the other H atom could not be located because of the disorder. All other H atoms were fixed at ideal positions.

Data collection: Nicolet *P*3 software (Nicolet XRD Corporation, 1985). Cell refinement: Nicolet *P*3 software. Data reduction: *SHELXTL* (Sheldrick, 1985). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1017). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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β-Cyclodextrin Nonanoic Acid 1:1 Complex

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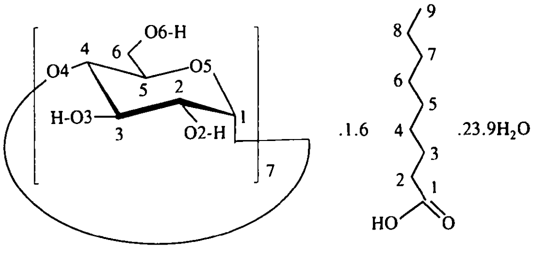
Abstract

The structure of the complex of cyclomaltoheptaose (β-CD) with nonanoic acid, β-cyclodextrin–nonanoic acid (1/1) water solvate, $2C_{42}H_{70}O_{35}.1.6C_9H_{18}O_2.23.9H_2O$, has been determined at room temperature. Two independent molecules of β-CD form a dimer which encloses two molecules of nonanoic acid. The guest molecules penetrate the β-CD cavities to different depths. Their carboxylic groups point towards the primary hydroxyl region of β-CDs forming direct dimers through hydrogen bonds with the carboxylic groups of adjacent guests along the *a* axis and interact with water molecules and disordered primary hydroxyl O atoms.

Comment

The structure determination of the title compound, (I), is a part of a systematic investigation involving the inclusion of linear molecules by β-CD or its derivatives (Mentzafos, Mavridis & Schenk, 1994).



(I)

The numbering scheme for the host and guest molecules is given in Fig. 1; C(*A* or *B*)*mn* or O(*A* or *B*)*mn* denotes the *m*th atom within the *n*th glucosidic residue of the crystallographically independent β-CD molecules

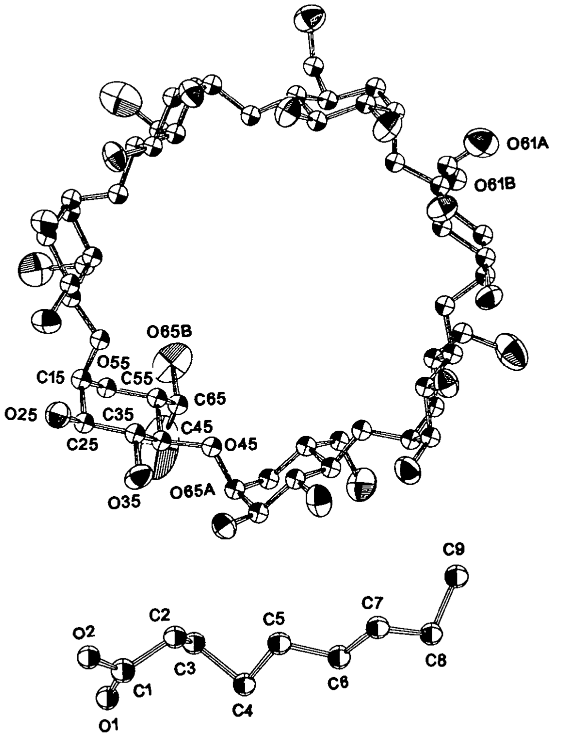


Fig. 1. The host and guest molecule *A* with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% level.

A or *B*. The β-CD molecules *A* and *B* form dimers *via* O*A*3*n*···O*B*3(8−*n*) hydrogen bonds (Mavridis & Hadjoudis, 1992). Two guest molecules are accommodated in the cavity of the β-CD dimer (Fig. 2). There is no distortion of the sevenfold symmetry of β-CD due to complexation: the average O4*n*···O4(*n*+1) distance is 4.38 (6) Å, the average O4(*n*−1)···O4*n*···O4(*n*+1) angle is 129 (2)° and the deviation of the O4*n* atoms from their optimum plane is 0.00 (2) Å.

