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

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Key indicators

Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.103
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 molecule of the title compound, $C_{30}H_{48}N_4O_4$, displays approximate twofold symmetry. The four amide groups are arranged such that four intramolecular N—H⋯O hydrogen bonds are formed.

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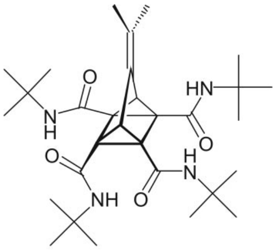
Comment

This structure determination is the final part of a short series of structures of norbornadiene and quadricyclane tetra-amides. Introductory material was presented in the first paper (Winkler *et al.*, 2003*a*). Here we present the structure of the title compound, (I), a tetrakis(secondary amide) derivative of quadricyclane.



(I)

The molecule (Fig. 1) possesses approximate twofold symmetry. Molecular dimensions, including those of the strained quadricyclane system (Table 1), may be considered normal. The four amide groups are arranged, as in all the analogous secondary amides (*e.g.* the tetrakis(isopropyl) analogue; Winkler *et al.*, 2003*b*), such that four intramolecular hydrogen bonds (Table 2) of the type N—H⋯O are formed.

Presumably because of the bulky *tert*-butyl groups, this is the only compound in the series that does not display weak intermolecular hydrogen bonds; there are no H⋯O contacts < 2.9 Å.

Experimental

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

Crystal data

$C_{30}H_{48}N_4O_4$
M_r = 528.72
Triclinic, $P\overline{1}$
a = 9.9831 (8) Å
b = 12.2324 (10) Å
c = 13.9644 (10) Å
α = 92.574 (3)°
β = 106.831 (3)°
γ = 105.554 (3)°
V = 1558.5 (2) Å^3
Z = 2
D_x = 1.127 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 5756 reflections
θ = 2–28°
μ = 0.08 mm^{-1}
T = 143 (2) K
Prism, colourless
0.40 × 0.29 × 0.17 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: none
14409 measured reflections
6352 independent reflections

4684 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 26.4^\circ$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.03$
6352 reflections
373 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.2051P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.5291 (18)	C3—C8	1.3309 (18)
C1—C7	1.5546 (18)	C3—C4	1.4767 (17)
C1—C5	1.5597 (16)	C4—C5	1.5237 (17)
C2—C3	1.4772 (18)	C4—C6	1.5316 (17)
C2—C7	1.5237 (17)	C5—C6	1.5505 (17)
C11—C1—C2	121.94 (11)	C4—C5—C6	59.76 (8)
C11—C1—C7	131.31 (11)	C16—C5—C1	133.63 (11)
C2—C1—C7	59.22 (8)	C4—C5—C1	103.53 (10)
C11—C1—C5	128.87 (11)	C6—C5—C1	89.62 (9)
C2—C1—C5	103.65 (10)	C21—C6—C4	121.26 (10)
C7—C1—C5	90.15 (9)	C21—C6—C5	133.07 (10)
C3—C2—C7	110.03 (10)	C4—C6—C5	59.25 (8)
C3—C2—C1	110.17 (10)	C21—C6—C7	128.32 (11)
C7—C2—C1	61.23 (8)	C4—C6—C7	103.36 (9)
C8—C3—C4	129.46 (12)	C5—C6—C7	90.53 (9)
C8—C3—C2	129.68 (12)	C26—C7—C2	119.96 (10)
C4—C3—C2	100.86 (10)	C26—C7—C6	133.30 (11)
C3—C4—C5	110.45 (10)	C2—C7—C6	104.02 (10)
C3—C4—C6	110.26 (10)	C26—C7—C1	124.74 (11)
C5—C4—C6	60.99 (8)	C2—C7—C1	59.56 (8)
C16—C5—C4	119.22 (10)	C6—C7—C1	89.69 (9)
C16—C5—C6	126.12 (10)		
C2—C1—C11—O1	−130.25 (13)	C4—C6—C21—O3	−127.28 (13)
C7—C1—C11—O1	154.52 (13)	C5—C6—C21—O3	157.28 (13)
C5—C1—C11—O1	19.0 (2)	C7—C6—C21—O3	18.26 (19)
C2—C1—C11—N1	47.65 (17)	C4—C6—C21—N3	49.60 (16)
C7—C1—C11—N1	−27.58 (19)	C5—C6—C21—N3	−25.85 (19)
C5—C1—C11—N1	−163.06 (12)	C7—C6—C21—N3	−164.86 (11)
C4—C5—C16—O2	−31.00 (17)	C2—C7—C26—O4	−23.46 (18)
C6—C5—C16—O2	41.16 (18)	C6—C7—C26—O4	178.49 (12)
C1—C5—C16—O2	174.36 (12)	C1—C7—C26—O4	48.36 (17)
C4—C5—C16—N2	149.03 (11)	C2—C7—C26—N4	158.29 (11)
C6—C5—C16—N2	−138.80 (12)	C6—C7—C26—N4	0.2 (2)
C1—C5—C16—N2	−5.6 (2)	C1—C7—C26—N4	−129.89 (13)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H01···O4	0.866 (16)	1.906 (17)	2.7481 (17)	163.7 (15)
N2—H02···O1	0.930 (16)	1.790 (16)	2.7116 (15)	170.5 (14)
N3—H03···O2	0.854 (16)	1.951 (16)	2.7799 (16)	163.2 (14)
N4—H04···O3	0.903 (15)	1.851 (15)	2.7350 (14)	165.8 (13)

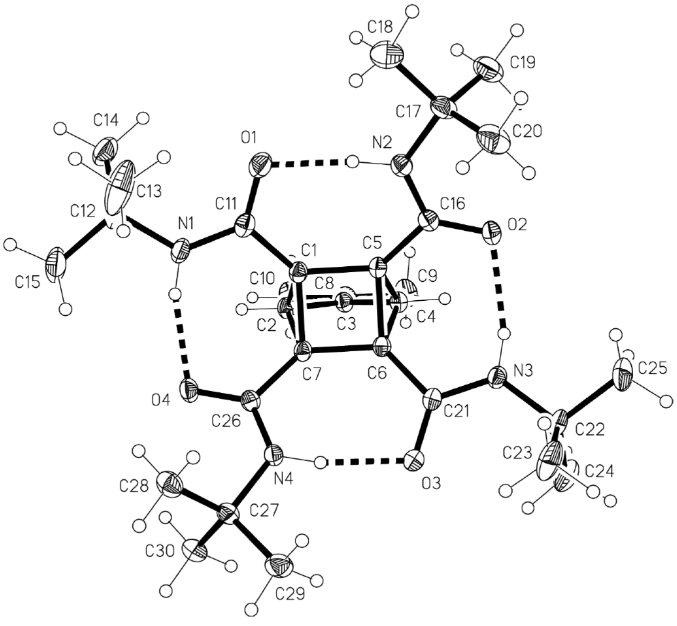


Figure 1
The molecule of the title compound in the crystal. Displacement ellipsoids are drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.

H atoms bonded to nitrogen were refined freely. Methyl H atoms were located in a difference synthesis, idealized (C—H 0.98 Å, H—C—H 109.5°) and refined on the basis of rigid groups allowed to rotate but not tip. Other H atoms were included using a riding model, with fixed C—H bond lengths of 1.00 Å; U(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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