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A tetrakis(secondary amide) derivative of quadricyclane: 3-isopropylidenetetracyclo[3.2.0.0^{2,7}.0^{4,6}]heptane-1,5,6,7-tetracarboxylic acid tetrakis(cyclohexylamide)

Torsten Winkler,[a]† Rainer Herges,[a]† Peter G. Jones[b]* and Ina Dix[a]‡

[a]Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [b]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

† Current address: Institut für Organische Chemie, Universität Kiel, Otto-Hahn-Platz 4, 24098 Kiel, Germany
‡ Current address: Bruker AXS GmbH, PO Box, 76181 Karlsruhe, Germany

Correspondence e-mail: p.jones@tu-bs.de

The molecule of the title compound, $C_{38}H_{56}N_4O_4$, displays approximate twofold symmetry. The four amide groups are arranged such that four intramolecular N—H···O hydrogen bonds are formed. The molecules are linked into dimers by bifurcated $(C—H)_2$···O interactions.

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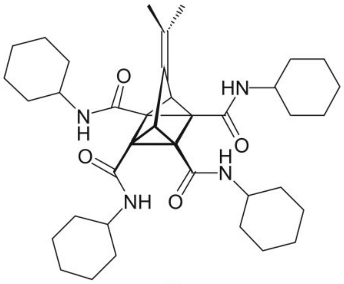
Key indicators

Single-crystal X-ray study
T = 143 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 16.4

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

Comment

The present structure determination forms part of a short series of structures of norbornadiene and quadricyclane tetraamides. Introductory material was presented in the first paper (Winkler *et al.*, 2003*a*). We present here the structure of the title compound, (I), a tetrakis(secondary amide) derivative of quadricyclane.



(I)

The molecule of (I) (Fig. 1) possesses approximate twofold symmetry, but the orientations of the cyclohexyl rings depart appreciably from the ideal symmetry. Molecular dimensions, including those of the strained quadricyclane system (Table 1), may be considered normal. The four amide groups are arranged, as in the tetrakis(isopropyl) analogue (Winkler *et al.*, 2003*b*), such that four intramolecular hydrogen bonds (Table 2) of the N—H···O type are formed.

The molecules are connected to form inversion-symmetric dimers by a bifurcated intermolecular hydrogen-bond system $(C—H)_2$···O3, involving a methine and a methyl H atom, of graph-set $R_2^1(7)$. The dimers are linked into chains parallel to the *a* axis by a further C—H···O interaction from a cyclohexyl H atom (Table 2).

Experimental

The title compound was prepared according to the method of Winkler (2002) and recrystallized by evaporation from a dichloromethane/methanol solution.

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Crystal data

$C_{38}H_{56}N_4O_4$
$M_r = 632.87$
Triclinic, $P\overline{1}$
$a = 10.1995\ (14)$ Å
$b = 11.6467\ (16)$ Å
$c = 15.206\ (2)$ Å
$\alpha = 87.126\ (3)^\circ$
$\beta = 77.132\ (3)^\circ$
$\gamma = 81.300\ (3)^\circ$
$V = 1740.5\ (4)$ Å^3

$Z = 2$
$D_x = 1.208$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6008 reflections
$\theta = 2$–28°
$\mu = 0.08$ mm^{-1}
$T = 143\ (2)$ K
Tablet, colourless
0.38 × 0.33 × 0.19 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: none
15921 measured reflections
7098 independent reflections

