

cis-[4a]-transoid-[4a,4b]-cis-[4b]-1,3,6,8,8a,8b-Hexamethylperhydro-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

Correspondence e-mail: cnaether@ac.uni-kiel.de

Key indicators

Single-crystal X-ray study

T = 170 K

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

R factor = 0.042

wR factor = 0.121

Data-to-parameter ratio = 17.0

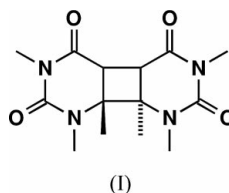
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{14}\text{H}_{20}\text{N}_4\text{O}_4$, 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 opposite sides of the cyclobutane ring (*trans*) and that the monomers are oriented head-to-head (*syn*).

Received 27 February 2002

Accepted 8 March 2002

Online 15 March 2002

**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

$\text{C}_{14}\text{H}_{20}\text{N}_4\text{O}_4$
 $M_r = 308.34$
 Triclinic, $P\bar{1}$
 $a = 7.6398 (6) \text{ \AA}$
 $b = 8.6267 (7) \text{ \AA}$
 $c = 12.308 (1) \text{ \AA}$
 $\alpha = 71.483 (9)^\circ$
 $\beta = 80.13 (1)^\circ$
 $\gamma = 72.521 (9)^\circ$
 $V = 731.09 (10) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.401 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 8000 reflections
 $\theta = 10.5\text{--}27^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 170 (2) \text{ K}$
 Irregular polyhedron, colourless
 $0.4 \times 0.2 \times 0.2 \text{ mm}$

Data collection

Stoe Imaging Plate Diffraction
 System diffractometer
 φ scans
 Absorption correction: none
 8266 measured reflections
 3499 independent reflections

3019 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 28.1^\circ$
 $h = -10 \rightarrow 10$
 $k = -11 \rightarrow 11$
 $l = -16 \rightarrow 16$

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.121$
 $S = 1.05$
 3499 reflections
 206 parameters
 H-atom parameters constrained
Refinement

$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.1861P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*

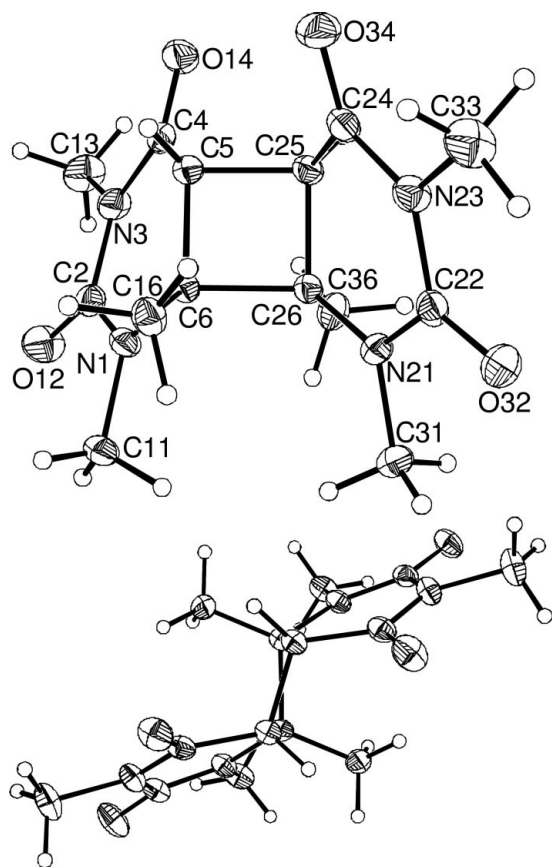


Figure 1
Top view (top) and side view (bottom) of the molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level.

The methine 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: *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*.

This work is supported by the state of Schleswig-Holstein and the Deutsche Forschungsgemeinschaft. We are very thankful to Professor Dr Wolfgang Bensch for the opportunity to use his experimental equipment.

References

- Bruker (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Krüger, O. (2002). PhD Thesis, University of Kiel, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (1998). *IPDS Program Package*. Version 2.89. Stoe & Cie, Darmstadt, Germany.