Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dimethyl 2,6-di-*tert*-butyl-4,8-dioxo-1,2,5,6-tetrahydro-2,3a,4a,6,7a,8a-hexaazacyclopenta-[*def*]fluorene-8b,8c-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.148
Data-to-parameter ratio = 14.2

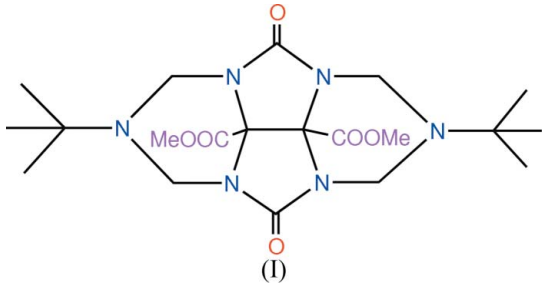
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 October 2005
Accepted 10 November 2005
Online 16 November 2005

The title compound, $C_{20}H_{32}N_6O_6$, is a glycoluril derivative. The molecule is built up from four fused rings, namely two nearly planar imidazole five-membered rings and two non-planar triazine six-membered rings. In the five-membered imidazole rings, the $N-C_{carbonyl}$ bond distances are much shorter than the other N—C distances, indicating π–π conjugation within the nearly planar ring. Both six-membered rings display chair conformations.

Comment

Glycoluril derivatives have applications in many fields, such as explosives, slow-release fertilizers, crosslinkers, iodogens, stabilisers of organic compounds against photodegradation and reagents in combinatorial chemistry (Wu *et al.*, 2002). As part of our ongoing investigation of glycoluril derivatives (Li & Wu, 2005), we present here the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule is built up from four fused rings, namely two nearly planar imidazole five-membered rings and two non-planar triazine six-membered rings. Within the nearly planar five-membered ring, the $N-C_{carbonyl}$ bond distances are much shorter than the other N—C distances (Table 1). This is due to the occurrence of π–π conjugation.

Experimental

tert-Butylamine (0.73 g, 10 mmol) and formaldehyde (1.2 g, 40 mmol) were added to a stirred solution of 2,5-dioxotetrahydroimidazo[4,5-*d*]imidazole-3a,6a-dicarboxylic acid dimethyl ester (1.29 g, 5 mmol) in acetonitrile (50 ml) under a nitrogen atmosphere. The mixture was stirred overnight at room temperature. The solvent was evaporated to dryness and the compound was purified by column-chromatography to yield (I) (2.03 g, 90%) as a colourless solid. Colourless block-like crystals of (I) suitable for data collection were obtained by slow evaporation of an ethyl acetate solutio at 283 K.

Crystal data

$C_{20}H_{32}N_6O_6$
M_r = 452.52
Monoclinic, $P2_1/c$
a = 14.6448 (15) Å
b = 12.4388 (12) Å
c = 13.2805 (14) Å
β = 98.362 (2)°
V = 2393.5 (4) Å^3
Z = 4
D_x = 1.256 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5173 reflections
θ = 2.3–27.8°
μ = 0.09 mm^{-1}
T = 292 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
11778 measured reflections
4210 independent reflections
3444 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°
$h = -16 \rightarrow 17$
$k = -11 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.148
S = 1.06
4210 reflections
297 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 0.5234P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N1	1.497 (3)	C8—N3	1.368 (2)
C5—N1	1.443 (3)	C8—N5	1.393 (3)
C5—N2	1.477 (2)	C9—N3	1.442 (2)
C7—N2	1.368 (2)	C9—N2	1.443 (2)
C7—N4	1.375 (3)		
N2—C7—N4	108.57 (15)	N3—C8—N5	108.26 (15)

All H atoms were treated as riding on their parent C atoms. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), whereas methylene H atoms were constrained to C—H = 0.97 Å with U_{iso}(H) = 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

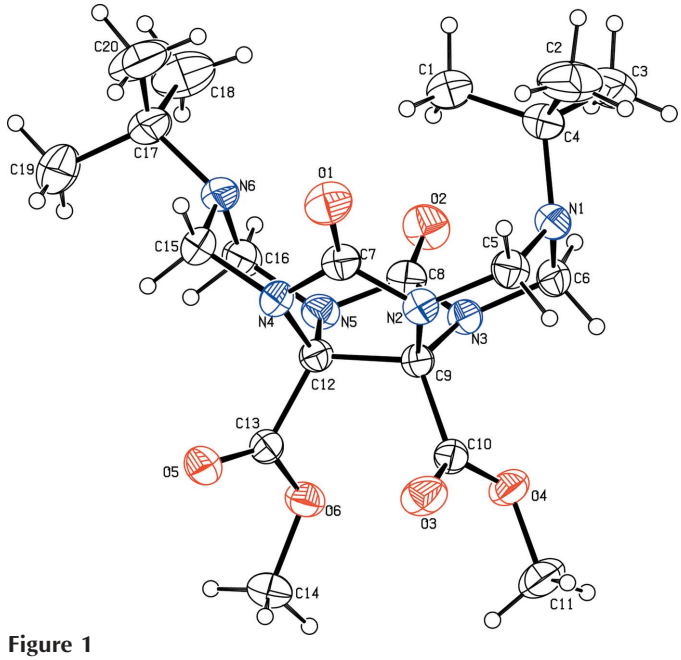


Figure 1
The molecular structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented as spheres of arbitrary radii.

The authors thank Xiang-Gao Meng for crystal data collection.

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