

Acta Crystallographica Section C

**Crystal Structure
Communications**

ISSN 0108-2701

1,7,8-Tri-O-acetyl-6-O-benzoylcastanospermine

Graeme J. Gainsford *et al.*

Electronic paper

This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

© 2000 International Union of Crystallography • Printed in Great Britain – all rights reserved

1,7,8-Tri-*O*-acetyl-6-*O*-benzoyl-castanospermine

Graeme J. Gainsford,* Peter C. Tyler and Richard H. Furneaux

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

Received 20 June 2000

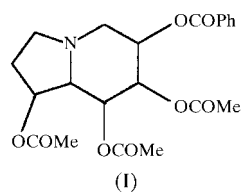
Accepted 13 July 2000

Data validation number: IUC0000192

The title compound (alternatively 1,7,8-triacetoxyperhydroindolizin-6-yl benzoate), $C_{21}H_{25}NO_8$, was obtained during studies of castanospermine derivatives. The crystal structure consists of independent molecules with only van der Waals contacts. The fused six- and five-membered rings adopt chair and twist conformations, respectively.

Comment

Crystals of the title compound, (I) [(7) in Furneaux *et al.* (1994)], were isolated as an acylated precursor in the preparation of substituted castanospermine derivatives. The structure consists of independent molecules with normal bond



lengths and angles (Orpen *et al.*, 1992). The small deviations from normality of the C2–C3 and N4–C3 bond lengths [1.565 (7) and 1.451 (7) Å, respectively] are consistent with those found in the previous crystal structures of 1,6,7,8-tetra-*O*-benzylcastanospermine (Mulzer *et al.*, 1992) and to a lesser extent in castanospermine (Hempel *et al.*, 1993, hereafter BEDLEB), 7-*epi*-7-*C*-methylcastanospermine (Furneaux *et al.*, 1997, hereafter NAMVIG) and 1-*C*-methylcastanospermine hydrochloride (Furneaux *et al.*, 1997). The conformation of the indolizine ring is close to ideal chair, with atoms C5, C6, C8 and C9 coplanar [± 0.008 (3) Å] and atoms N4 and C7 0.710 (7) and -0.675 (8) Å, respectively, from the plane [$Q = 0.606$ (6) Å, $\theta = 3.9$ (5)° and $\varphi = 340$ (8)°; Cremer & Pople, 1975], as has been found in BEDLEB and NAMVIG. The five-membered fused ring (C1/C2/C3/N4/C9) adopts a twist conformation on C9–N4 with $Q(2) = 0.411$ (6) Å and $\varphi = 160.6$ (8)°, similar to the values of 0.448 Å and 169.1° found in NAMVIG. The similarity of the fused-ring conformations is

best illustrated by the dihedral angles C9–C1–C2–C3 and C5–N4–C3–C2, which are -14.1 (5) and 158.3 (5)° here, -16.1 and 161.8 ° in NAMVIG, and -12.1 and 159.2 ° in BEDLEB. The benzoyl phenyl ring (C13–C18) makes an angle of 87.4 (2)° with the mean plane through the C5–C9/N4 ring.

The closest H···O intermolecular contacts with potential as C–H···O hydrogen bonds [e.g. C5–H5B···O16 2.44 (5) Å; O16 at $-1 + x, y, z$] are longer than several intramolecular contacts, e.g. C6–H6···O16 2.28 Å, suggesting only van der Waals packing forces are present for the molecules.

Experimental

Crystals of (I) were obtained as described for compound (7) in Furneaux *et al.* (1994).

Crystal data

$C_{21}H_{25}NO_8$
 $M_r = 419.42$
Orthorhombic, $P2_12_12_1$
 $a = 6.004$ (2) Å
 $b = 7.675$ (4) Å
 $c = 45.564$ (3) Å
 $V = 2099.6$ (13) Å³
 $Z = 4$
 $D_x = 1.327$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 17$ – 33 °
 $\mu = 0.102$ mm⁻¹
 $T = 143$ (2) K
Needle, translucent colourless
 $0.75 \times 0.25 \times 0.20$ mm

Data collection

Siemens $R3m$ diffractometer
 ω scans
2219 measured reflections
2219 independent reflections
1373 reflections with $I > 3\sigma(I)$
 $\theta_{max} = 24.65$ °

$h = 0 \rightarrow 7$
 $k = 0 \rightarrow 8$
 $l = 0 \rightarrow 53$
3 standard reflections every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R(F) = 0.048$
 $wR(F^2) = 0.093$
 $S = 1.126$
1373 reflections
271 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 0.5110P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.17$ e Å⁻³
 $\Delta\rho_{min} = -0.21$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O10–C10	1.348 (6)	N4–C9	1.475 (7)
O10–C1	1.457 (6)	N4–C5	1.486 (6)
O14–C10	1.200 (6)	C5–C6	1.516 (7)
C1–C2	1.517 (7)	C6–C7	1.518 (7)
C1–C9	1.519 (7)	C7–C8	1.524 (7)
C2–C3	1.565 (7)	C8–C9	1.503 (7)
C3–N4	1.451 (7)	C10–C11	1.490 (8)
C10–O10–C1	116.2 (4)	C9–N4–C5	111.4 (4)
O10–C1–C2	111.5 (4)	C5–C6–C7	111.2 (5)
C2–C1–C9	104.1 (4)	N4–C9–C8	109.2 (5)
C1–C2–C3	105.3 (4)	N4–C9–C1	102.2 (4)
N4–C3–C2	103.7 (4)	C8–C9–C1	117.8 (4)
C3–N4–C9	105.5 (4)	O14–C10–O10	122.6 (5)
C3–N4–C5	114.2 (4)	O14–C10–C11	125.9 (5)
C10–O10–C1–C2	83.1 (6)	C2–C3–N4–C5	158.3 (5)
C9–C1–C2–C3	–14.1 (5)	C9–N4–C5–C6	–62.1 (6)
C1–C2–C3–N4	–12.6 (5)	C2–C1–C9–N4	35.4 (5)
C2–C3–N4–C9	35.7 (5)	C2–C1–C9–C8	155.0 (5)

Methyl and all other H atoms were constrained to an isotropic displacement parameter 1.5 and 1.2 times, respectively, that of the U_{eq} of their parent atom.

Data collection: *R3m Software* (Siemens, 1983); cell refinement: *R3m Software*; data reduction: *R3m Software*; program(s) used to solve structure: *SHELXS85* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

References

- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Furneaux, R. H., Gainsford, G. J., Mason, J. M. & Tyler, P. C. (1994). *Tetrahedron*, **50**, 2131–2160.
- Furneaux, R. H., Gainsford, G. J., Mason, J. M., Tyler, P. C., Hartley, O. & Winchester, B. G. (1997). *Tetrahedron*, **53**, 245–268.
- Hempel, A., Camerman, N., Mastropaolo, D. & Camerman, A. (1993). *J. Med. Chem.* **36**, 4082–4086.
- Mulzer, J., Dehmlow, H., Buschmann, J. & Luger, P. (1992). *J. Org. Chem.* **57**, 3194–3202.
- Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1992). *International Tables for Crystallography*, Vol. C, pp. 685–706. Dordrecht: Kluwer Academic Publishers.
- Sheldrick, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Siemens (1983). *R3m Software*. Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.