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A Reinvestigation of the Structure of N-Dichloroacetyl-S,S-diethylsulphilimine

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Abstract. $C_6H_{11}Cl_2NOS$, monoclinic, $P2_1/n$, $a = 6\cdot950$ (1), $b = 11\cdot322$ (1), $c = 12\cdot460$ (2) Å, $\beta = 95\cdot32$ (1)°, $V = 976\cdot2$ (4) Å^3, $Z = 4$, $D_c = 1\cdot470$, $D_m = 1\cdot468$ Mg m^{-3}, $F(000) = 448$. The structure of $Et_2SNCOCHCl_2$, DEDAS, has been reinvestigated with diffractometer data. The atomic parameters obtained from film data ($R = 0\cdot103$) have been refined by least squares to $R = 0\cdot034$ for 2253 independent reflexions ($R_{tot} = 0\cdot056$ for 2846 intensities). The $S^{IV}–N$ bond length of $1\cdot664$ (1) Å and the $S\cdots O$ close contact of $2\cdot838$ (1) Å are discussed.

Introduction. A preliminary report on the X-ray structure determination of DEDAS was published (Kálmán, Sasvári & Kucsman, 1971) to confirm the conclusion obtained from IR (Kucsman, Ruff, Kapovits & Fischer, 1967; *cf.* Kucsman, Ruff & Tanács, 1976) and extended Hückel molecular-orbital (EHMO) studies (Mezey, 1970) that the $S^{IV}–N$ bond is weaker in N-carbonylsulphilimines than in N-sulphonyl derivatives. The structure analysis of S,S-dimethyl-N-trichloroacetylsulphilimine (Me$_2$SNCOCCl$_3$, DMTAS; Kálmán, Sasvári & Kucsman, 1973) and that of N-benzoyl-S,S-dimethylsulphilimine [Me$_2$SNCOPh; DMBES; reported as N-benzoyliminosulphur-(IV) by Cameron, Duncanson & Morris, 1976] have led to the same result. In all three N-carbonylsulphilimines the conjugated SNCO moiety is practically planar with the S and carbonyl O atoms being in *cis* arrangement. Since this conformation gives rise to an S$\cdots$O close contact (Kálmán & Párkányi, 1980)

associated with a four-membered ring an accurate structure refinement of DEDAS was performed.

Experimental. The compound was recrystallized from methanol. A crystal $0\cdot4 \times 0\cdot4 \times 0\cdot3$ mm was mounted on an Enraf–Nonius CAD-4 computer-controlled diffractometer. The lattice parameters obtained from precession photographs were refined from the setting angles of 25 reflexions. 2846 independent intensities were collected with graphite-monochromated Mo $K\alpha$ ($\lambda = 0\cdot71073$ Å) radiation. No absorption ($\mu = 0\cdot91$ mm^{-1}) correction was performed.

Anisotropic refinement was started with the parameters obtained for non-hydrogen atoms in the first analysis ($R = 0\cdot103$ for 963 Weissenberg observations). Full-matrix least-squares refinement of 101 variables reduced R to $0\cdot053$ for 2035 reflexions which were above the level $F - 10\sigma(F) > 0$. At this stage all H-atom positions could be located in a difference map. A further three cycles of least-squares adjustment in which H-atom positions were refined gave a final $R = 0\cdot034$ ($R_w = 0\cdot048$) for the 2253 reflexions with the $F - 6\sigma(F) > 0$ criterion ($R_{tot} = 0\cdot056$).* Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). All calculations were per-

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36680 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* ($\times 10^5$ *for non-hydrogen atoms and* $\times 10^3$ *for* H *atoms*)

E.s.d.'s are in parentheses. $B_{eq} = \frac{4}{3} \sum_i \sum_j b_{ij}(\mathbf{a}_i \cdot \mathbf{a}_j)$.

