Acta Cryst. (1974). B30, 527

Oxisuran

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(*Received* 14 *August* 1973; *accepted* 25 *September* 1973)

Abstract. 2-[(Methylsulfinyl)acetyl]pyridine, $C_5H_4NCOCH_2SOCH_3$, $P2_1/a$, $a=10{\cdot}107\pm0{\cdot}007$, $b=9{\cdot}991\pm0{\cdot}010$, $c=9{\cdot}298\pm0{\cdot}010$ Å, $\beta=105{\cdot}6°\pm0{\cdot}1°$, $Z=4$. The molecule has a fully extended chain and is approximately planar except for the O atom in the sulfoxide group. The sulfoxide group is pyramidal. Carbonyl groups of adjacent molecules are aligned in an antiparallel manner with C···O separations of 3·18 Å.

Introduction. Oxisuran, a synthetic compound, has the property of inhibiting the rejection of organ transplants (Freedman, Fox, Shavel & Morrison, 1972; *Chemical and Engineering News*, 1972). In animals, the drug suppresses cell-mediated immune reactions but does not inhibit antibody production. It appears to be a selective, noncytotoxic immunosuppressive agent. For this reason, it was deemed important to examine the structure of the molecule. Furthermore, the structures of relatively few sulfoxides have been determined.

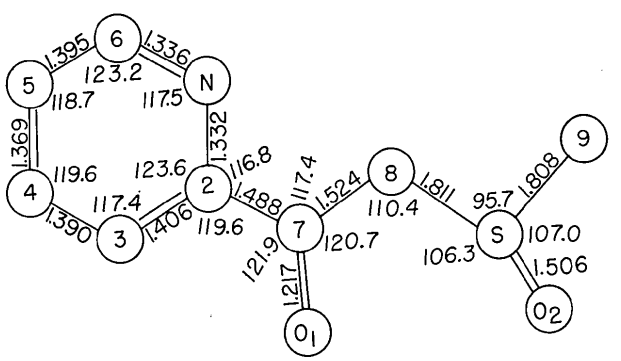

Fig. 1. Bond lengths and angles in oxisuran. The standard deviations for the bond lengths are 0·004 Å and for the bond angles 0·25°.

Crystals of oxisuran were provided by Dr John Shavel Jr of the Warner–Lambert Research Institute. An irregular fragment, roughly in the shape of a cube with ~0·3 mm on a side, was used to collect diffraction data. The space group is $P2_1/a$ with $a=10{\cdot}107\pm0{\cdot}007$, $b=9{\cdot}991\pm0{\cdot}010$, $c=9{\cdot}298\pm0{\cdot}010$ Å, $\beta=105{\cdot}6\pm0{\cdot}1°$, and $Z=4$. 1488 reflections were measured on a four-circle diffractometer with Mo radiation using a scan of $1{\cdot}5°+2\theta(\alpha_2)-2\theta(\alpha_1)$ and a scan speed of 1° min^{-1}. The structure was solved by the symbolic addition procedure. Hydrogen atoms were located in a difference map and their parameters were kept constant in the least-squares refinement with anisotropic thermal parameters and weights based on counting statistics. The final R index is 6·7%.* Fractional coordinates are shown in Tables 1 and 2. Bond lengths and angles are shown in Fig. 1.

* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. 30233 (7 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional coordinates and anisotropic thermal parameters for oxisuran*

The thermal parameters are of the form

$$T=\exp\left[-\tfrac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)\right].$$

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
N(1)	0·6322	0·4059	0·3676	4·35	3·62	3·67	0·67	0·58	−0·14
C(2)	0·5427	0·3716	0·2401	3·16	2·96	3·56	0·09	1·39	0·16
C(3)	0·5394	0·2446	0·1740	3·79	3·46	5·42	−0·24	1·91	−0·40
C(4)	0·6354	0·1510	0·2475	4·74	3·25	6·88	0·55	2·82	−0·15
C(5)	0·7281	0·1849	0·3787	5·26	4·42	5·72	1·16	2·21	0·75
C(6)	0·7238	0·3139	0·4347	6·04	5·01	4·33	1·71	0·83	0·45
C(7)	0·4447	0·4768	0·1640	2·81	3·46	3·46	−0·35	0·87	−0·14
C(8)	0·4549	0·6134	0·2389	3·11	2·72	4·28	0·42	0·63	−0·05
C(9)	0·3809	0·8710	0·2429	3·76	2·80	7·32	−0·10	1·36	−0·07
O(1)	0·3610	0·4564	0·0452	4·33	4·11	4·73	0·12	−0·05	−0·79
O(2)	0·1911	0·6783	0·1868	2·67	3·92	5·48	−0·22	1·50	0·51
S	0·3162	0·7208	0·1397	2·91	2·83	4·07	0·14	1·03	0·56
Standard deviations									
C, N	0·0003	0·0003	0·0004						
O	0·0002	0·0002	0·0003						
S	0·0001	0·0001	0·0001						

