

Fig. 1. A perspective view of (2) with atomic labelling.

The structures of the related cross-conjugated heterocyclic mesomeric betaines (5) (Kratky & Kappe, 1981) (6) (Debaerdemaeker & Friedrichsen, 1982) and (7) (Raston & White, 1984) and also of the pseudo cross-conjugated heterocyclic mesomeric betaine (8) (Potts, Murphy, DeLuca &

Kuehnlung, 1988), have all been confirmed by X-ray investigations.

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Structure of 2-Azacycloheptanone Azine

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Abstract. $C_{12}H_{22}N_4$, $M_r = 222.34$, triclinic, $P\bar{1}$, $a = 10.80$ (1), $b = 6.00$ (1), $c = 5.410$ (5) Å, $\alpha = 113.16$ (6), $\beta = 77.21$ (5), $\gamma = 92.84$ (7)°, $V = 313.04$ Å³, $Z = 1$, $D_x = 1.175$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.685$ cm⁻¹, $F(000) = 122.0$, $T = 290$ K, $R = 0.039$ for 881 unique observed reflexions. The molecule lies on a centre of symmetry. The central N—N bond of the azine system is 1.419 (2) Å and the N—C bond to the ring is 1.297 (2) Å; the (N=CNC)₂ system is planar to within 0.02 Å, which together with the shortened distances indicates a weak π system. This results in a close intramolecular NH...N approach, H...N = 2.18 (2), N...N = 2.572 (2) Å.

Experimental. The compound was supplied by Dr S. W. Breuer. Crystals were readily deformable thin plates, tabular on (100), showing the forms {100}, {010}, {001}, {011}. Cell dimensions, initially from Weissenberg and precession photographs [$\lambda(\text{Cu } K\alpha) = 1.542$ Å], refined from setting angles of 20 reflexions in the range $10 < 2\theta < 40^\circ$ on Stadi-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$).

This was also used for measurement of 1122 unique intensities, from a crystal of dimensions $0.3 \times 0.14 \times 0.07$ mm, for the layers $hk0$ to $hk6$, $h - 11$ to 12, $k - 7$ to 6, max. $\sin\theta/\lambda = 0.6$ Å⁻¹; variable ω scan, $2\theta'$ fixed, stationary background count, standard measured every 20 reflexions, intensity variation $< 5\%$ corrected for by linear interpolation. Lp corrections but not absorption corrections were applied, C- and N-atom positions were obtained by the use of the direct-methods program *EEES* of *SHELX76* (Sheldrick, 1976), which was used for all calculations. H-atom positions were obtained from ΔF maps. Full-matrix least-squares refinement of coordinates, U_{ij} of C and N atoms and U_{iso} of H atoms, using F values of 881 reflexions for which $I > 3\sigma(I)$ proceeded until Δ/σ was < 0.003 . At an intermediate stage of the refinement interlayer scale factors were refined. At the conclusion of the refinement of 123 parameters the largest features on the F map lay between -0.18 and 0.13 e Å⁻³, $R = 0.039$, $wR = 0.045$, $S = 1.514$, observation/parameter ratio $Q = 7.16$ and $w = 2.018/[\sigma^2(F) + 0.00098F^2]$. Atomic scattering factors from *International Tables for X-ray*

Table 1. Fractional atomic coordinates and U_{eq} values (\AA^2)

	x	y	z	U_{eq}
N(1)	0.0217 (1)	0.1015 (2)	0.9689 (2)	0.044 (1)
N(2)	0.1432 (1)	-0.1976 (3)	0.5758 (3)	0.052 (1)
C(1)	0.1022 (1)	0.0356 (3)	0.7333 (3)	0.041 (1)
C(2)	0.1536 (2)	0.2298 (3)	0.6370 (3)	0.046 (1)
C(3)	0.2987 (2)	0.2424 (3)	0.5812 (3)	0.054 (1)
C(4)	0.3675 (2)	0.0568 (4)	0.3048 (3)	0.063 (1)
C(5)	0.3531 (2)	-0.2070 (4)	0.2686 (4)	0.061 (1)
C(6)	0.2171 (2)	-0.2878 (3)	0.2949 (3)	0.053 (1)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

