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Bis[*N*¹-(2-pyrimidinyl)sulphanilamido]zinc–Ammonia (1/2),* [Zn(C₁₀H₉N₄O₂S)₂].2NH₃

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Abstract. $M_r = 597.98$, orthorhombic, $Pn2_1a$ (non-standard setting of $Pna2_1$), $a = 13.887$ (4), $b = 14.217$ (4), $c = 12.603$ (4) Å, $V = 2488.23$ Å³, $Z = 4$, $D_m = 1.62$ (1), $D_x = 1.596$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 1.23$ mm⁻¹, $F(000) = 1232$, $T = 294$ K, $R = 0.041$ for 2407 observed diffractometer reflexions [$I > \sigma(I)$]. The Zn atom is coordinated tetrahedrally to four N atoms, one from each of the two sulphadiazine molecules and two of the ammonias of crystallization. The two rings of each molecule are inclined to each other at +95.71 (5) and +85.57 (5)°, and are linked to each other and to the two ammonia molecules by a three-dimensional system of hydrogen bonds, of lengths between 2.833 and 3.194 (10) Å. Bond lengths and interbond angles are within the usual range.

Introduction. The title compound is one of a series of sulphonamides which have been investigated for the treatment and control of infection in burns. The structures of silver sulphadiazine (Cook & Turner, 1975), [*N*¹-(3-pyridyl)sulphanilamido]silver (Cook & Turner, 1976) and *N*¹-phenylsulphanilamide (Brown, Cook & Sengier, 1984) have already been published from these laboratories, and an extensive literature on the crystal structures of sulpha drugs has appeared in recent years. This investigation was undertaken to determine the coordination of the Zn atom and to

ascertain the role of the NH₃ of crystallization in the molecular structural arrangement.

Experimental. Samples supplied by both Professor C. L. Fox of Columbia University, New York, and by Smith & Nephew Ltd, Harlow, Essex; m.p. 509 K (dec.); analysis gave Zn 10.96, C 39.59, H 4.03, N 23.17% (required for C₂₀H₂₄N₁₀O₄S₂Zn: Zn 10.93, C 40.17, H 4.05, N 23.42%). White needle-shaped crystals ca 0.1 × 0.1 × 0.8 mm from 10% ammonia solution in the dark. D_m by flotation in NaI solution. Lattice parameters initially from rotation photographs, subsequently refined on the diffractometer. Intensities measured on CAD-4 diffractometer with Mo $K\alpha$ radiation; $2\theta_{\max} = 54^\circ$, index range h 0→17, k 0→18, l 0→16; 2 standard reflexions, no variation; 2814 observed reflexions, 407 of which with $I < \sigma(I)$; corrections for Lp but not for absorption or extinction; structure solved by Patterson synthesis followed by successive Fourier syntheses; refinement by least squares on F using NRC programs (Ahmed, Hall, Pippy & Huber, 1970) on our DEC-20 computer; NH₃ molecules from difference Fourier map, other H atoms by calculation by bisecting bond angles or on direct line of H bonds where appropriate; H used in F calculations with $B_{\text{iso}} = 7.0$ Å² but not refined; non-H atoms refined using B_{iso} initially and finally B_{ij} until all shifts $< 0.2\sigma$; residual $\Delta\rho$ in final difference Fourier synthesis within $0.2 e \text{ \AA}^{-3}$ (except +0.9 at Zn and +0.5 $e \text{ \AA}^{-3}$ at S atoms); scattering factors from *International Tables for X-ray Crystallography* (1974); $R = 0.041$, $wR = 0.094$, $\sqrt{w} = 1/F_o$.

* Bis[4-amino-*N*-(2-pyrimidinyl)benzenesulphonamido]zinc–ammonia (1/2); zinc sulphadiazine–ammonia (1/2).

