

Chemistry of the Phenoxathiins and Isostructurally Related
Heterocycles. XXX [1]. The Crystal and Molecular Structure of 2-Aza-
phenoxathiin 2-Oxide: Further Evidence in Support of the Relationship
Between the ^{13}C -NMR Chemical Shift of C-10a Resonance and the
Molecular Dihedral Angle

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The synthesis of 2-azaphenoxathiin 2-oxide (**4**) and the total assignment of the ^{13}C -nmr spectrum is described. Facile assignment was obtained from the assigned spectrum of 2-azaphenoxathiin with additivities obtained from a comparison of 1-azaphenoxathiin and its *N*-oxide. The crystal structure has also been determined from three dimensional X-ray diffraction data. The compound crystallizes in the monoclinic space group $P2_1/n$ with $a = 17.50(2)\text{\AA}$, $b = 7.147(3)\text{\AA}$, $c = 16.044(7)\text{\AA}$, $\beta = 110.60(5)$ and $Z = 8$. Intensity data were collected at $-135 \pm 2^\circ\text{C}$ on an automatic diffractometer using nickel-filtered $\text{CuK}\alpha$ radiation. The structure was solved by direct methods and was refined by least-squares techniques to give a final R-value of 0.045 for all 3831 independent reflections.

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There are two crystallographically independent molecules in one asymmetric unit of the crystal. The two molecules have significantly different folding angles and C-O-C bond angles within the central ring. The dihedral angle between the least squares planes of the two non-

central rings are 160.8° and 151.3° , and the C-O-C bond angles within the central ring are 120.0° and 117.8° , respectively, for the two crystallographically independent molecules.

Evaluation of the observed dihedral angles with respect to the ^{13}C -nmr chemical shift of the sulfur bearing carbon of the pyridyl ring (C-10a), shows that the data reflects further the sensitivity of the chemical shift of this carbon to alterations in electronic properties which are presumably responsible for control of the dihedral angle in these systems. Considering the present data with that previously available shows a linear relationship with a correlation coefficient $r = 0.967$.

Syntheses of 1-azaphenoxathiin [3] and a variety of substituted analogs have been reported [4-7]. Data obtained

Table 1

Bond Angles ($^\circ$). The standard deviations are given in parentheses and refer to the last digit of respective values

| Angle | Molecule A | Molecule B |
|------------------|------------|------------|
| C(9a)—S—C(1a) | 99.1(1) | 98.4(1) |
| C(5a)—O(5)—C(4a) | 120.0(2) | 117.8(1) |
| C(1)—N—C(3) | 119.9(2) | 120.0(2) |
| C(1)—N—O(2) | 119.7(2) | 120.2(2) |
| C(3)—N—O(2) | 120.4(2) | 119.7(2) |
| C(1a)—C(1)—N | 121.1(2) | 120.6(2) |
| S—C(1a)—C(1) | 118.0(1) | 119.8(1) |
| S—C(1a)—C(4a) | 122.7(1) | 120.8(1) |
| C(1)—C(1a)—C(4a) | 119.2(2) | 119.4(2) |
| N—C(3)—C(4) | 121.0(2) | 121.0(2) |
| C(3)—C(4)—C(4a) | 119.6(2) | 119.8(2) |
| O(5)—C(4a)—C(4) | 116.9(2) | 117.8(2) |
| O(5)—C(4a)—C(1a) | 123.9(2) | 123.0(2) |
| C(4)—C(4a)—C(1a) | 119.1(2) | 119.2(2) |
| O(5)—C(5a)—C(6) | 115.1(2) | 116.5(2) |
| O(5)—C(5a)—C(9a) | 123.8(2) | 122.2(2) |
| C(6)—C(5a)—C(9a) | 121.1(2) | 121.2(2) |
| C(5a)—C(6)—C(7) | 119.2(2) | 119.2(2) |
| C(6)—C(7)—C(8) | 120.6(2) | 120.1(2) |
| C(7)—C(8)—C(9) | 119.9(2) | 120.4(2) |
| C(8)—C(9)—C(9a) | 119.9(2) | 120.0(2) |
| S—C(9a)—C(5a) | 122.4(2) | 121.2(1) |
| S—C(9a)—C(9) | 118.4(2) | 119.7(2) |
| C(9)—C(9a)—C(5a) | 119.2(2) | 119.1(2) |