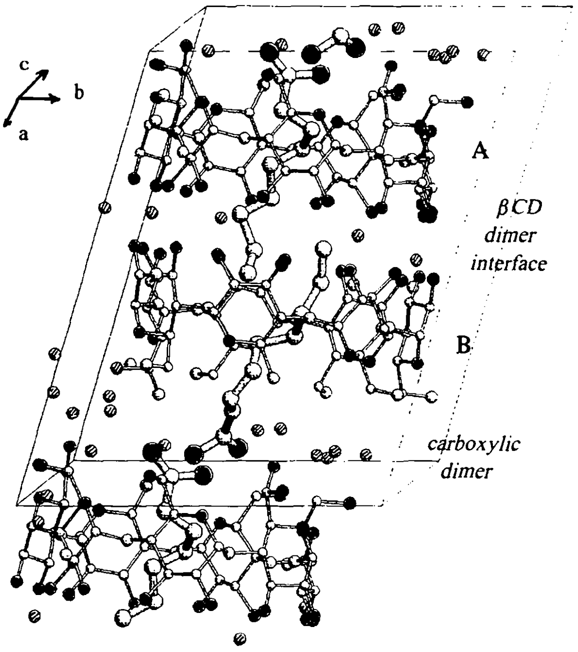


Fig. 2. The packing of complex molecules as viewed along the *a* axis. The O atoms of the host and guest molecules are fully shaded. Water O atoms are line shaded.

The primary hydroxyl groups have a *gauche–gauche* orientation pointing outside the cavity [mean torsion angles C4—C5—C6—O6 and O5—C5—C6—O6 are 56 (3) and −64 (4)°, respectively]. Atoms O61 and O65 of β-CD *A* and O67 of β-CD *B* are disordered over two positions. The major orientation is *gauche–gauche* (site-occupancy factors 0.62, 0.51 and 0.74, respectively) and the other is *gauche–trans* pointing inwards [mean torsion angles C4—C5—C6—O6 168.7, 180.0, 171.5° and O5—C5—C6—O6 50.4, 57.7, 50.5°, respectively]. The dimers form layers almost parallel to the *bc* plane, the dimer axis forming an angle of 18.3° with the crystallographic *a* axis. The distance between the projections of two consecutive dimers onto the O4*n* mean plane is 5.96 Å.

The nonanoic acid molecules are enclosed in the β-CD dimeric cavity with their carboxylic groups pointing towards the primary hydroxyl edges of β-CD. The carboxylic group of guest molecule *A* is located at the level of the primary hydroxyl region of β-CD molecule *A*, while its aliphatic chain extending through the intradimer space enters the secondary hydroxyl site of the β-CD molecule *B*. Guest molecule *B* does not penetrate the β-CD cavity as much: Its aliphatic end is located at the level of the secondary hydroxyl groups of β-CD molecule *B* and the carboxylic group protruding from the primary hydroxyl region extends into the interdimer space. Thus, it approaches the carboxylic group of guest molecule *A* of the consecutive dimeric layer forming a conventional carboxylic dimer (Fig. 3). The structure is also stabilized by additional hydrogen bonds between the carboxylic O atoms of both guest molecules and water molecules O262 and O26*B* as well as the *gauche–trans* disordered hydroxyl groups O65*B* and O67*B* (Table 2), therefore justifying their inward orientation. The β-CD dimers form a channel-like matrix (intermediate packing mode; Mentzafos, Mavridis, Le Bas & Tsoucaris, 1991) that allows the nonanoic acid molecules to form dimers along the *a* axis of the crystal.

