

These unusual features seem to result from the strong intermolecular hydrogen bonding with Me₂SO. The presence of this enhanced hydrogen-bonding donor may explain the plant-growth activity of this molecule.

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Structure of the 1:1 Complex of 1,4-Dithiintetracarboxylic *N,N'*-Dimethyldiimide and Acridine, [C₁₀H₆N₂O₄S₂][C₁₃H₉N]

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Abstract. $M_r = 461.52$, monoclinic, space group Pn , $a = 13.701$ (11), $b = 10.244$ (4), $c = 7.208$ (2) Å, $\beta = 92.20$ (5)°, $V = 1010.9$ (9) Å³, $Z = 2$, $D_m = 1.515$, $D_x = 1.511$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.2998$ mm⁻¹, $F(000) = 482$, $T = 298$ K, final $R = 0.039$ for 1544 observed reflections. The title complex contains 1,4-dithiintetracarboxylic *N,N'*-dimethyldiimide and acridine which have planar structures. These almost parallel molecules are alternately stacked along the c axis.

Introduction. 1,4-Dithiins have the boat structure, e.g. 1,4-dithiin (Howell, Curtis & Lipscomb, 1954) and thianthrene (Rowe & Post, 1956, 1958; Lynton & Cox, 1956). Recently, it has been reported that 1,4-dithiintetracarboxylic *N,N'*-dimethyldiimide (*A*) reacts with various anthracenes *via* charge-transfer (CT) complexes to give Diels–Alder adducts and that, in contrast, the reaction of *A* with acridine (*B*) gives a stable 1:1 complex which does not give rise to any adducts (Hayakawa, Mibu, Osawa & Kanematsu, 1982). This complex in chloroform shows no new ultraviolet absorption bands indicative of the formation of a CT complex. To explain these facts, the X-ray structure of the title complex was determined.

Experimental. Green pillars grown from chloroform, density measured by flotation in KI solution, crystal data collected from single crystal of approximately 0.2 × 0.15 × 0.3 mm on an automated Syntex P1 diffractometer, using graphite-monochromatized Mo $K\alpha$, cell parameters determined by least squares from the setting angles of 15 reflections, 1802 reflections measured using θ – 2θ variable scans (4.8–24.0° min⁻¹ for 2θ) up to $2\theta = 55^\circ$, range of hkl : $-16 \leq h \leq 16$, $-12 \leq k \leq 0$, $0 \leq l \leq 8$, three standard reflections (150, 241, 140) measured every 100 reflections (intensity variation 10%), data corrected for geometrical factors and for monitored intensities but not for absorption, 1544 independent reflections [$I > 3.0\sigma(I)$] considered observed and used for the analysis; heavy-atom method; the 100 peaks were selected from a Patterson synthesis and the S atoms located with the *RMINH* 80 program (Kawano, 1983); all non-H atoms determined by a Fourier synthesis, all H atoms determined by a difference Fourier synthesis, all coordinates, anisotropic thermal parameters for the non-H atoms and isotropic ones for H atoms refined by the block-diagonal least-squares procedure, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1.0$, final $R = 0.039$, $wR = 0.058$, $S = 1.09$, final $\Delta\rho$ excursions < 0.3 e Å⁻³; atomic scattering factors involving the anomalous-dispersion terms for non-H atoms from *International*

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Tables for X-ray Crystallography (1974), those for H atoms from Stewart, Davidson & Simpson (1965); calculations performed on a FACOM M-200 computer at the Computer Center of Kyushu University using the UNICS III program system (Sakurai & Kobayashi, 1979).

Discussion. The final atomic parameters for the non-H atoms are listed in Table 1.* The bond distances are shown in Figs. 1 and 2 with the atom-numbering scheme. Table 2 shows the angles. Fig. 3 shows a projection of the crystal structure along the *b* axis (ORTEP; Johnson, 1965).

* Lists of structure factors, anisotropic thermal parameters of the non-H atoms, positional parameters of the H atoms and the calculations of the least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38841 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates with their estimated standard deviations in parentheses

