Table 2. *Selected intermolecular and intramolecular bond lengths (Å) and angles (°)*

MgA–W(1)A	2·079 (12)	MgB–W(1)B	2·065 (17)	
MgA–W(2)A	2·118 (15)	MgB–W(2)B	2·124 (14)	
MgA–W(3)A	2·034 (15)	MgB–W(3)B	2·053 (13)	
W(1)A–MgA–W(2)A	91·1 (5)	W(1)B–MgB–W(2)B	88·2 (7)	
W(1)A–MgA–W(3)A	91·0 (5)	W(1)B–MgB–W(3)B	88·3 (7)	
W(2)A–MgA–W(3)A	88·4 (5)	W(2)B–MgB–W(3)B	91·6 (6)	
Cd–W(4)	2·246 (9)	Cd–O(16)	2·370 (9)	
Cd–N(8)	2·382 (9)	Cd–O(12)	2·457 (7)	
Cd–N(11)	2·414 (7)	Cd–O(20)	2·336 (6)	
		Cd–O(24)	2·396 (9)	
N(8)–Cd–W(4)	132·8 (5)	O(12)–Cd–W(4)	81·6 (4)	
N(8)–Cd–N(11)	76·2 (3)	O(12)–Cd–O(16)	109·6 (3)	
N(8)–Cd–O(16)	70·6 (3)	O(12)–Cd–O(20)	78·4 (2)	
N(8)–Cd–O(12)	68·3 (3)	O(12)–Cd–O(24)	159·9 (30)	
N(8)–Cd–O(20)	102·4 (3)	O(16)–Cd–W(4)	87·6 (4)	
N(8)–Cd–O(24)	131·7 (5)	O(16)–Cd–O(20)	165·1 (2)	
N(11)–Cd–W(4)	148·6 (4)	O(16)–Cd–O(24)	80·4 (3)	
N(11)–Cd–O(12)	126·8 (2)	O(20)–Cd–W(4)	106·3 (4)	
N(11)–Cd–O(16)	93·6 (3)	O(2)–Cd–O(24)	96·0 (3)	
N(11)–Cd–O(20)	71·8 (2)	O(24)–Cd–W(4)	81·5 (4)	
N(11)–Cd–O(24)	67·8 (3)			
W(1)A[i]···O(12)	2·707 (12)	W(6)[iv]···W(1)B	3·086 (14)	
W(1)A[ii]···O(22)	2·759 (12)	W(6)[i]···O(24)	2·612 (13)	
W(2)A[iii]···O(14)	2·711 (15)	W(7)[ix]···O(26)	2·935 (14)	
W(2)A[iv]···O(14)	2·938 (15)	W(7)[v]···W(6)	2·813 (14)	
W(3)A[v]···W(5)	2·721 (15)	W(1)B[x]···O(26)	2·752 (15)	
W(3)[iv]···W(5)	2·748 (15)	W(1)B[xi]···O(26)	2·730 (15)	
W(4)[vi]···O(18)	2·677 (13)	W(2)B[x]···O(16)	2·734 (15)	
W(4)[vii]···O(22)	2·686 (13)	W(2)B[i]···W(7)	2·898 (15)	
W(5)[viii]···O(20)	2·682 (15)	W(3)B[x]···O(18)	2·721 (13)	
W(5)[iv]···O(14)	2·799 (15)	W(3)B[iv]···W(7)	2·934 (14)	

Symmetry code: (i) $1-x,1-y,-z$; (ii) $1-x,2-y,-z$; (iii) $x,y,z-1$; (iv) $1-x,1-y,1-z$; (v) $x,y-1,z$; (vi) $x,y,z+1$; (vii) $x,1+y,z$; (viii) $x,1+y,z-1$; (ix) $1+x,y,z$; (x) x,y,z; (xi) $2-x,2-y,1-z$.

The role of each water molecule linked to an Mg^{2+} ion in the molecular packing is different. The water molecules of cation *B* donate protons with the same geometry as those observed in the cation of the previously mentioned Mg–Mg crystal structure (Passer *et al.*, 1977), while in cation *A*, $W(3)A$ acts as donor to two hydrate molecules and $W(1)A$ to a coordinated O and a non-coordinated O of the EDTA ligand.