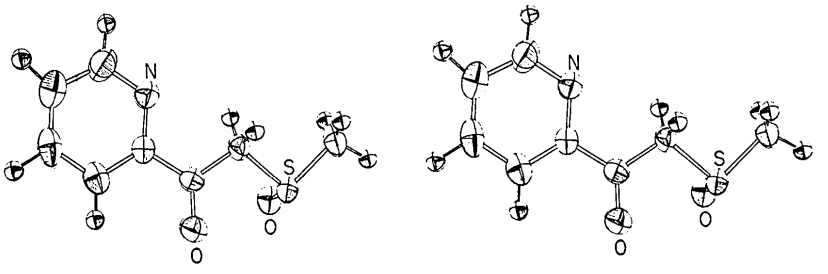

Fig. 2. Stereodiagram of oxisuran with thermal ellipsoids at the 50% level. Hydrogen atoms are indicated by the small spheres.

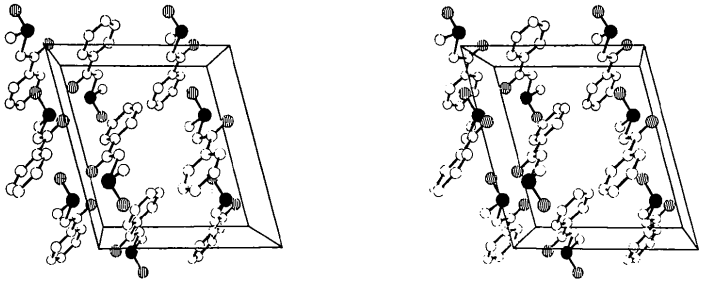

Fig. 3. Stereodiagram of the molecular packing. The axial directions are $a\nwarrow$, $c\rightarrow$ and b up from the plane of the page. The S atoms are dark while the O atoms are lined.

Table 2. *Approximate fractional coordinates for the hydrogen atoms as determined from a difference map*

	x	*y*	*z*
H(3)	0·4561	0·2188	0·0874
H(4)	0·6415	0·0630	0·1853
H(5)	0·8258	0·1356	0·4208
H(6)	0·7795	0·3411	0·5395
H(81)	0·5527	0·6548	0·2140
H(82)	0·4613	0·6086	0·3545
H(91)	0·4840	0·8794	0·2355
H(92)	0·3058	0·9541	0·1983
H(93)	0·3749	0·8664	0·3286

Discussion. Bond lengths and angles in oxisuran are very similar to those observed in similar moieties in other molecules. The angles in the pyridyl ring alternate between a smaller and a larger value, with extremes of 117·4° and 123·6°. The CNC angle is 117·5°. Similar alternations in angular values in a pyridyl group have been observed in picolinamide (Takano, Sasada & Kakudo, 1966) and pyridine.LiCl (Durant, Verkist & Van Meerssche, 1966). The S atom is pyramidal and the bond lengths and angles are very near those observed in methyl *p*-tolyl sulfoxide (de la Camp & Hope, 1970). The atoms in the oxisuran molecule are approximately coplanar except for O(2), Fig. 2. The pyridyl ring is planar to within ±0·005 Å and the carboxyl group with C(2) and C(8) is planar to within ±0·005 Å. Values for the torsional angles in the chain are: C(3)C(2)C(7)C(8) = −178·7°, C(2)C(7)C(8)S = −174·1° and C(7)C(8)SC(9) = −171·8° (where ±180° is equivalent to a planar *trans* conformation). The torsional angle which measures the out-of-plane O(2) atom, C(7)C(8)SO(2), is +78·6.

The packing of the molecules is illustrated in Fig. 3. The molecules are stacked in rows parallel to **c**. Within each row, adjacent molecules are related by a center of symmetry. In these rows, C=O groups of adjacent molecules are aligned antiparallel to each other with a pair of C(7)···O(1) distances at 3·18 Å. Similar antiparallel arrangements of C=O groups with a C···O separation of ~3·15 Å have been observed in a number of compounds (see *e.g.* Prout & Wallwork, 1966; Przybylska, 1972). Other close approaches between molecules in oxisuran are O(1)···S′ (at $\frac{1}{2}-x$, $-\frac{1}{2}+y$, $-z$) = 3·17 Å and O(1)···C(9′) (at $\frac{1}{2}-x$, $-\frac{1}{2}+y$, $-z$) = 3·23 Å.

References

Camp, U. de la & Hope, H. (1970). *Acta Cryst.* B**26**, 846–853.

Chemical and Engineering News (1972). Sept. 11, p. 22.

Durant, F., Verkist, J. & Van Meerssche, M. (1966). *Bull. Soc. Chim. Belges*, **75**, 806.

Freedman, H. H., Fox, A. E., Shavel, J. Jr & Morrison, G. C. (1972). *Proc. Coc. Exp. Biol. Med.* **139**, 909–912.

Prout, C. K. & Wallwork, S. C. (1966). *Acta Cryst.* **21**, 449–450.

Przybylska, M. (1972). *Acta Cryst.* B**28**, 2814–2820.

Takano, T., Sasada, Y. & Kakudo, M. (1966). *Acta Cryst.* **21**, 514–522.