5126 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\rm max} = 26.4^\circ$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.105$
$S = 1.03$
7098 reflections
433 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.1495P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.5259 (19)	C3—C8	1.3343 (19)
C1—C7	1.5543 (19)	C3—C4	1.4791 (19)
C1—C5	1.5572 (18)	C4—C5	1.5268 (18)
C2—C3	1.4788 (18)	C4—C6	1.5298 (18)
C2—C7	1.5175 (19)	C5—C6	1.5545 (19)
C11—C1—C2	119.79 (11)	C4—C5—C6	59.53 (8)
C11—C1—C7	128.02 (11)	C18—C5—C1	128.07 (11)
C2—C1—C7	59.02 (9)	C4—C5—C1	103.73 (10)
C11—C1—C5	132.38 (11)	C6—C5—C1	90.21 (10)
C2—C1—C5	103.77 (10)	C25—C6—C4	118.91 (11)
C7—C1—C5	89.55 (10)	C25—C6—C7	132.13 (11)
C3—C2—C7	110.32 (11)	C4—C6—C7	103.97 (11)
C3—C2—C1	109.86 (11)	C25—C6—C5	129.19 (12)
C7—C2—C1	61.42 (9)	C4—C6—C5	59.33 (8)
C8—C3—C2	128.92 (12)	C7—C6—C5	89.78 (10)
C8—C3—C4	130.05 (12)	C32—C7—C2	120.34 (11)
C2—C3—C4	101.04 (11)	C32—C7—C6	128.80 (12)
C3—C4—C5	110.15 (11)	C2—C7—C6	103.83 (10)
C3—C4—C6	109.42 (11)	C32—C7—C1	132.80 (12)
C5—C4—C6	61.14 (9)	C2—C7—C1	59.56 (9)
C18—C5—C4	121.36 (11)	C6—C7—C1	90.45 (10)
C18—C5—C6	133.19 (12)		
C2—C1—C11—O1	38.25 (18)	C4—C6—C25—O3	42.36 (18)
C7—C1—C11—O1	−34.0 (2)	C7—C6—C25—O3	−167.02 (13)
C5—C1—C11—O1	−168.41 (13)	C5—C6—C25—O3	−30.1 (2)
C2—C1—C11—N1	−140.90 (13)	C4—C6—C25—N3	−135.48 (13)
C7—C1—C11—N1	146.87 (13)	C7—C6—C25—N3	15.1 (2)
C5—C1—C11—N1	12.4 (2)	C5—C6—C25—N3	152.06 (13)
C4—C5—C18—O2	117.87 (14)	C2—C7—C32—O4	117.39 (15)
C6—C5—C18—O2	−166.16 (13)	C6—C7—C32—O4	−28.3 (2)
C1—C5—C18—O2	−28.3 (2)	C1—C7—C32—O4	−167.58 (13)
C4—C5—C18—N2	−58.95 (17)	C2—C7—C32—N4	−59.03 (17)
C6—C5—C18—N2	17.0 (2)	C6—C7—C32—N4	155.32 (13)
C1—C5—C18—N2	154.90 (13)	C1—C7—C32—N4	16.0 (2)

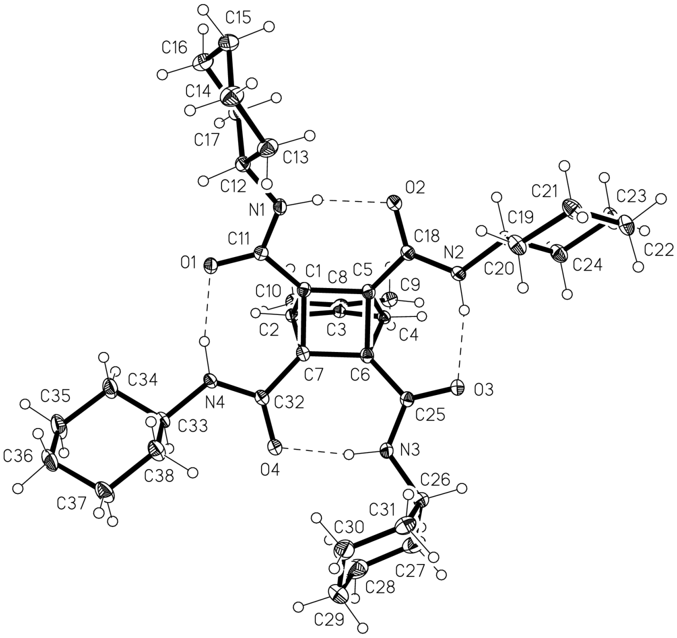

Figure 1
The molecule of the title compound in the crystal. Displacement ellipsoids are drawn at the 30% probability level. H-atom radii are arbitrary. Hydrogen bonds are indicated by dashed lines.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H01···O2	0.900 (18)	1.824 (18)	2.7091 (15)	167.4 (17)
N2—H02···O3	0.895 (18)	1.907 (19)	2.7841 (16)	166.2 (16)
N3—H03···O4	0.927 (17)	1.804 (17)	2.7153 (15)	167.2 (15)
N4—H04···O1	0.953 (19)	1.81 (2)	2.7490 (16)	166.5 (16)
C4—H4···O3$^{\rm i}$	1.00	2.39	3.3890 (16)	174
C9—H9A···O3$^{\rm i}$	0.98	2.50	3.4717 (18)	170
C14—H14B···O4$^{\rm ii}$	0.99	2.50	3.367 (2)	146

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $x, y - 1, z$.

H atoms bonded to nitrogen were refined freely. Methyl H atoms were located in difference syntheses, idealized (C—H = 0.98 Å and H—C—H = 109.5°) and refined on the basis of rigid groups allowed to rotate but not to tip. Other H atoms were included using a riding model with fixed C—H bond lengths of 1.00 (methine) or 0.99 Å (methylene); $U_{\rm iso}$(H) values were fixed at 1.2 times $U_{\rm eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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