

title compound average $112.7 [2.0]^\circ$. The correction, $-\delta l = 0.018 \delta \Phi$, would require a $\delta \Phi$ of 1.9° to yield a C—C distance of 1.54 \AA , and thus a corrected C—C—C angle of 110.8° . (Applying this same correction to the averaged literature values gives an average angle of 112.4° to correspond to correcting the average observed distance to 1.540 \AA .)

Perhaps the best comparison is between the title compound and (\pm)-hexestrol (Weeks, Pokrywiecki & Duax, 1973) [(3*R*,4*S*)-3,4-bis(*p*-hydroxyphenyl)-hexane]. In hexestrol the central C—C bond distance is $1.553 (6) \text{ \AA}$, the 2,3 and 4,5 C—C bond distances are $1.539 (6)$ and $1.535 (6) \text{ \AA}$, and the terminal 1,2 and 5,6 C—C bond distances are $1.504 (7)$ and $1.512 (8) \text{ \AA}$. The C—C_{ar} distances in hexestrol are $1.522 (6)$ and $1.523 (6) \text{ \AA}$ as compared to $1.511 (3)$ and $1.522 (3) \text{ \AA}$ in the title compound. The C—C—C bond angles in hexestrol average $112.5 [1.2]^\circ$ compared to 112.7° in the title compound. In both hexestrol and the title compounds the phenyl groups have adopted a *gauche* orientation rather than *trans*. The main six-carbon-atom chain can be described as *gauche,trans,trans* in the title compound compared to approximately *trans-trans,trans* in hexestrol.

Finally, the C \equiv N distance [$1.136 (3) \text{ \AA}$] and the C—C_{CN} distance [$1.483 (3) \text{ \AA}$] appear to lie close to the values normally found for these bond lengths.

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4-Cyano-3-hydroxy-6*H*-1,2,6-thiadiazine 1,1-Dioxide

BY C. ESTEBAN-CALDERÓN, M. MARTÍNEZ-RIPOLL AND S. GARCÍA-BLANCO

Departamento de Rayos X, Instituto Rocasolano, Serrano 119, Madrid 6, Spain

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Abstract. C₄H₃N₃O₃S, $M_r = 173.16$, monoclinic, $P2_1/c$, $a = 8.519 (1)$, $b = 5.5024 (2)$, $c = 14.791 (2) \text{ \AA}$, $\beta = 114.50 (1)^\circ$, $U = 630.9 (2) \text{ \AA}^3$, $Z = 4$, $D_c = 1.822 \text{ Mg m}^{-3}$, $\mu(\text{Mo } K\alpha) = 0.462 \text{ mm}^{-1}$. The structure has been refined to $R = 0.043$ for 1331 independent reflexions. The ring presents an envelope conformation, the S atom being at the flap. The molecules are held together by hydrogen bonds.

Introduction. Crystals of the title compound were supplied by Drs Stud and Goya (Instituto de Química Médica, CSIC, Madrid). 1832 unique reflexions were collected up to $\theta = 30^\circ$ at 295 K on a PW 1100 diffractometer equipped with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069 \text{ \AA}$). An $\omega/2\theta$ scan technique was used. No crystal decomposition was detected. An absorption correction was performed with ORABS (Schwarzenbach, 1972). After Lorentz and

polarization corrections, 1331 reflexions were considered observed with $I > 2\sigma(I)$ and were used in the calculations. Scattering factors for neutral atoms and anomalous-dispersion corrections for S were taken from *International Tables for X-ray Crystallography* (1974).

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Anisotropic full-matrix refinement of non-H atoms with unit weights led to $R = 0.050$. The H atoms were located on a difference synthesis calculated for reflexions with $\sin \theta/\lambda < 0.5 \text{ \AA}^{-1}$. A good weighting scheme to prevent bias on $\langle \Delta F \rangle$ vs $\langle F_o \rangle$ or $\langle \sin \theta/\lambda \rangle$ was $w = w_1 w_2$, where $w_1 = K/\sigma_1^2$ and $w_2 = 1/\sigma_2^2$ with $\sigma_1 = a + b|F_o|$ and $\sigma_2^2 = c + d \sin \theta/\lambda$ (coefficients given in Table 1), calculated with *PESOS* (Martínez-Ripoll & Cano, 1975). Final refinement, with isotropic temperature factors for H atoms, gave $R = 0.043$ and $R_w =$

