The Ni complex can be contrasted with the Cu complex (Haendler, 1989) in which two chelated molecules are in each of four asymmetric units. In one molecule, the N atoms are in a *cis* position with the Cu in a planar configuration. In the second molecule, the N atoms are in the *trans* position with the Cu atom intermediate between a distorted pentacoordinated trigonal bipyramid and a distorted rectangular pyramid. The metal–ligand distances are greater in the Ni complex than in the Cu complex and the O—*M*—N angle is slightly smaller.

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Structure of Sodium 6-Hydroxy-2-methyl-3-thioxo-2*H*-1,2,4-triazin-5(4*H*)-onate 1.25-Hydrate

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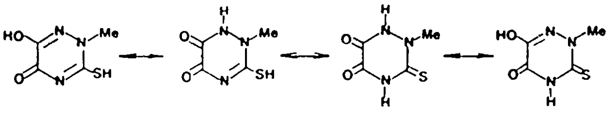
Abstract. $Na^+.C_4H_4N_3O_2S^-.1.25H_2O$, $M_r = 202.61$, monoclinic, $P2_1/c$, $a = 11.032\,(2)$, $b = 9.793\,(1)$, $c = 15.867\,(2)$ Å, $\beta = 103.53\,(1)°$, $V = 1666.7\,(7)$ Å^3, $Z = 8$, $D_m = 1.618\,(5)$, $D_x = 1.623$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.396$ mm^{-1}, $F(000) = 836$, $T = 293\,(2)$ K, final $R = 0.035$ and $wR = 0.042$ for 2465 observed reflections. There are two formula units in the asymmetric unit. The Na atoms are sixfold coordinated with two different arrangements of ligands. The two triazine units are equal within standard deviations. The structure can be regarded as being constituted of layers linked together by Na coordination polyhedra along the *yz* plane. One water molecule has a population of 0.5 linking layers with weak hydrogen bonds.

Introduction. The title compound was investigated in connection with studies on the synthesis of pharmaceutical compounds. It is an intermediate for the synthesis of ceftriaxone, which belongs to the third generation of cephalosporin antibiotics (Neu, Meropol & Fu, 1981). Infrared spectroscopy, mass spectrometry or proton magnetic resonance cannot permit complete identification of which of the four possible tautomeric forms, (i)–(iv) (Štefanič, Valenčič, Tišler, Kobal, Vitezič & Japelj, 1990), is present in this compound. Therefore we have confirmed the structure by X-ray investigation. The compound was isolated as a hydrated Na salt and the crystals for diffraction measurement were grown by slow evaporation of cold water–methanol solution.



(i) (ii) (iii) (iv)

Experimental. A prismatic crystal with approximate dimensions 0.18 × 0.25 × 0.65 mm was used for data

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collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were determined by a least-squares treatment of 75 carefully centred θ values in the range $8 < \theta < 15°$. A hemisphere of data with index ranges $-14 \leq h \leq 14$, $-12 \leq k \leq 12$ and $-20 \leq l \leq 0$ was measured at room temperature [293 (2) K] to $\theta_{\max}$ of 28° by the ω–2θ-scan technique with minimum and maximum scan rates of 1.04 and 5.55° $\min^{-1}$ and a scan width $(0.7 + 0.3\tan\theta)°$ with aperture $(2.4 + 0.9\tan\theta)$ mm; maximum scan time 60 s. Background intensities were measured at one quarter of the scan at each limit. Crystal stability was monitored by periodic measurement of three standard reflections ($50\bar{2}$, $15\bar{2}$ and $\bar{2}2\bar{5}$) every 20 000 s of scan time; there was no evidence of crystal deterioration. A change of +1.89% in the intensities of the standard reflections was observed and a correction applied. Owing to the low value of the linear absorption coefficient (0.396 mm^{-1}), no absorption correction was applied. 8152 reflections were collected. Averaging gave 4002 unique reflections with $R_{\rm int} = 0.023$, 2465 of which were observed [$I > 2.5\sigma(I)$].