SCHEME 1

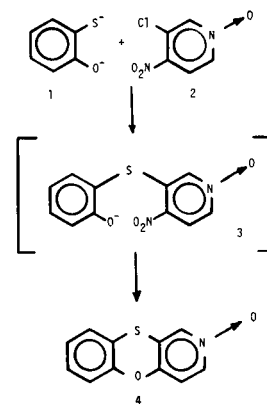


Table 2
Conformational Angles

| Angle | Molecule A | Molecule B |
|------------------------|------------|------------|
| S—C(1a)—C(4a)—O(5) | −3.7 | 1.0 |
| C(1a)—C(4a)—O(5)—C(5a) | −22.4 | −35.9 |
| C(4a)—O(5)—C(5a)—C(9a) | 24.2 | 34.1 |
| O(5)—C(5a)—C(9a)—S | 0.4 | 2.0 |
| C(5a)—C(9a)—S—C(1a) | −20.2 | −28.5 |
| C(9a)—S—C(1a)—C(4a) | 21.9 | 27.1 |

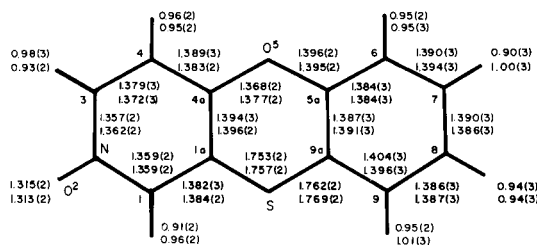


Figure 1. Bond lengths of the two crystallographically independent molecules of 2-azaphenoxathiin 2-oxide. Upper numbers refer to molecule A, the lower numbers refer to molecule B.

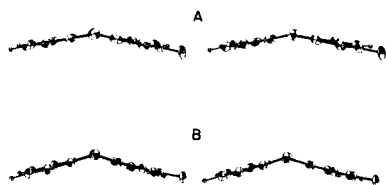


Figure 2. Stereoscopic side view of molecules A and B.

from our studies of the ^{13}C -nmr spectroscopy of these compounds, in conjunction with single crystal X-ray diffraction studies, has resulted in the observation of a linear relationship between the ^{13}C -nmr chemical shift of the alpha carbon and the dihedral angle in this series [8]. As a part of an effort intended to broaden the data base upon which this relationship is founded, we recently reported the synthesis of 2-azaphenoxathiin and the total assignment of its ^{13}C -nmr spectrum [9]. Repeated efforts to obtain a suitable crystal of this compound for X-ray diffraction studies, however, proved to be fruitless. On this basis, we undertook the isolation of the 2-azaphenoxathiin 2-oxide which is the immediate precursor of 2-azaphenoxathiin (Scheme I). Sublimation of crude 2-azaphenoxathiin 2-oxide provided crystals of suitable quality for X-ray diffraction and the results of these studies are reported with the assignment of the ^{13}C -nmr spectrum of the compound.

The synthesis of 2-azaphenoxathiin 2-oxide was based on the condensation of 3-chloro-4-nitropyridine-*N*-oxide

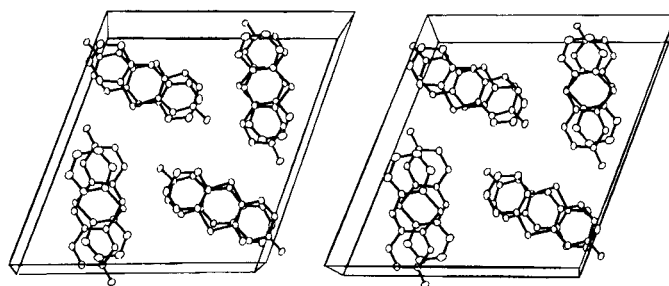


Figure 3. Stereoscopic view of the molecular packing of 2-azaphenoxathiin 2-oxide.

with the disodium salt of *o*-mercaptophenol as previously described [8], with the exception that the dianion was generated according to the procedure of Elliott [7] using sodium hydride. The isolated crude brownish material was purified for crystallography by linear thermal gradient sublimation to provide colorless monoclinic crystals.