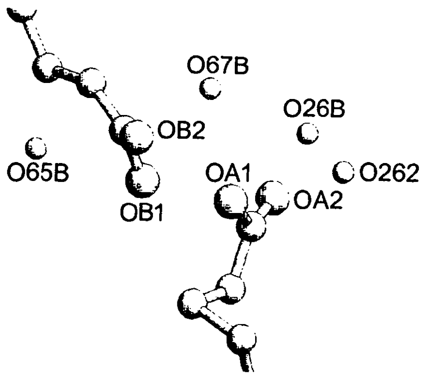


Fig. 3. Detail of the interacting atoms at the vicinity of the carboxylic dimers (O262 and O26*B* are water O atoms and O67*B* and O65*B* disordered hydroxyl O atoms).

There are 23.9 water molecules in the unit cell distributed over 27 sites. 25 of them are within hydrogen-bonding distance of primary and secondary hydroxyl groups of β-CDs.

Experimental

The complex was prepared from an aqueous solution of β-CD to which an approximately tenfold excess of nonanoic acid was added. It was then dissolved on heating to 339 K and the solution was allowed to cool slowly to 276 K over a 9 day period when colourless crystals were formed.

Crystal data

$2C_{42}H_{70}O_{35}.1.6C_9H_{18}O_2.$-
$23.9H_2O$
$M_r = 2953.55$
Triclinic

Mo *Kα* Nb-filtered radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections

$P1$
$a = 18.056$ (3) Å
$b = 15.446$ (2) Å
$c = 15.452$ (2) Å
$\alpha = 103.159$ (5)°
$\beta = 112.994$ (5)°
$\gamma = 99.352$ (5)°
$V = 3707.8$ (9) Å^3
$Z = 1$
$D_x = 1.323$ Mg m^{-3}
D_m not measured

$\theta = 5.5$–11.5°
$\mu = 0.117$ mm^{-1}
$T = 293$ (2) K
Diamond
0.8 × 0.4 × 0.2 mm
Colourless

Data collection

Syntex $P2_1$ diffractometer
θ–2θ with 4.5° min^{-1} scans
Absorption correction:
none
8325 measured reflections
8325 independent reflections
5872 observed reflections
$[I > 2\sigma(I)]$
$\theta_{\max} = 21.00°$

$h = -18 \rightarrow 16$
$k = -15 \rightarrow 15$
$l = 0 \rightarrow 15$
3 standard reflections
monitored every 67
reflections
frequency: 95 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0783$
$wR(F^2) = 0.2939$
$S = 1.094$
8311 reflections
1096 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.1816P)^2 + 6.3005P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -1.626$

$\Delta\rho_{\max} = 0.573$ e Å^{-3}
$\Delta\rho_{\min} = -0.375$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration:
Flack (1983)
Flack parameter =
−1.31 (241)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (*Å^2*)

