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### 7-Amino-2,4-dimethyl-2*H*,4*H*-1,2,3-triazolo[4,5-*c*] [1,2,6]thiadiazine 5,5-Dioxide

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**Abstract.** C<sub>7</sub>H<sub>8</sub>N<sub>6</sub>O<sub>2</sub>S, *M<sub>r</sub>* = 216.22, triclinic, *P*1̄, *Z* = 2, *a* = 7.430 (1), *b* = 8.639 (1), *c* = 7.357 (1) Å, α = 105.24 (1), β = 103.12 (1), γ = 76.55 (1)°, *U* = 436.2 (1) Å<sup>3</sup>, *D<sub>c</sub>* = 1.65 Mg m<sup>-3</sup>, μ(Mo *K*α) = 0.340 mm<sup>-1</sup>. The structure was refined to *R* = 0.046 for 1402 observed reflexions. The thiadiazine ring was found in the expected envelope conformation. A considerable electron delocalization is detected in the molecular rings. The crystal packing is mainly due to N–H···O and N–H···N hydrogen bonds.

**Introduction.** This compound has been prepared by Drs Goya, Ochoa and Stud (Inst. Química Médica, CSIC, Madrid) as a part of a research project on heterocyclic compounds containing the thiadiazine group, aimed at obtaining substances with biological activity. The crystal structures of other closely related heterocycles have been reported recently (Foces-Foces, Cano & García-Blanco, 1975*a,b*; Foces-Foces, Cano, García-Blanco & Rodríguez, 1975; Smith-Verdier, Florencio, García-Blanco & Rodríguez, 1977).

A colourless single crystal of dimensions 0.08 × 0.09 × 0.13 mm was used to collect the intensities of 2543 independent reflexions up to θ = 30°. A four-circle diffractometer, equipped with Mo *K*α radiation (λ = 0.71069 Å), and an ω/2θ scan

technique were used. An absorption correction was made using the *ORABS* program (Schwarzenbach, 1972). 1402 reflexions were considered as observed by the criterion *I* > 2σ(*I*) and used in the remaining calculations.

The structure was solved by the multiresolution tangent-formula program *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and successive Fourier syntheses. Scattering factors for neutral atoms and anomalous-dispersion corrections for S were taken from *International Tables for X-ray Crystallography* (1974). The H atoms were located on a difference map calculated after least-squares refinement. The last cycles of anisotropic refinement (isotropically fixed H atoms) were performed using a weighting scheme of type *w* = *w*<sub>1</sub>·*w*<sub>2</sub>, where *w*<sub>1</sub> = 1/(*a* + *b*|*F*<sub>o</sub>|)<sup>2</sup> and *w*<sub>2</sub> = 1/(*c* + *d* sin θ/λ). Final disagreement indices are *R* = 0.046 and *R<sub>w</sub>* = 0.048. Table 1 shows the atomic parameters.\*

\* Lists of coefficients for the weighting scheme, structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36798 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )
$$U_{eq} = \frac{1}{3} \sum_i \sum_j a_i^* a_j^* (a_i \cdot a_j).$$

	x	y	z	$U_{eq}/U$
N(1)	0.0539 (3)	0.1565 (3)	0.1829 (4)	30 (1)
N(2)	0.2235 (3)	0.1220 (3)	0.1415 (3)	30 (1)
N(3)	0.2454 (3)	0.1751 (3)	-0.0077 (3)	30 (1)
N(4)	0.0185 (4)	0.3349 (4)	-0.2095 (3)	33 (1)
S(5)	-0.2123 (1)	0.3684 (1)	-0.2942 (1)	30 (0)
O(51)	-0.2555 (3)	0.2306 (3)	-0.4422 (3)	43 (1)
O(52)	-0.2545 (4)	0.5201 (3)	-0.3521 (4)	46 (1)
N(6)	-0.3148 (4)	0.3887 (4)	-0.1180 (4)	37 (1)
C(7)	-0.2416 (4)	0.3146 (4)	0.0263 (4)	30 (1)
C(8)	-0.0476 (4)	0.2384 (4)	0.0493 (4)	27 (1)
C(9)	0.0718 (4)	0.2495 (4)	-0.0651 (4)	28 (1)
N(10)	-0.3463 (4)	0.3183 (4)	0.1514 (4)	41 (1)
C(11)	0.3854 (5)	0.0457 (5)	0.2613 (6)	40 (1)
C(12)	0.1475 (6)	0.3126 (7)	-0.3431 (6)	50 (2)
H(101)	-0.460 (10)	0.370 (8)	0.140 (9)	63
H(102)	-0.292 (9)	0.278 (8)	0.255 (8)	63
H(111)	0.441 (9)	0.131 (8)	0.348 (9)	63
H(112)	0.340 (8)	-0.013 (8)	0.328 (9)	63
H(113)	0.471 (9)	-0.015 (8)	0.192 (9)	63
H(121)	0.099 (9)	0.381 (8)	-0.427 (9)	63
H(122)	0.154 (9)	0.204 (8)	-0.415 (10)	63
H(123)	0.271 (9)	0.325 (8)	-0.270 (9)	63