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
S(1)A	0.3785 (1)	0.0751 (2)	0.4886 (3)	2.84 (4)
S(2)A	0.6217 (1)	-0.0745 (2)	0.5126 (3)	2.67 (4)
O(1)A	0.2594 (4)	-0.1708 (6)	0.3540 (9)	3.95 (16)
O(2)A	0.4375 (4)	0.3548 (6)	0.6264 (9)	3.79 (16)
O(3)A	0.7369 (4)	0.1701 (6)	0.6463 (9)	4.01 (17)
O(4)A	0.5622 (4)	-0.3543 (6)	0.3747 (9)	3.68 (15)
N(1)A	0.3993 (5)	-0.2980 (6)	0.3468 (9)	2.90 (16)
N(2)A	0.6005 (5)	0.2956 (7)	0.651 (1)	3.13 (17)
C(1)A	0.5025 (6)	0.2741 (7)	0.613 (1)	2.60 (18)
C(2)A	0.4929 (5)	0.1372 (8)	0.555 (1)	2.71 (18)
C(3)A	0.4190 (5)	-0.0825 (8)	0.437 (1)	2.50 (17)
C(4)A	0.3467 (6)	-0.1845 (7)	0.375 (1)	2.51 (17)
C(5)A	0.3563 (7)	-0.4199 (9)	0.297 (1)	4.16 (25)
C(6)A	0.4983 (6)	-0.2765 (8)	0.385 (1)	2.99 (20)
C(7)A	0.5074 (5)	-0.1355 (7)	0.445 (1)	2.21 (16)
C(8)A	0.5815 (5)	0.0823 (7)	0.560 (1)	2.56 (17)
C(9)A	0.6536 (6)	0.1827 (9)	0.624 (1)	3.21 (20)
C(10)A	0.6427 (7)	0.4238 (8)	0.708 (1)	3.70 (23)
N(1)B	0.4127 (5)	0.0592 (8)	-0.009 (1)	3.46 (18)
C(1)B	0.4158 (6)	-0.0652 (8)	-0.060 (1)	2.99 (19)
C(2)B	0.5094 (6)	-0.131 (1)	-0.051 (1)	3.74 (23)
C(3)B	0.5146 (7)	-0.263 (1)	-0.110 (1)	4.13 (25)
C(4)B	0.4324 (8)	-0.326 (1)	-0.173 (2)	5.15 (31)
C(5)B	0.3420 (8)	-0.263 (1)	-0.176 (2)	4.94 (29)
C(6)B	0.3321 (7)	-0.1369 (9)	-0.125 (1)	3.76 (23)
C(7)B	0.5838 (6)	0.0642 (9)	0.059 (1)	3.26 (21)
C(8)B	0.4912 (7)	0.1272 (8)	0.050 (1)	3.22 (21)
C(9)B	0.4853 (8)	0.261 (1)	0.106 (2)	5.20 (31)
C(10)B	0.5653 (9)	0.326 (1)	0.162 (2)	5.11 (31)
C(11)B	0.6585 (8)	0.262 (1)	0.176 (1)	4.37 (26)
C(12)B	0.6670 (6)	0.136 (1)	0.122 (1)	4.40 (27)
C(13)B	0.5971 (6)	-0.0670 (9)	0.009 (1)	3.43 (21)

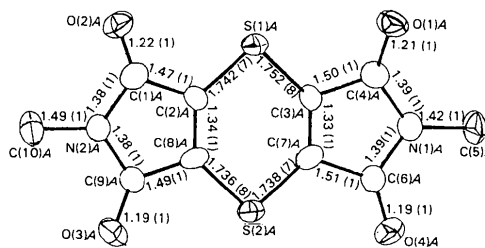


Fig. 1. Interatomic distances (Å) of molecule A.

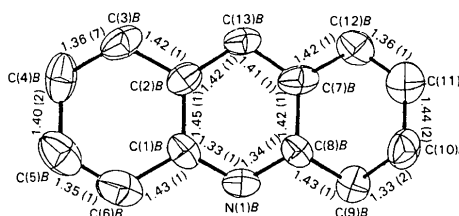


Fig. 2. Interatomic distances (Å) of molecule B.

Table 2. Interatomic angles for molecules A and B (°)

C(2)A-S(1)A-C(3)A	96.1 (4)	C(4)A-C(3)A-C(7)A	108.7 (7)
C(7)A-S(2)A-C(8)A	95.7 (3)	O(1)A-C(4)A-N(1)A	126.6 (7)
C(4)A-N(1)A-C(5)A	124.1 (7)	O(1)A-C(4)A-C(3)A	126.7 (7)
C(4)A-N(1)A-C(6)A	110.3 (6)	N(1)A-C(4)A-C(3)A	106.7 (7)
C(5)A-N(1)A-C(6)A	125.5 (7)	O(4)A-C(6)A-N(1)A	126.6 (8)
C(1)A-N(2)A-C(9)A	110.6 (7)	O(4)A-C(6)A-C(7)A	127.3 (7)
C(1)A-N(2)A-C(10)A	124.2 (7)	N(1)A-C(6)A-C(7)A	106.1 (6)
C(9)A-N(2)A-C(10)A	125.2 (7)	S(2)A-C(7)A-C(3)A	132.6 (6)
O(2)A-C(1)A-N(2)A	125.7 (7)	S(2)A-C(7)A-C(6)A	119.2 (5)
O(2)A-C(1)A-C(2)A	127.7 (7)	C(3)A-C(7)A-C(6)A	108.3 (7)
N(2)A-C(1)A-C(2)A	106.6 (6)	S(2)A-C(8)A-C(2)A	132.7 (6)
S(1)A-C(2)A-C(1)A	119.8 (6)	S(2)A-C(8)A-C(9)A	119.3 (5)
S(1)A-C(2)A-C(8)A	131.3 (6)	C(2)A-C(8)A-C(9)A	108.0 (7)
C(1)A-C(2)A-C(8)A	108.8 (7)	O(3)A-C(9)A-N(2)A	126.5 (8)
S(1)A-C(3)A-C(4)A	119.7 (5)	O(3)A-C(9)A-C(8)A	127.5 (8)
S(1)A-C(3)A-C(7)A	131.6 (6)	N(2)A-C(9)A-C(8)A	106.0 (7)
C(1)B-N(1)B-C(8)B	123.7 (8)	C(8)B-C(7)B-C(12)B	119.0 (8)
N(1)B-C(1)B-C(2)B	118.2 (8)	C(8)B-C(7)B-C(13)B	123.1 (8)
N(1)B-C(1)B-C(6)B	123.5 (8)	C(12)B-C(7)B-C(13)B	117.9 (8)
C(2)B-C(1)B-C(6)B	118.3 (8)	N(1)B-C(8)B-C(7)B	118.9 (8)
C(1)B-C(2)B-C(3)B	119.0 (8)	N(1)B-C(8)B-C(9)B	122.3 (9)
C(1)B-C(2)B-C(13)B	122.4 (9)	C(7)B-C(8)B-C(9)B	118.8 (8)
C(3)B-C(2)B-C(13)B	118.6 (8)	C(8)B-C(9)B-C(10)B	121 (1)
C(2)B-C(3)B-C(4)B	120.1 (9)	C(9)B-C(10)B-C(11)B	121 (1)
C(3)B-C(4)B-C(5)B	121 (1)	C(10)B-C(11)B-C(12)B	120 (1)
C(4)B-C(5)B-C(6)B	122 (1)	C(7)B-C(12)B-C(11)B	120.7 (9)
C(1)B-C(6)B-C(5)B	119.6 (9)	C(2)B-C(13)B-C(7)B	113.8 (8)

