 0.3385(5) | 0.3554(5) | 0.0701(12) |
| H13 | 0.4233(5) | 0.4305(5) | 0.3164(5) | 0.093 |
| C14 | 0.4900(6) | 0.2342(5) | 0.3451(5) | 0.0720(12) |
| H14 | 0.4720(6) | 0.1964(5) | 0.2377(5) | 0.096 |
| C15 | 0.6531(6) | 0.3271(5) | 0.4001(5) | 0.0707(12) |
| C16 | 0.6628(6) | 0.3753(5) | 0.1352(5) | 0.0720(12) |
| H16a | 0.6918(6) | 0.4689(5) | 0.0959(5) | 0.096 |
| H16b | 0.5513(6) | 0.3445(5) | 0.1104(5) | 0.096 |
| C17 | 0.7204(5) | 0.2594(5) | 0.0567(5) | 0.0624(10) |
| C18 | 0.7186(6) | 0.2615(6) | -0.0950(5) | 0.0793(13) |
| H18 | 0.6826(6) | 0.3339(6) | -0.1472(5) | 0.105 |

| | | | | |
|------|-----------|------------|------------|------------|
| C19 | 0.7696(8) | 0.1575(7) | -0.1698(6) | 0.098(2) |
| H19 | 0.7708(8) | 0.1619(7) | -0.2711(6) | 0.130 |
| C20 | 0.8185(7) | 0.0478(7) | -0.0959(6) | 0.097(2) |
| H20 | 0.8498(7) | -0.0244(7) | -0.1473(6) | 0.129 |
| C21 | 0.8209(7) | 0.0450(6) | 0.0543(6) | 0.093(2) |
| H21 | 0.8547(7) | -0.0292(6) | 0.1052(6) | 0.124 |
| C22 | 0.7739(6) | 0.1506(5) | 0.1311(5) | 0.0795(13) |
| H22 | 0.7782(6) | 0.1485(5) | 0.2339(5) | 0.106 |
| C23 | 0.8745(6) | 0.5027(6) | 0.3591(6) | 0.0834(14) |
| H23a | 0.9330(6) | 0.5001(6) | 0.2840(6) | 0.111 |
| H23b | 0.9289(6) | 0.4703(6) | 0.4502(6) | 0.111 |
| C24 | 0.8668(6) | 0.6614(5) | 0.3965(5) | 0.0716(12) |
| C25 | 0.7452(6) | 0.6940(5) | 0.4413(6) | 0.0854(14) |
| H25 | 0.6612(6) | 0.6166(5) | 0.4417(6) | 0.114 |
| C26 | 0.7435(7) | 0.8387(5) | 0.4859(6) | 0.088(2) |
| H26 | 0.6589(7) | 0.8568(5) | 0.5151(6) | 0.117 |
| C27 | 0.8644(7) | 0.9543(6) | 0.4872(6) | 0.091(2) |
| H27 | 0.8635(7) | 1.0513(6) | 0.5182(6) | 0.121 |
| C28 | 0.9867(8) | 0.9265(6) | 0.4427(6) | 0.097(2) |
| H28 | 1.0698(8) | 1.0052(6) | 0.4433(6) | 0.129 |
| C29 | 0.9892(7) | 0.7821(7) | 0.3963(6) | 0.089(2) |
| H29 | 1.0732(7) | 0.7654(7) | 0.3647(6) | 0.119 |
| C30 | 0.2175(6) | 0.2709(6) | 0.2576(7) | 0.093(2) |
| H30a | 0.156(2) | 0.341(2) | 0.264(4) | 0.139 |
| H30b | 0.174(2) | 0.181(3) | 0.294(3) | 0.139 |
| H30c | 0.2191(8) | 0.249(5) | 0.1537(11) | 0.139 |

| | | | | |
|------|------------|-----------|-----------|----------|
| C31 | 0.4623(8) | 0.1012(6) | 0.4322(8) | 0.103(2) |
| H31a | 0.3553(14) | 0.049(3) | 0.401(4) | 0.155 |
| H31b | 0.491(6) | 0.1351(8) | 0.5394(8) | 0.155 |
| H31c | 0.523(5) | 0.036(3) | 0.411(5) | 0.155 |