The S-atom positions were determined by Patterson map. All remaining non-H atoms were located from successive Fourier maps. All H atoms were located on an intermediate difference map and were refined with isotropic thermal factors. Their positions do not differ significantly from idealized positions. Two-block-matrix least-squares refinement on F magnitudes was performed with anisotropic temperature factors for all non-H atoms, using the empirical weighting function $w = 6.0 \times W_f \times W_s$ where W_f ($|F_o| < 6.0) = |F_o|/6.0$, W_f ($|F_o| > 11.0) = (11.0/|F_o|)^{1.5}$, W_f $(6.0 < F_o < 11.0) = 1.0$ and W_s $(\sin\theta < 0.37) = (\sin\theta/0.37)^2$, W_s $(\sin\theta > 0.95) = (0.95/\sin\theta)^2$, and W_s $(0.37 < \sin\theta < 0.95) = 1.0$, to keep $\sum w(\Delta F)^2$ uniform over the ranges of $\langle\sin\theta/\lambda\rangle$ and $\langle F_o\rangle$. In the final least-squares cycle there were 3095 (m) contributing reflections (unobserved reflections for which F_c was greater than F_o were included) and 282 (n) refined parameters; data-to-variable ratio (m/n) 11.0; scale factor 0.998; S $\{= [\sum w(\Delta F)^2/(m-n)]^{1/2}$ 0.89; average and maximum shift/e.s.d. 0.019 and 0.298. Maximum and minimum densities in the final difference map were 0.4 and -0.3 e $\mathrm{\AA}^{-3}$, respectively. The final R and wR are 0.035 and 0.042, respectively.

Atomic scattering factors for H atoms were taken from Stewart, Davidson & Simpson (1965) and for other neutral atoms from Cromer & Mann (1986), with dispersion corrections from Cromer & Liberman (1970). All calculations were performed on a DEC-10 computer at RCU-Ljubljana using the *XRAY76* system of crystallographic programs (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. A minimum-overlap view of the asymmetric unit with atom-numbering scheme is shown in Fig. 1; a stereoview of the unit cell is presented in Fig. 2. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1. Interatomic distances and angles are given in Table 2.*

The compound has two formula units in the asymmetric unit (indicated with a and b), with small differences in interatomic distances (0.01 Å) and angles (0.9°) for two triazine units; average values are used in the discussion.

The N(1)—N(2) single bond with value of 1.393 (4) Å is similar to the corresponding value in a compound with the same triazine structure [1.394 Å (Ayato, Tanaka, Yamane, Ashida, Sasaki, Minamoto & Harada, 1981)] and is comparable with the value for N—N planar bonds [1.401 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987)]. The N(2)—C(4) bond length is 1.465 (1) Å and lies within the range quoted by Allen *et al.* (1987) for C(sp^2)—N(sp^2) planar bonds (1.454–1.464 Å).

The N(2)—C(1) bond with value of 1.321 (2) Å is shorter than normal C(sp^2)—N(sp^2) planar bonds

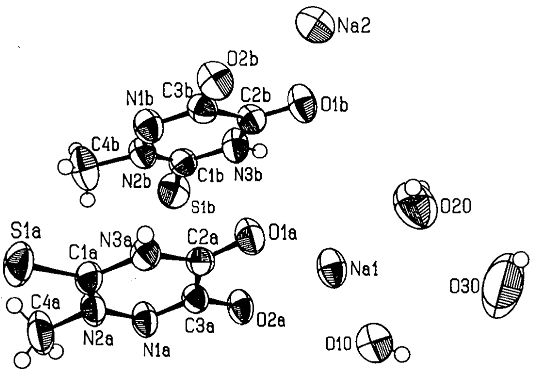

Fig. 1. Minimum-overlap view of the asymmetric unit.

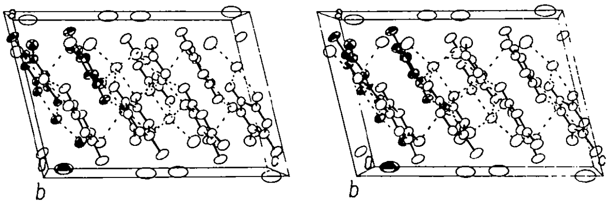

Fig. 2. Stereoview of the molecular packing.

