

Fig. 1. A PLUTO (Motherwell & Clegg, 1978) diagram of the structure of $[\text{Co}_2(\text{CO})_{10}(\mu_4\text{-S})_2]$. The molecule lies about a crystallographic inversion centre.

are given in Table 1, and selected structural parameters in Table 2. The structure is illustrated in Fig. 1.*

Related literature. This determination gives parameters an order of magnitude more precise than those previously determined using visually estimated photographic data ($R = 0.092$) (Wei & Dahl, 1975).

* Lists of structure factors, full bond parameters and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55307 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1006]

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Structure of Monosodium N^1 -(4,6-Dimethyl-2-pyrimidyl)sulfanilamidate Dihydrate

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Abstract. $\text{Na}^+\text{C}_{12}\text{H}_{13}\text{N}_4\text{O}_2\text{S}^- \cdot 2\text{H}_2\text{O}$, $M_r = 336.34$, triclinic, $P\bar{1}$, $a = 11.570$ (3), $b = 12.725$ (3), $c = 5.790$ (4) Å, $\alpha = 101.28$ (3), $\beta = 102.35$ (4), $\gamma = 70.01$ (3)°, $V = 775.6$ Å³, $Z = 2$, $D_m = 1.46$, $D_x = 1.44$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 23.06$ cm⁻¹, $F(000) = 352$, $T = 298$ K, $R = 0.074$ for 1538 reflections with $F > 5\sigma(F)$. The Na^+ ion is directly coordinated to the sulfonyl O atoms, one water molecule and the amide N atom. The molecules are held together by a three-dimensional network of hydrogen bonds of type N—H...O, O—H...O and O—H...N. Molecular packing shows the presence of alternate layers of hydrophilic and hydrophobic regions along the a direction.

Some significant changes have been found in the present determination. Compounds with a square-bipyramidal core $\mu_4\text{-E}_2\text{M}_4$ are of structural [see Albright, Yee, Saillard, Kahlal, Halet, Leigh & Whitmire (1991) for a recent list] and of theoretical interest (Halet, Hoffmann & Saillard, 1985; Halet & Saillard, 1987).

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Experimental. Colourless needle-shaped crystals were obtained from aqueous solution; crystal size $0.22 \times 0.12 \times 0.40$ mm. Symmetry $P\bar{1}$ was determined from oscillation and Weissenberg photographs. Density was measured by flotation in benzene–bromofrom. Data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Cell parameters were refined by least-squares method on the basis of 25 independent 2θ values ($37 < 2\theta < 62^\circ$). Intensities of 2052 independent reflections with $I > 3\sigma(I)$ ($-11 \leq h \leq 12$, $-13 \leq k \leq 13$, $0 \leq l \leq 6$, $3 \leq 2\theta \leq 126^\circ$) were measured by $\omega/2\theta$ -scan technique with scan speed 4° min^{-1} . Three standard reflections monitored every 200 reflections

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j, \text{ where } U_{ij} \text{ are defined by the expression} \\ \exp[-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + \dots + 2U_{23}b^*c^*kl + \\ 2U_{13}a^*c^*hl + 2U_{12}a^*b^*hk)].$$