Table 1. Final atomic parameters and e.s.d.'s

$$B_{eq} = \frac{4}{3}[(\beta_{11}/a^{*2}) + (\beta_{22}/b^{*2}) + (\beta_{33}/c^{*2})].$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Zn	0.25748 (6)	0.29738	0.92273 (7)	4.22 (1)
S(1)	0.3008 (1)	0.0970 (1)	1.0739 (1)	3.34 (2)
S(2)	0.3993 (1)	0.2200 (1)	0.6496 (1)	3.82 (2)
O(1)	0.2679 (3)	0.0574 (4)	1.1740 (4)	3.92 (7)
O(2)	0.2747 (3)	0.0564 (3)	0.9712 (4)	3.91 (6)
O(3)	0.4793 (3)	0.1579 (4)	0.6723 (5)	4.32 (8)
O(4)	0.3053 (5)	0.1820 (5)	0.6230 (4)	6.05 (9)
N(1)	0.2612 (3)	0.2061 (5)	1.0524 (6)	4.38 (9)
N(2)	0.7184 (4)	0.1108 (7)	1.0859 (6)	4.63 (10)
N(3)	0.2506 (4)	0.3577 (6)	1.0995 (5)	4.21 (8)
N(4)	0.2787 (6)	0.2488 (4)	1.2406 (4)	4.65 (8)
N(5)	0.3668 (4)	0.2856 (5)	0.7523 (6)	5.26 (11)
N(6)	0.5281 (4)	0.4458 (4)	0.2792 (5)	3.02 (8)
N(7)	0.5328 (3)	0.3152 (4)	0.8003 (5)	4.54 (8)
N(8)	0.4038 (4)	0.3346 (3)	0.9192 (3)	2.37 (6)
N(9)	0.1944 (4)	0.1949 (5)	0.8360 (5)	4.40 (9)
N(10)	0.1770 (5)	0.4134 (6)	0.8778 (6)	6.16 (12)
C(1)	0.4275 (3)	0.1026 (4)	1.0786 (4)	3.14 (7)
C(2)	0.4783 (4)	0.1103 (3)	0.9834 (3)	2.11 (5)
C(3)	0.5781 (6)	0.1151 (4)	0.9800 (6)	3.81 (10)
C(4)	0.6194 (5)	0.1085 (4)	1.0796 (5)	3.41 (9)
C(5)	0.5796 (4)	0.0999 (8)	1.1790 (6)	5.92 (10)
C(6)	0.4796 (8)	0.0989 (6)	1.1718 (8)	5.17 (13)
C(7)	0.2653 (3)	0.2707 (7)	1.1371 (5)	3.41 (9)
C(8)	0.2473 (5)	0.4269 (6)	1.1710 (10)	7.06 (11)
C(9)	0.2564 (5)	0.4161 (6)	1.2790 (5)	4.44 (11)
C(10)	0.2718 (4)	0.3237 (3)	1.3042 (3)	2.57 (6)
C(11)	0.4388 (4)	0.2946 (4)	0.5481 (4)	3.12 (7)
C(12)	0.4242 (3)	0.3912 (3)	0.5442 (4)	2.64 (6)
C(13)	0.4553 (9)	0.4426 (7)	0.4573 (9)	5.78 (12)
C(14)	0.4983 (7)	0.3999 (6)	0.3701 (10)	5.73 (14)
C(15)	0.5118 (5)	0.3028 (10)	0.3766 (7)	5.66 (12)
C(16)	0.4839 (5)	0.2490 (7)	0.4639 (6)	5.12 (9)
C(17)	0.4389 (4)	0.3131 (3)	0.8239 (5)	2.97 (7)
C(18)	0.5952 (6)	0.3426 (6)	0.8747 (6)	4.15 (12)
C(19)	0.5563 (5)	0.3647 (3)	0.9720 (3)	2.30 (7)
C(20)	0.4593 (5)	0.3636 (6)	0.9993 (4)	4.38 (9)

Table 2. Bond lengths (\AA) and interbond angles ($^\circ$)