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

	x	y	z	U_{eq}
CA11	0.2095	0.0353	0.2526	0.053 (4)
CA21	0.2773 (10)	0.0349 (12)	0.2176 (13)	0.061 (4)
OA21	0.3497 (8)	0.0284 (8)	0.2931 (9)	0.061 (3)
CA31	0.2927 (10)	0.1188 (11)	0.1851 (12)	0.053 (4)
OA31	0.3533 (8)	0.1184 (9)	0.1496 (9)	0.065 (3)
CA41	0.2112 (9)	0.1220 (11)	0.1065 (11)	0.046 (3)
OA41	0.2291 (7)	0.2106 (8)	0.0902 (8)	0.051 (2)
CA51	0.1445 (10)	0.1169 (11)	0.1408 (12)	0.052 (3)
OA51	0.1336 (7)	0.0394 (8)	0.1751 (9)	0.057 (3)
CA61	0.0598 (15)	0.1080 (17)	0.0623 (19)	0.091 (6)
O61A*	−0.0024 (15)	0.0917 (16)	0.0829 (18)	0.071 (9)
O61B*	0.0334 (16)	0.042 (2)	−0.005 (2)	0.084 (11)
CA12	0.2080 (9)	0.2101 (11)	−0.0087 (11)	0.047 (3)
CA22	0.2867 (10)	0.2673 (11)	−0.0062 (12)	0.053 (4)
OA22	0.3540 (7)	0.2244 (9)	0.0251 (10)	0.065 (3)
CA32	0.3091 (9)	0.3638 (10)	0.0607 (11)	0.044 (3)
OA32	0.3811 (7)	0.4203 (8)	0.0614 (9)	0.058 (3)
CA42	0.2369 (9)	0.4035 (10)	0.0224 (11)	0.043 (3)
OA42	0.2576 (7)	0.4935 (8)	0.0933 (8)	0.050 (2)
CA52	0.1573 (10)	0.3428 (11)	0.0123 (12)	0.052 (4)
OA52	0.1413 (7)	0.2476 (8)	−0.0453 (9)	0.055 (2)
CA62	0.0785 (10)	0.3697 (12)	−0.0460 (12)	0.054 (4)
OA62	0.0729 (8)	0.3768 (9)	−0.1371 (10)	0.070 (3)
CA13	0.2464 (9)	0.5682 (10)	0.0569 (12)	0.046 (3)
CA23	0.3287 (9)	0.6442 (11)	0.1074 (11)	0.048 (3)
OA23	0.3898 (7)	0.6086 (8)	0.0880 (9)	0.057 (3)
CA33	0.3538 (9)	0.6838 (10)	0.2173 (11)	0.046 (3)
OA33	0.4289 (7)	0.7588 (8)	0.2627 (9)	0.058 (3)
CA43	0.2843 (9)	0.7180 (11)	0.2289 (11)	0.047 (3)
OA43	0.3074 (7)	0.7522 (7)	0.3337 (8)	0.045 (2)
CA53	0.2032 (9)	0.6398 (11)	0.1777 (12)	0.050 (3)
OA53	0.1830 (7)	0.6028 (8)	0.0743 (8)	0.056 (2)
CA63	0.1306 (11)	0.6707 (12)	0.1818 (14)	0.064 (4)
OA63	0.1224 (9)	0.7465 (12)	0.1450 (12)	0.107 (5)
CA14	0.3058 (9)	0.8439 (10)	0.3748 (11)	0.046 (3)
CA24	0.3898 (9)	0.8932 (10)	0.4627 (10)	0.041 (3)
OA24	0.4569 (7)	0.8966 (8)	0.4369 (8)	0.049 (2)
CA34	0.3992 (9)	0.8470 (11)	0.5411 (11)	0.044 (3)
OA34	0.4762 (7)	0.8938 (8)	0.6298 (8)	0.056 (3)
CA44	0.3262 (9)	0.8452 (10)	0.5664 (11)	0.043 (3)
OA44	0.3330 (7)	0.7889 (8)	0.6303 (8)	0.050 (2)
CA54	0.2427 (9)	0.8034 (11)	0.4739 (11)	0.048 (3)
OA54	0.2392 (7)	0.8459 (8)	0.3996 (8)	0.050 (2)
CA64	0.1716 (11)	0.8181 (12)	0.4952 (14)	0.063 (4)
OA64	0.1789 (9)	0.9143 (10)	0.5355 (11)	0.086 (4)
CA15	0.3297 (10)	0.8261 (11)	0.7213 (12)	0.051 (3)
CA25	0.4080 (10)	0.8208 (11)	0.8048 (12)	0.054 (4)
OA25	0.4823 (7)	0.8720 (8)	0.8064 (9)	0.060 (3)
CA35	0.4065 (9)	0.7212 (10)	0.7924 (11)	0.047 (3)
OA35	0.4780 (7)	0.7147 (8)	0.8747 (9)	0.059 (3)