	x	y	z	U_{eq}
Na	-557 (2)	6382 (2)	3803 (4)	39 (1)
S	1867 (1)	3928 (1)	2182 (2)	24 (1)
O(1)	1273 (3)	4849 (3)	3955 (7)	32 (1)
O(4)	1552 (4)	4227 (3)	-0212 (7)	32 (1)
N(1)	1484 (4)	2897 (4)	2497 (8)	26 (1)
N(2)	1543 (4)	1073 (4)	1849 (8)	32 (2)
C(1)	1931 (5)	1847 (5)	1276 (10)	28 (2)
C(3)	1984 (6)	17 (5)	772 (13)	44 (3)
C(4)	2761 (7)	-244 (5)	-855 (13)	50 (3)
C(5)	3106 (6)	598 (5)	-1374 (11)	39 (2)
N(6)	2703 (5)	1661 (4)	-326 (9)	35 (2)
C(7)	3983 (8)	386 (7)	-3133 (13)	63 (3)
C(8)	1553 (9)	-857 (7)	1504 (17)	75 (4)
C(9)	3477 (5)	3657 (5)	2997 (9)	24 (2)
C(10)	4083 (5)	3218 (5)	5107 (10)	35 (2)
C(11)	5338 (6)	3057 (6)	5792 (11)	40 (2)
C(12)	6030 (5)	3367 (5)	4510 (10)	31 (2)
C(13)	5410 (6)	3787 (5)	2356 (11)	37 (3)
C(14)	4147 (6)	3921 (5)	1588 (10)	33 (2)
N(14)	7281 (5)	3238 (5)	5255 (10)	47 (2)
W(1)	-52 (5)	8041 (4)	4508 (10)	60 (2)
W(2)	-1499 (5)	3546 (4)	10692 (10)	65 (2)

Table 2. Bond lengths (\AA), bond angles ($^\circ$), selected torsion angles ($^\circ$) and contact distances (\AA) with the Na atom, and angles ($^\circ$) about the Na atom

S—O(1)	1.476 (3)	C(4)—C(5)	1.366 (11)
S—N(1)	1.573 (6)	C(3)—C(8)	1.522 (14)
S—C(9)	1.749 (5)	C(5)—C(7)	1.512 (12)
S—O(4)	1.447 (4)	C(9)—C(10)	1.389 (7)
N(1)—C(1)	1.366 (6)	C(10)—C(11)	1.374 (8)
N(2)—C(1)	1.332 (9)	C(11)—C(12)	1.389 (11)
N(2)—C(3)	1.339 (7)	C(12)—C(13)	1.404 (8)
C(1)—N(6)	1.355 (8)	C(13)—C(14)	1.393 (9)
N(6)—C(5)	1.335 (7)	C(9)—C(14)	1.391 (10)
C(3)—C(4)	1.357 (11)	C(12)—N(14)	1.380 (7)
N(1)—S—C(9)	110.5 (2)	C(3)—C(4)—C(5)	118.5 (6)
O(1)—S—C(9)	106.9 (2)	C(4)—C(5)—C(7)	122.3 (6)
O(1)—S—N(1)	104.1 (2)	N(6)—C(5)—C(4)	121.9 (6)
O(4)—S—N(1)	115.2 (2)	N(6)—C(5)—C(7)	115.7 (6)
O(1)—S—O(4)	113.3 (2)	S—C(9)—C(14)	120.2 (4)
N(1)—C(1)—N(6)	120.7 (5)	C(10)—C(9)—C(14)	119.7 (5)
N(2)—C(1)—N(6)	125.7 (5)	C(13)—C(12)—C(11)	116.9 (6)
S—N(1)—C(1)	122.1 (4)	C(12)—C(11)—C(10)	122.5 (5)
C(1)—N(2)—C(3)	116.3 (5)	C(11)—C(12)—N(14)	122.1 (5)
C(1)—N(6)—C(5)	115.6 (5)	C(13)—C(12)—N(14)	120.8 (5)
N(2)—C(3)—C(8)	115.3 (6)	C(9)—C(10)—C(11)	119.6 (5)
N(2)—C(3)—C(4)	121.7 (6)	C(12)—C(13)—C(14)	121.2 (5)
C(4)—C(3)—C(8)	122.9 (6)		
N(1)—S—C(9)—C(14)	135.9 (5)	O(1)—S—C(9)—C(14)	-111.2 (5)
N(1)—S—C(9)—C(10)	-46.5 (5)	O(1)—S—N(1)—C(1)	-175.1 (4)
C(9)—S—N(1)—C(1)	-60.6 (5)	S—N(1)—C(1)—N(6)	-1.9 (8)
O(1)—S—C(9)—C(10)	66.1 (5)	S—N(1)—C(1)—N(2)	177.3 (4)
Na...O(1 ⁱⁱ)	2.336 (3)	O(1 ⁱ)—Na—O(1 ⁱⁱ)	82.7 (5)
Na...O(1 ⁱⁱ)	2.639 (5)	O(1 ⁱ)—Na—O(4 ⁱⁱⁱ)	97.7 (5)
Na...O(4 ⁱⁱⁱ)	2.282 (4)	O(1 ⁱ)—Na—N(1 ^{iv})	117.2 (5)
Na...N(1 ^{iv})	2.475 (5)	O(1 ⁱ)—Na—W(1 ^v)	109.2 (2)
Na...W(1 ^v)	2.316 (6)	O(1 ⁱⁱ)—Na—N(1 ^{iv})	55.8 (4)
		O(4 ⁱⁱⁱ)—Na—N(1 ^{iv})	125.1 (3)
		O(4 ⁱⁱⁱ)—Na—W(1 ^v)	122.9 (2)
		N(1 ^{iv})—Na—W(1 ^v)	85.2 (9)

