

# A tetrakis(secondary amide) derivative of quadricyclane: *N,N',N'',N'''*-tetrakispropyl-3-isopropylidenetetracyclo[3.2.0.0<sup>2,7</sup>.0<sup>4,6</sup>]-heptane-1,5,6,7-tetracarboxamide

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## Key indicators

Single-crystal X-ray study

*T* = 143 K

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

*R* factor = 0.038

*wR* factor = 0.104

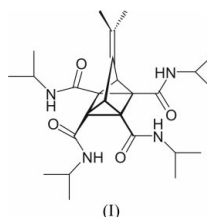
Data-to-parameter ratio = 16.5

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

The title compound,  $\text{C}_{26}\text{H}_{40}\text{N}_4\text{O}_4$ , displays approximate twofold symmetry. The four amide groups are arranged such that four intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds are formed. The molecules are linked into layers by bifurcated  $(\text{C}-\text{H})_2\cdots\text{O}$  interactions.

## Comment

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



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 such that four intramolecular hydrogen bonds (Table 2) of the type  $\text{N}-\text{H}\cdots\text{O}$  are formed, in the same pattern as reported in the previous paper.

The molecules are connected to form layers perpendicular to the *bc* plane by two bifurcated intermolecular hydrogen bond systems  $(\text{C}-\text{H})_2\cdots\text{O}$  of graph set  $R_2^1(7)$  (Fig. 2). One of the  $\text{H}\cdots\text{O}$  interactions,  $\text{H}4\cdots\text{O}3$ , is very short, with a normalized  $\text{H}\cdots\text{O}$  distance of only 2.21 Å.

## Experimental

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

### Crystal data

$\text{C}_{26}\text{H}_{40}\text{N}_4\text{O}_4$   
*M<sub>r</sub>* = 472.62  
 Monoclinic,  $P2_1/c$   
*a* = 13.3354 (18) Å  
*b* = 12.1987 (16) Å  
*c* = 16.981 (2) Å  
 $\beta$  = 104.454 (4)°  
*V* = 2674.9 (6) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.174 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 6314 reflections  
 $\theta$  = 2.2–28.5°  
 $\mu$  = 0.08 mm<sup>-1</sup>  
*T* = 143 (2) K  
 Prism, yellow  
 0.42 × 0.17 × 0.16 mm

Data collection

Bruker SMART 1000 CCD  
diffractometer  
 $\omega$  scans  
Absorption correction: none  
35207 measured reflections  
5478 independent reflections

4203 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$   
 $\theta_{\text{max}} = 26.4^\circ$   
 $h = -16 \rightarrow 16$   
 $k = -15 \rightarrow 15$   
 $l = -21 \rightarrow 21$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.104$   
 $S = 1.04$   
5478 reflections  
333 parameters  
H atoms treated by a mixture of  
independent and constrained  
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.6537P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.25 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C1—C2	1.5209 (18)	C3—C8	1.3345 (18)
C1—C5	1.5563 (17)	C3—C4	1.4765 (18)
C1—C7	1.5707 (18)	C4—C5	1.5224 (17)
C2—C3	1.4828 (18)	C4—C6	1.5245 (17)
C2—C7	1.5217 (18)	C5—C6	1.5630 (18)
C11—C1—C2	119.99 (11)	C4—C5—C1	103.92 (10)
C11—C1—C5	132.62 (11)	C15—C5—C6	133.58 (11)
C2—C1—C5	103.91 (10)	C4—C5—C6	59.20 (8)
C11—C1—C7	126.88 (11)	C1—C5—C6	90.16 (9)
C2—C1—C7	58.95 (8)	C19—C6—C4	118.36 (10)
C5—C1—C7	89.67 (9)	C19—C6—C7	132.61 (11)
C3—C2—C1	109.47 (10)	C4—C6—C7	104.07 (10)
C3—C2—C7	109.64 (10)	C19—C6—C5	128.68 (11)
C1—C2—C7	62.16 (9)	C4—C6—C5	59.07 (8)
C8—C3—C4	128.71 (12)	C7—C6—C5	90.12 (9)
C8—C3—C2	130.00 (12)	C23—C7—C2	122.01 (11)
C4—C3—C2	101.29 (10)	C23—C7—C6	128.12 (11)
C3—C4—C5	109.70 (10)	C2—C7—C6	103.91 (10)
C3—C4—C6	109.56 (10)	C23—C7—C1	132.62 (11)
C5—C4—C6	61.73 (8)	C2—C7—C1	58.89 (8)
C15—C5—C4	120.42 (11)	C6—C7—C1	90.05 (9)
C15—C5—C1	128.51 (11)		
C2—C1—C11—O1	38.23 (18)	C4—C6—C19—O3	44.37 (17)
C5—C1—C11—O1	-166.51 (13)	C7—C6—C19—O3	-164.94 (13)
C7—C1—C11—O1	-33.62 (19)	C5—C6—C19—O3	-27.21 (19)
C2—C1—C11—N1	-141.00 (13)	C4—C6—C19—N3	-132.78 (13)
C5—C1—C11—N1	14.3 (2)	C7—C6—C19—N3	17.9 (2)
C7—C1—C11—N1	147.16 (13)	C5—C6—C19—N3	155.65 (12)
C4—C5—C15—O2	121.03 (13)	C2—C7—C23—O4	125.20 (14)
C1—C5—C15—O2	-24.4 (2)	C6—C7—C23—O4	-23.1 (2)
C6—C5—C15—O2	-164.08 (13)	C1—C7—C23—O4	-159.44 (13)
C4—C5—C15—N2	-55.89 (17)	C2—C7—C23—N4	-51.96 (18)
C1—C5—C15—N2	158.64 (12)	C6—C7—C23—N4	159.75 (13)
C6—C5—C15—N2	19.0 (2)	C1—C7—C23—N4	23.4 (2)

