C26	−0.3592 (6)	0.6339 (9)	1.1530 (3)	0.072 (2)
C27	−0.3542 (5)	0.4039 (11)	1.2611 (3)	0.070 (2)
C28	0.5318 (5)	−0.4887 (9)	0.6425 (3)	0.035 (2)
C29	0.4497 (4)	−0.6243 (9)	0.6192 (3)	0.0342 (14)
C30	0.3739 (4)	−0.5494 (8)	0.5685 (3)	0.033 (2)
C31	0.3763 (4)	−0.3442 (8)	0.5423 (2)	0.0231 (13)
C32	0.4588 (4)	−0.2158 (8)	0.5648 (3)	0.039 (2)
C33	0.5377 (5)	−0.2873 (9)	0.6142 (3)	0.037 (2)
C34	0.6107 (5)	−0.5653 (9)	0.7001 (3)	0.052 (2)

Table 2. *Selected geometric parameters (Å, °)*

S—O1	1.415 (3)	C20—C21	1.519 (6)
S—O2	1.414 (3)	C20—C22	1.519 (6)
S—O3	1.567 (4)	C22—C23	1.504 (7)
S—C31	1.722 (5)	C23—C24	1.504 (6)
O1—C3	1.469 (5)	C24—C25	1.496 (7)
C5—C6	1.312 (6)	C25—C27	1.494 (6)
C17—C20	1.518 (7)	C25—C26	1.505 (7)
O3—S—O2	119.4 (2)	C21—C20—C22	110.1 (4)
O3—S—O1	104.1 (2)	C23—C22—C20	116.8 (5)
O2—S—O1	110.2 (2)	C22—C23—C24	115.4 (5)
O3—S—C31	110.6 (2)	C25—C24—C23	116.6 (5)
O2—S—C31	108.8 (3)	C27—C25—C24	111.0 (5)
O1—S—C31	102.4 (2)	C27—C25—C26	111.6 (5)
C17—C20—C21	114.4 (4)	C24—C25—C26	112.4 (5)
C17—C20—C22	112.2 (4)		
C31—S—O1—C3	68.6 (4)		

The unit-cell and intensity data were collected on a Delft Instruments FAST diffractometer using the routines *ENDEX*, *REFINE* and *MADONL* in the *MADNES* software (Pflugrath & Messerschmidt, 1989) and processed using *ABSMAD* (Karaulov, 1992); detailed procedures are described by Darr, Drake, Hursthouse & Malik (1993). The non-H atoms were refined with anisotropic displacement parameters and H atoms were allowed to ride on their attached C atoms with a common isotropic displacement parameter.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1989). Cell refinement: *ABSMAD* (Karaulov, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1066). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Naphtho[2,3-*b*]cholestane

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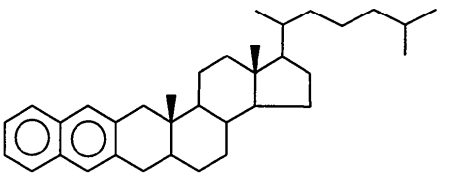
Abstract

The crystal structure of the title compound, naphtho-[2,3-*b*]cholestane, $C_{35}H_{50}$, is composed of independent molecules with normal molecular dimensions and no unusual contacts shorter than van der Waals distances.

Comment

In the course of our studies on the direct asymmetric introduction of a tricarbonylchromium moiety on prochiral arenes, we required pure optically active naphthalene ligands. The title compound, (1), has eight asymmetric centres and fulfills some of the requirements for a good chiral auxiliary: (i) good leaving-group ability, (ii) stable chiral information and (iii) easy recovery. In this paper, we report the crystal structure of the title compound. We did not determine the absolute configuration by X-ray methods, but the absolute configuration of the dibromocholestane has been established (Geise & Romers, 1966) and the structure and coordinates reported here refer to the same absolute configuration.