The compound crystallizes with two molecules in the asymmetric unit and there are, therefore, two independent determinations of both the geometry and conformation of the compound. The numbering of the atoms and the bond lengths are shown in Figure 1. For both molecules, the bond angles are listed in Table 1, conformational angles for both central rings are shown in Table 2, while the equations of the least-squares planes through the aromatic rings in both molecules are listed in Table 3.

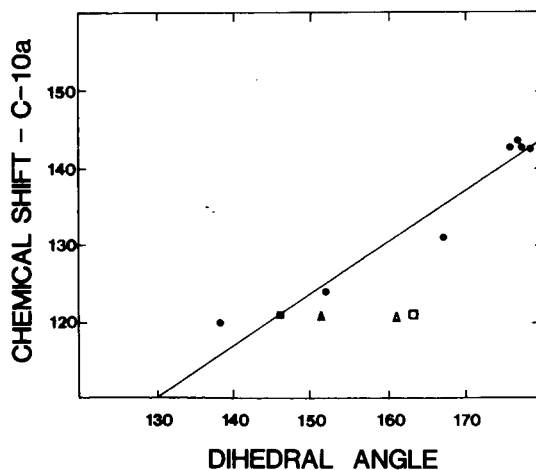


Figure 4. Correlation of the ^{13}C -nmr chemical shift of the C-10a resonance of phenoxathiin and azaphenoxathiins with the crystallographically determined dihedral angle. Points corresponding to 2-azaphenoxathiin 2-oxide are denoted by \blacktriangle and \triangle ; points corresponding to 1-nitrophenoxathiin are denoted by \blacksquare and \square . Linear regression analysis of the nine data pairs plotted gave a correlation coefficient, $r = 0.967$. Points represented by \triangle and \square were ignored in the computation [28].

Table 3

Least-Squares Planes
Equation of Planes: $Ax + By + Cz = D$, where x, y, z are in Å

| Plane | Atoms Defining Plane | A | B | C | D |
|-------|--|-------|--------|--------|--------|
| 1 | N, C(1), C(1a), C(3) C(4), C(4a) | 2.242 | 6.914 | 2.564 | 7.328 |
| 2 | C(5a), C(6), C(7) C(8), C(9), C(9a) | 2.813 | -7.044 | -1.715 | -5.376 |
| 3 | N', C'(1), C'(1a) C'(3), C'(4), C'(4a) | 0.401 | -6.920 | 3.613 | -4.355 |
| 4 | C'(5a), C'(6), C'(7) C'(8), C'(9), C'(9a) | 0.652 | -6.920 | -3.923 | -6.128 |

Angle between planes 1 and 2 = 160.8°; Angle between planes 3 and 4 = 151.3°.

Table 4

Comparison of C-S and C-O bond lengths, C-S-C and C-O-C bond angles and dihedral angles of phenoxathiin derivatives

