

(Johnson, 1976) shows the structure of (I) and the atom-numbering scheme used.

Related literature. The structure of (I) was determined during the course of developing efficient synthetic approaches to all stereoisomers of 2-(hydroxymethyl)pyrrolidine-3,4-diols, which have important biological properties, especially in the stimulation of the immune response (Meng & Hesse, 1991).

Professor M. Hesse and Mr Q. Meng are thanked for providing the crystals.

References

- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 MENG, Q. & HESSE, M. (1991). *Helv. Chim. Acta*, **74**, 445–450.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELX86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Acta Cryst. (1992). **C48**, 1516–1518

Structure of 3,4-Diethylcanthine-5,6-dione

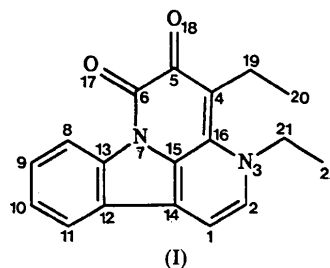
BY XIAOLING SUI AND PENELOPE W. CODDING*

Department of Chemistry, University of Calgary, Calgary, Alberta, Canada T2N 1N4

(Received 23 September 1991; accepted 7 January 1992)

Abstract. 3,4-Diethylindolo[3,2,1-*de*][1,5]naphthyridine-5,6-dione, C₁₈H₁₆N₂O₂, *M_r* = 292.34, triclinic, *P* $\bar{1}$, *a* = 8.4118 (2), *b* = 11.9934 (4), *c* = 7.9901 (4) Å, α = 83.493 (3), β = 114.946 (3), γ = 101.598 (3)°, *V* = 715.61 (5) Å³, *Z* = 2, *D_x* = 1.356 g cm⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.54178 Å, μ = 6.8 cm⁻¹, *F*(000) = 308, *T* = 293 K, *R* = 0.039 and *wR* = 0.045 for 2690 unique observed reflections. The canthine-5,6-dione skeleton is planar (r.m.s. = 0.031 Å and χ^2 = 1.06 for 16 atoms). The side chains are oriented so that the terminal C—C bonds of the ethyl groups are perpendicular to the plane of the canthine-5,6-dione with one group above and the other below the plane [the torsion angles are $\tau_1 = \text{C}(5)\text{—C}(4)\text{—C}(19)\text{—C}(20) = 89.2$ (2) and $\tau_2 = \text{C}(16)\text{—N}(3)\text{—C}(21)\text{—C}(22) = -88.6$ (2)°]. In the crystal, the molecules that are related by the center of symmetry stack in head-to-tail pairs with a distance between the planes of the aromatic rings of adjacent molecules of 3.384 Å. Owing to the lack of hydrogen-bond donors in the crystal, there are no hydrogen-bond interactions.

eters and orientation matrix were determined by least-squares refinement of the angular positions of 25 reflections with $35 < \theta < 50^\circ$. During the data collection the intensities of three reflections (116, 045, 132) were measured every 2000 s of X-ray exposure time; these reflections showed no significant variation in intensity. Intensities for 3073 reflections were collected [*h* -10→10, *k* 0→15, *l* -10→10, maximum ($\sin\theta/\lambda$) = 0.6265 Å⁻¹], of which 2928 reflections were unique (*R_{int}* = 0.044) and 2690 reflections with $I \geq 2.5\sigma(I)$ were regarded as observed and included in the refinement. Lorentz and polarization corrections were applied and absorption correction was not necessary.



Experimental. The title compound was provided by Dr Janos Fischer of Gedeon Richter Ltd, Budapest, Hungary. A single crystal of dimensions 0.36 × 0.37 × 0.14 mm was obtained by slow evaporation from dichloromethane. X-ray intensity data were collected at room temperature on an Enraf-Nonius CAD-4F automated diffractometer with Ni-filtered Cu *K*α radiation, using an $\omega/2\theta$ scan mode. The cell param-

The structure was solved by direct methods with *SHELXS86* (Sheldrick, 1986) and refined on *F* by full-matrix least-squares techniques with *XRAY76* (Stewart, 1976). Most of the H atoms were located in difference Fourier syntheses and the remaining H atoms were placed in calculated idealized positions. The parameters varied in the final refinement cycles consisted of the coordinates of all the atoms, the

* Author to whom correspondence should be addressed.