(1)

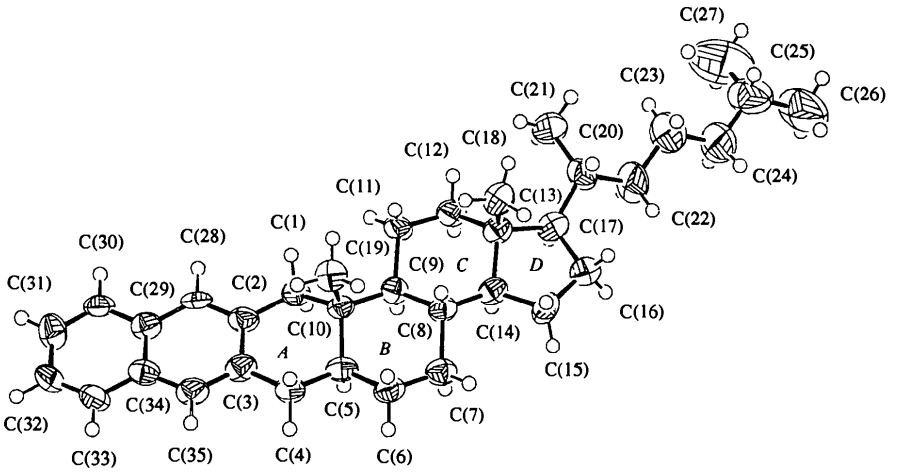


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level; H atoms have been assigned arbitrary radii.

Fig. 1 shows the *ORTEP*II (Johnson, 1976) drawing of (1) in which a naphthyl group has been fused to cholestane at ring *A*. The molecular dimensions in (1) are unexceptional with mean bond distances: Csp^3—Csp^3 1.53 (3), Csp^3—Csp^2 1.50 (1) and C—C(aromatic) 1.39 (3) Å. The bond angles are also within error limits of the expected values. The molecular dimensions in (1) correspond closely with the molecular dimensions reported for 2,3-dibromo- and 2,3-dichlorocholestane (Geise & Romers, 1966).

The naphthyl moiety is essentially planar with maximum deviation of atoms from the least-squares planes being 0.03 (1) Å. The ring *A* of cholestane adopts a flattened chair conformation owing to the rigid naphthalene ring. The rings *B* and *C* of the steroid are in almost ideal chair conformations. The cyclopentane ring *D* adopts a C(13)-envelope conformation. The aliphatic chain that extends off ring *D* is a fully extended arm and has a *transoid* conformation along the entire chain.

Experimental

The title compound was prepared from 5α-cholestan-3-one in four steps according to a modified literature method (Tius & Gomez-Saleno, 1986). Hydroxymethylation with ethyl formate followed by treatment with the trimethylchlorosilane-tertiary amine system gave 5α-2-[{(trimethylsilyl)oxy}methylene]-cholestan-3-one. The 1,2-addition of benzyl magnesium chloride followed by treatment with pyridinium *p*-toluenesulfonate gave 5α-3-benzyl-2-formyl-cholest-2-ene, which is easily aromatized in the presence of $TiCl_4$ to the title compound. Purification by flash chromatography (SiO_2/hexanes) and subsequent recrystallization from hexane gave naphtho-[2,3-*b*]cholestane, (1).

Crystal data

$C_{35}H_{50}$
M_r = 470.78
Monoclinic
$P2_1$
a = 6.553 (4) Å
b = 10.893 (4) Å
c = 20.492 (2) Å
β = 94.09 (2)°
V = 1459.0 (8) Å^3
Z = 2
D_x = 1.072 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 21 reflections
θ = 9.0–12.0°
μ = 0.059 mm^{-1}
T = 296 K
Plate
0.60 × 0.45 × 0.15 mm
Colourless

Data collection

Rigaku AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ-scans (North, Phillips & Mathews, 1968)
T_{min} = 0.781, T_{max} = 1.000
2981 measured reflections
2730 independent reflections
1175 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.0453
θ_{max} = 25.0°
h = 0 → 7
k = 0 → 12
l = −24 → 24
3 standard reflections monitored every 200 reflections
intensity decay: 0.12%