| Compound | C-S (Å) | C-O (Å) | C-S-C (°) | C-O-C (°) | Dihedral Angle |
|--------------------------------|-----------|-----------|--------------|---------------|----------------|
| 2-Azaphenoxathiin 2-oxide | 1.762(2) | 1.396(2) | 99.1 | 120.0 | 160.8 |
| | 1.753(2) | 1.368(2) | — | — | — |
| | 1.757(2) | 1.377(2) | 98.4 | 117.8 | 151.3 |
| | 1.769(2) | 1.395(2) | — | — | — |
| Phenoxathiin [25] | 1.753(43) | 1.401(54) | 97.68(3) [a] | 117.63(5) [a] | 138.4 |
| | 1.751(43) | 1.386(52) | — | — | — |
| 7-Chloro-1-azaphenoxathiin [6] | 1.761(3) | 1.385(3) | 100.8(1) | 123.2(2) | 175.7 |
| | 1.757(2) | 1.370(3) | — | — | — |
| 8-Chloro-1-azaphenoxathiin [6] | 1.764(2) | 1.384(2) | 100.47(9) | 122.2(2) | 176.78 |
| | 1.768(2) | 1.376(2) | — | — | — |
| 3-Azaphenoxathiin [26] | 1.764 | 1.385 | 100.3 | 121.2 | 167.07 |
| | 1.749 | 1.377 | — | — | — |
| 9-Nitro-1-azaphenoxathiin [12] | 1.769(7) | 1.342(10) | 100.6(3) | 126.7(6) | 178.0 |
| | 1.767(7) | 1.348(10) | — | — | — |
| | 1.253(7) | 1.373(9) | 101.2(4) | 123.1(6) | 176.8 |
| | 1.758(7) | 1.365(9) | — | — | — |
| 1-Nitrophenoxathiin [12] | 1.753(3) | 1.386(3) | 100.3(1) | 121.4(2) | 163.8 |
| | 1.761(3) | 1.379(3) | — | — | — |
| | 1.761(3) | 1.386(3) | 99.0(1) | 117.4(2) | 145.7 |
| | 1.763(3) | 1.383(3) | — | — | — |

[a] The standard deviations in these angles are probably underestimated.

Table 5

Short Intermolecular Distances (Å)

| | |
|-------------------------------|-------|
| O(2) ... H'(4 ^{''}) | 2.266 |
| O(2) ... H(1 [']) | 2.309 |
| O'(2) ... H(4') | 2.382 |
| O'(2) ... H'(1 [']) | 2.232 |

Roman numerals as superscripts refer to the following equivalent positions relative to the reference molecule at x, y, z :

| | |
|-----|--|
| I | x, y, z |
| II | $1 - x, 2 - y, 1 - z$ |
| III | $\frac{1}{2} + x, 1\frac{1}{2} - y, \frac{1}{2} + z$ |
| IV | $1 - x, 1 - y, 2 - z$ |

The dihedral angle, which is the angle between the two aromatic rings or wings of the molecule, differ significantly in the two molecules (Figure 2) and are 160.8° and 151.3°, respectively, for molecules A and B. Similar observations have been made for the dihedral angles of the A and B forms of triflupromazine [10] (134.4 and 141.0°), *N*-isopropylphenothiazine [11] (136.1 and 146.8°), 1-nitrophenoxathiin [12] (145.7 and 163.8°) and for 9-nitro-1-azaphenoxathiin [12] (177.1 and 178.2°), which also crystallize with two molecules in the asymmetric unit. The increase in dihedral angle for molecule A with respect to B is not associated with changes in bond lengths, which are closely

Table 6
Crystallographic Data

| | |
|--|--------------------|
| Formula | $C_{11}H_7NSO_2$ |
| F.W. | 217.2 |
| Crystal System | monoclinic |
| a, Å | 17.50(2) |
| b, Å | 7.147(3) |
| c, Å | 16.044(7) |
| β , deg | 110.60(5) |
| Volume, Å ³ | 1878 |
| Space Group | P2 ₁ /n |
| Z | 8 |
| F(0,0,0) | 896 |
| D_{calc} g/cm ³ | 1.53 |
| $\lambda(\text{CuK}\alpha)$, Å | 1.5418 |
| (for intensity data) | |
| $\mu(\text{CuK}\alpha)$, cm ⁻¹ | 27.8 |
| No. of independent reflections | 3831 |
| Maximum 2 θ , deg | 150 |
| Temperature, °C | -135 ± 2 |
| $\lambda(\text{CuK}\alpha_1)$ (for unit cell data) | 1.5405 Å |