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* List of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55429 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$)

	x	*y*	*z*	U_{eq}
Na(1)	3339 (1)	1770 (1)	924 (1)	49.7 (4)
Na(2)	4086 (1)	4328 (1)	4047 (1)	46.5 (4)
S(1*a*)	8074.7 (5)	6552.1 (5)	751.7 (4)	54.6 (3)
N(1*a*)	6865 (1)	2750 (1)	156 (1)	44 (1)
N(2*a*)	7550 (1)	3955 (2)	270 (1)	43 (1)
C(1*a*)	7257 (2)	5081 (2)	640 (1)	37 (1)
N(3*a*)	6201 (1)	4998 (1)	955 (1)	37 (1)
C(2*a*)	5474 (1)	3867 (2)	919 (1)	32 (1)
C(3*a*)	5858 (1)	2676 (2)	458 (1)	34 (1)
O(1*a*)	4569 (1)	3841 (1)	1244 (1)	45 (1)
O(2*a*)	5195 (1)	1610 (1)	381 (1)	42 (1)
C(4*a*)	8674 (2)	3876 (3)	− 69 (2)	67 (1)
S(1*b*)	8081.2 (5)	44.9 (6)	2377.7 (4)	58.9 (3)
N(1*b*)	7328 (1)	3903 (2)	2891 (1)	44 (1)
N(2*b*)	7874 (1)	2711 (2)	2670 (1)	42 (1)
C(1*b*)	7402 (2)	1471 (2)	2652 (1)	40 (1)
N(3*b*)	6277 (1)	1380 (1)	2881 (1)	45 (1)
C(2*b*)	5643 (2)	2445 (2)	3108 (1)	37 (1)
C(3*b*)	6250 (2)	3807 (2)	3102 (1)	37 (1)
O(1*b*)	4643 (1)	2312 (1)	3308 (1)	47 (1)
O(2*b*)	5686 (1)	4828 (1)	3304 (1)	47 (1)
C(4*b*)	9079 (2)	2955 (3)	2458 (2)	66 (1)
O(10)	2455 (2)	129 (2)	− 126 (1)	58 (1)
O(20)	1895 (2)	1796 (3)	1787 (2)	86 (1)
O(30)	15 (7)	306 (15)	707 (8)	155 (8)

Table 2. *Interatomic distances* (Å), *bond angles* (°) *and hydrogen-bond distances* (Å)

Molecule (I)		Molecule (II)	
Na(1)—O(20)	2.332 (3)	Na(2)—O(2*a*^{iii})	2.368 (2)
Na(1)—O(10)	2.355 (2)	Na(2)—O(2*b*)	2.392 (2)
Na(1)—O(2*b*^{i})	2.378 (1)	Na(2)—O(1*b*)	2.448 (2)
Na(1)—O(2*a*)	2.404 (2)	Na(2)—O(2*a*^{iv})	2.472 (1)
Na(1)—O(1*a*)	2.427 (1)	Na(2)—O(10^{iii})	2.518 (3)
Na(1)—S(1*a*^{ii})	3.204 (1)	Na(2)—S(1*b*^{iv})	2.965 (1)
N(1*a*)—N(2*a*)	1.390 (2)	N(1*b*)—N(2*b*)	1.395 (2)
N(2*a*)—C(4*a*)	1.465 (3)	N(2*b*)—C(4*b*)	1.464 (3)
N(2*a*)—C(1*a*)	1.324 (2)	N(2*b*)—C(1*b*)	1.319 (2)
C(1*a*)—S(1*a*)	1.687 (2)	C(1*b*)—S(1*b*)	1.689 (2)
C(1*a*)—N(3*a*)	1.373 (3)	C(1*b*)—N(3*b*)	1.375 (3)
N(3*a*)—C(2*a*)	1.361 (2)	N(3*b*)—C(2*b*)	1.351 (2)
C(2*a*)—O(1*a*)	1.227 (2)	C(2*b*)—O(1*b*)	1.224 (2)
C(2*a*)—C(3*a*)	1.490 (2)	C(2*b*)—C(3*b*)	1.494 (2)
C(3*a*)—O(2*a*)	1.264 (2)	C(3*b*)—O(2*b*)	1.259 (2)
C(3*a*)—N(1*a*)	1.312 (3)	C(3*b*)—N(1*b*)	1.312 (3)
C(3*a*)—N(1*a*)—N(2*a*)	118.6 (2)	C(3*b*)—N(1*b*)—N(2*b*)	118.3 (1)
N(1*a*)—N(2*a*)—C(1*a*)	125.7 (2)	N(1*b*)—N(2*b*)—C(1*b*)	125.7 (2)
N(1*a*)—N(2*a*)—C(4*a*)	112.8 (2)	N(1*b*)—N(2*b*)—C(4*a*)	112.8 (2)
C(4*a*)—N(2*a*)—C(1*a*)	121.5 (2)	C(4*b*)—N(2*b*)—C(1*b*)	121.4 (2)
N(2*a*)—C(1*a*)—S(1*a*)	125.3 (2)	N(2*b*)—C(1*b*)—S(1*b*)	124.7 (2)
S(1*a*)—C(1*a*)—N(3*a*)	119.4 (1)	S(1*b*)—C(1*b*)—N(3*b*)	119.8 (1)
N(2*a*)—C(1*a*)—N(3*a*)	115.3 (1)	N(2*b*)—C(1*b*)—N(3*b*)	115.5 (2)
C(1*a*)—N(3*a*)—C(2*a*)	125.1 (1)	C(1*b*)—N(3*b*)—C(2*b*)	125.2 (2)
N(3*a*)—C(2*a*)—C(3*a*)	115.2 (2)	N(3*b*)—C(2*b*)—C(3*b*)	115.3 (2)
N(3*a*)—C(2*a*)—O(1*a*)	121.9 (2)	N(3*b*)—C(2*b*)—O(1*b*)	122.8 (2)
O(1*a*)—C(2*a*)—C(3*a*)	122.9 (1)	O(1*b*)—C(2*b*)—C(3*b*)	122.0 (2)
C(2*a*)—C(3*a*)—N(1*a*)	119.9 (1)	C(2*b*)—C(3*b*)—N(1*b*)	120.0 (2)
O(2*a*)—C(3*a*)—N(1*a*)	122.0 (2)	O(2*b*)—C(3*b*)—N(1*b*)	122.8 (2)
C(2*a*)—C(3*a*)—O(2*a*)	118.0 (2)	C(2*b*)—C(3*b*)—O(2*b*)	117.3 (2)
N(3*a*)···O(1*b*^{iv})	2.807 (2)	O(10)···O(30^{vi})	2.698 (8)
N(3*b*)···O(2*b*^{i})	2.937 (2)	O(20)···N(1*b*^{i})	2.970 (3)
O(10)···N(1*b*^{v})	2.921 (2)	O(20)···O(30)	2.78 (1)

