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

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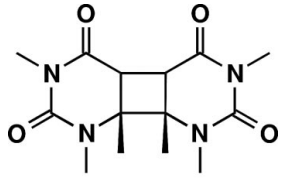
Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.124
Data-to-parameter ratio = 16.1

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

The title compound, $C_{14}H_{20}N_4O_4$, was prepared by [2+2]-photocycloaddition of 1,3,6-trimethyluracil 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 are located on the same side of the cyclobutane ring (*cis*) and that the monomers are oriented head-to-head (*syn*).



(I)

Experimental

The title compound was prepared by [2+2]-photocycloaddition of 1,3,6-trimethyluracil in acetone. 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

$C_{14}H_{20}N_4O_4$
M_r = 308.34
Triclinic, $P\overline{1}$
a = 7.0365 (6) Å
b = 8.0400 (6) Å
c = 13.02 (1) Å
α = 81.74 (1)°
β = 75.76 (1)°
γ = 84.02 (1)°
V = 704.58 (10) Å^3
Z = 2
D_x = 1.453 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 10–27.5°
μ = 0.11 mm^{-1}
T = 170 (2) K
Irregular block, colourless
0.4 × 0.4 × 0.3 mm

Data collection

Stoe Imaging Plate Diffraction System diffractometer
φ scans
Absorption correction: none
6641 measured reflections
3323 independent reflections
2973 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.1°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.124
S = 1.05
3323 reflections
206 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.1990P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.094 (12)

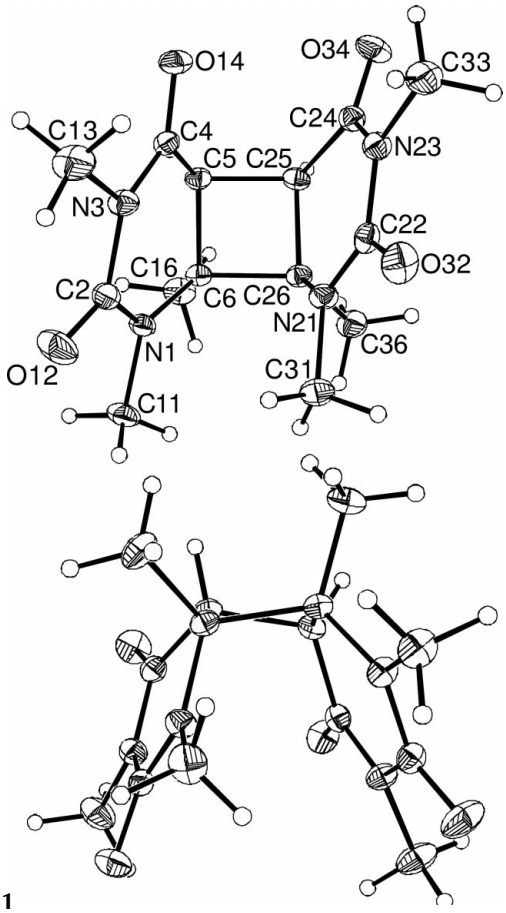


Figure 1
Top view (top) and side view (bottom) of the molecular structure of the title compound, with labelling and displacement ellipsoids drawn at the 50% probability level.

The methine H atoms were positioned with idealized geometry (C—$H_{methine}$ = 1.00 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 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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