The geometry of the anion is more complex. The Cd atom is heptacoordinated by four O atoms and two N

atoms of the EDTA ligand, the coordination being completed by a water molecule. These coordinating atoms lie at the vertices of a pentagonal bipyramid. This coordination is similar to those obtained in other heptacoordinated EDTA complexes (Richards, Pedersen, Silverton & Hoard, 1964; Lind, Hamor, Hamor & Hoard, 1964; Stezowski, Countryman & Hoard, 1973; Passer *et al.*, 1977; Solans *et al.*, 1983b) which changes from the monocapped trigonal prism to the pentagonal bipyramid.

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Monopotassium Salt of 3-Hydroxy-2-methyl-4-nitro-2H-1,2,6-thiadiazine 1,1-Dioxide, $C_4H_4N_3O_5S^-.K^+$

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Abstract. $M_r = 245·27$, triclinic, $P\bar{1}$, $a = 8·933$ (6), $b = 7·645$ (5), $c = 7·168$ (7) Å, $\alpha = 110·7$ (1), $\beta = 106·3$ (1), $\gamma = 70·3$ (1)°, $V = 424·0$ (5) Å³, $Z = 2$, $D_x = 1·92$ Mg m⁻³, $\lambda(Mo\,K\alpha) = 0·71069$ Å, $\mu =$

0·857 mm^{-1}, $F(000) = 248$. Final $R = 0·057$ for 1120 observed reflexions. The observed K—O distances are 5·6% elongated with respect to the expected ones. This fact could be related to the different cationic changeability shown by the potassium and sodium salts. The cationic interaction with the bidentate ligand does not seem to be the reason for the irreversible binding of the sodium compounds.

Introduction. As reported by Goya & Stud (1978), some 4-substituted 1,2,6-thiadiazine 1,1-dioxides show a great tendency to bind alkaline ions. The crystal structures of several compounds related with that binding feature have been published recently. Thus, 4NO2 (Esteban-Calderón, Martínez-Ripoll & Garcia-Blanco, 1982c) binds Na$^+$ ions giving rise to 4NO2-NA (Esteban-Calderón, Martínez-Ripoll & García-Blanco, 1982a) and neither acid treatment nor resin exchange can release 4NO2. However, the free heterocyclic compound 4NO2 can be obtained from 4NO2-K (Esteban-Calderón et al., 1982a). Similar irreversible retention of Na$^+$ ions is shown by 4CN-NA (Esteban-Calderón, Martínez-Ripoll & García-Blanco, 1982b) which does not release 4CN (Esteban-Calderón, Martínez-Ripoll & Garcia-Blanco, 1979). On the other hand, a total exchangeability of K$^+$ ions exists between 4CN and 4CN-K (Esteban-Calderón et al., 1982b).

These studies suggest an explanation for the ionic retention in terms of a direct or an indirect contact of the cation with the bidentate ligand. In fact, in the crystal structures of the above-mentioned compounds, the Na$^+$ ions are bonded to the bidentate ligand, whereas in the potassium salts all K$^+$—anion contacts are with different anionic units. This retention could also be explained in terms of a higher intensity of interaction in the Na$^+$—anion bonds than in K$^+$—anion. In order to obtain more structural results which could explain the different cationic exchangeability, we report here the structure of the title compound.

Experimental. Sample of the title compound supplied by Drs Stud and Goya (Instituto de Química Médica, CSIC, Madrid) and crystallized from aqueous solution as transparent colourless single crystals; intensities collected on a four-circle diffractometer equipped with graphite-monochromated Mo $K\alpha$ radiation using an $\omega/2\theta$ scan; no crystal decomposition observed; an absorption correction made using ORABS (Schwarzenbach, 1972), the absorption factors varying between 1·04 and 1·09; 2482 independent reflexions measured up to $\theta = 30°$, 1120 of which were considered observed with $I > 2\sigma(I)$ and used in the calculations; MULTAN (Main, Lessinger, Woolfson, Germain & Declercq, 1977) not successful for structure solution; interpretation of the Patterson map and subsequent use of DIRDIF (Beurskens, Bosman, Doesburg, Gould, Van der Hark & Prick, 1980) revealed the whole