CA45	0.3268 (9)	0.6700 (10)	0.7897 (11)	0.040 (3)
OA45	0.3238 (7)	0.5723 (8)	0.7690 (8)	0.047 (2)
CA55	0.2511 (10)	0.6779 (12)	0.7085 (13)	0.057 (4)
OA55	0.2576 (8)	0.7766 (8)	0.7214 (9)	0.061 (3)
CA65	0.1708 (14)	0.6375 (15)	0.7080 (17)	0.087 (6)
O65A*	0.178 (2)	0.673 (2)	0.804 (4)	0.18 (2)
O65B*	0.0987 (14)	0.648 (2)	0.628 (3)	0.094 (16)
CA16	0.3161 (9)	0.5286 (10)	0.8376 (11)	0.040 (3)
CA26	0.3862 (9)	0.4822 (10)	0.8677 (11)	0.043 (3)
OA26	0.4652 (7)	0.5495 (8)	0.9162 (8)	0.052 (2)
CA36	0.3765 (9)	0.4102 (10)	0.7776 (12)	0.048 (3)
OA36	0.4408 (7)	0.3643 (8)	0.8046 (10)	0.064 (3)
CA46	0.2920 (9)	0.3409 (10)	0.7324 (11)	0.046 (3)
OA46	0.2813 (7)	0.2789 (8)	0.6386 (8)	0.049 (2)
CA56	0.2220 (9)	0.3882 (10)	0.7064 (11)	0.041 (3)
OA56	0.2370 (7)	0.4620 (8)	0.7948 (8)	0.052 (2)
CA66	0.1371 (10)	0.3236 (12)	0.6744 (13)	0.060 (4)
OA66	0.1350 (8)	0.2798 (9)	0.7456 (11)	0.077 (3)
CA17	0.2602 (10)	0.1823 (11)	0.6249 (12)	0.053 (4)
CA27	0.3262 (10)	0.1437 (11)	0.6068 (11)	0.049 (4)
OA27	0.4054 (8)	0.1887 (8)	0.6872 (9)	0.065 (3)
CA37	0.3242 (10)	0.1534 (11)	0.5116 (12)	0.051 (4)
OA37	0.3859 (7)	0.1123 (8)	0.4906 (9)	0.058 (3)
CA47	0.2394 (10)	0.0998 (11)	0.4277 (12)	0.050 (3)
OA47	0.2381 (7)	0.1143 (8)	0.3379 (8)	0.051 (2)
CA57	0.1730 (10)	0.1367 (12)	0.4497 (12)	0.060 (4)
OA57	0.1817 (7)	0.1355 (8)	0.5450 (9)	0.060 (3)
CA67	0.0852 (11)	0.0802 (13)	0.3798 (14)	0.071 (5)
OA67	0.0713 (9)	−0.0144 (10)	0.3853 (11)	0.099 (4)
CB11	0.6831 (9)	0.1190 (10)	0.5057 (11)	0.039 (3)
CB21	0.6110 (9)	0.1011 (10)	0.5338 (11)	0.040 (3)
OB21	0.5317 (7)	0.0714 (8)	0.4476 (9)	0.056 (2)
CB31	0.6189 (9)	0.1879 (10)	0.6089 (11)	0.043 (3)
OB31	0.5538 (7)	0.1721 (9)	0.6379 (9)	0.063 (3)
CB41	0.7033 (9)	0.2131 (10)	0.6971 (11)	0.042 (3)
OB41	0.7138 (7)	0.3033 (8)	0.7637 (8)	0.047 (2)
CB51	0.7731 (9)	0.2246 (10)	0.6647 (11)	0.043 (3)
OB51	0.7588 (7)	0.1425 (8)	0.5881 (8)	0.049 (2)
CB61	0.8596 (10)	0.2386 (11)	0.7497 (12)	0.057 (4)
OB61	0.8600 (8)	0.1691 (9)	0.7950 (9)	0.068 (3)
CB12	0.7329 (9)	0.3111 (10)	0.8628 (11)	0.043 (3)
CB22	0.6676 (9)	0.3441 (10)	0.8880 (11)	0.045 (3)
OB22	0.5874 (7)	0.2782 (8)	0.8245 (9)	0.061 (3)
CB32	0.6678 (9)	0.4381 (10)	0.8782 (11)	0.045 (3)
OB32	0.6083 (7)	0.4723 (8)	0.9071 (9)	0.054 (2)
CB42	0.7548 (9)	0.5032 (10)	0.9487 (11)	0.043 (3)
OB42	0.7557 (7)	0.5913 (8)	0.9343 (8)	0.046 (2)
CB52	0.8196 (9)	0.4669 (10)	0.9262 (12)	0.048 (3)
OB52	0.8139 (7)	0.3747 (8)	0.9284 (9)	0.053 (2)
CB62	0.9103 (11)	0.5217 (12)	1.0074 (13)	0.061 (4)
OB62	0.9229 (8)	0.5181 (9)	1.0995 (10)	0.082 (3)