Table 7

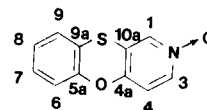
Positional ($\times 10^4$) Parameters for Non-Hydrogen Atoms.
Calculated standard deviations for the last digit are in parentheses

| Molecule A | x/a | y/b | z/c |
|------------|-----------|-----------|-----------|
| S | 2760.1(3) | 6778.9(7) | 8572.5(3) |
| O(2) | 5702(1) | 5243(2) | 9237(1) |
| O(5) | 2831(1) | 7202(2) | 6664(1) |
| N | 5008(1) | 5755(2) | 8627(1) |
| C(1) | 4328(1) | 5914(3) | 8845(1) |
| C(1a) | 3587(1) | 6416(2) | 8215(1) |
| C(3) | 4969(1) | 6124(3) | 7784(1) |
| C(4) | 4238(1) | 6584(3) | 7128(1) |
| C(4a) | 3535(1) | 6708(3) | 7338(1) |
| C(5a) | 2075(1) | 6819(3) | 6735(1) |
| C(6) | 1426(1) | 6747(3) | 5934(1) |
| C(7) | 643(1) | 6454(3) | 5940(1) |
| C(8) | 512(1) | 6197(3) | 6738(2) |
| C(9) | 1165(1) | 6250(3) | 7537(1) |
| C(9a) | 1956(1) | 6592(2) | 7539(1) |

| Molecule B | x/a | y/b | z/c |
|------------|-----------|-----------|-----------|
| S' | 3632.5(3) | 7656.1(7) | 2397.4(3) |
| O'(2) | 4359(1) | 9367(2) | 5628(1) |
| O'(5) | 1924(1) | 7575(2) | 2526(1) |
| N' | 3779(1) | 9022(2) | 4862(1) |
| C'(1) | 3968(1) | 8702(3) | 4122(1) |
| C'(1a) | 3365(1) | 8223(2) | 3325(1) |
| C'(3) | 2986(1) | 8982(3) | 4811(1) |
| C'(4) | 2370(1) | 8555(3) | 4028(1) |
| C'(4a) | 2554(1) | 8131(2) | 3279(1) |
| C'(5a) | 1960(1) | 8089(2) | 1701(1) |
| C'(6) | 1221(1) | 8389(3) | 1022(1) |
| C'(7) | 1216(1) | 8861(3) | 176(1) |
| C'(8) | 1945(1) | 9033(3) | 24(1) |
| C'(9) | 2683(1) | 8712(3) | 705(1) |
| C'(9a) | 2695(1) | 8216(3) | 1552(1) |

Table 8

Calculated vs. observed ¹³C-nmr chemical shifts of 2-azaphenoxathiin 2-oxide in deuteriochloroform at 25.158 MHz.



| Carbon | 2-Azaphenoxathiin [a] | | 2-Azaphenoxathiin 2-Oxide | |
|------------------|-----------------------|--------------------|-------------------------------|-----------------------------|
| | $\delta^{13}\text{C}$ | $\Delta\delta$ [a] | $\delta^{13}\text{C}$ -calcd. | $\delta^{13}\text{C}$ -obs. |
| 10a (α) | 117.24 | + 2.0 | 119.2 | 120.20 |
| 9a (α') | 117.24 | --- | 117.2 | 115.33 |
| 4a (β) | 158.01 | - 10.7 | 147.3 | 149.63 |
| 5a (β') | 149.87 | --- | 149.9 | 149.12 |
| 1 | 148.71 | - 11.3 | 137.4 | 137.85 |
| 3 | 146.53 | - 11.3 | 135.2 | 135.41 |
| 4 | 112.32 | + 2.0 | 114.3 | 113.78 |
| 6 | 117.60 | --- | 117.6 | 117.56 |
| 7 | 126.81 | --- | 126.8 | 126.59 |
| 8 | 125.16 | --- | 125.2 | 125.32 |
| 9 | 127.84 | --- | 127.8 | 128.28 |

[a] $\Delta\delta$ values were obtained from the work of Sojka and co-workers [17] and represent the difference in chemical shifts produced by the introduction of the N-oxide moiety onto the pyridine ring.

similar in both molecules (Figure 1), but the increase is correlated with both significant increases of the endocyclic bond angles of the central ring (Table 2), especially the C-O-C angles (120.0(2)° for A and 117.8(1)° for B), and with significant decreases of the conformational angles of the central ring (Table 3). These statements are also applicable when a more general comparison is made with other related compounds (Table 4). An attempt to explain the differences in dihedral angle between molecules A and B as due to packing forces was inconclusive. The only close intermolecular contacts involve the oxygen atom of the pyridine oxide. There are two for each molecule and they are listed in Table 5. However, their vector direction with respect to the pyridine rings do not allow a definite conclusion. A packing drawing is shown in Figure 3.