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$(\sum w\Delta^2 F / \sum w|F_o|^2)^{1/2} = 0.041$. The final difference synthesis had no electron density $>0.2 \text{ e } \text{Å}^{-3}$. Table 2 shows the final atomic parameters.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34622 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coefficients for the weighting scheme*

	<i>a</i>	<i>b</i>		<i>c</i>	<i>d</i>
$F_o \leq 9.00$	1.17	-0.90	$\sin \theta/\lambda \leq 0.46 \text{ Å}^{-1}$	4.23	-8.62
$F_o > 9.00$	0.33	0.01	$\sin \theta/\lambda > 0.46$	-0.39	2.13
$K = 0.60$					

Table 2. *Atomic positional parameters* ($\times 10^5$, for $H \times 10^3$) for $C_4H_3N_3O_3S$

	<i>x</i>	<i>y</i>	<i>z</i>
S	19409 (7)	34640 (10)	70275 (3)
C(1)	34116 (28)	70541 (41)	82846 (16)
C(2)	29039 (27)	60835 (38)	89648 (15)
C(3)	18688 (29)	39273 (39)	87288 (16)
C(4)	33865 (30)	72030 (42)	99095 (17)
N(1)	29906 (28)	60861 (40)	73897 (15)
N(2)	13635 (28)	27676 (37)	78868 (14)
N(3)	38054 (33)	80776 (49)	106753 (17)
O(1)	31161 (23)	17098 (37)	69647 (13)
O(2)	4620 (21)	39567 (32)	61236 (11)
O(3)	14101 (27)	31538 (41)	94200 (13)
H(C1)	405 (4)	843 (6)	839 (2)
H(N1)	332 (4)	672 (7)	699 (2)
H(O3)	89 (4)	195 (7)	924 (2)

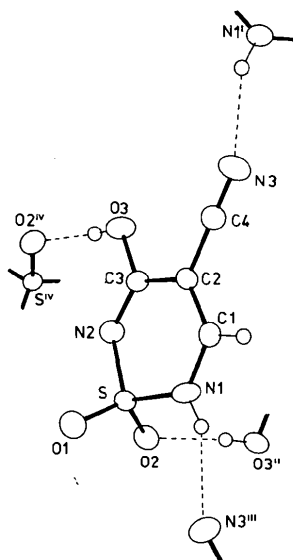


Fig. 1. Perspective drawing (Johnson, 1965) of the molecule. Some symmetrically related atoms are also shown. Dashed lines represent hydrogen bonds. For symmetry-operation codes see Table 6.

Discussion. The study of this compound forms part of a research project of Goya & Stud (1978) aimed at obtaining potential antimetabolites.

Fig. 1 is a perspective drawing of the molecule. Considerable electron delocalization within the ring can be deduced from an inspection of the bond distances and angles (Table 3). The length of S—N(1), 1.665 Å, is close to the value for a S—N single bond (Gillespie, Ireland & Vekris, 1975); the distance S—N(2), 1.588 Å, implies a bond order of ~ 1.5 . The S—O lengths are typical for most thiadiazine compounds (Cabezuelo, Cano, Foces-Foces & Garcia-Blanco, 1977, and references therein). C(3)—O(3), 1.308 Å, lies between the accepted values of double and single bonds, and consequently a bond order of ~ 1.5 can be assigned. From the above-mentioned consideration and from the distribution of bond lengths among the remaining

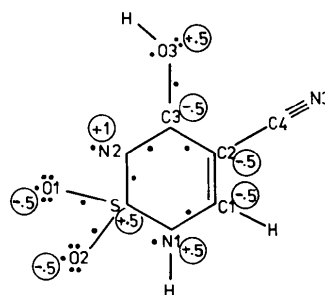


Fig. 2. Major electron distribution.

Table 3. *Bond lengths* (Å) *and angles* ($^\circ$)

E.s.d.'s are in parentheses.