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for the non-H atoms with e.s.d.'s in parentheses

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

	x	y	z	U_{eq}
C(1)	4946 (2)	4475 (1)	2535 (2)	501 (5)
C(2)	5210 (2)	3397 (1)	2556 (2)	491 (5)
N(3)	4106 (1)	2626 (1)	1214 (1)	425 (4)
C(4)	1348 (2)	2194 (1)	-1748 (2)	397 (5)
C(5)	-102 (2)	2676 (1)	-3141 (2)	440 (5)
C(6)	-263 (2)	3914 (1)	-3123 (2)	440 (5)
N(7)	1063 (1)	4521 (1)	-1670 (1)	397 (4)
C(8)	244 (2)	6489 (1)	-2157 (2)	521 (6)
C(9)	768 (2)	7536 (1)	-1338 (3)	613 (8)
C(10)	2219 (2)	7744 (1)	341 (3)	631 (8)
C(11)	3225 (2)	6915 (1)	1267 (2)	556 (7)
C(12)	2745 (2)	5855 (1)	474 (2)	432 (5)
C(13)	1254 (2)	5658 (1)	-1210 (2)	421 (5)
C(14)	3481 (2)	4816 (1)	1034 (2)	416 (5)
C(15)	2421 (1)	4034 (1)	-297 (2)	376 (4)
C(16)	2618 (2)	2894 (1)	-308 (2)	373 (4)
O(17)	-1473 (1)	4346 (1)	-4283 (1)	623 (4)
O(18)	-1331 (1)	2127 (1)	-4434 (1)	617 (4)
C(19)	1267 (2)	959 (1)	-2053 (2)	514 (6)
C(20)	2205 (3)	791 (2)	-3242 (3)	689 (9)
C(21)	4589 (2)	1477 (1)	1633 (2)	526 (6)
C(22)	3709 (4)	785 (2)	2795 (3)	756 (10)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) for the non-H atoms with e.s.d.'s in parentheses

C(1)—C(2)	1.352 (2)	N(7)—C(15)	1.384 (1)
C(1)—C(14)	1.403 (2)	C(8)—C(9)	1.385 (1)
C(2)—N(3)	1.381 (2)	C(8)—C(13)	1.383 (2)
N(3)—C(16)	1.393 (1)	C(9)—C(10)	1.387 (2)
N(3)—C(21)	1.476 (2)	C(10)—C(11)	1.376 (2)
C(4)—C(5)	1.433 (2)	C(11)—C(12)	1.394 (2)
C(4)—C(16)	1.411 (2)	C(12)—C(13)	1.403 (2)
C(4)—C(19)	1.511 (2)	C(12)—C(14)	1.444 (2)
C(5)—C(6)	1.520 (2)	C(14)—C(15)	1.377 (2)
C(5)—O(18)	1.244 (1)	C(15)—C(16)	1.411 (2)
C(6)—O(17)	1.212 (2)	C(19)—C(20)	1.519 (3)
N(7)—C(6)	1.376 (1)	C(21)—C(22)	1.502 (3)
N(7)—C(13)	1.413 (2)		
N(3)—C(16)—C(15)	111.6 (1)	C(1)—C(14)—C(12)	135.0 (1)
N(3)—C(16)—C(4)	129.2 (1)	C(15)—C(14)—C(12)	107.0 (1)
C(15)—C(16)—C(4)	119.2 (1)	C(11)—C(12)—C(14)	133.1 (1)
C(16)—N(3)—C(2)	122.8 (1)	C(11)—C(12)—C(13)	119.6 (1)
C(16)—N(3)—C(21)	123.8 (1)	C(14)—C(12)—C(13)	107.3 (1)
C(2)—N(3)—C(21)	113.2 (1)	C(8)—C(13)—N(7)	129.9 (1)
N(3)—C(2)—C(1)	123.6 (1)	C(8)—C(13)—C(12)	122.6 (1)
C(2)—C(1)—C(14)	117.1 (1)	N(7)—C(13)—C(12)	107.5 (1)
C(10)—C(11)—C(12)	118.2 (1)	N(7)—C(6)—C(5)	115.1 (1)
C(11)—C(10)—C(9)	121.2 (2)	N(7)—C(6)—O(17)	121.7 (1)
C(10)—C(9)—C(8)	122.1 (2)	C(5)—C(6)—O(17)	123.2 (1)
C(9)—C(8)—C(13)	116.3 (1)	C(6)—C(5)—C(4)	121.7 (1)
C(15)—N(7)—C(13)	108.2 (1)	C(6)—C(5)—O(18)	114.7 (1)
C(15)—N(7)—C(6)	122.6 (1)	C(4)—C(5)—O(18)	123.6 (1)
C(13)—N(7)—C(6)	129.1 (1)	C(16)—C(4)—C(5)	118.2 (1)
C(16)—C(15)—N(7)	123.2 (1)	C(16)—C(4)—C(19)	128.9 (1)
C(16)—C(15)—C(14)	126.9 (1)	C(5)—C(4)—C(19)	112.9 (1)
N(7)—C(15)—C(14)	109.9 (1)	C(4)—C(19)—C(20)	113.8 (1)
C(1)—C(14)—C(15)	118.0 (1)	N(3)—C(21)—C(22)	110.8 (2)