**Discussion.** Fig. 1 shows a perspective drawing of the molecule with the nearest neighbouring atoms. Table 2 shows the atomic deviations from mean planes. It can be seen that the five-membered ring is clearly a plane (plane 1). The thiadiazine ring (plane 2) exhibits an envelope conformation, the S atom being at the flap, as commonly occurs in similar compounds (Foces-Foces, Cano & Garcia-Blanco, 1975*a,b*; Foces-Foces, Cano, Garcia-Blanco & Rodriguez, 1975; Smith-Verdier, Florencio, Garcia-Blanco & Rodriguez, 1977).

A certain electronic delocalization within the rings can be derived from an observation of the bond lengths (Table 3), as previously reported for 7-amino-2*H*,4*H*-1,2,3-triazolo[4,5-*c*][1,2,6]thiadiazine 5,5-dioxide (ATT) and its monohydrate (ATTM) (Foces-Foces,

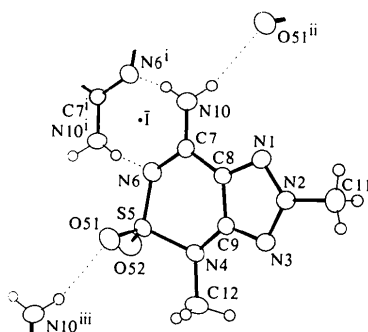


Fig. 1. Perspective drawing (Johnson, 1965). Some symmetrically related atoms are also shown. Dashed lines represent hydrogen bonds. For symmetry-operation codes see Table 4. Thermal ellipsoids are scaled to 50% probability.

Table 2. Atomic deviations ( $\text{\AA}$ ) from least-squares planes (Nardelli, Musatti, Domiano & Andreotti, 1965)

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

Plane 1		Plane 2	
N(1)	-0.003 (3)	N(4)	-0.022 (3)
N(2)	0.002 (3)	N(6)	0.030 (3)
N(3)	-0.000 (3)	C(7)	-0.040 (3)
C(8)	0.004 (3)	C(8)	0.009 (3)
C(9)	-0.002 (3)	C(9)	0.023 (3)
*S(5)	0.375 (1)	*S(5)	0.461 (1)
*N(4)	-0.086 (3)	*O(51)	1.881 (3)
*N(6)	-0.030 (3)	*O(52)	-0.292 (3)
*C(7)	-0.063 (3)	*N(10)	-0.211 (3)
*N(10)	-0.214 (4)	*C(12)	0.247 (5)
*C(12)	0.615 (5)	*N(1)	-0.034 (3)
*C(11)	-0.127 (4)	*N(2)	-0.029 (3)
		*N(3)	0.002 (3)
		*C(11)	-0.192 (4)

Angle between planes 1/2 = 1.6 (3)°.