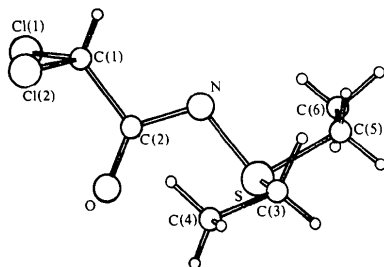|	x	y	z	B_{eq}/B_{iso} (Å^2)
Cl(1)	50074 (7)	3274 (4)	85498 (4)	3·4 (1)
Cl(2)	9185 (9)	537 (5)	87662 (5)	4·4 (2)
S	9144 (5)	21107 (3)	53567 (3)	3·7 (1)
O	24092 (22)	22780 (10)	75497 (10)	3·2 (4)
N	15861 (19)	9628 (11)	61367 (9)	2·1 (3)
C(1)	26885 (24)	1905 (13)	78420 (11)	2·1 (4)
C(2)	22132 (21)	12929 (13)	71497 (11)	1·9 (5)
C(3)	−16932 (24)	19714 (16)	52052 (14)	2·6 (4)
C(4)	−25254 (32)	22146 (21)	62563 (18)	3·7 (2)
C(5)	14228 (24)	15329 (14)	40673 (12)	2·3 (4)
C(6)	35751 (29)	14588 (21)	39867 (17)	3·7 (2)
H(5A)	85 (3)	205 (2)	351 (2)	4·2 (6)
H(5B)	89 (2)	85 (2)	398 (2)	4·6 (9)
H(6A)	413 (3)	101 (2)	448 (2)	6·8 (7)
H(6B)	393 (4)	98 (3)	331 (2)	8·0 (8)
H(6C)	414 (3)	215 (2)	402 (3)	7·4 (8)
H(3A)	−193 (2)	112 (2)	481 (2)	3·6 (7)
H(3B)	−211 (3)	249 (2)	467 (2)	5·9 (7)
H(4A)	−378 (3)	216 (2)	616 (2)	5·9 (7)
H(4B)	−227 (4)	293 (2)	651 (2)	9·3 (10)
H(4C)	−204 (3)	162 (2)	679 (2)	5·4 (7)
H(1)	262 (2)	−51 (2)	755 (1)	2·9 (5)

Table 2. *Bond distances* (Å) *and angles* (°)

	(I)	(II)
S^{IV}−N	1·664 (1)	1·673 (10)
S−C(3)	1·811 (2)	1·837 (13)
S−C(5)	1·800 (1)	1·788 (13)
N−C(2)	1·349 (2)	1·344 (16)
C(2)−O	1·224 (2)	1·212 (16)
C(2)−C(1)	1·536 (2)	1·518 (17)
C(1)−Cl(1)	1·771 (2)	1·795 (12)
C(1)−Cl(2)	1·768 (2)	1·760 (13)
C(3)−C(4)	1·505 (3)	1·494 (21)
C(5)−C(6)	1·511 (3)	1·519 (23)
N−S−C(3)	102·5 (1)	103·8 (5)
N−S−C(5)	99·8 (1)	99·2 (5)
C(3)−S−C(5)	98·8 (1)	99·3 (5)
S−N−C(2)	112·3 (2)	112·6 (8)
N−C(2)−O	130·3 (2)	130·1 (11)
C(1)−C(2)−O	120·1 (2)	121·8 (11)
N−C(2)−C(1)	109·5 (2)	108·0 (10)
S−C(3)−C(4)	110·8 (2)	109·8 (10)
S−C(5)−C(6)	110·9 (2)	111·1 (10)
C(2)−C(1)−Cl(1)	110·3 (2)	109·0 (8)
C(2)−C(1)−Cl(2)	108·1 (2)	107·9 (8)
Cl(1)−C(1)−Cl(2)	109·8 (1)	109·0 (6)

Column (I): obtained from diffractometer data.
Column (II): obtained from film data.

Fig. 1. A view of a molecule of DEDAS with the atomic numbering.

Table 3. *Typical bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *in N-carbonylsulphilimines*

E.s.d.'s are in parentheses; e.s.d.'s for torsion angles in (II) and (III) are <1°. C* represents the carbon atoms of S-ethyl groups taking part in a C−S···O-type interaction.

	(I)	(II)	(III)
S=N	1·664 (1)	1·667 (7)	1·659 (2)
N−C	1·349 (2)	1·320 (10)	1·344 (3)
C=O	1·224 (2)	1·227 (9)	1·246 (3)
S···O	2·838 (1)	2·769 (8)	2·708 (2)
S−N−C	112·3 (3)	110·6 (6)	110·4 (1)
N−C−O	130·3 (2)	130·7 (8)	125·9 (1)
C*−S···O	143·0 (1)	154·7 (6)	155·2 (2)
CSNC	−107·6 (2)	−85·1	87·4
C*SNC	151·1 (2)	172·3	−168·6
SNCO	−2·6 (2)	7·6	−0·5
SNCC	176·5 (2)	−171·6	−179·4

Column (I):   DEDAS; Et₂SNCOCHCl₂; Present work.
Column (II): DMTAS; Me₂SNCOCCl₃; Kálmán, Sasvári & Kucsman (1973).
Column (III): DMBES; Me₂SNCOPh; Cameron, Duncanson & Morris (1976).

formed on a PDP-11/34 minicomputer with the Enraf–Nonius SDP-34 system. The final coordinates for the non-hydrogen and H atoms are given in Table 1. A perspective view of the molecule is given in Fig. 1.