structure; remaining calculations made with XRAY 70 (Stewart, Kundell & Baldwin, 1970); all H atoms appeared on a difference synthesis; refinement by full-matrix least squares minimizing $\sum w|\Delta F|^2$ with $w = 1$ until convergence obtained, $R = 0·057$ and $R_w = 0·061$; last steps of refinement carried out with weights given by $w = w_1.w_2$, with $w_1 = K/(a + b|F_o| + c|F_o|^2)^2$ and $w_2 = 1/(d + e \sin\theta/\lambda + f \sin^2\theta/\lambda^2)$ to give flat dependence of $\langle w|\Delta F|^2\rangle$ vs $\langle F_o\rangle$ and vs $\langle \sin\theta/\lambda\rangle$; the coefficients used are $a = 1·78$, $b = -0·25$, $c = 0·05$ for $F_o < 5·7$, $a = 3·37$, $b = -0·29$, $c = 0·01$ for $F_o > 5·7$, $K = 0·59$, $d = 3·39$, $e = -11·33$ and $f = 12·14$ for all reflexions (Martínez-Ripoll & Cano, 1975); scattering factors for neutral atoms and anomalous-dispersion corrections for S and K taken from International Tables for X-ray Crystallography (1974).

Discussion. Table 1 shows the final atomic parameters.*

Fig. 1 shows a partial view of this crystal structure. Some symmetrically related atoms are also shown for clarity. Table 2 lists the bond lengths and angles. The geometrical features of the molecule are very similar to those reported for 4NO2-K, the molecular ring being described as an envelope with the S atom at the flap [0·16 (1) Å out of the plane]. Consequently, the same electron-delocalization scheme given for that compound can also be proposed for the present one. The methyl substituent does not modify the molecular geometry. The K$^+$ ions are surrounded by seven O atoms at distances in the range 2·70 to 2·89 Å forming a laterally capped trigonal prism. Different molecules are held together by the K—O bonds.

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* Lists of structure factors, anisotropic thermal parameters, atomic least-squares planes, H-atom coordinates, and torsional angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38292 (22 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* ($\text{Å}^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

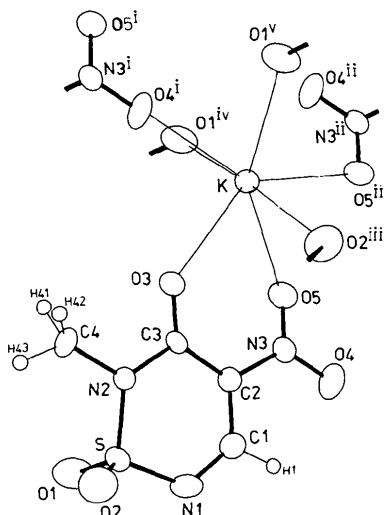|	x	y	z	U_{eq}
K	−0·3942 (2)	0·8737 (3)	−0·2975 (2)	36 (1)
S	0·2977 (2)	0·6320 (3)	0·2558 (3)	32 (1)
C(1)	0·0410 (10)	0·7241 (15)	0·3918 (13)	50 (4)
C(2)	−0·0577 (8)	0·7850 (11)	0·2277 (10)	31 (3)
C(3)	−0·0020 (7)	0·7619 (11)	0·0495 (10)	29 (3)
C(4)	0·2295 (9)	0·6526 (15)	−0·1205 (13)	51 (4)
N(1)	0·1991 (9)	0·6538 (15)	0·4185 (11)	65 (4)
N(2)	0·1635 (7)	0·6811 (11)	0·0570 (9)	38 (3)
N(3)	−0·2250 (7)	0·8614 (9)	0·2391 (9)	31 (3)
O(1)	0·3914 (8)	0·7678 (10)	0·3329 (10)	63 (3)
O(2)	0·3894 (8)	0·4373 (9)	0·1929 (11)	58 (3)
O(3)	−0·0832 (6)	0·8004 (9)	−0·1067 (8)	42 (3)
O(4)	−0·2725 (7)	0·8438 (11)	0·3756 (10)	59 (3)
O(5)	−0·3183 (6)	0·9474 (9)	0·1197 (8)	46 (3)

Fig. 1. Perspective drawing of the structure (Johnson, 1965). Some symmetrically related atoms are also shown. Thermal ellipsoids scaled to 50% probability. Symmetry code: (i) $x,y,-1 + z$; (ii) $-1-x,2-y,-z$; (iii) $-x,1-y,-z$; (iv) $-x,2-y,-z$; (v) $-1 + x,y,-1 + z$.