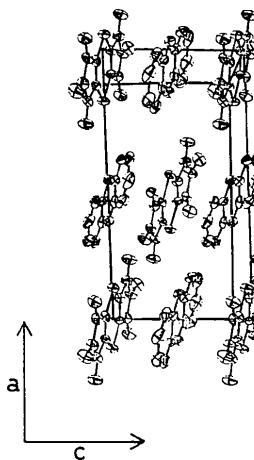


Fig. 3. A Projection of the crystal structure along the *b* axis.

1,4-Dithiins are known to have the boat structure, as mentioned above. However, molecules *A* and *B* of this complex are planar with respective maximum shifts of 0.0736 Å [C(5)*A*] and 0.0303 Å [C(4)*B*] from their least-squares planes. The C=C–S angles of molecule *A* are larger than that of 1,4-dithiin (124°) (Howell, Curtis & Lipscomb, 1954). The C–S–C angles of molecule *A* are somewhat smaller than those of 1,4-dithiin (101°), thianthrene (100°) (Rowe & Post, 1956, 1958; Lynton & Cox, 1956) and 1,4-dithiane (99°) (Marsh, 1955). The C–S and C=C bond distances of molecule *A* are normal. The dithiin ring of molecule *A* is flattened out along the S...S direction by the face-to-face stacking of molecules *A* and *B*.

The intermolecular distances C(4)*A*...C(5)*B* and C(6)*A*...C(4)*B* are respectively 3.34 (2) and 3.38 (2) Å and are shorter than the van der Waals distance (3.40 Å). This suggests that π–π interactions between molecules *A* and *B* exist in these parts and the molecules form a paired molecular group. The dihedral angle between the least-squares planes of molecules *A* and *B* is 2.3°. Distances between paired groups are larger than the van der Waals distances. The minimum distance is 3.50 (1) Å [C(1)*A*...C(8)*B*]. The stacking of paired molecules is in accordance with the poor electrical conductivity of this complex (Hayakawa, Mibu, Ōsawa & Kanematsu, 1982).

Although the crystal structure of this complex is similar to those of charge-transfer complexes, the

interactions between molecules *A* and *B* in this complex seem to be fairly weak. No CT band could be observed in chloroform solution.

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Structure of 1-(*p*-Dimethylaminophenyl)-3-(mercaptoacetyl)urea, C₁₁H₁₅N₃O₂S

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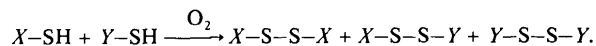
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Abstract. $M_r = 253.32$, monoclinic, $P2_1/n$, $a = 20.981$ (1), $b = 8.1402$ (4), $c = 7.2535$ (4) Å, $\beta = 94.375$ (6)°, $V = 1235.2$ (1) Å³, $D_m = 1.38$, $D_x = 1.363$ g cm⁻³, $Z = 4$, $T = 293$ K, $F(000) = 134$, $\mu(\text{Cu } K\alpha) = 21.40$ cm⁻¹, $\lambda = 1.54184$ Å, $R = 0.054$ for 1779 observed reflections. The molecule is roughly planar, except the SH group. Two molecules around an inversion center are connected by two N–H...O hydrogen bonds to form a dimer. Two molecules

around another inversion center are stacked, the distance between the central acylurea moieties being 3.392 (3) Å.

Introduction. It has been reported that in the following oxidation, the selectivity, defined by the ratio of the yield of the unsymmetrical disulfide ($X-S-S-Y$) to that of the symmetrical disulfides ($X-S-S-X$ and $Y-S-S-Y$), depends on the substituents, X and Y :



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