Symmetry code: (i) x, y, z ; (ii) $-x, 1-y, 1-z$; (iii) $-x, 1-y, -z$.

showed no significant variation in intensity during data collection. $R_{int} = 0.07$. Lorentz and polarization corrections were applied, but absorption correction was ignored. The structure was solved by direct method using *SHELXS86* (Sheldrick, 1986).

Refinement (on F) was carried out using *SHELX76* (Sheldrick, 1976) with anisotropic thermal parameters for the non-H atoms. Most of the H atoms were located from a difference synthesis and included in the refinement with isotropic temperature factors with values equal to those of the atoms to which they are attached; remaining H atoms were included in the riding model at idealized positions (C—H = 1.08 \AA) with fixed isotropic temperature factors equal to those of their parent atoms. For 1538 reflections with $F > 5\sigma(F)$, final $R = 0.074$, $wR = 0.076$, $S = 0.588$, for 247 refined parameters, quantity minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1/[\sigma^2(F_o) + 0.043F_o^2]$. The R value is rather high because of poor crystallinity. $(\Delta/\sigma)_{max} = 0.78$. Peak heights in the difference Fourier map were in the range -0.23 – 0.25 e \AA^{-3} . Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1. Bond lengths, bond angles, selected torsion angles, contact distances with the Na atom and angles

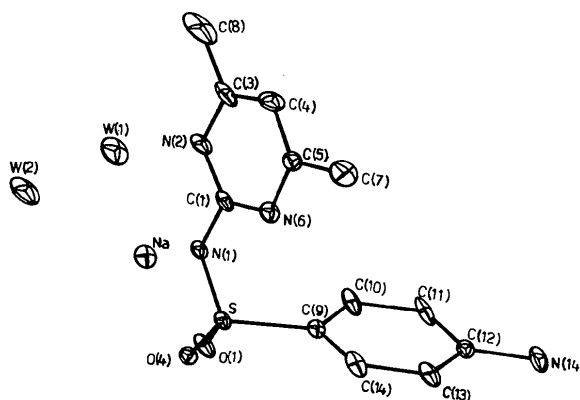


Fig. 1. Molecular structure of the title compound.

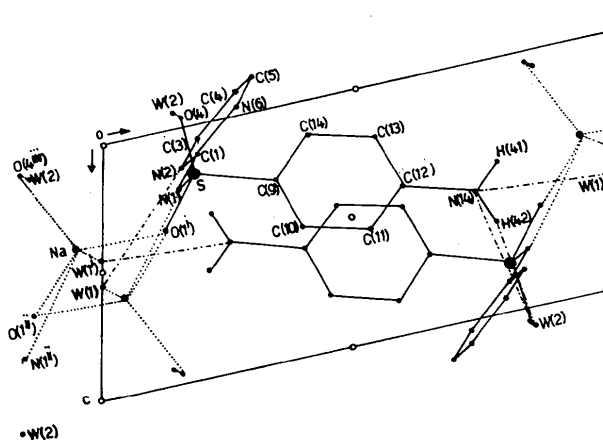


Fig. 2. Projection of the unit cell down the b axis showing numbering of atoms, packing of molecules, coordination and hydrogen bonds. All the H atoms except H(41) and H(42) are omitted for clarity.

around the Na atom are listed in Table 2.* The molecular structure is shown in Fig. 1. The molecular packing viewed along the *b* axis, showing atomic numbering and molecular conformation, is depicted in Fig. 2.