Discussion. The results of the earlier analysis (Kálmán, Sasvári & Kucsman, 1971) have been essentially confirmed (Table 2). Due to π-electron delocalization in the SNCO moiety, the S^{IV}−N bond distance in DEDAS is relatively long [1·664 (1) Å]. The refined bonding parameters in DEDAS are similar to those in DMTAS and DMBES (Table 3). The mean of the S^{IV}−N bond lengths measured for DEDAS and DMBES (1·663 Å) is 0·035 Å longer than that of N-sulphonylsulphilimines (1·628 Å; Kálmán, Párkányi & Kucsman, 1980). A similar value (0·037 Å) was

given earlier by Cameron, Duncanson & Morris (1976).

As shown by the S−N−C−O torsion angles (Table 3), the four-membered SNCO ring is practically planar and the *cis* arrangement of the S^{IV} and carbonyl O atoms pertains to an S···O close contact of 2·708–2·838 Å. This conformation also allows the favourable

trans arrangement for the lone pair of the N atom and the π bond of the C=O group. Data in Table 3 indicate that the geometry of the four-membered SNCO ring closed by an S···O interaction is nearly the same in all three sulphilimines. Nevertheless, the shortest S···O distance (2·708 Å) in DMBES is accompanied by a long C=O bond (1·246 Å) and by a relatively wide C(alkyl)—S···O angle (155·2°), while the longest S···O distance (2·838 Å) in DEDAS is associated with an unusually wide N—C—O bond angle (130·3°) and with the less collinear C(alkyl)—S···O angle (143·0°). Although the C=O bond in DEDAS is not so markedly elongated (1·224 Å), the low ν(CO) data (1622 and 1624 cm⁻¹ in KBr and CHCl₃, respectively), which are not a consequence of a coupled vibration of the SNCO moiety as they exhibit no shifting on ¹⁵N substitution (Kucsman, Ruff & Tanács, 1967), also point out a significant S···O interaction (*cf.* Schmidt & Tulinsky, 1967; Pinel, Mollier, Llaguno & Paul, 1971).

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*N*¹-(3,4-Dimethyl-5-isoxazolyl)sulfanilamide (Sulfisoxazole)

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Abstract. $C_{11}H_{13}N_3O_3S$, $M_r = 267\cdot3$, orthorhombic, *Pbca*, $Z = 8$, $a = 14\cdot917\,(4)$, $b = 14\cdot506\,(4)$, $c = 11\cdot570\,(2)$ Å, $V = 2503\cdot6$ Å³, $D_c = 1\cdot418$, D_m (flotation) $= 1\cdot402$ Mg m⁻³, $F(000) = 1120$, μ(Cu *K*α) $= 2\cdot24$ mm⁻¹. Refinement with 1765 diffractometer data gave a final $R = 0\cdot069$. Estimated standard deviations for bond lengths and angles involving nonhydrogen atoms average 0·005 Å and 0·3° respectively. The crystal structure is stabilized by hydrogen bonds of the types NH···OS and NH···N.

Introduction. Sulfisoxazole [*N*¹-(3,4-dimethyl-5-isoxazolyl)sulfanilamide] is a sulfonamide drug. Its chemical structure is given in Fig. 1. Though less active than sulfadiazine, it is better tolerated because of its high solubility and rapid excretion. Its significant activity against *Proteus vulgaris* and *E. coli* makes it useful in urinary-tract infections. Preliminary results of the crystal structure of this drug have been reported earlier (Chatterjee, Dattagupta & Saha, 1979) and the structure refinement is reported here.
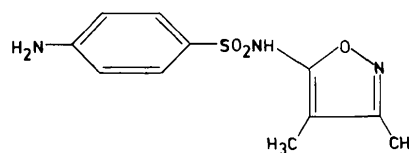

Fig. 1. Sulfisoxazole molecule.

Needle-shaped single crystals of sulfisoxazole were grown by slow evaporation of a solution of the compound in 1:1 water–alcohol mixture at room temperature, and a crystal of dimensions 0·38 × 0·36 × 0·40 mm was used for collecting intensity data. The structure was initially solved by the heavy-atom method (Chatterjee, Dattagupta & Saha, 1979) using visual data. For further refinement of the structure, diffractometric data were collected on an automatic Stoe diffractometer and 1769 reflections were measured using Ni-filtered Cu *K*α radiation. The θ/2θ scan technique was employed and three standard reflections were monitored after each group of 100 measure-