Zn-N(1)	2.087 (8)	Zn-N(9)	2.022 (7)
Zn-N(10)	2.071 (9)	Zn-N(8)	2.100 (6)
S(1)-O(1)	1.455 (6)	S(2)-O(3)	1.448 (6)
S(1)-O(2)	1.463 (5)	S(2)-O(4)	1.453 (8)
S(1)-N(1)	1.668 (8)	S(2)-N(5)	1.658 (8)
S(1)-C(1)	1.763 (6)	S(2)-C(11)	1.749 (6)
N(1)-C(7)	1.410 (12)	N(5)-C(17)	1.404 (10)
N(2)-C(4)	1.377 (9)	N(6)-C(14)	1.382 (14)
N(3)-C(7)	1.339 (14)	N(7)-C(17)	1.338 (8)
N(3)-C(8)	1.335 (14)	N(7)-C(18)	1.335 (10)
N(4)-C(7)	1.354 (10)	N(8)-C(17)	1.332 (8)
N(4)-C(10)	1.336 (8)	N(8)-C(20)	1.335 (9)
C(1)-C(2)	1.397 (7)	C(11)-C(12)	1.389 (8)
C(1)-C(6)	1.381 (12)	C(11)-C(16)	1.393 (10)
C(2)-C(3)	1.388 (10)	C(12)-C(13)	1.386 (13)
C(3)-C(4)	1.384 (11)	C(13)-C(14)	1.391 (18)
C(4)-C(5)	1.375 (11)	C(14)-C(15)	1.396 (19)
C(5)-C(6)	1.391 (14)	C(15)-C(16)	1.395 (15)
C(8)-C(9)	1.376 (15)	C(18)-C(19)	1.377 (9)
C(9)-C(10)	1.368 (11)	C(19)-C(20)	1.391 (10)
O(1)-S(1)-O(2)	122.5 (3)	O(3)-S(2)-O(4)	120.5 (4)
O(1)-S(1)-N(1)	113.4 (3)	O(3)-S(2)-N(5)	113.5 (4)
O(1)-S(1)-C(1)	107.6 (3)	O(3)-S(2)-C(11)	105.9 (3)
O(2)-S(1)-N(1)	98.2 (3)	O(4)-S(2)-N(5)	98.3 (4)
O(2)-S(1)-C(1)	107.1 (3)	O(4)-S(2)-C(11)	109.8 (4)
N(1)-S(1)-C(1)	107.0 (3)	N(5)-S(2)-C(11)	108.4 (3)
S(1)-N(1)-C(7)	118.0 (6)	S(2)-N(5)-C(17)	117.7 (6)
C(7)-N(3)-C(8)	116.6 (8)	C(17)-N(7)-C(18)	118.9 (6)
C(7)-N(4)-C(10)	112.6 (6)	C(17)-N(8)-C(20)	122.8 (6)
S(1)-C(1)-C(2)	118.6 (4)	S(2)-C(11)-C(12)	125.4 (5)
S(1)-C(1)-C(6)	123.4 (6)	S(2)-C(11)-C(16)	114.6 (5)
C(2)-C(1)-C(6)	118.0 (6)	C(12)-C(11)-C(16)	119.9 (6)
C(1)-C(2)-C(3)	122.4 (5)	C(11)-C(12)-C(13)	120.3 (7)
C(2)-C(3)-C(4)	112.5 (7)	C(12)-C(13)-C(14)	121.9 (1.0)
N(2)-C(4)-C(3)	117.7 (7)	N(6)-C(14)-C(13)	125.3 (1.0)
N(2)-C(4)-C(5)	110.5 (7)	N(6)-C(14)-C(15)	118.4 (1.0)
C(3)-C(4)-C(5)	131.8 (7)	C(13)-C(14)-C(15)	116.3 (1.1)
C(4)-C(5)-C(6)	110.1 (8)	C(14)-C(15)-C(16)	123.5 (1.1)
C(1)-C(6)-C(5)	125.3 (9)	C(11)-C(16)-C(15)	118.1 (8)
N(1)-C(7)-N(3)	109.1 (7)	N(5)-C(17)-N(7)	123.9 (6)
N(1)-C(7)-N(4)	125.8 (7)	N(5)-C(17)-N(8)	112.5 (6)
N(3)-C(7)-N(4)	125.1 (8)	N(7)-C(17)-N(8)	123.5 (6)
N(3)-C(8)-C(9)	125.6 (9)	N(7)-C(18)-C(19)	115.9 (7)
C(8)-C(9)-C(10)	110.6 (7)	C(18)-C(19)-C(20)	126.8 (6)
N(4)-C(10)-C(9)	129.6 (6)	N(8)-C(20)-C(19)	112.1 (6)

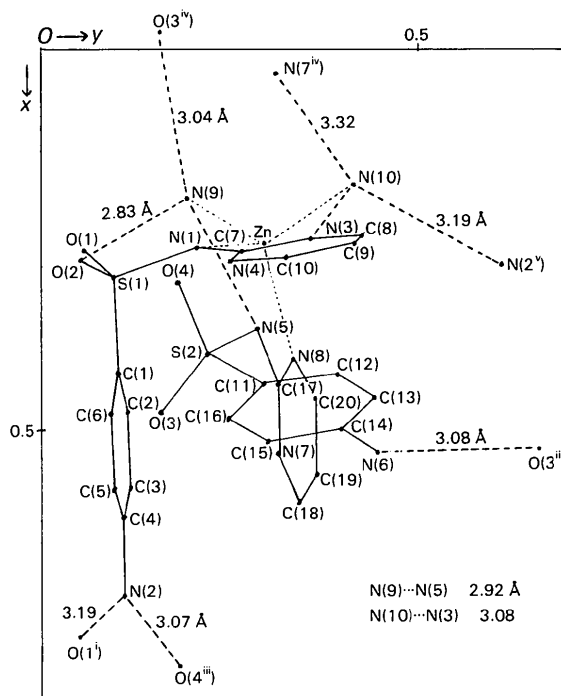


Fig. 1. Arrangement of the molecule in (001) projection of the unit cell showing numbering of atoms, coordination of Zn (dotted lines) and H-bonding system (dashed lines). E.s.d.'s in H-bond distances are 0.01 Å. Symmetry code: (i) $\frac{1}{2} + x, y, \frac{1}{2} - z$; (ii) $1 - x, \frac{1}{2} + y, 1 - z$; (iii) $\frac{1}{2} + x, y, 1\frac{1}{2} - z$; (iv) $x - \frac{1}{2}, y, 1\frac{1}{2} - z$; (v) $1 - x, \frac{1}{2} + y, -z$.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,* and bond lengths and interbond angles are in Table 2.