Related literature. The structural parameters can be compared with those of 2-sulfanilamido-5-methoxypyrimidine (Giuseppetti, Tadini, Bettinetti & Giordano, 1977), 4-amino-*N*-(4,6-dimethyl-2-pyrimidinyl)benzenesulfonamide (Basak, Mazumdar & Chaudhuri, 1983), tolbutamide (Donaldson, Leary, Ross, Thomas & Smith, 1981) and *p*-

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and bond angles involving H atoms, and deviations from least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55262 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0507]

aminobenzenesulfonacetamide sodium monohydrate (Ghosh, Basak & Mazumdar, 1987).

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Bis-Adducts of Trimethylaluminium and Trimethylgallium with *N,N,N',N'*-Tetramethylethylenediamine

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Abstract. μ -(*N,N,N',N'*-Tetramethylethylenediamine)-bis(trimethylaluminium) (*1a*), $[\text{Al}_2(\text{CH}_3)_6(\text{C}_6\text{H}_{16}\text{N}_2)]$, $M_r = 260.38$, monoclinic, $P2_1/n$, $a = 10.719$ (3), $b = 7.648$ (2), $c = 12.224$ (3) Å, $\beta = 110.32$ (2)°, $V = 939.7$ (5) Å³, $Z = 2$, $D_x = 0.92$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, graphite monochromator, $\mu = 1.4$ cm⁻¹, $F(000) = 292$, $T = 294$ K, final $R = 0.0487$ for 1132 observed [$I > 3\sigma(I)$] reflections. The molecule is situated about an inversion center coincident with the midpoint of the C—C bond. The Al—N bond distance is 2.074 (2) Å. μ -(*N,N,N',N'*-Tetramethylethylenediamine)-bis(trimethylgallium) (*1b*), $[\text{Ga}_2(\text{CH}_3)_6(\text{C}_6\text{H}_{16}\text{N}_2)]$, $M_r = 345.86$, monoclinic, $P2_1/n$, $a = 10.735$ (3), $b = 7.656$ (2), $c = 12.310$ (4) Å, $\beta = 110.70$ (2)°, $V = 946.4$ (4) Å³, $Z = 2$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, graphite monochromator, $\mu = 28.2$ cm⁻¹, $F(000) = 364$, $T = 294$ K, final $R = 0.0800$ for 909 observed [$I > 3\sigma(I)$] reflections. Compound (*1b*) is

isomorphous with (*1a*). The Ga—N bond distance is 2.174 (8) Å.

Experimental. Data quality crystals of (*1a*) and (*1b*) were prepared by heating (393 K) stoichiometric amounts of the corresponding trimethylmetal derivative with *N,N,N',N'*-tetramethylethylenediamine in heptane, followed by slow cooling of the reaction mixture. All manipulations were carried out under an atmosphere of argon gas. Colorless parallelepiped crystals of (*1a*) (0.2 × 0.5 × 0.7 mm) and (*1b*) (0.1 × 0.3 × 0.5 mm), mounted in sealed glass capillaries under argon, were used for data collection on a Nicolet *R3m/V* diffractometer using $\omega/2\theta$ scans of 2–15° min⁻¹. Unit-cell dimensions were determined by least-squares fit to settings for a number of high-order reflections [34 with $27 < 2\theta < 40^\circ$ for (*1a*) and 46 with $27 < 2\theta < 36^\circ$ for (*1b*)]. Lorentz and polarization corrections were applied. Three standards monitored after every 100 measurements showed $\pm 2\%$ fluctuation for (*1a*) (18.3 h of X-ray exposure;

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