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

Table 3. Refined Thermal Parameters (U's) for Compound 770

| Atom | U ₁₁ | U ₂₂ | U ₃₃ | U ₂₃ | U ₁₃ | U ₁₂ |
|------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| S1 | 0.0925(8) | 0.0586(6) | 0.0849(7) | 0.0067(5) | 0.0459(7) | 0.0218(6) |
| O1 | 0.143(3) | 0.089(2) | 0.092(2) | 0.032(2) | 0.045(2) | 0.064(2) |
| O2 | 0.099(3) | 0.067(2) | 0.147(3) | -0.017(2) | 0.040(3) | 0.003(2) |
| O3 | 0.172(4) | 0.096(2) | 0.094(2) | 0.037(2) | 0.079(3) | 0.071(3) |
| O4 | 0.105(3) | 0.116(3) | 0.063(2) | 0.005(2) | 0.020(2) | 0.047(2) |
| N1 | 0.080(2) | 0.057(2) | 0.057(2) | 0.0075(14) | 0.027(2) | 0.028(2) |
| N2 | 0.073(2) | 0.073(2) | 0.067(2) | -0.004(2) | 0.024(2) | 0.027(2) |
| C1 | 0.080(3) | 0.058(2) | 0.058(2) | 0.006(2) | 0.021(2) | 0.030(2) |
| C2 | 0.101(3) | 0.067(3) | 0.066(3) | 0.009(2) | 0.036(2) | 0.036(2) |
| C3 | 0.124(5) | 0.097(4) | 0.097(4) | 0.009(3) | 0.045(3) | 0.064(4) |
| C4 | 0.148(6) | 0.078(3) | 0.085(3) | -0.002(3) | 0.024(3) | 0.060(4) |
| C5 | 0.120(5) | 0.071(3) | 0.099(4) | -0.020(3) | 0.003(3) | 0.031(3) |
| C6 | 0.101(4) | 0.062(3) | 0.115(4) | 0.006(3) | 0.032(3) | 0.018(3) |
| C7 | 0.145(5) | 0.096(4) | 0.065(3) | -0.011(3) | 0.006(3) | 0.066(4) |
| C8 | 0.338(14) | 0.176(7) | 0.059(3) | -0.003(4) | 0.041(5) | 0.149(9) |
| C9 | 0.154(7) | 0.126(5) | 0.107(5) | -0.029(4) | -0.036(5) | 0.073(5) |
| C10 | 0.141(5) | 0.085(3) | 0.125(4) | 0.019(3) | 0.092(4) | 0.045(3) |
| C11 | 0.095(3) | 0.076(3) | 0.067(3) | 0.015(2) | 0.038(2) | 0.033(2) |
| C12 | 0.106(4) | 0.088(3) | 0.073(3) | 0.011(2) | 0.046(3) | 0.049(3) |
| C13 | 0.083(3) | 0.067(3) | 0.071(3) | 0.008(2) | 0.035(2) | 0.027(2) |
| C14 | 0.089(3) | 0.069(3) | 0.068(2) | -0.001(2) | 0.034(2) | 0.028(2) |
| C15 | 0.085(3) | 0.074(3) | 0.066(3) | 0.001(2) | 0.030(3) | 0.036(2) |
| C16 | 0.086(3) | 0.075(3) | 0.068(2) | 0.011(2) | 0.026(2) | 0.038(2) |
| C17 | 0.063(2) | 0.067(2) | 0.061(2) | 0.003(2) | 0.023(2) | 0.020(2) |
| C18 | 0.096(3) | 0.091(3) | 0.059(3) | 0.012(2) | 0.027(2) | 0.034(3) |
| C19 | 0.120(5) | 0.124(4) | 0.064(3) | 0.000(3) | 0.039(3) | 0.045(4) |
| C20 | 0.108(4) | 0.113(4) | 0.076(3) | -0.015(3) | 0.020(3) | 0.054(4) |
| C21 | 0.121(4) | 0.095(3) | 0.077(3) | 0.009(3) | 0.018(3) | 0.063(3) |
| C22 | 0.104(4) | 0.088(3) | 0.061(2) | 0.011(2) | 0.025(2) | 0.049(3) |
| C23 | 0.074(3) | 0.093(3) | 0.087(3) | -0.003(3) | 0.028(3) | 0.027(3) |
| C24 | 0.074(3) | 0.080(3) | 0.060(2) | 0.005(2) | 0.015(2) | 0.022(2) |
| C25 | 0.078(3) | 0.069(3) | 0.105(4) | -0.007(2) | 0.024(3) | 0.014(2) |
| C26 | 0.102(4) | 0.067(3) | 0.096(3) | -0.002(2) | 0.028(3) | 0.025(3) |
| C27 | 0.111(4) | 0.072(3) | 0.086(3) | 0.002(2) | 0.025(3) | 0.018(3) |
| C28 | 0.118(5) | 0.077(3) | 0.094(4) | 0.019(3) | 0.036(3) | 0.010(3) |
| C29 | 0.092(4) | 0.108(4) | 0.075(3) | 0.023(3) | 0.034(3) | 0.025(3) |
| C30 | 0.082(3) | 0.095(4) | 0.103(4) | 0.013(3) | 0.029(3) | 0.019(3) |
| C31 | 0.130(5) | 0.071(3) | 0.129(5) | 0.015(3) | 0.055(4) | 0.043(3) |

The form of the anisotropic displacement parameter is:

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

Table 4. Bond Distances in Compound 770, Å

| | | | | | |
|---------|----------|---------|----------|---------|----------|
| S1-O2 | 1.422(4) | S1-O1 | 1.427(4) | S1-N1 | 1.685(4) |
| S1-C10 | 1.792(5) | O3-C11 | 1.215(6) | O4-C15 | 1.228(5) |
| N1-C11 | 1.387(5) | N1-C1 | 1.473(5) | N2-C15 | 1.359(6) |
| N2-C16 | 1.461(6) | N2-C23 | 1.461(6) | C1-C6 | 1.531(7) |
| C1-C2 | 1.538(6) | C2-C10 | 1.507(8) | C2-C3 | 1.535(7) |
| C2-C7 | 1.552(8) | C3-C4 | 1.532(9) | C4-C5 | 1.522(9) |
| C5-C6 | 1.521(8) | C5-C7 | 1.523(9) | C7-C9 | 1.546(9) |
| C7-C8 | 1.552(9) | C11-C12 | 1.498(7) | C12-C13 | 1.532(6) |
| C13-C30 | 1.534(7) | C13-C14 | 1.556(6) | C14-C15 | 1.502(7) |
| C14-C31 | 1.522(8) | C16-C17 | 1.516(6) | C17-C18 | 1.382(6) |
| C17-C22 | 1.378(7) | C18-C19 | 1.378(7) | C19-C20 | 1.366(8) |
| C20-C21 | 1.367(7) | C21-C22 | 1.376(7) | C23-C24 | 1.516(7) |
| C24-C25 | 1.371(7) | C24-C29 | 1.399(7) | C25-C26 | 1.385(7) |
| C26-C27 | 1.358(8) | C27-C28 | 1.359(9) | C28-C29 | 1.389(8) |

Table 5. Bond Angles in Compound 770, °

| | | | | | |
|-------------|----------|-------------|----------|-------------|----------|
| O2-S1-O1 | 117.2(2) | O2-S1-N1 | 108.9(2) | O1-S1-N1 | 110.2(2) |
| O2-S1-C10 | 109.9(3) | O1-S1-C10 | 113.3(3) | N1-S1-C10 | 95.1(2) |
| C11-N1-C1 | 119.0(3) | C11-N1-S1 | 122.3(3) | C1-N1-S1 | 112.9(3) |
| C15-N2-C16 | 125.3(4) | C15-N2-C23 | 118.9(4) | C16-N2-C23 | 115.7(4) |
| N1-C1-C6 | 118.0(4) | N1-C1-C2 | 107.5(3) | C6-C1-C2 | 103.0(4) |
| C10-C2-C3 | 116.8(5) | C10-C2-C1 | 107.8(4) | C3-C2-C1 | 105.2(4) |
| C10-C2-C7 | 120.7(5) | C3-C2-C7 | 101.1(4) | C1-C2-C7 | 103.5(4) |
| C4-C3-C2 | 103.0(5) | C5-C4-C3 | 103.1(4) | C6-C5-C4 | 108.4(5) |
| C6-C5-C7 | 102.0(4) | C4-C5-C7 | 103.4(6) | C5-C6-C1 | 102.8(5) |
| C5-C7-C9 | 114.6(7) | C5-C7-C8 | 114.4(5) | C9-C7-C8 | 107.4(6) |
| C5-C7-C2 | 92.6(4) | C9-C7-C2 | 116.2(4) | C8-C7-C2 | 111.5(7) |
| C2-C10-S1 | 107.1(4) | O3-C11-N1 | 118.7(4) | O3-C11-C12 | 123.7(4) |
| N1-C11-C12 | 117.4(4) | C11-C12-C13 | 113.9(4) | C12-C13-C30 | 111.4(4) |
| C12-C13-C14 | 110.2(4) | C30-C13-C14 | 112.3(4) | C15-C14-C31 | 111.4(5) |
| C15-C14-C13 | 107.6(3) | C31-C14-C13 | 113.8(4) | O4-C15-N2 | 119.6(5) |
| O4-C15-C14 | 120.4(5) | N2-C15-C14 | 119.7(4) | N2-C16-C17 | 113.6(3) |
| C18-C17-C22 | 118.4(4) | C18-C17-C16 | 119.2(4) | C22-C17-C16 | 122.3(4) |
| C19-C18-C17 | 120.7(5) | C20-C19-C18 | 120.3(5) | C21-C20-C19 | 119.4(5) |
| C20-C21-C22 | 120.8(5) | C21-C22-C17 | 120.4(4) | N2-C23-C24 | 113.0(4) |
| C25-C24-C29 | 116.7(5) | C25-C24-C23 | 122.1(5) | C29-C24-C23 | 121.1(5) |
| C24-C25-C26 | 122.0(5) | C27-C26-C25 | 120.6(5) | C28-C27-C26 | 119.1(5) |
| C27-C28-C29 | 120.9(6) | C28-C29-C24 | 120.8(6) | | |

References

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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.
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