Refinement

Refinement on F
R = 0.0595
wR = 0.0611
S = 3.158
1175 reflections
317 parameters
H atoms riding, C—H 0.95 Å
$w = 1/[\sigma^2(F_o) + 0.020(F_o^2)]$
$(\Delta/\sigma)_{max}$ = 0.031
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: Zachariasen type 2 Gaussian isotropic (Zachariasen, 1968)
Extinction coefficient: 13.41504
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C(1)	0.9657 (9)	0.0888 (6)	0.8272 (3)	0.047 (2)
C(2)	0.9204 (9)	−0.0299 (6)	0.8612 (3)	0.050 (2)
C(3)	0.7392 (9)	−0.0436 (6)	0.8958 (3)	0.053 (2)
C(4)	0.5822 (9)	0.0564 (6)	0.8932 (3)	0.063 (2)
C(5)	0.6625 (9)	0.1823 (5)	0.8718 (3)	0.048 (2)
C(6)	0.4911 (9)	0.2756 (6)	0.8662 (3)	0.057 (2)
C(7)	0.5638 (9)	0.4019 (6)	0.8468 (3)	0.054 (2)
C(8)	0.6753 (9)	0.3956 (6)	0.7840 (3)	0.049 (2)
C(9)	0.8541 (9)	0.3019 (6)	0.7931 (3)	0.047 (2)
C(10)	0.7825 (9)	0.1709 (5)	0.8115 (3)	0.044 (2)
C(11)	0.9786 (9)	0.3018 (6)	0.7303 (3)	0.063 (2)
C(12)	1.0522 (9)	0.4337 (6)	0.7142 (3)	0.056 (2)
C(13)	0.8761 (9)	0.5221 (6)	0.7039 (3)	0.050 (2)
C(14)	0.7606 (9)	0.5204 (5)	0.7666 (3)	0.050 (2)
C(15)	0.6159 (9)	0.6293 (6)	0.7592 (3)	0.065 (2)
C(16)	0.7355 (9)	0.7261 (6)	0.7245 (3)	0.059 (2)
C(17)	0.9285 (9)	0.6603 (6)	0.6993 (3)	0.053 (2)
C(18)	0.7339 (9)	0.4872 (6)	0.6418 (3)	0.063 (2)
C(19)	0.6480 (9)	0.1113 (6)	0.7526 (3)	0.058 (2)
C(20)	1.0021 (10)	0.7164 (6)	0.6374 (3)	0.066 (2)
C(21)	1.1732 (11)	0.6470 (7)	0.6078 (4)	0.090 (3)
C(22)	1.0650 (11)	0.8508 (7)	0.6505 (4)	0.095 (3)
C(23)	1.1242 (14)	0.9208 (8)	0.5896 (4)	0.112 (3)
C(24)	1.1774 (14)	1.0539 (8)	0.6036 (5)	0.120 (4)
C(25)	1.2751 (16)	1.1233 (9)	0.5528 (4)	0.122 (4)
C(26)	1.2745 (21)	1.2539 (9)	0.5634 (5)	0.210 (6)
C(27)	1.4855 (16)	1.0731 (14)	0.5439 (5)	0.206 (6)
C(28)	1.0600 (9)	−0.1263 (6)	0.8637 (3)	0.046 (2)
C(29)	1.0342 (9)	−0.2362 (6)	0.8993 (3)	0.051 (2)
C(30)	1.1759 (9)	−0.3325 (6)	0.9017 (3)	0.051 (2)
C(31)	1.1445 (10)	−0.4347 (6)	0.9359 (3)	0.064 (2)
C(32)	0.9662 (10)	−0.4493 (6)	0.9691 (3)	0.059 (2)
C(33)	0.8246 (10)	−0.3588 (7)	0.9664 (3)	0.060 (2)
C(34)	0.8540 (9)	−0.2493 (6)	0.9316 (3)	0.049 (2)
C(35)	0.7112 (9)	−0.1511 (7)	0.9282 (3)	0.059 (2)

Space group $P2_1$ or $P2_1/m$ from the systematic absences $0k0$, $k = 2n + 1$; the former was chosen and confirmed by successful solution and refinement of the structure. The terminal C atoms C(26) and C(27) of the aliphatic chain exhibit large thermal motions showing a small degree of disorder.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1167). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6-(3-Methylbenzyl)-2-(2-methylpropyl)thio-4(3*H*)-pyrimidinone (DABO 622)

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Abstract

The title compound, $C_{16}H_{20}ON_2S$, shows the pyrimidine and benzene rings arranged in 'butterfly-like' conformation as observed for TIBO, nevirapine and other non-nucleoside inhibitors of HIV-1 Reverse Transcriptase.

Comment

Virally encoded reverse transcriptase (RT) of human immunodeficiency virus type 1 (HIV-1) catalyses the retrotranscription of single-stranded viral RNA into double-stranded DNA before the viral genome is integrated into the DNA of the host cell (Mitsuya, Yarchoan & Broder, 1990). Currently two classes of *anti*-AIDS agents targeted at RT have been developed, *i.e.* nucleoside analogues such as AZT, ddC, ddI and D4T, which have been approved for the treatment of AIDS, and non-nucleoside inhibitors (NNRTIs). Among these, the first compounds described were TIBO, HEPT and nevirapine, followed by BHAP, PETT α-APA and, more recently, by DABO