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The Monopotassium Salt of 2*H*-1,2,6-Thiadiazine-3,5(4*H*,6*H*)-dione 1,1-Dioxide Monohydrate

By M. D. Cabezuelo, C. Foces-Foces, F. H. Cano and S. García-Blanco

Departamento de Rayos X, Instituto de Química-Física 'Rocasolano', Serrano 119, *Madrid*-6, *Spain*

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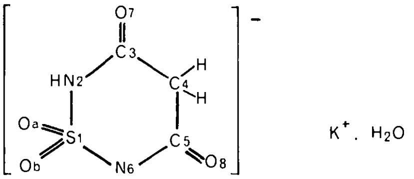
Abstract. $C_3H_3N_2O_4SK.H_2O$, monoclinic, *Cm*, $a = 9{\cdot}3743$ (3), $b = 15{\cdot}7092$ (6), $c = 5{\cdot}3693$ (1) Å, $\beta = 104{\cdot}47$ (2)°, $Z = 4$, $V = 765{\cdot}61$ (3) Å^3, $M_r = 220{\cdot}2$, $D_x = 1{\cdot}91$ g cm^{-3}. The structure was solved by Patterson and Fourier syntheses and refined by least-squares methods to $R = 0{\cdot}024$ for 1145 observed reflections. In the unit cell there are two K cations, eight and seven coordinating respectively. The thiadiazine ring has a flattened boat conformation.

Introduction. This compound, $K^+.C_3H_3N_2O_4S^-.H_2O$ (I), is one of a series of *S*-dioxo analogues of uracil and barbituric acid (Ochoa & Stud, 1978). The structure has been determined to eliminate doubts about its conformation.



(I)

Intensity data were collected from a crystal of dimensions 0·21 × 0·11 × 0·29 mm on a Philips PW 1100 four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å). The ω–2θ scan technique was employed. Of the 1166 independent reflections, 1145 had $I > 2\sigma(I)$. Lorentz, polarization and absorption corrections were applied ($\mu = 9{\cdot}38$ cm^{-1}). The statistical distribution of intensities tended to eliminate the centrosymmetric space group *C*2/*m*; the concentration of peaks only in the Harker sections $(0,v,0)$ and $(\frac{1}{2},v,0)$ and the subsequent structure solution confirmed space group *Cm*. The structure was solved by Patterson and Fourier syntheses and refined by full-matrix least-squares calculations. H atoms were found in a difference map and included in the final refinement. The weighting scheme was: $w = K/|f(F_o)|^2 f(S)$ with $f(F_o) = 0{\cdot}53 - 0{\cdot}03F_o$ for $F_o < 25$ and $f(F_o) = 0{\cdot}83 - 0{\cdot}03F_o$ otherwise and $f(S) = 12{\cdot}01 - 52{\cdot}61S + 61{\cdot}72S^2$ for $S < 0{\cdot}48$ and $f(S) = 10{\cdot}58 - 34{\cdot}30S + 29{\cdot}67S^2$ otherwise, with $K = 0{\cdot}586$ and $S = \sin\theta/\lambda$. Both R and R_w for the observed reflections were 0·024, where $R_w = (\sum w\Delta^2/\sum w|F_o|^2)^{1/2}$. There were no peaks larger than 0·42 e Å^{-3} in the final difference map. Table 1 lists the fractional coordinates.[*] Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. Table 2 contains the main molecular parameters. Distances and angles in the ring are consistent with values reported for other thiadiazine rings, *e.g.* Foces-Foces, Cano & García-Blanco (1975*a*, *b*); Smith-Verdier, Florencio & García-Blanco (1977); Cabezuelo, Cano, Foces-Foces & García-Blanco (1977); Foces-Foces, Smith-Verdier, Florencio-Sabaté & García-Blanco (1975). The packing and the numbering are illustrated in Fig. 1, which shows that the cations have two different geometrical environments, making use of atoms in symmetry-related molecules. The K(1) cation is surrounded by eight atoms which form a distorted *mm* (C_{2v}) hendeca-

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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 33639 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates*

	x	*y*	*z*
S(1)	0	0·15937 (2)	0
K(1)	0·3710 (1)	0	0·3204 (2)
K(2)	−0·1807 (1)	0	0·4094 (1)
N(2)	−0·0034 (2)	0·2652 (1)	−0·0248 (3)
C(3)	0·0705 (2)	0·3163 (1)	0·1698 (3)
C(4)	0·1558 (2)	0·2717 (1)	0·4081 (3)
C(5)	0·2236 (2)	0·1872 (1)	0·3663 (3)
N(6)	0·1577 (2)	0·1366 (1)	0·1707 (3)
O(7)	0·0673 (2)	0·3939 (1)	0·1499 (3)
O(8)	0·3426 (2)	0·1660 (1)	0·5145 (3)
O(*a*)	−0·0152 (2)	0·1285 (1)	−0·2571 (3)
O(*b*)	−0·1160 (2)	0·1350 (1)	0·1164 (4)
O(*W*1)	−0·3708 (4)	0	−0·2634 (6)
O(*W*2)	0·2405 (5)	0	−0·2544 (8)
H(2)	−0·058 (4)	0·290 (2)	−0·164 (7)
H(4*a*)	0·079 (5)	0·258 (3)	0·501 (8)
H(4*b*)	0·233 (5)	0·311 (3)	0·496 (9)
H(*W*1)	−0·377 (7)	0·047 (4)	−0·171 (13)
H(*W*2)	0·213 (6)	0·052 (4)	−0·197 (10)