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

N(1)—N(1')	1.419 (2)	N(1')—N(1)—C(1)	111.5 (1)
N(1)—C(1)	1.297 (2)	C(6)—N(2)—C(1)	126.1 (2)
N(2)—C(1)	1.356 (2)	N(2)—C(1)—C(2)	119.0 (1)
N(2)—C(6)	1.449 (2)	N(1)—C(1)—N(2)	123.5 (1)
C(1)—C(2)	1.491 (2)	N(1)—C(1)—C(2)	117.5 (1)
C(2)—C(3)	1.533 (2)	C(1)—C(2)—C(3)	114.8 (1)
C(3)—C(4)	1.520 (2)	C(2)—C(3)—C(4)	114.3 (1)
C(4)—C(5)	1.521 (3)	C(3)—C(4)—C(5)	115.4 (2)
C(5)—C(6)	1.517 (2)	C(4)—C(5)—C(6)	114.6 (1)
		C(5)—C(6)—N(2)	113.8 (1)

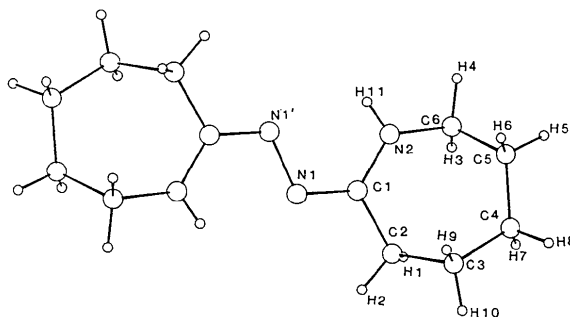
Symmetry code: (') $-x, -y, 2-z$.

Fig. 1. View of molecule with atomic labelling.

Crystallography (1974, Vol. IV). Atomic parameters are given in Table 1,* bond distances and angles in Table 2. The molecule with atomic labelling is shown in Fig. 1.

Related literature. The azacycloheptane ring occurs in 1-azacycloheptane hydrochloride (Cameron & Scheeren, 1977). Numerous structures involving the

* Lists of structure factors, H-atom parameters, bond distances involving H atoms, anisotropic vibrational factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54352 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(2-aminoethyl)amine Perchlorate

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Abstract. $[\text{N}(\text{CH}_2\text{CH}_2\text{NH}_3)_3](\text{ClO}_4)_3$, $M_r = 447.6$, cubic, $P2_13$, $a = 11.858 (5) \text{\AA}$, $V = 1667.4 \text{\AA}^3$, $Z = 4$, $D_x = 1.78 \text{ g cm}^{-3}$, $\text{Mo K}\alpha$, $\lambda = 0.71073 \text{\AA}$, $\mu = 5.49 \text{ cm}^{-1}$, $F(000) = 928$, $T = 293 \text{ K}$, $R = 0.038$ for 646 reflections with $I > 3\sigma(I)$. The cations and

azine $\text{C}=\text{N}-\text{N}=\text{C}$ grouping have been described; only in a few of these are the C atoms part of a ring. In one of these there is an N atom in the 2-position of the ring, 4,5-dihydro-6-(2-thienyl)-3(2*H*)-pyridazinone azine (Will, Farag & El-Kordy, 1984). Others involving carbon rings are *N,N'*-bis[2-(4-chlorophenylamino)-2-cyclopenten-1-ylidene]hydrazine (Villa, Manfredotti, Guastini & Pocar, 1980) and bis(1,2,3,4-tetrahydro-6-methoxy-2-naphthylidene)hydrazinecarboximidamide sulfate monohydrate (Pitzele, Moormann, Gulliken, Albin, Bianchi, Palicharla, Sanguinetti & Walters, 1988).

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anions of the title compound all lie on a threefold axis of the cubic unit cell. There are significant hydrogen-bonding interactions between cation- NH_3^+ groups and perchlorate-O atoms, but no such cation-cation interactions.