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cis-[4a]-*cisoid*-[4a,4b]-*cis*-[4b]-4b-Ethyl-1,3,5,7,8b-hexamethylperhydro-1,3,5,7-tetraazabiphenylene-2,4,6,8-tetraone

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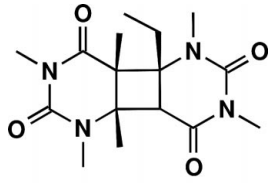
Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.111
Data-to-parameter ratio = 18.6

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

The title compound, $C_{16}H_{24}N_4O_4$, was prepared by [2+2]-photocycloaddition of 1,3-dimethyl-6-ethyluracil in acetone. The structure determination was undertaken in order to determine the stereoconfiguration of the product, which could not be extracted from NMR data. Single-crystal X-ray analysis revealed that the methyl groups and the ethyl group are located on the same side of the cyclobutane ring (*cis*) and that the monomers are oriented head-to-tail (*anti*).



(I)

Experimental

The title compound was formed as a byproduct in the [2+2]-photocycloaddition of 1,3-dimethyl-6-ethyluracil in acetone, by a mechanism which is not yet clear. The products were separated by column chromatography. Single crystals were obtained by slow evaporation of *n*-pentane into a saturated solution of the title compound in dichloromethane. Details of the synthesis are given by Krüger (2002).

Crystal data

$C_{16}H_{24}N_4O_4$
M_r = 336.39
Orthorhombic, *Pbca*
a = 15.2661 (9) Å
b = 14.4367 (8) Å
c = 15.7200 (12) Å
V = 3464.6 (4) Å^3
Z = 8
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 17–23.5°
μ = 0.09 mm^{-1}
T = 170 (2) K
Irregular block, colourless
0.5 × 0.3 × 0.3 mm

Data collection

Stoe Imaging Plate Diffraction System diffractometer
φ scans
Absorption correction: none
27222 measured reflections
4171 independent reflections
3642 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.1°
$h = -20 \rightarrow 20$
$k = -17 \rightarrow 18$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.04
4171 reflections
224 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 0.9107P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0088 (11)

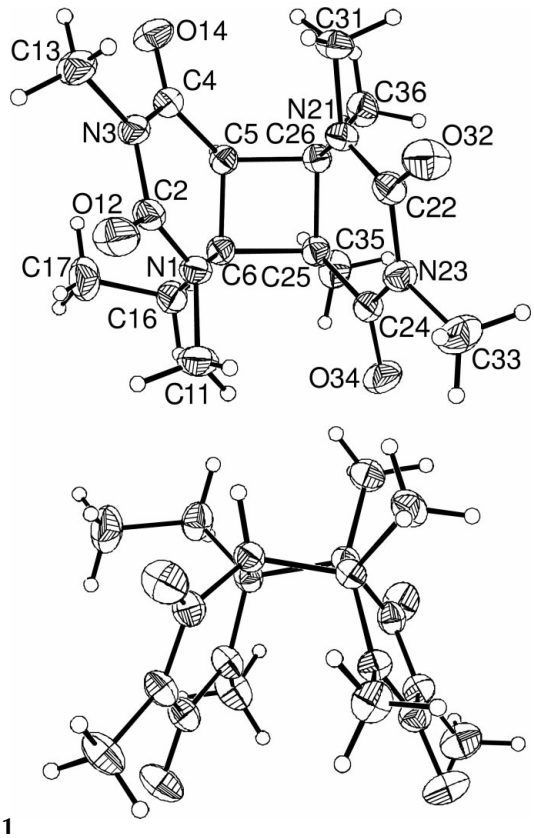


Figure 1
Top view (top) and side view (bottom) of the molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level.

The methine and methylene H atoms were positioned with idealized geometry ($C-H_{methine}$ = 1.00 Å and $C-H_{methylene}$ = 0.99 Å). The positions of the methyl H atoms were idealized (C—H = 0.98 Å), then refined as rigid groups allowed to rotate but not tip. All H atoms were refined with fixed isotropic displacement parameters using a riding model with U_{iso} = $1.2U_{eq}$(C) and $1.5U_{eq}$(C) for methine/methylene and methyl H atoms, respectively.

Data collection: *IPDS Program Package* (Stoe & Cie, 1998); cell refinement: *IPDS Program Package*; data reduction: *IPDS Program Package*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXL*97.

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References

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