C*B*13	0.7852 (9)	0.6725 (10)	1.0192 (12)	0.048 (3)
C*B*23	0.7164 (9)	0.7200 (11)	1.0050 (12)	0.046 (3)
O*B*23	0.6445 (7)	0.6625 (8)	0.9955 (9)	0.057 (3)
C*B*33	0.7033 (9)	0.7561 (10)	0.9184 (11)	0.046 (3)
O*B*33	0.6429 (7)	0.8078 (9)	0.9082 (10)	0.066 (3)
C*B*43	0.7837 (10)	0.8181 (11)	0.9359 (12)	0.048 (3)
O*B*43	0.7701 (7)	0.8407 (8)	0.8447 (8)	0.049 (2)
C*B*53	0.8514 (10)	0.7661 (11)	0.9527 (12)	0.050 (3)
O*B*53	0.8598 (7)	0.7308 (8)	1.0329 (8)	0.053 (2)
C*B*63	0.9366 (12)	0.8224 (14)	0.9782 (15)	0.077 (5)
O*B*63	0.9586 (10)	0.8987 (12)	1.0630 (15)	0.127 (6)
C*B*14	0.7922 (9)	0.9370 (10)	0.8546 (11)	0.044 (3)
C*B*24	0.7151 (9)	0.9523 (11)	0.7790 (11)	0.045 (3)
O*B*24	0.6462 (7)	0.9355 (9)	0.8049 (9)	0.061 (3)
C*B*34	0.6935 (9)	0.8955 (11)	0.6777 (11)	0.045 (3)
O*B*34	0.6220 (7)	0.9134 (8)	0.6060 (9)	0.055 (2)
C*B*44	0.7674 (9)	0.9185 (11)	0.6554 (11)	0.043 (3)
O*B*44	0.7454 (7)	0.8529 (8)	0.5595 (8)	0.052 (2)
C*B*54	0.8449 (9)	0.9088 (11)	0.7336 (11)	0.045 (3)
O*B*54	0.8597 (7)	0.9585 (8)	0.8306 (8)	0.051 (2)
C*B*64	0.9235 (10)	0.9478 (12)	0.7255 (13)	0.059 (4)
O*B*64	0.9318 (9)	1.0400 (9)	0.7265 (12)	0.090 (4)
C*B*15	0.7567 (10)	0.8853 (11)	0.4876 (12)	0.052 (4)
C*B*25	0.6752 (10)	0.8542 (11)	0.3949 (12)	0.049 (4)
O*B*25	0.6131 (7)	0.8873 (8)	0.4144 (9)	0.054 (2)
C*B*35	0.6504 (10)	0.7508 (11)	0.3486 (12)	0.050 (3)
O*B*35	0.5750 (7)	0.7209 (8)	0.2574 (9)	0.058 (3)
C*B*45	0.7212 (9)	0.7246 (10)	0.3295 (11)	0.045 (3)
O*B*45	0.6965 (7)	0.6233 (8)	0.2904 (8)	0.048 (2)
C*B*55	0.8004 (10)	0.7576 (12)	0.4256 (13)	0.060 (4)
O*B*55	0.8205 (8)	0.8573 (8)	0.4667 (9)	0.063 (3)
C*B*65	0.8744 (13)	0.7364 (15)	0.4131 (16)	0.085 (5)
O*B*65	0.8851 (12)	0.7706 (13)	0.3403 (17)	0.139 (7)
C*B*16	0.6963 (9)	0.5827 (10)	0.1976 (11)	0.045 (3)
C*B*26	0.6140 (9)	0.5135 (10)	0.1306 (11)	0.047 (3)
O*B*26	0.5463 (7)	0.5556 (8)	0.1150 (9)	0.055 (2)
C*B*36	0.6035 (10)	0.4377 (11)	0.1769 (12)	0.050 (3)
O*B*36	0.5269 (7)	0.3664 (8)	0.1116 (10)	0.063 (3)
C*B*46	0.6745 (9)	0.3964 (10)	0.1917 (11)	0.040 (3)
O*B*46	0.6682 (7)	0.3327 (8)	0.2468 (8)	0.050 (2)
C*B*56	0.7592 (9)	0.4688 (10)	0.2518 (12)	0.049 (3)
O*B*56	0.7620 (7)	0.5431 (8)	0.2088 (9)	0.052 (2)
C*B*66	0.8317 (11)	0.4333 (13)	0.2533 (13)	0.066 (4)
O*B*66	0.8220 (10)	0.3923 (10)	0.1560 (13)	0.094 (4)
C*B*17	0.6714 (10)	0.2414 (11)	0.2085 (12)	0.056 (4)
C*B*27	0.5938 (10)	0.1735 (12)	0.1941 (12)	0.058 (4)
O*B*27	0.5208 (8)	0.1873 (9)	0.1264 (9)	0.068 (3)
C*B*37	0.5927 (9)	0.1837 (10)	0.2936 (11)	0.046 (3)
O*B*37	0.5210 (7)	0.1153 (8)	0.2800 (9)	0.065 (3)
C*B*47	0.6742 (10)	0.1701 (11)	0.3651 (12)	0.050 (3)