anisotropic thermal parameters of non-H atoms and the isotropic thermal parameters of H atoms.

The final agreement factors were $R = 0.039$, $wR = 0.045$ [$w^{-1} = \sigma^2(F) + 0.00002F^2$] and $S = 1.062$ for 264 variables and 2690 observed reflections. An empirical isotropic extinction parameter (g) converged at $3.85(8) \times 10^{-3}$ (Zachariasen, 1967; Larson, 1967). The maximum Δ/σ in the last refinement cycle was 0.0019 and the final difference Fourier map showed residual densities from 0.21 to $-0.17 e \text{\AA}^{-3}$. The scattering factors for non-H atoms were taken

from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965).

The fractional coordinates and B_{eq} values for the non-H atoms are given in Table 1.* Bond lengths and angles are listed in Table 2. The atomic labeling scheme (ORTEPII; Johnson, 1976) and the crystal-packing diagram (PLUTO; Motherwell, 1979) are shown in Figs. 1 and 2, respectively.

Related literature. Bond lengths and angles are in good accord with expected values. The differences between the angles around the equivalent atoms C(14) and C(12), namely C(1)—C(14)—C(15)/C(11)—C(12)—C(13) = 118.0(1)/119.6(1) $^\circ$ and C(1)—C(14)—C(12)/C(11)—C(12)—C(14) = 135.0(1)/

* Lists of anisotropic thermal parameters for the non-H atoms, H-atom coordinates and isotropic thermal parameters, bond distances and angles involving H atoms, least-squares-planes data and structure factors, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55004 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0392]

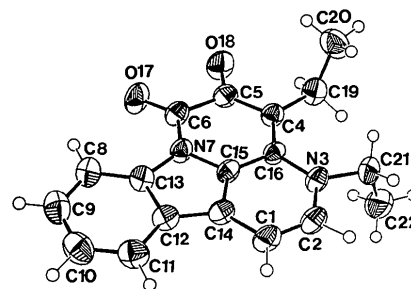


Fig. 1. A view of the title molecule showing the labeling of the non-H atoms. Thermal ellipsoids are drawn at the 50% probability level.

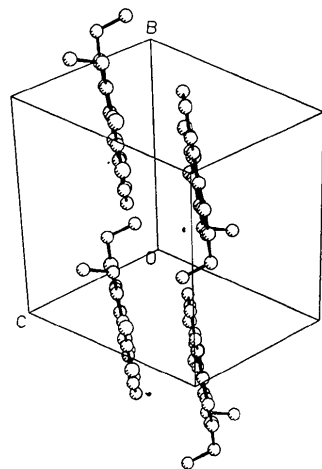


Fig. 2. A portion of the molecular packing in the unit cell, showing the stacking of aromatic units.

133.1 (1)°, are smaller than those in similar structures found in the Cambridge Structural Database (Clarke, Jewers & Jones 1980; Codding, Szkaradzinska & Roszak 1988; Fukamiya, Okano & Aratani 1986).