Symmetry code: (i) $1-x,\ -\frac{1}{2}+y,\ \frac{1}{2}-z$; (ii) $1-x,\ 1-y,\ -z$; (iii) $x,\ \frac{1}{2}-y,\ \frac{1}{2}+z$; (iv) $1-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$; (v) $1-x,\ -y,\ -z$; (vi) $-x,\ -y,\ -z$.

(1.355 Å). The opposite situation is found for C(1)—S(1) [1.688 (1) Å]. The value is longer than expected for a C(sp^2)—S double bond [1.671 Å (Allen *et al.*, 1987)]. The reason for these differences lies in delocalization of the π electrons between these bonds and the S atom coordinating to the Na ion.

Comparison of lengths of chemically similar bonds C(1)—N(3) and C(2)—N(3) in the triazine moiety with those tabulated in the literature (Allen *et al.*, 1987) or with values in similar compounds [1.358–1.389 Å (Haltiwanger, Kolonko, Shapiro & Sievers, 1979; Kutschabsky, Kretschmer, Kircheiss & Bauwe, 1981) shows no significant differences. Their values [1.374 (1) and 1.356 (7) Å] show that they all have bonding character in the range for C(sp^2—N(sp^2) planar bonds with less than 20% double-bond character (Wheatley, 1955).

The only C—C bond, C(2)—C(3), is longer than normal C(sp^2)—C(sp^2) bonds. The value is 1.492 (2) Å, compared to literature values of about 1.465 Å (Allen *et al.*, 1987). Also, the C(2)—O(1) double bond with length of 1.225 (2) Å is slightly longer than literature values of 1.210–1.221 Å (Allen *et al.*, 1987). The single bond between C(3)—O(2) with a value of 1.261 (3) Å is shorter than a normal C(sp^2)—O single bond. These differences result from the formation of the chelate ring between those bonds and the Na ion.

The length of C(3)—N(1) is 1.312 (3) Å and is comparable with values for such bonds in imidazole (1.313 Å) and has significant double-bond character.

The two Na ions have different sixfold coordination. One Na ion has three O atoms at distances 2.378 (1), 2.404 (2) and 2.427 (1) Å, two water molecules at distances 2.332 (3) and 2.355 (2) Å and one S atom at distance 3.204 (1) Å. The second Na ion has four O atoms at distances 2.368 (2), 2.392 (2), 2.448 (2) and 2.472 (1) Å, one water molecule at 2.518 (3) Å and one S atom at 2.965 (1) Å.