O*B*47	0.6746 (7)	0.1878 (7)	0.4598 (8)	0.048 (2)
C*B*57	0.7483 (11)	0.2364 (13)	0.3719 (13)	0.063 (4)
O*B*57	0.7436 (8)	0.2249 (9)	0.2736 (10)	0.068 (3)
C*B*67	0.8318 (14)	0.2152 (16)	0.4310 (17)	0.090 (6)
O67*A**	0.8299 (15)	0.127 (2)	0.3906 (19)	0.140 (11)
O67*B**	0.895 (4)	0.260 (4)	0.424 (4)	0.08 (2)
O161	0.094 (4)	0.890 (4)	0.905 (4)	0.30 (3)
O162	0.035 (4)	0.535 (3)	0.830 (3)	0.33 (3)
O163	0.109 (4)	0.730 (4)	0.963 (3)	0.37 (4)
O164	0.1835 (15)	−0.0067 (14)	0.7190 (16)	0.150 (7)
O16*A*	0.270 (3)	0.833 (5)	−0.028 (4)	0.35 (3)
O16*B*	1.074 (4)	0.816 (3)	0.692 (5)	0.42 (4)
O166	0.0635 (10)	0.0925 (10)	0.6854 (15)	0.112 (5)
O167	0.0367 (9)	0.8433 (10)	0.2249 (11)	0.095 (4)
O121	0.401 (3)	0.9059 (18)	0.185 (3)	0.248 (17)
O122	0.342 (2)	0.095 (2)	0.850 (2)	0.240 (14)
O123	0.469 (2)	0.7598 (18)	0.0578 (17)	0.212 (13)
O125	0.576 (4)	0.978 (3)	0.008 (3)	0.46 (4)
O127	0.4801 (16)	0.0596 (12)	0.7515 (15)	0.174 (10)
O261	0.8981 (12)	0.1942 (14)	−0.0106 (13)	0.123 (6)
O262	−0.001 (2)	0.368 (2)	0.129 (3)	0.221 (13)
O263	0.889 (4)	−0.004 (5)	0.170 (5)	0.10 (2)
O265	0.9679 (12)	0.6814 (15)	0.2518 (15)	0.110 (5)
O266	0.7904 (16)	0.2054 (17)	0.0694 (18)	0.142 (7)
O26*A*	−0.042 (3)	0.021 (4)	0.482 (3)	0.32 (4)
O26*B*	−0.113 (3)	0.150 (4)	0.236 (3)	0.29 (2)
O222	0.5069 (18)	0.2424 (17)	0.9329 (16)	0.168 (12)
O223	0.602 (4)	0.781 (3)	0.109 (4)	0.25 (2)
O224	0.643 (2)	0.104 (2)	0.918 (3)	0.225 (13)
O225	0.544 (2)	0.932 (2)	0.244 (2)	0.155 (9)
O227	0.395 (4)	0.009 (5)	−0.002 (5)	0.34 (3)
O1	0.847 (12)	−0.104 (16)	0.211 (17)	0.31 (11)
O2	0.248 (7)	−0.041 (8)	0.895 (8)	0.25 (4)
C*A*1	0.096 (2)	0.400 (3)	0.313 (3)	0.66 (4)
C*A*2	0.186 (2)	0.403 (3)	0.373 (3)	0.43 (4)
C*A*3	0.243 (2)	0.499 (3)	0.430 (3)	0.40 (4)
C*A*4	0.282 (2)	0.496 (3)	0.352 (3)	0.41 (4)
C*A*5	0.322 (2)	0.414 (2)	0.365 (3)	0.47 (4)
C*A*6	0.405 (2)	0.446 (2)	0.362 (2)	0.55 (4)
C*A*7	0.445 (2)	0.367 (2)	0.357 (3)	0.49 (4)
C*A*8	0.537 (2)	0.434 (2)	0.391 (2)	0.47 (4)
C*A*9	0.602 (2)	0.425 (4)	0.487 (2)	0.47 (4)
O*A*1	0.091 (3)	0.486 (4)	0.327 (5)	0.52 (3)
O*A*2	0.029 (3)	0.322 (3)	0.282 (4)	0.45 (3)
C*B*1	1.011 (2)	0.493 (3)	0.496 (3)	0.43 (4)
C*B*2	0.944 (2)	0.509 (2)	0.535 (2)	0.39 (4)
C*B*3	0.937 (3)	0.484 (3)	0.626 (3)	0.53 (4)
C*B*4	0.866 (3)	0.523 (3)	0.638 (3)	0.53 (4)
C*B*5	0.789 (2)	0.513 (2)	0.552 (3)	0.51 (4)
C*B*6	0.769 (2)	0.610 (2)	0.594 (3)	0.40 (4)
C*B*7	0.724 (3)	0.608 (3)	0.668 (3)	0.53 (4)
C*B*8	0.636 (3)	0.635 (3)	0.615 (4)	0.59 (4)
C*B*9	0.556 (3)	0.611 (4)	0.627 (4)	0.44 (4)
O*B*1	1.056 (3)	0.423 (3)	0.496 (4)	0.50 (3)
O*B*2	1.037 (3)	0.550 (4)	0.464 (4)	0.55 (3)

