

cis-[4a]-cisoid-[4a,4b]-cis-[4b]-8a,8b-Diethyl-1,3,6,8-tetramethylperhydro-1,3,6,8-tetraazabiphenylene-2,4,5,7-tetraone**Christian Näther,^{a*} Oliver Krüger^b and Uta Wille^b**^aInstitut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstraße 40, D-24098 Kiel, Germany, and ^bInstitut für Organische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstraße 40, D-24098 Kiel, Germany

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Key indicators

Single-crystal X-ray study

T = 170 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

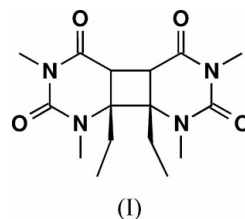
R factor = 0.049

wR factor = 0.140

Data-to-parameter ratio = 17.8

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

The title compound, $\text{C}_{16}\text{H}_{24}\text{N}_4\text{O}_4$, 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 are located on the same side of the cyclobutane ring (*cis*) and that the monomers are oriented head-to-head (*syn*). There are two crystallographically independent molecules in the asymmetric unit; these differ only slightly in geometry.

**Experimental**

The title compound was prepared by [2+2]-photocycloaddition of 1,3-dimethyl-6-ethyluracil 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

$\text{C}_{16}\text{H}_{24}\text{N}_4\text{O}_4$
 $M_r = 336.39$
 Triclinic, $P\bar{1}$
 $a = 8.5284 (6) \text{ \AA}$
 $b = 12.2178 (9) \text{ \AA}$
 $c = 16.2102 (12) \text{ \AA}$
 $\alpha = 91.481 (9)^\circ$
 $\beta = 101.269 (8)^\circ$
 $\gamma = 90.518 (8)^\circ$
 $V = 1655.8 (2) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.349 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 8000 reflections
 $\theta = 10.5\text{--}27^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 170 (2) \text{ K}$
 Needle, colourless
 $0.40 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Stoe Imaging Plate Diffraction System diffractometer
 φ scans
 Absorption correction: none
 15745 measured reflections
 7846 independent reflections

5552 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 28.1^\circ$
 $h = -11 \rightarrow 11$
 $k = -16 \rightarrow 16$
 $l = -21 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.140$
 $S = 1.02$
 7846 reflections
 442 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.1459P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.035 (2)

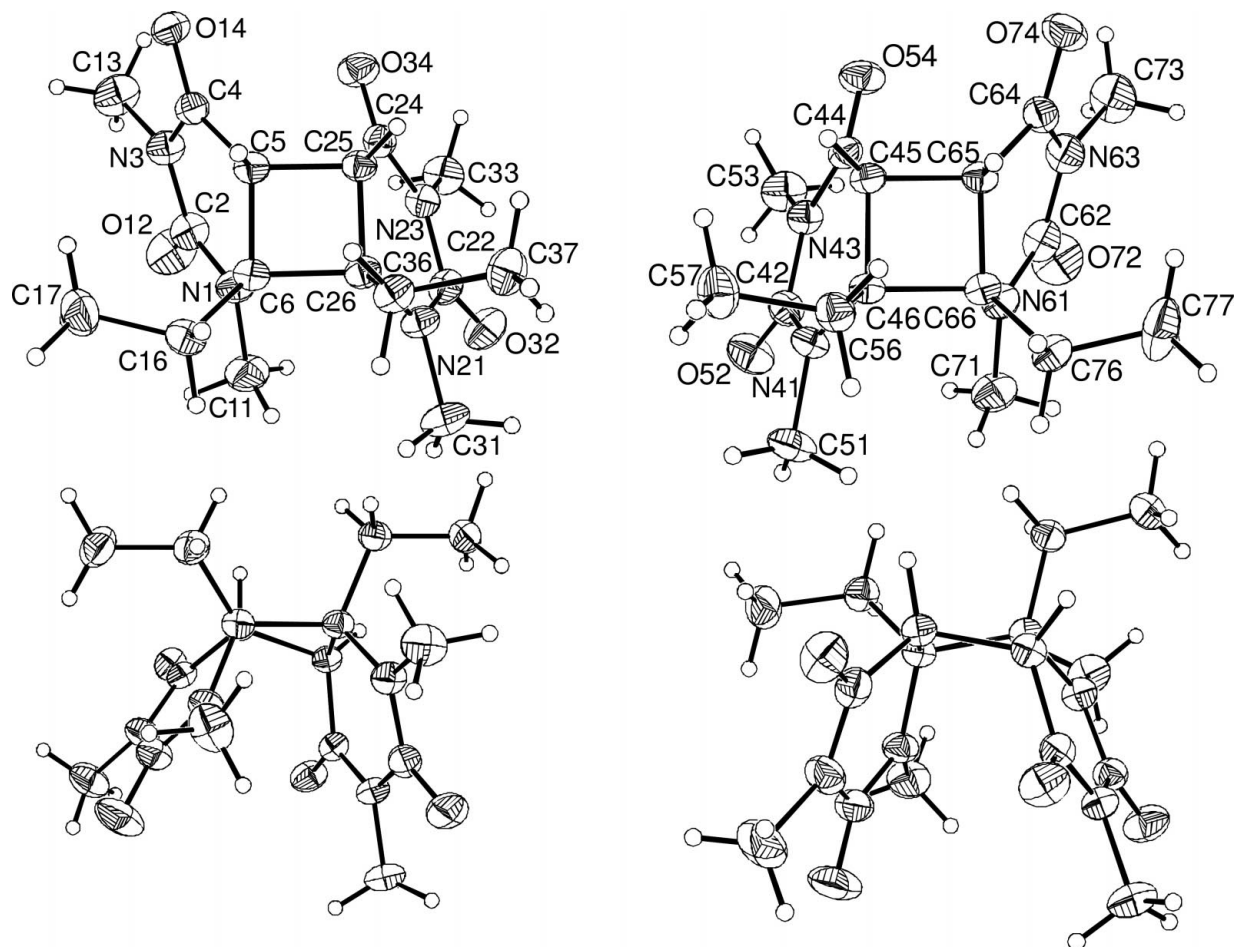


Figure 1

Top view (top) and side view (bottom) of the structures of the two independent molecules 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 ($C-H_{\text{methine}} = 1.00 \text{ \AA}$ and $C-H_{\text{methylene}} = 0.99 \text{ \AA}$). The positions of the methyl H atoms were idealized ($C-H = 0.98 \text{ \AA}$), 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 $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C})$ 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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXL97*.

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References

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