It is interesting to note that O...H distances listed in Table 5 are considerably less than the sum of the van der Waals' radii (2.6 Å). Similar short distances between the O-atom of the N-O group and atomic H-atoms have been observed before as, for instance, 2.19 Å in 3-methyl-4-nitropyridine N-oxide [13] in 2.22 Å in p-nitropyridine N-oxide [14].

The possibility existed that the difference in dihedral angle between the molecules was a result of the temperature (-135°C) at which the data was taken. Therefore, a room temperature data set was taken and refined. The dihedral angles for molecules A and B show small changes with respect to their values at low temperature 161.2°

(160.8° at -135°C) and 152.8° (151.3° at -135°C), but the important and significant difference in dihedral angles between molecules A and B persists.

It is interesting to note that the benzene and pyridine rings affect the central ring in different ways. This is especially clear in the significant difference between the C(5a)-O(5) and C(4a)-O(5) bond distances for which the averages are 1.395 and 1.372 Å, respectively.

The N-O bond lengths for molecules A and B are 1.315(2) Å and 1.313(2) Å, respectively, which are shorter than the N-O bond length of 1.34 Å in pyridine 1-oxide [15] but longer than the N-O bond length of 1.26 Å in 4-nitropyridine *N*-oxide [16]. The estimated π -bond character for the N-O bond is 15-20% for the present compound.

The decoupled ¹³C-nmr spectrum of 2-azaphenoxathiin 2-oxide showed eleven clearly distinct resonances which were readily separable into protonated and non-protonated resonances from relaxation considerations. Calculated ¹³C-nmr chemical shifts were obtained by incrementation of the assigned chemical shifts of 2-azaphenoxathiin [9] with the additivities from pyridine *N*-oxide [17]. Although attempts were made to utilize the additivities determined from 1-azaphenoxathiin 1-oxide [18], they were found to be less accurate than the more general model, probably because of the profound interaction between the sulfur and the *N*-oxide in 1-azaphenoxathiin. On this basis, the calculated ¹³C-nmr chemical shifts shown in Table 8 were obtained. Assignment of the observed resonances of 2-azaphenoxathiin 2-oxide was reasonably straightforward since there was no perturbation of the benzenoid carbon resonances relative to 2-azaphenoxathiin itself [9] (Table 8).

Evaluation of the relationship between the ¹³C-nmr chemical shift of the C-10a resonance and the molecular dihedral angles for 2-azaphenoxathiin 2-oxide opens some interesting possibilities for speculation. Specifically, the molecule with a dihedral angle of 151.2° (Δ , Figure 4) conforms reasonably well to the least squares linear fit of the data ($n = 9$, $r = 0.967$). In contrast, the molecule with a dihedral angle of 160.8° deviates considerably from the least squares fitted line. A similar situation exists in the case of 1-nitrophenoxathiin [12,21] (compare \blacksquare and \square , Figure 4). It is attractive to speculate that in solution both 1-nitrophenoxathiin and 2-azaphenoxathiin 2-oxide exist in a single preferred conformation. Ideally, we may further assume that the preferred solution dihedral angle is near that proscribed by the least squares fitted line shown in Figure 4. Further work on the nature of this relationship and on the expansion of the data base for it is underway in these laboratories and will be forthcoming.

EXPERIMENTAL

The sample of 2-azaphenoxathiin 2-oxide utilized in this study was prepared as described previously in the synthesis of 2-azaphenoxathiin [9] as shown in Scheme I. The sample utilized in both the ¹³C-nmr and the X-ray crystallographic portions of this work was purified by linear thermal gradient sublimation at 160°/0.5 mm Hg to afford colorless monoclinic crystals.

Anal. Calcd. for C₁₁H₇NO₂S: C, 60.83; H, 3.23; N, 6.45. Found: C, 60.69; H, 3.20; N, 6.47.

