Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1,7-Bis(1,3-dimethyluracil-6-yl)-1,4,7,10-tetraazacyclododecane acetonitrile disolvate at 100 K

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Received 26 December 2006
Accepted 5 January 2007

Key indicators

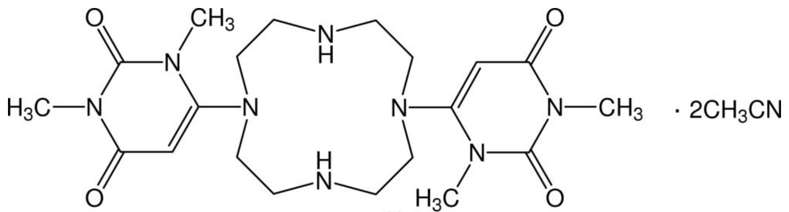
Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.094
Data-to-parameter ratio = 17.3

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

The title compound [systematic name: 1,1′,3,3′-tetramethyl-1,1′,2,2′,3,3′,4,4′-octahydro-6,6-(1,4,7,10-tetraazacyclododecane-1,7-diyl)dipyrimidine-2,2′,4,4′-tetraone acetonitrile disolvate], $C_{20}H_{32}N_8O_4\cdot 2CH_3CN$, an *N*-uracil-substituted cyclen derivative, is located on a crystallographic twofold rotation axis running through the center of the 12-membered ring and adopts a [3333] quadrangular conformation with C atoms occupying the corners.

Comment

Cyclen (1,4,7,10-tetraazacyclododecane) is one of the most important macrocycles as a metal-chelating ligand (Curtis, 2004). Numerous *N*-alkyl cyclen derivatives have been reported to improve their affinity, modify their metal-chelating properties, prepare MRI contrast reagents (Gries & Schering, 2002), and so on. However, there are few reports on *N*-aryl-substituted cyclen derivatives because an efficient general synthetic approach is lacking (Michael & Burkhart, 2001). We report here the structure of compound, (I), prepared in the course of our studies on new *N*-aryl-substituted cyclen derivatives.



(I)

The molecule possesses twofold rotation symmetry, the axis of which passes through the center of the 12-membered cyclen ring (Fig. 1). The cyclen ring assumes a [3333] quadrangular conformation with four repeating units, each containing two succesive *gauche* and one *anti* conformations (Table 1). All four N atoms of the cyclen ring lie on one side of the mean plane through the macrocycle. Due to the steric hindrance caused by the dimethyluracil group at N1, the N1—C1 and N1—C4^{i} distances are significantly longer [1.4814 (13) and 1.4972 (13) Å, respectively; symmetry code: (i) $1 - x, y, \frac{1}{2} - z$] than the N2—C2 and N2—C3 distances [1.4659 (13) and 1.4627 (13) Å, respectively], and C5 of the uracil ring lies out of the mean plane by 0.440 (1) Å.

Experimental

A solution of cyclen (3.53 mmol), 1,3-dimethyl-6-chlorouracil (7.50 mmol) and sodium carbonate (7.03 mmol) in acetonitrile

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(25 ml) was stirred overnight under reflux. After filtration of the inorganic salt, a white solid of (I) precipitated from the filtrate (yield 61%, 2.15 mmol). Single crystals were obtained from an acetonitrile solution.

Crystal data

$C_{20}H_{32}N_8O_4 \cdot 2C_2H_3N$
$M_r = 530.64$
Monoclinic, $C2/c$
$a = 24.2718\ (18)$ Å
$b = 7.5477\ (6)$ Å
$c = 14.3193\ (11)$ Å
$\beta = 92.4510\ (10)^\circ$
$V = 2620.8\ (3)$ Å^3
$Z = 4$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 100\ (1)$ K
Block, colorless
$0.40 \times 0.40 \times 0.25$ mm

Data collection

Bruker APEX-II CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.963$, $T_{max} = 0.977$
7243 measured reflections
3096 independent reflections
2681 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 28.7^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.094$
$S = 1.04$
3096 reflections
179 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 1.6563P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—C4	1.5232 (14)	C2—C1	1.5287 (14)
C1—N1—C4^i	112.48 (8)	C3—N2—C2	113.65 (8)
N2—C3—C4—N1^i	73.27 (11)	C4^i—N1—C1—C2	−150.35 (8)
C4—C3—N2—C2	−170.94 (8)	N2—C2—C1—N1	60.59 (11)
C1—C2—N2—C3	70.45 (11)	C1—N1—C4^i—C3^i	70.80 (10)

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

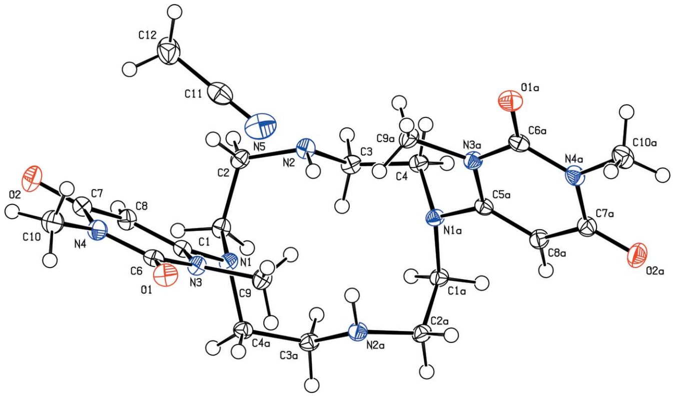

Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radius. The suffix a corresponds to symmetry code (i) in Table 1.

All H atoms were located in a difference Fourier map. Except for the imino H atom, they were repositioned in calculated positions (C—H = 0.93–0.98Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. For the imino H atom, the N—H distance was restrained to 0.86 (1)Å and the isotropic displacement parameter was refined freely.

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *APEX2*; data reduction: *SAINT-Plus* (Bruker–Nonius, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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