

X-Ray Structure Determination

(Change the highlighted values with the values from your own experiment)

A colorless tablet-like crystal of (XYZ), approximate dimensions .50mm x .45mm x .15mm, was used for X-ray crystallographic analysis. The X-ray intensity data were measured at 100K on a Bruker SMART 1000 CCD-based X-ray diffractometer system equipped with a Mo-target X-ray tube ($\lambda = 0.71073 \text{ \AA}$) operated at 2250 watts power. The detector was placed at a distance of 4.986 cm from the crystal.

A total of 1800 frames were collected with a scan width of 0.3° in ω , with an exposure time of 20 sec./frame. The total data collection time was 18.6 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a monoclinic unit cell yielded a total of 19136 reflections to a maximum 2θ angle of 56.56° , of which 6913 were independent (redundancy 2.79, $R_{\text{int}} = 2.7\%$, $R_{\text{sig}} = 3.2\%$) and 5760 (83%) were greater than 4σ (F). The final cell constants of $a = 11.8029(17) \text{ \AA}$, $b = 11.8080(16) \text{ \AA}$, $c = 20.768(3) \text{ \AA}$, $\beta = 95.773(2)^\circ$, volume = $2879.7(7) \text{ \AA}^3$, are based upon the refinement of the XYZ centroids of 4500 reflections above $20\sigma(I)$. Analysis of the data showed negligible decay during data collection.

The structure was refined using the Bruker SHELXTL (Version 6.12) Software Package, using the space group $P2(1)/n$, with $Z = 4$ for the formula unit xyz. The final anisotropic full-matrix least-squares refinement on F^2 converged at $R1 = 2.74\%$, $wR2 = 7.37\%$ and a goodness-of-fit of 1.044. The largest peak on the final difference map was 0.510 e/ \AA^3 . The calculated density for XYZ is 1.302 g/cm^3 and $F(000)$ is 1184 e.