The present structure shows an interesting feature not found in the above-mentioned potassium salts: the K^+ ions are bonded to the bidentate ligand. As this compound can release the free heterocycle, it is concluded that this type of interaction does not seem to be the reason for the cationic retention. Thus, from the structural point of view, the only explanation should be encountered in the different intensity of interaction between anion and cations. In fact, from the observed $K-O$ bond lengths, one can deduce a lower intensity of interaction in $K-O$ bonds than in $Na-O$. Assuming the same interaction model of cation–dipole type as described for 4NO2-K, the mean observed $K-O$ distances for the present compound are 5·6% elongated, somewhat less than in 4NO2-K (6·8%). However, K^+ is here bonded to seven anionic sites instead of five as occurs in 4NO2-K, which could indicate a decrease in the intensity of the individual $K-O$ interactions.

Table 2. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

For symmetry codes see Fig. 1.

S—O(1)	1·42 (1)	C(1)—C(2)	1·38 (1)
S—O(2)	1·41 (1)	C(2)—C(3)	1·43 (1)
S—N(1)	1·58 (1)	C(3)—O(3)	1·23 (1)
S—N(2)	1·65 (1)	K—O(3)	2·70 (1)
N(1)—C(1)	1·32 (1)	K—O(2^{iii})	2·72 (1)
N(2)—C(3)	1·39 (1)	K—O(4^i)	2·76 (1)
N(2)—C(4)	1·47 (1)	K—O(5)	2·77 (1)
N(3)—C(2)	1·42 (1)	K—O(1^v)	2·83 (1)
N(3)—O(4)	1·23 (1)	K—O(1^{iv})	2·85 (1)
N(3)—O(5)	1·22 (1)	K—O(5^{ii})	2·89 (1)
O(1)—S—O(2)	113·8 (4)	O(3)—K—O(5)	59·5 (2)
O(1)—S—N(1)	110·3 (5)	O(3)—K—O(1^v)	144·6 (2)
O(1)—S—N(2)	108·6 (5)	O(3)—K—O(1^{iv})	93·3 (2)
O(2)—S—N(1)	109·5 (6)	O(3)—K—O(2^{iii})	82·6 (2)
O(2)—S—N(2)	107·6 (4)	O(3)—K—O(5^{ii})	128·2 (2)
N(1)—S—N(2)	106·8 (4)	O(3)—K—O(4^i)	85·5 (2)
S—N(1)—C(1)	121 (1)	O(5)—K—O(1^v)	149·2 (3)
S—N(2)—C(3)	126 (1)	O(5)—K—O(1^{iv})	100·1 (2)
S—N(2)—C(4)	116 (1)	O(5)—K—O(2^{iii})	70·0 (2)
C(3)—N(2)—C(4)	118 (1)	O(5)—K—O(5^{ii})	68·9 (2)
C(2)—N(3)—O(4)	119 (1)	O(5)—K—O(4^i)	141·9 (2)
C(2)—N(3)—O(5)	121 (1)	O(1^v)—K—O(1^{iv})	97·1 (2)
O(4)—N(3)—O(5)	121 (1)	O(1^v)—K—O(5^{ii})	85·3 (2)
C(2)—C(1)—N(1)	127 (1)	O(1^v)—K—O(4^i)	68·8 (2)
C(1)—C(2)—C(3)	124 (1)	O(1^{iv})—K—O(2^{iii})	170·0 (2)
C(1)—C(2)—N(3)	115 (1)	O(1^{iv})—K—O(5^{ii})	92·1 (2)
C(3)—C(2)—N(3)	121 (1)	O(1^{iv})—K—O(4^i)	64·8 (2)
C(2)—C(3)—N(2)	115 (1)	O(2^{iii})—K—O(5^{ii})	83·4 (2)
C(2)—C(3)—O(3)	128 (1)	O(2^{iii})—K—O(4^i)	123·6 (2)
N(2)—C(3)—O(3)	118 (1)	O(5^{ii})—K—O(4^i)	141·6 (2)

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