Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SH1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Glycoluril

SHIMIN XU, PETER K. GANTZEL AND LEIGH B. CLARK

Department of Chemistry, University of California, San Diego, 9500 Gilman Drive, La Jolla, California 92093-0359, USA

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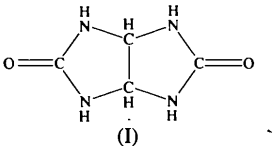
Abstract

The crystal structure of glycoluril [tetrahydroimidazo-[4,5-*d*]imidazole-2,5(1*H*,3*H*)-dione, $C_4H_6N_4O_2$] has been determined. The molecule has two equivalent planes containing urea moieties, and hence exhibits C_{2v} symmetry. The dihedral angle is found to be 124.1 (4)°.

Comment

Glycoluril (I) is a small molecule with relatively high symmetry and has a structure closely related to that of urea.



(I)

A recent study of the electronic spectrum of urea (Campbell & Clark, 1989) obtained experimental results that were contradictory to previous theoretical electronic structure calculations. In order to clarify the situation, polarized electronic spectra of single crystals of glycoluril have been measured (Xu & Clark, 1994). Interpretation of the polarized spectra requires a knowledge of the disposition of the molecules in the crystal. Although glycoluril has no important chemical applications except as a slow release fertilizer (Addiscott & Thomas, 1979; Shimizu, 1987), the fact that it is composed of two symmetrically disposed urea linkages is of significant value with respect to studies of the electronic structure of the basic chromophore. The structure of glycoluril has been investigated previously, but atomic co-

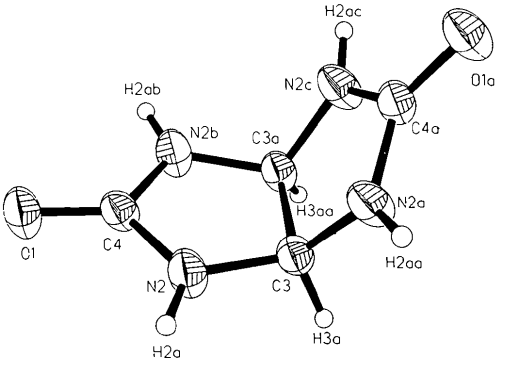


Fig. 1. A view of glycoluril with displacement ellipsoids at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

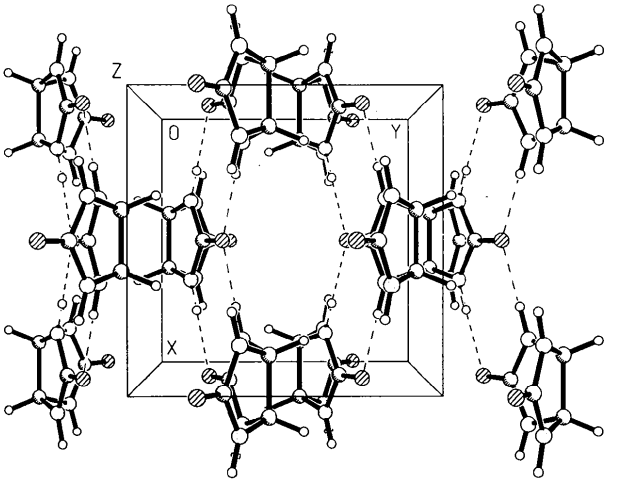


Fig. 2. A packing diagram viewed down the *c* axis showing the hydrogen bonding (O1···H2—N2 2.86 Å). The (010) face is determined to be a cleavage plane.

ordinates have not been published (Blessing & Detilta, 1979; Gilardi, 1982; Pierrot, Baldy, Wimmer, Gallo & Boileau, 1985).

Experimental

Glycoluril was purchased from Aldrich and was used without further purification. Crystals were grown by the slow cooling of a filtered hot aqueous solution. The (010) face is predominant and shows perfect cleavage. This observation is consistent with the sheet-like arrangement of the structure (see Fig. 2).

Crystal data

$C_4H_6N_4O_2$
M_r = 142.12
Orthorhombic
Cmcm
a = 7.373 (2) Å
b = 7.546 (3) Å
c = 10.579 (3) Å
V = 588.6 (3) Å^3
Z = 4
D_x = 1.604 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 16 reflections
θ = 4–13.5°
μ = 0.131 mm^{-1}
T = 299 K
Prism
1.0 × 0.4 × 0.4 mm
Colorless

Data collection

Siemens *R3m/V* diffractometer
Wyckoff scans
Absorption correction: none
550 measured reflections
486 independent reflections
403 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0165
θ_{max} = 29.94°
$h = -1 \rightarrow 10$
$k = 0 \rightarrow 10$
$l = -14 \rightarrow 0$
3 standard reflections monitored every 197 reflections
intensity variation: 0.35%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0405
$wR(F^2)$ = 0.1163
S = 1.114
486 reflections
31 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0785P)^2 + 0.1473P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.268 e Å^{-3}
$\Delta\rho_{min}$ = −0.264 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0	0.2245 (2)	0.46658 (11)	0.0393 (4)
N2	0.14924 (12)	0.3510 (2)	0.63534 (9)	0.0358 (3)
C3	0.1059 (2)	0.4450 (2)	3/4	0.0274 (4)
C4	0	0.3016 (2)	0.57014 (15)	0.0285 (4)

Table 2. *Selected geometric parameters* (Å, °)

O1—C4	1.241 (2)	C3—C3^{i}	1.561 (3)
N2—C4	1.3512 (13)	C4—N2^{ii}	1.3512 (13)
N2—C3	1.4409 (13)		
C4—N2—C3	112.64 (11)	O1—C4—N2	125.47 (7)
N2^{iii}—C3—N2	114.67 (13)	N2—C4—N2^{ii}	109.05 (14)
N2^{iii}—C3—C3^{i}	102.82 (7)		

Symmetry codes: (i) $-x, y, \frac{3}{2} - z$; (ii) $-x, y, z$; (iii) $x, y, \frac{3}{2} - z$.

Data collection, cell refinement and data reduction: *SHELXTL-Plus* (Sheldrick, 1990*a*). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: KA1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A 3-Phenylsulfinyl-1-phenylsulfonylindole

ROY L. BEDDOES, JASON G. KETTLE AND JOHN A. JOULE*

Chemistry Department, University of Manchester, Manchester M13 9PL, England

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Abstract

The title compound 2-(3,4-dihydro-2*H*-pyrrol-5-yl)-1-phenylsulfonylindol-3-yl phenyl sulfoxide, $C_{24}H_{20}N_2O_3S_2$, was isolated, together with 3-chloro-2-(3,4-dihydro-2*H*-pyrrol-5-yl)-1-phenylsulfonylindole, from the treatment of 2-(3,4-dihydro-2*H*-pyrrol-5-yl)indole with sodium hydride and phenylsulfonylchloride, showing that the latter can act as an electrophilic chlorinating agent and as a source of an electrophilic phenylsulfinylating species.