$C_{11}H_{13}NO_3$

Monoclinic
$P2_1$
$a = 12.653 (3)$ Å
$b = 8.0849 (6)$ Å
$c = 5.5247 (9)$ Å
$\beta = 95.408 (8)°$
$V = 562.7 (2)$ Å^3
$Z = 2$
$D_x = 1.223$ Mg m^{-3}
D_m not measured

Cell parameters from 25
  reflections
$\theta = 9.78–14.03°$
$\mu = 0.089$ mm^{-1}
$T = 293 (2)$ K
Plate
$0.4 \times 0.3 \times 0.2$ mm
Colourless

Data collection

Enraf–Nonius CAD-4
  diffractometer
ω–2θ scans
Absorption correction: none
2062 measured reflections
1891 independent reflections
1243 reflections with
  $I > 2\sigma(I)$

$R_{int} = 0.018$
$\theta_{max} = 30.98°$
$h = -18 \rightarrow 18$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 7$
3 standard reflections
  frequency: 120 min
  intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.188$
$S = 0.801$
1891 reflections
138 parameters
H-atom parameters
  constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Extinction correction: none
Scattering factors from
  International Tables for
  Crystallography (Vol. C)

Table 1. *Selected torsion angles (°) for (I)*

C1—O2—C3—O4	−2.1 (7)	C5—N6—C8—C9	−138.8 (4)
O4—C3—C5—N6	13.2 (6)	C5—N6—C8—C10	96.5 (4)
O2—C3—C5—N6	−168.4 (4)	O7—N6—C8—C9	42.3 (5)
C3—C5—N6—O7	2.6 (6)	O7—N6—C8—C10	−82.5 (4)
C3—C5—N6—C8	−176.2 (3)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989).
Cell refinement: *CAD-4 Software*. Data reduction: *MolEN*
(Fair, 1990). Program(s) used to solve structure: *SHELXS86*
(Sheldrick, 1990). Program(s) used to refine structure:
SHELXL93 (Sheldrick, 1993). Molecular graphics: *ORTEPII*
(Johnson, 1976).

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius,
  Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for
  Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Hara, J., Inouye, Y. & Kakisawa, H. (1981). *Bull. Chem. Soc. Jpn*,
  52, 3763–3764.
Inouye, Y. (1983). *Bull. Chem. Soc. Jpn*, **56**, 244–247.
Inouye, Y. (1984). *Acta Cryst.* C40, 140–142.
Inouye, Y., Hara, J. & Kakisawa, K. (1980). *Chem. Lett.* pp. 1407–
  1410.

Inouye, Y., Takaya, K. & Kakisawa, K. (1983). *Bull. Chem. Soc. Jpn*,
  56, 3541–3542.
Inouye, Y., Takaya, K. & Kakisawa, K. (1985). *Magn. Reson. Chem.*
  23, 101–103.
Inouye, Y., Watanabe, Y., Takahashi, S. & Kakisawa, H. (1979). *Bull.
  Chem. Soc. Jpn*, **52**, 3763–3764.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge
  National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of
  Crystal Structures*. University of Göttingen, Germany.
Tamura, O., Yamaguchi, T., Okabe, T. & Sakamoto, M. (1994).
  Synlett, pp. 620–622.
Tufariello, J. J. (1984). *1,3-Dipolar Cycloaddition Chemistry*, Vol. 2,
  edited by A. Padwa, pp. 83–168. New York: John Wiley & Sons.

Mirror symmetry in 7,9-dibenzyl-1-*tert*-butoxycarbonyl-1,7,9-triazaspiro[4.5]dec-3-en-2-one

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Abstract

The crystal structure of *tert*-butyl 7,9-dibenzyl-2-oxo-
1,7,9-triazaspiro[4.5]dec-3-ene-1-carboxylate, $C_{26}H_{31}$-
N_3O_3, is reported. The molecule has imposed mirror
symmetry and the hexahydropyrimidine ring adopts a
chair conformation with two benzyl substituents bonded
equatorially to the ring N atoms. The 3-pyrrolin-2-one is
attached as a spiro-skeleton to the hexahydropyrimidine
ring by one axial C—C and one equatorial C—N bond.