¹³C-NMR Spectroscopy.

The ¹³C-nmr spectra of 2-azaphenoxathiin 2-oxide were acquired in deuteriochloroform on a Varian XL-100-15 spectrometer operating in the Fourier transform mode at an observation frequency of 25.158 MHz for ¹³C. The spectrometer was equipped with a Nicolet 1180 data system and a TT-760 decoupler with a decoupling power of $\gamma H_2/2\pi = 2.9$ KHz applied at δ 7.2 in the proton spectral window. Typical instrument parameters were: pulse width = 8 μ sec (40°); pulse delay = 4.0 sec; acquisition time = 0.8192 sec (8K data points); sweep width = 5 KHz; apodization = 1 Hz.

Crystallographic Measurements.

The crystal used for the X-ray diffraction studies was a colorless prismatic needle with the *b*-axis parallel to the needle axis. A crystal of approximately 0.65 mm \times 0.13 mm \times 0.11 mm in size was used for all cell parameters and data collection. The least squares cell dimensions were obtained from the averages of the $+2\theta$ and -2θ values of 24 reflections. The crystal data are summarized in Table 6.

The unit cell dimensions and the intensity data were collected at -135 \pm 2°C with a CAD-4 counter diffractometer (Enraf-Nonius) controlled by a PDP 8/e computer fitted with a low temperature apparatus. A $\theta - 2\theta$ scan technique with variable scan rates and Ni-filtered CuK α radiation ($\lambda = 1.5418$ Å) was used to measure 3831 independent reflections with 2θ values $\leq 150^\circ$. The variable scan width was $(0.70 + 0.14 \tan \theta)^\circ$ for each reflection. A receiving aperture with a variable width of $(3.00 + 0.86 \tan \theta)$ mm and a constant height of 6 mm was located 173 mm from the crystal. The maximum scan time for each reflection was 50 seconds. Reflection (4, 1, 3) was taken as the standard reflection. During the data collection, the standard intensity varied less than 3%. Of the 3831 independent reflections measured 232 reflections were considered unobserved (with $I \leq 2.0\sigma(I)$). All intensity data were scaled by a standard reflection and according to their scan rate. Lorentz, polarization corrections and absorption corrections [19] ($\mu = 27.8$ cm⁻¹) were applied to the data. Each amplitude was assigned a weight given by $w_r = 1/\sigma_r^2$, where the standard deviations were obtained from intensity statistics [20]. A second data set was taken, but in this set all intensity data were taken at room temperature. The parameters used in this data set were the same as those described for the one taken at low temperature. The room temperature data set was only used to calculate the dihedral angles of the molecules at this temperature.

The structure was solved by direct methods using the program MULTAN [21]. The phases of 250 reflections with the highest E values ($E \leq 1.83$) were generated by the tangent formula [22] and refined. The largest peaks in the E map revealed the positions of the non-hydrogen atoms. The atomic positions from the E map were refined by block-diagonal least-squares minimization of $\sum w(|kF_o| - |F_c|)^2$, first isotropically and then at a later stage with anisotropic thermal parameters. The hydrogen atoms were included in the least-squares refinement with isotropic temperature parameters. The effect of anomalous dispersion by the sulfur atoms was included in F_c by using $\Delta f'$ and $\Delta f''$ from the International Tables for X-ray Crystallography [23]. The refinement converged to an $R = (\sum |F_o| - |F_c|/|F_o|)$ of 0.038 for the 3526 reflections that were included in the least-squares calculations and 0.045 for all 3831 reflections. Final shifts in all the parameters were less than 0.2σ . The scattering factors for S, O, N and C atoms were taken from the International Tables for X-ray Crystallography [24].

The final positional parameters for the S, O, C and N atoms are given in Table 7. The anisotropic thermal parameters, the positional and isotropic parameters for the H atoms and a listing of the observed and calculated structure factors are submitted as supplementary material.

Supplementary Material Available.

Anisotropic thermal parameters, H atom parameters and structure factors are available from the authors.

REFERENCES AND NOTES

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