The authors thank A. W. Roszak for technical assistance, the Alberta Heritage Foundation for Medical Research for a studentship to XS and the Medical Research Council of Canada for financial support (grant to PWC).

References

CLARKE, P. J., JEWERS, K. & JONES, H. F. (1980). *J. Chem. Soc. Perkin Trans. 1*, pp. 1614–1617.

- CODDING, P. W., SZKARADZINSKA, M. B. & ROSZAK, A. W. (1988). *Can. J. Chem.* **66**, 2981–2988.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 FUKAMIYA, N., OKANO, M. & ARATANI, T. (1986). *J. Nat. Prod.* **49**, 428–434.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
 MOTHERWELL, W. D. S. (1979). *PLUTO*. Program for plotting molecular and crystal structures. University Chemical Laboratory, Cambridge, England.
 SHELDRICK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
 STEWART, J. M. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1992). **C48**, 1518–1519

Structure of *N,N'*-Bis(2-hydroxyethyl)piperazine

BY KOJUN OKAMOTO,* SATOSHI FUJII AND KEN-ICHI TOMITA

Department of Physical Chemistry, Faculty of Pharmaceutical Sciences, Osaka University, 1-6 Yamada-Oka, Suita City, Osaka 565, Japan

AND SHOJI ARAI AND YUKIHIRO TSUTSUMI

Chemical Institute, Tohsoh Chemical Co. Ltd, Ouaza-Tomita, Shinnan-Yo City, Yamaguchi 746, Japan

(Received 1 March 1991; accepted 6 January 1992)

Abstract. 1,4-Piperazinediethanol, C₈H₁₈N₂O₂, *M_r* = 174.24, orthorhombic, *Pbca*, *a* = 9.328 (1), *b* = 12.106 (2), *c* = 8.253 (1) Å, *V* = 931.9 (4) Å³, *Z* = 4, *D_m* = 1.236, *D_x* = 1.239 g cm⁻³, λ(Cu Kα) = 1.5418 Å, μ = 7.32 cm⁻¹, *F*(000) = 384, *T* = 293 K, final *R* = 0.052 for 911 independent reflections. The molecule is centrosymmetric with the piperazine ring in a chair form. In the ring the mean N—C length is 1.471 Å and the C—N—C angle is 108.2°. The hydroxyethyl groups are in equatorial positions.

Experimental. Colorless prisms from an ethyl acetate solution. Crystal 0.4 × 0.35 × 0.6 mm (m.p. 408.3–409.1 K). *D_m* measured by flotation. Rigaku AFC-5 four-circle diffractometer, graphite-monochromated Cu Kα radiation. Intensity data collected in ω–2θ scan mode (2θ_{max} = 120°). Unit-cell dimensions by least-squares procedure based on 2θ values (29 < 2θ < 56°) of 48 reflections. Total of 95 reflections measured of which 911 independent (0 ≤ *h* ≤ 10, –13 ≤ *k* ≤ 0, 0 ≤ *l* ≤ 9). No systematic fluctuation in

333, 400 and 004 standard reflections, monitored every 200 reflections. No absorption correction.

Structure determined using *SHELXS86* (Sheldrick, 1986). Block-diagonal and full-matrix least-squares refinement (*HBL5-V*; Ashida, 1979). Σ*w*(*F_o* – *k|F_c|*)² minimized. Weighting scheme: *w* = 0.0 for *F_o* = 0.0, *w* = 1 for |*F_o*| < 10.0, *w* = (10.0/|*F_o*|)² for |*F_o*| > 10.0. Subsequent difference Fourier maps revealed positions for all H atoms. Non-H atoms refined with anisotropic temperature factors, and H atoms with isotropic ones. Final *R* = 0.052, *wR* = 0.065, *S* = 0.25, using 704 observations with |*F_o*| ≥

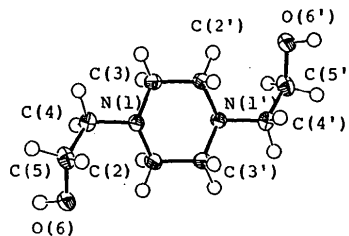


Fig. 1. Numbering scheme and the thermal ellipsoids. Thermal vibration ellipsoids are set to 50% probability (Johnson, 1965).

* Author to whom correspondence should be addressed.