The Zn atom coordinates to N(1), N(8), N(9) and N(10) at a mean distance of 2.070 (7) Å, with mean N-Zn-N angle 110.0°. There is also a Zn-N(3) distance of 2.375 (7) Å, but this is probably outside the sphere of coordination.

The intramolecular bond lengths and angles all agree well with those found in similar compounds (see *Introduction*) and in sulphamethazine (Basak, Mazumdar & Chaudhuri, 1983). The four molecular rings are each effectively planar, the r.m.s. deviations being for C(1)-(6) 0.009, C(7)-(10), N(3), N(4) 0.009, C(11)-(16) 0.009, and C(17)-(20), N(7), N(8) 0.008 Å. The dihedral angles between the normals to these planes are (1:2) +95.71 (5), (1:3) +81.51 (5), (1:4) +158.01 (5), (2:3) +149.64 (5), (2:4) +86.52 (5), (3:4) +85.57 (5)°.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, mean planes and H-bonding geometry have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42041 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Only eight of the ten available H atoms are used in definite H bonding (*cf.* Fig. 1); there is a ninth possible H bond shown, N(10)⋯N(7), but this is 3.316 (10) Å, and probably outside the range. As the electron density around the N atoms in the difference map was somewhat diffuse, it is not possible to be absolutely certain as to the H-atom positions, so confirmation of the H-bonding scheme will need to await a neutron analysis.

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The Structure of 3,5-Di-*O*-benzoyl-1,2-dideoxy-1-phenyl-β-D-ribofuranose, C₂₅H₂₂O₅

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Abstract. $M_r = 402.4$, orthorhombic, $P2_12_12_1$, $a = 4.946$ (1), $b = 15.887$ (2), $c = 26.555$ (2) Å, $V = 2086.7$ (5) Å³, $Z = 4$, $D_x = 1.28$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 6.868$ cm⁻¹, $F(000) = 848$, $T = 293$ K, final $R = 0.054$ for 648 observed reflections. The molecule is propeller shaped. The benzoyl groups act as protecting groups and the phenyl group is a base substitute. The crystal structure does not involve any intermolecular stacking interactions between the phenyl groups. The molecules pack in typical herring-bone-like arrays. The sugar has a β-D configuration with C(2′)-endo–C(3′)-exo pucker (²T₃), pseudorotation angle $P = 172$ (2)°, degree of pucker $\tau_m = 39$ (2)°.

Introduction. This molecule was synthesized as a protected base analogue for eventual insertion into an oligonucleotide strand in order to investigate its effects on DNA duplex stability (Millican, Mock, Eaton, Patel, Chauncey, Mann, Gunning, Cutbush & Neidle, 1984).

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This forms an initial part of a series of experiments to design a ‘universal’ base, which would obviate the necessity for synthesizing multiple gene probes in cases where there are ambiguities in the genetic code.

Experimental. Recrystallization from diethyl ether as colourless needles, Enraf–Nonius CAD-4 diffractometer, crystal fragment 0.2 × 0.1 × 0.05 mm, Ni-filtered Cu $K\alpha$ radiation. Unit cell from θ values of 25 reflections, $\omega/2\theta$ mode, max. scan time 120 s, 1424 measured reflections, unique set of 648 with $I > 2\sigma(I)$, $1.5 < \theta < 50^\circ$, index range h 0/4, k 0/15, l 0/25; L_p corrections. Structure solved by direct methods with *MULTAN80* (Main *et al.*, 1980); H in calculated positions and not refined; common B of 5.0 Å² assigned. Full-matrix least-squares refinement (with Enraf–Nonius *SDP* package; Frenz, 1978); anisotropic temperature factors only for O atoms, due to small number of observations; isotropic temperature factors for all other non-hydrogen atoms, $R = 0.054$, $wR = 0.055$ with $w = 1/\sigma^2(F)$, max. $\Delta/\sigma = 0.01$, max. and min. densities in final difference synthesis 0.3 and -0.2 e Å⁻³, minimization of $\sum w(|F_o| - |F_c|)^2$. Atom