Comment

During the course of synthetic studies with *N*-methyl-
eneamine equivalents generated from 1,3,5-trialkyl-
hexahydro-1,3,5-triazines, the spiro product 1-*tert*-
butoxycarbonyl-7,9-dialkyl-1,7,9-triazaspiro[4,5]dec-3-
en-2-one, was obtained from their reaction with 1-*tert*-
butoxycarbonyl-2-*tert*-butyldimethylsilyloxypyrrole (Ha
et al., 1999). When the alkyl substituent is benzyl, the
product is 1-*tert*-butoxycarbonyl-7,9-dibenzyl-1,7,9-tri-
azaspiro[4,5]dec-3-en-2-one, (I), and its structure was
determined by X-ray diffraction. In this title compound,
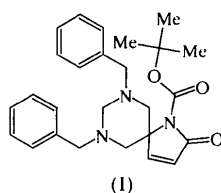
the hexahydropyrimidine ring adopts a chair conformation, and two benzyl substituents on nitrogen are bonded equatorially to the ring. The chair conformation with two equatorial pendants attached to the N atom was observed in the crystal structure of 1,3-dicyanomethyl-hexahydropyrimidine (Shoja & Saba, 1993). In the title compound, the 3-pyrrolin-2-one ring is perfectly flat, maximizing π-conjugation with the olefin, nitrogen and carbonyl groups. Two 3-pyrrolin-2-one and hexahydropyrimidine rings share a spiro-skeleton C atom and are almost perpendicular [88.3 (2)°]. As a whole the molecule has crystallographic mirror symmetry, bisected by 3-pyrrolin-2-one, making the two benzyl substituents spatially indistinguishable and chemically equivalent.
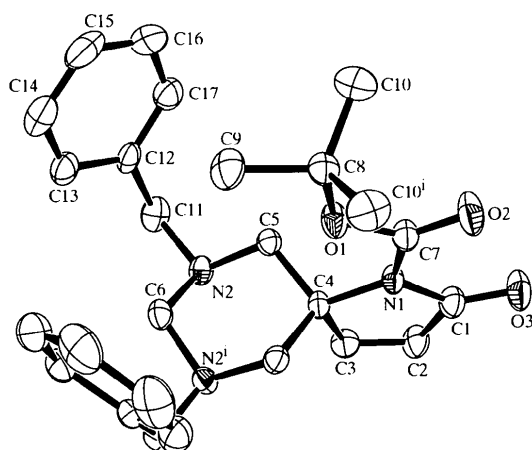

Fig. 1. An *ORTEP*-3 (Farrugia, 1998) drawing of the title compound with the atomic labelling scheme. Displacement ellipsoids are shown at the 30% probability level, and H atoms are omitted for clarity. Symmetry code: (i) x, $\frac{1}{2} - y$, z.

Experimental

The title compound was prepared from the reaction of 1,3,5-tribenzylhexahydro-1,3,5-triazine with 1-*tert*-butoxycarbonyl-2-*tert*-butyldimethylsilyloxypyrrole. The initial reaction products were purified by column chromatography on silica gel. The title compound was recrystallized from ethyl acetate to afford single crystals suitable for X-ray diffraction analysis.

Crystal data

$C_{26}H_{31}N_3O_3$
$M_r = 433.60$

Orthorhombic
Pnma
$a = 16.482\,(5)$ Å
$b = 14.258\,(5)$ Å
$c = 9.838\,(4)$ Å
$V = 2312.0\,(1)$ Å^3
$Z = 4$
$D_x = 1.246$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 18 reflections
$\theta = 10$–$14°$
$\mu = 0.082$ mm^{-1}
$T = 293\,(2)$ K
Prism
$0.50 \times 0.26 \times 0.24$ mm
Colourless

Data collection

MXC3 diffractometer
ω–2θ scans
Absorption correction: none
2266 measured reflections
2199 independent reflections
1221 reflections with
$\quad I > 2\sigma(I)$
$R_{int} = 0.127$

$\theta_{max} = 27.48°$
$h = -21 \rightarrow 0$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 12$
2 standard reflections
$\quad$ every 100 reflections
$\quad$ intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.116$
$S = 1.009$
2199 reflections
172 parameters
H atoms treated by a
$\quad$ mixture of independent
$\quad$ and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2]$
$\quad$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.243$ e Å^{-3}
$\Delta\rho_{min} = -0.141$ e Å^{-3}
Extinction correction:
$\quad$ *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient:
$\quad$ 0.0141 (10)
Scattering factors from
$\quad$ *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—O3	1.206 (4)	C6—N2	1.456 (3)
C1—N1	1.407 (4)	C7—O2	1.193 (4)
C1—C2	1.459 (5)	C7—O1	1.326 (4)
C2—C3	1.311 (5)	C7—N1	1.400 (4)
C3—C4	1.495 (5)	C8—O1	1.484 (4)
C4—N1	1.487 (4)	C11—N2	1.475 (3)
C4—C5	1.532 (3)	C11—C12	1.512 (3)
C5—N2	1.466 (3)		
O3—C1—N1	126.6 (4)	O2—C7—N1	124.3 (3)
O3—C1—C2	127.9 (4)	O1—C7—N1	109.1 (3)
N1—C1—C2	105.5 (3)	O1—C8—C10	109.1 (2)
C3—C2—C1	110.6 (3)	O1—C8—C9	101.9 (3)
C2—C3—C4	112.1 (3)	N2—C11—C12	115.4 (2)
N1—C4—C3	100.9 (3)	C7—O1—C8	122.7 (3)
N1—C4—C5	111.9 (2)	C7—N1—C1	122.7 (3)
C3—C4—C5	110.3 (2)	C7—N1—C4	126.3 (3)
C5—C4—C5^i	111.2 (3)	C1—N1—C4	111.0 (3)
N2—C5—C4	109.3 (2)	C6—N2—C5	109.8 (2)
N2^i—C6—N2	109.8 (3)	C6—N2—C11	111.0 (2)
O2—C7—O1	126.5 (3)	C5—N2—C11	111.2 (2)

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

Only H atoms H9*A* and H9*B* were refined.