During the structure refinement the formula was originally thought to correspond to a monohydrate with two general positions occupied by water molecules in the coordination spheres of the Na ions. A difference map showed an additional peak of half the height for an O atom among the layers of the structure, denoted here as O(30). During refinement, the occupancy of this water molecule was 0.48 (4) and in the final stage of refinement it was fixed to 0.50, as indicated also by density measurements. It links layers of the structure by weak hydrogen bonds to O(10) [2.698 (8) Å] and to O(20) [2.78 (1) Å].

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Synthesis and Structure of Diaquabis(glycolato-*O*,*O''*)germanium(IV)

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Abstract. Diaquabis(oxoacetato-*O*,*O''*)germanium(IV), $[Ge(C_2H_2O_3)_2(H_2O)_2]$, was prepared from germanium dioxide and glycolic acid in refluxed H_2O and identified with IR, ^1H NMR, ^{13}C NMR, mass spectrum, elemental analysis, and X-ray single-crystal structure determination. $C_4H_8GeO_8$, $M_r = 256.7$, monoclinic, $P2_1/n$, $a = 4.862$ (2), $b = 5.952$ (2), $c = 13.448$ (4) Å, $\beta = 95.91$ (2)°, $V = 387.1$ (2) Å^3, $Z = 2$, $D_x = 2.20$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 3.92$ mm^{-1}, $F(000) = 256$, $T = 298$ K, $R = 0.030$ for 462 observed reflections. The title compound has a distorted octahedral structure with two aqua groups on the axial position and two bidentate glycolate groups on the equatorial position. The Ge—O bond lengths for the aqua groups are longer than those for the anion ligands.

Introduction. The adduct of germanium dioxide and glycolic acid was first published by Clark (1959) and its approximate composition ratio, Ge–glycolic acid 1:3, was identified mainly by conductimetric, polarimetric and pH measurement. Subsequently, Mikanova & Bartusek (1981) studied the less-stable mononuclear 1:3 chelates of Ge formed by the reaction of Ge^{IV} with glycolic acid in dilute aqueous solution with potentiometric titration. Because Ge complexes are attracting particular interest in their specific biological activity (Brutkiewicz & Suzuki, 1987) and it is important to elucidate the reaction between germanium dioxide and glycolic acid in aqueous solution, the title compound has been synthesized and the crystal structure determination has been undertaken (Chiang, Lin & Ueng, 1992).

Experimental. The IR spectrum was recorded using a potassium bromide pellet and a Jasco Model 700 spectrophotometer. The NMR spectrum was recorded in D_2O solution on a Jeol JNM-EX 400 MHz spectrophotometer. The mass spectrum was obtained using the electron-impact method on a Jeol JMS-D300 instrument. Microanalysis was performed by the National Science Council, Taipei, Taiwan. The title compound was prepared by adding germanium dioxide (0.52 g, 5.0 mmol) to an aqueous solution (100 cm^3) of glycolic acid (1.52 g, 20.0 mmol) and refluxing the acidic mixture (pH = 1.5) for 4 h. After reducing to about 5 ml, the solution was allowed to stand for 2 d, which yielded colourless square crystals which were filtered, washed with water and dried; m.p. 439–440 K (dec); yield 1.08 g (85%). Analysis found: C 18.69, H 2.93%. Analysis calculated for $C_4H_8GeO_8$: C 18.69, H 3.11%. IR ν_{max}: 1678*s* (C=O), 1084*s* and 930*m* (Ge—OC), and 561*m* cm^{-1} (Ge—OH$_2$). ^1H NMR δ_H (D_2O): 4.22. (2H, *s*, CH$_2$). ^{13}C NMR δ_C (D_2O): 62.8 (CH$_2$) and 179.8. (C=O). Mass spectrum *m*/*Z*: 222 ($M - 2H_2O$) and 106 ($M - 2H_2O - 2CH_2CO_2$).

A crystal suitable for X-ray diffraction (CAD-4 diffractometer) was prepared from H_2O by evaporation and had dimensions 0.25 × 0.30 × 0.40 mm. The unit cell was determined using 24 reflections with 2θ 18.76–29.08°. $\theta/2\theta$ scans, with θ scan width of $(1.00 + 0.35\tan\theta)$°, and Mo $K\alpha$ radiation were used for data collection. Three standard reflections were monitored every hour and showed variation on $I < 2.0\%$. 507 unique reflections were measured ($2.0 < 2\theta < 44.9$°; h − 5 to 5, k 0 to 6, l 0 to 14; $R_{int} = 0.008$) of which 462 were observed with $I > 2.0\sigma(I)$. Absorption corrections were made according to

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