Table 2

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

D—H...A	D—H	H...A	D...A	D—H...A
N1—H01...O2	0.892 (17)	1.849 (18)	2.7279 (15)	168.3 (16)
N2—H02...O3	0.881 (17)	1.910 (17)	2.7686 (15)	164.3 (15)
N3—H03...O4	0.919 (18)	1.785 (18)	2.6959 (16)	170.6 (15)
N4—H04...O1	0.888 (18)	1.858 (18)	2.7331 (16)	168.1 (16)
C9—H9B...O2 <sup>i</sup>	0.98	2.54	3.4176 (17)	149
C10—H10C...O2 <sup>i</sup>	0.98	2.58	3.4461 (18)	148
C4—H4...O3 <sup>ii</sup>	1.00	2.29	3.2723 (16)	167
C9—H9A...O3 <sup>ii</sup>	0.98	2.51	3.4581 (17)	164

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

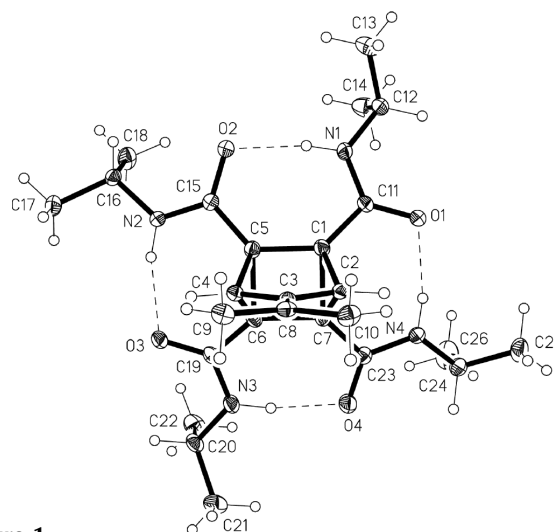


Figure 1

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.

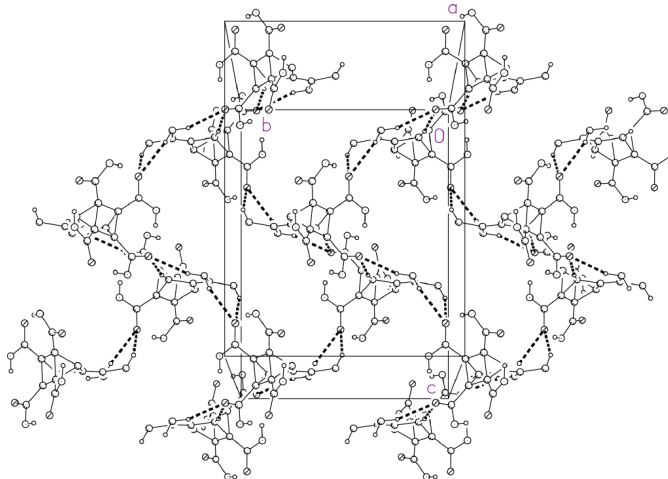


Figure 2

Packing diagram of the title compound, viewed perpendicular to the  $bc$  plane. Isopropyl groups have been omitted for clarity, as have other H atoms not involved in hydrogen bonds. Intermolecular hydrogen bonds are shown as thick dashed lines and intramolecular hydrogen bonds have been omitted.

H atoms bonded to N atoms were refined freely. Methyl H atoms were located in difference syntheses, idealized ( $\text{C—H } 0.98 \text{ \AA}$  and  $\text{H—C—H } 109.5^\circ$ ) 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  $\text{C—H}$  bond lengths of  $1.00 \text{ \AA}$ ;  $U_{\text{iso}}(\text{H})$  values were fixed at  $1.2U_{\text{eq}}$  of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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