Data collection: *MXC Diffractometer Control Software* (MacScience Corporation, 1994). Cell refinement: *MXC Diffractometer Control Software*. Data reduction: *MXC Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*-3 (Farrugia, 1998). Software used to prepare material for publication: *SHELXL*97.

References

Farrugia, L. J. (1998). *ORTEP-3 for Windows*. University of Glasgow, Scotland.

Ha, H.-J., Suh, J.-M., Ahn, Y.-G., Dong, Y. & Yun, H. (1999). *Heterocycles*, **50**, 203–214.

MacScience Corporation (1994). *MXC Diffractometer Control Software*. MacScience Corporation, Tokyo, Japan.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Shoja, M. & Saba, S. (1993). *Acta Cryst.* C**49**, 354–355.

2-*tert*-Butyl-9,10-dibromoanthracene

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Abstract

The title compound, $C_{18}H_{16}Br_2$, crystallized on a crystallographic mirror plane. There was disorder of the ^{t}Bu group with a major orientation at 59%. Interplanar spacings are 3.509 (4) Å.

Comment

The title compound, (I), was structurally characterized in order to measure interplanar distances for studies aimed at assessing the forces of attraction in this molecule, and to make comparisons with other molecules such as anthracene (Lehmann & Pawley, 1972) and 9,10-dibromoanthracene (Kitaigorodski, 1946; Trotter, 1958, 1986; Bulgarovskaya *et al.*, 1987) of a similar nature. We find that the interplanar spacing in (I) of 3.509 (4) Å is similar to that in 9,10-dibromoanthracene of 3.52 (1) Å (Trotter, 1986). It is also interesting to note that the packing arrangement in 9,10-dibromoanthracene, where the molecules are stacked on top of each other, is very different from that observed in (I). Clearly, the ^{t}Bu group is responsible for these differences. Additionally, the closest Br$\cdots$Br intermolecular distance of 4.004 (2) Å in (I) is smaller than that of 4.068 (1) Å in 9,10-dibromoanthracene (Trotter, 1968). It is likely that the skewed arrangement of the rings in (I) which

reduces Br–Br repulsions, also results in a decrease in the interplanar spacings in (I).

(I)
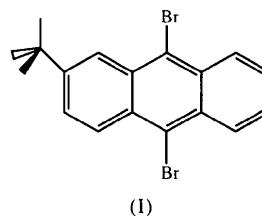
Molecules of (I) in the crystal are arranged in a herring-bone manner parallel to the *bc* plane and perpendicular to the *a* axis. The angle for the intramolecular Br$\cdots$Br vectors situated between adjacent coplanar rows is 90.9 (1)°. Adjacent pairs of molecules are skewed, with ^{t}Bu groups on opposite sides and displaced by one ring so that only the two fused rings furthest from the ^{t}Bu group are aligned on top of each other. The bond distance and angles within the anthracene rings in (I) and 9,10-dibromoanthracene are similar.
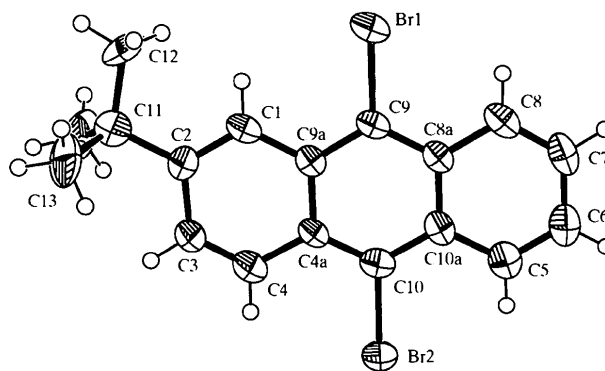

Fig. 1. View of the major contributor to (I) (50% probability displacement ellipsoids). H atoms, represented by circles of arbitrary radii, are not labeled.

Experimental

Crystals of (I) prepared as detailed previously (Lee & Mendenhall, 1988) were obtained by slow crystallization from an ethanol solution.

Crystal data

$C_{18}H_{16}Br_2$	Mo $K\alpha$ radiation
$M_r = 392.14$	$\lambda = 0.71069$ Å
Orthorhombic	Cell parameters from 25
$Pnma$	reflections
$a = 23.433 (5)$ Å	$\theta = 10.4$–$15.0°$
$b = 7.018 (4)$ Å	$\mu = 5.15$ mm^{-1}
$c = 9.554 (4)$ Å	$T = 293 (2)$ K
$V = 1571.2 (12)$ Å^3	Prism
$Z = 4$	$0.22 \times 0.20 \times 0.08$ mm
$D_x = 1.658$ Mg m^{-3}	Yellow
D_m not measured	