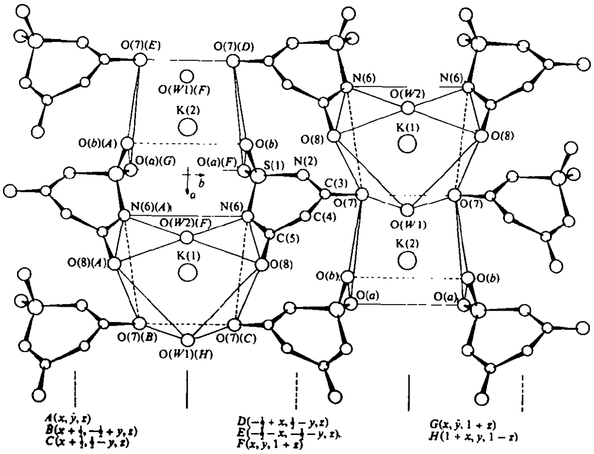

Fig. 1. The molecular structure and packing viewed along the *c* axis.

Table 2. *Molecular dimensions: distances* (Å), *angles and intra-annular torsion angles* (°)

The symmetry transformations are given in Fig. 1.

S(1)–O(*a*)	1·436 (2)	O(*a*)–S(1)–O(*b*)	115·1 (1)
S(1)–O(*b*)	1·435 (2)	O(*a*)–S(1)–N(2)	105·3 (1)
S(1)–N(2)	1·667 (2)	O(*a*)–S(1)–N(6)	109·7 (1)
N(2)–C(3)	1·362 (2)	O(*b*)–S(1)–N(2)	107·4 (1)
C(3)–C(4)	1·502 (2)	O(*b*)–S(1)–N(6)	112·9 (1)
C(3)–O(7)	1·224 (2)	N(2)–S(1)–N(6)	105·6 (1)
C(4)–C(5)	1·513 (3)	S(1)–N(2)–C(3)	122·1 (1)
C(5)–N(6)	1·338 (2)	N(2)–C(3)–C(4)	116·1 (2)
C(5)–O(8)	1·243 (2)	N(2)–C(3)–O(7)	121·6 (2)
N(6)–S(1)	1·574 (2)	C(4)–C(3)–O(7)	122·4 (2)
N(2)–H(2)	0·89 (3)	C(3)–C(4)–C(5)	115·9 (2)
C(4)–H(4*a*)	0·99 (5)	C(4)–C(5)–N(6)	120·6 (2)
C(4)–H(4*b*)	0·98 (5)	C(4)–C(5)–O(8)	118·9 (2)
		N(6)–C(5)–O(8)	120·5 (2)
		C(5)–N(6)–S(1)	120·3 (2)

K(1)–N(6)		2·906 (2)	K(2)–O(*a*)	(*F*)	2·881 (2)
K(1)–N(6)	(*A*)	2·906 (2)	K(2)–O(*a*)	(*G*)	2·881 (2)
K(1)–O(7)	(*B*)	2·801 (2)	K(2)–O(*b*)		2·796 (2)
K(1)–O(7)	(*C*)	2·801 (2)	K(2)–O(*b*)	(*A*)	2·796 (2)
K(1)–O(8)		2·845 (2)	K(2)–O(7)	(*D*)	2·942 (2)
K(1)–O(8)	(*A*)	2·845 (2)	K(2)–O(7)	(*E*)	2·942 (2)
K(1)–O(*W*1)	(*A*)	2·855 (3)	K(2)–O(*W*1)	(*F*)	2·797 (4)
K(1)–O(*W*2)	(*F*)	2·846 (5)			

S(1)–N(2)–C(3)–C(4)	0·1 (3)
N(2)–C(3)–C(4)–C(5)	33·9 (3)
C(3)–C(4)–C(5)–N(6)	−31·9 (3)
C(4)–C(5)–N(6)–S(1)	−6·9 (3)
C(5)–N(6)–S(1)–N(2)	36·4 (2)
N(6)–S(1)–N(2)–C(3)	−33·5 (2)

hedron (Lippard, 1967; Rosenzweig & Cromer, 1970). K(2) is at the centre of a monocapped face of a trigonal prism formed by seven O atoms (Drew, 1977; Andersen, Klewe & Thom, 1967). There is an intermolecular hydrogen bond N(2)–H(2)···O(8) with the N(2)–O(8) distance and N(2)–H(2)···O(8) angle 2·755 (2) Å and 171 (4)° respectively.

In the present work, the conformation of the ring can be considered as a flattened boat, whereas the thiadiazine ring exhibits an envelope conformation in the above mentioned literature.

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