Table 3. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

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

N(1)-N(2)	1.312 (4)	C(7)-C(8)	1.431 (4)
N(1)-C(8)	1.348 (4)	C(7)-N(10)	1.323 (5)
N(2)-N(3)	1.350 (4)	C(8)-C(9)	1.388 (5)
N(2)-C(11)	1.454 (4)	N(10)-H(101)	0.86 (7)
N(3)-C(9)	1.328 (4)	N(10)-H(102)	0.90 (7)
N(4)-S(5)	1.671 (3)	C(11)-H(111)	0.95 (7)
N(4)-C(9)	1.384 (5)	C(11)-H(112)	0.95 (8)
N(4)-C(12)	1.471 (6)	C(11)-H(113)	0.89 (7)
S(5)-O(51)	1.430 (2)	C(12)-H(121)	0.93 (7)
S(5)-O(52)	1.428 (3)	C(12)-H(122)	0.94 (6)
S(5)-N(6)	1.599 (3)	C(12)-H(123)	0.97 (6)
N(6)-C(7)	1.329 (4)		
N(2)-N(1)-C(8)	102.8 (3)	C(9)-C(8)-N(1)	108.4 (3)
N(1)-N(2)-N(3)	116.9 (2)	C(8)-C(9)-N(4)	124.4 (3)
N(1)-N(2)-C(11)	122.1 (3)	C(8)-C(9)-N(3)	110.1 (3)
N(3)-N(2)-C(11)	120.6 (3)	N(3)-C(9)-N(4)	125.4 (3)
N(2)-N(3)-C(9)	101.8 (3)	H(101)-N(10)-H(102)	121 (7)
S(5)-N(4)-C(9)	114.7 (3)	H(101)-N(10)-C(7)	120 (5)
S(5)-N(4)-C(12)	117.9 (2)	H(102)-N(10)-C(7)	119 (5)
C(9)-N(4)-C(12)	118.9 (3)	H(111)-C(11)-H(112)	110 (6)
N(4)-S(5)-N(6)	106.4 (1)	H(111)-C(11)-H(113)	107 (6)
N(4)-S(5)-O(51)	108.5 (1)	H(112)-C(11)-H(113)	114 (6)
N(4)-S(5)-O(52)	106.4 (2)	H(111)-C(11)-N(2)	107 (4)
O(51)-S(5)-O(52)	115.4 (2)	H(112)-C(11)-N(2)	107 (3)
N(6)-S(5)-O(51)	110.9 (2)	H(113)-C(11)-N(2)	111 (4)
N(6)-S(5)-O(52)	108.8 (2)	H(121)-C(12)-H(122)	107 (6)
S(5)-N(6)-C(7)	123.7 (2)	H(121)-C(12)-H(123)	115 (6)
N(6)-C(7)-C(8)	119.4 (3)	H(122)-C(12)-H(123)	108 (5)
N(6)-C(7)-N(10)	119.8 (3)	H(121)-C(12)-N(4)	110 (4)
C(8)-C(7)-N(10)	120.8 (3)	H(122)-C(12)-N(4)	107 (5)
C(7)-C(8)-C(9)	122.3 (3)	H(123)-C(12)-N(4)	109 (4)
C(7)-C(8)-N(1)	129.2 (3)		

Cano & Garcia-Blanco, 1975*a,b*). Several half-normal probability plots (Abrahams & Keve, 1971) have been calculated in order to compare the structure of the present compound with those reported for ATT and ATTM. Excluding the significant differences of S(5)-N(4) and S(5)-N(6), the plots are quite linear,

with correlation coefficients of 0.96 (in comparison with ATT) and 0.98 (ATTM), the slope of both suggesting an underestimation factor of 1.9 in the e.s.d.'s.

The significant difference between the two S–N distances could be due to some degree of polarization, leaving the S atom with an electronic deficiency. This could be neutralized by delocalization of the  $\pi$ -electron pair of the N(6)–C(7) double bond, leaving the C(7) atom with a deficiency of charge, which also could be neutralized by a localization of positive charge on N(10). This hypothesis could explain the planarity around N(10). The significant deviation from planarity of the atoms around N(4) (sum of bond angles = 351.5°) could be explained by assuming the existence of an electron pair on this atom. On the other hand, the sum of bond angles around N(2) (359.6°) could indicate no electron-pair localization on this atom. From all these observations, and taking into account the remaining bond lengths observed, one can give an approximate model for the major electron distribution in the molecule as shown in Fig. 2.

The molecular packing in the crystal involves the formation of hydrogen bonds in which the amino group, N(10), plays a dominant role (dashed lines in Fig. 1). The N(10)–H...O(51) bonds form infinite chains in the *c* direction. The N(10)–H...N(6) bonds form dimeric units around crystallographic symmetry centres. The geometrical features of the hydrogen bonds are given in Table 4, where other intermolecular contacts are also shown.

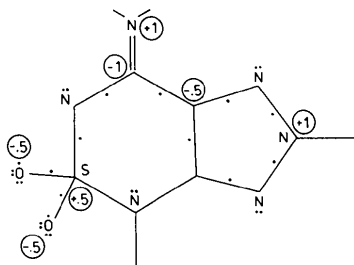


Fig. 2. Major electron-distribution model according to Linnett (1966).

Table 4. *Intermolecular contacts*

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

$X-H \cdots Y$	$X \cdots Y$	$X-H$	$H \cdots Y$	$X-H \cdots Y$
Hydrogen bonds				
N(10)–H(101)⋯N(6) <sup>(i)</sup>	3.145 (4) Å	0.86 (7) Å	2.37 (6) Å	151 (7)°
N(10)–H(102)⋯O(51) <sup>(ii)</sup>	3.163 (4)	0.90 (7)	2.31 (8)	159 (6)
Other contacts				
C(12)–H(121)⋯O(52) <sup>(iii)</sup>	3.286 (7)	0.93 (7)	2.60 (8)	132 (5)

Symmetry-operation codes: (i)  $-1 - x, 1 - y, -z$ ; (ii)  $x, y, 1 + z$ ; (iii)  $x, y, -1 + z$ ; (iv)  $-x, 1 - y, -1 - z$ .

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