S—O(1)	1.421 (2)	O(1)—S—O(2)	116.0 (1)
—O(2)	1.432 (1)	O(1)—S—N(1)	108.1 (1)
—N(1)	1.665 (2)	O(1)—S—N(2)	110.4 (1)
—N(2)	1.588 (3)	O(2)—S—N(1)	106.5 (1)
N(1)—C(1)	1.331 (3)	O(2)—S—N(2)	110.4 (1)
—H	0.83 (5)	N(1)—S—N(2)	104.8 (1)
N(2)—C(3)	1.303 (3)	S—N(1)—C(1)	123.7 (2)
N(3)—C(4)	1.143 (3)	S—N(1)—H	116 (3)
C(1)—C(2)	1.358 (4)	C(1)—N(1)—H	120 (3)
—H	0.91 (4)	S—N(2)—C(3)	123.9 (2)
C(2)—C(3)	1.432 (3)	N(1)—C(1)—C(2)	122.8 (2)
—C(4)	1.423 (3)	N(1)—C(1)—H	114 (2)
C(3)—O(3)	1.308 (4)	C(2)—C(1)—H	123 (2)
O(3)—H	0.78 (4)	C(1)—C(2)—C(3)	119.9 (2)
		C(3)—C(2)—C(4)	120.4 (2)
		C(3)—C(2)—C(4)	119.7 (2)
		N(2)—C(3)—C(2)	124.4 (3)
		N(2)—C(3)—O(3)	120.0 (2)
		C(2)—C(3)—O(3)	115.6 (2)
		C(3)—O(3)—H	109 (3)
		C(2)—C(4)—N(3)	178.6 (3)

Table 4. *Torsion angles* ($^{\circ}$)

E.s.d.'s are in parentheses.

N(2)—S—N(1)—C(1)	8.7 (2)
S—N(1)—C(1)—C(2)	-5.3 (3)
N(1)—C(1)—C(2)—C(3)	-0.1 (3)
C(1)—C(2)—C(3)—N(2)	0.5 (3)
C(2)—C(3)—N(2)—S	4.8 (3)
C(3)—N(2)—S—N(1)	-8.4 (2)
O(3)—C(3)—N(2)—S	-176.2 (2)
O(3)—C(3)—C(2)—C(4)	1.1 (3)
O(1)—S—N(1)—C(1)	-109.0 (2)
C(3)—N(2)—S—O(2)	-122.6 (2)
O(2)—S—N(1)—C(1)	125.7 (2)

Table 5. *Atomic deviations* (\AA) *from least-squares planes* (Nardelli, Musatti, Domiano & Andreetti, 1965)

Atoms marked with asterisks are not included in the calculation.
E.s.d.'s are in parentheses.

Plane 1		Plane 2	
C(1)	-0.001 (3)	O(1)	0.000 (1)
C(2)	-0.001 (3)	O(2)	0.000 (2)
C(3)	0.003 (3)	S	0.000 (2)
N(1)	0.001 (3)		
N(2)	-0.002 (3)	The angle between planes 1 and 2 = 90.23° .	
*S	-0.124 (2)		
*C(4)	0.002 (3)		
*N(3)	-0.023 (3)		
*O(3)	0.032 (3)		

atoms of the molecule (Table 3), we propose the electron distribution shown in Fig. 2 according to Linnett's (1966) model.

Table 4 shows a list of torsion angles. The ring can be described as envelope 2,3-diplanar according to the six-ring conformational model (Cano, Foces-Foces & Garcia-Blanco, 1977) (see also Table 5).

The molecules are held together through hydrogen bonds of types N(1)—H...N(3) and O(3)—H...O(2) (Fig. 1). Their geometrical features are listed in Table 6, where some other contacts are also given. The remaining intermolecular contacts are equal to or greater than the sum of the corresponding van der Waals radii.

Most computations were performed with XRAY 70 (Stewart, Kundell & Baldwin, 1970). Thanks are due to

Table 6. *Intermolecular contacts*

E.s.d.'s are in parentheses.

$X-H\cdots Y$	$X\cdots Y$	$X-H$	$H\cdots Y$	$\angle X-H\cdots Y$
Hydrogen bonds				
N(1)—H...N(3) ^{III}	2.930 (4) \AA	0.83 (5) \AA	2.16 (5) \AA	156 (4) $^{\circ}$
O(3)—H...O(2) ^{IV}	2.731 (3)	0.78 (4)	1.96 (4)	176 (5)
Other contacts				
C(1)—H...O(1) ^V	3.135 (4)	0.91 (4)	2.84 (4)	100 (3)
C(1)—H...O(1) ^{VI}	3.167 (3)	0.91 (4)	2.64 (4)	118 (2)
C(4)...O(2) ^{II}	3.140 (3)			
Symmetry operations				
(I)	$x, 1.5 - y,$	$0.5 + z$	(IV)	$-x, -0.5 + y, 1.5 - z$
(II)	$-x, 0.5 + y,$	$1.5 - z$	(V)	$1 - x, 0.5 + y, 1.5 - z$
(III)	$x, 1.5 - y,$	$-0.5 + z$	(VI)	$x, 1 + y, z$

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