* β-CD O atoms with partial occupancy.

Table 2. *Hydrogen-bonding contact distances of the carboxylic groups of guest molecules (Å)*

O*A*1^{i}···O*B*2	2.72
O*A*2^{i}···O*B*1	3.11
O*A*1···O262	2.83
O*A*2···O262	2.51
O*A*2···O26*B*	3.11
O*B*1···O67*B*	3.14
O*B*2···O65*B*	2.34

Symmetry code: (i) $1 + x, y, z$.

The crystal was sealed in a glass capillary to prevent water loss. The relatively poor quality of the crystal, which is very common in cyclodextrin complexes, reflects our inability to collect data over 21° in θ.

The structure was determined by using the coordinates of the atoms of β-CD molecules (apart from those of primary C and O atoms) of the isomorphous structure of β-CD with 4-*tert*-butylbenzoic acid (Rontoyianni, Mavridis, Hadjoudis & Duisenberg, 1994). Subsequent $\Delta\rho$ maps revealed the missing atoms of β-CD, water and guest molecules. H atoms were calculated for all C atoms of β-CD molecules using a riding model with U_{eq}(H) equal to $1.2U_{eq}$ of the parent C atom. Anisotropic displacement parameters were assigned to all O2, O3 and O6 and to the water O atoms which behaved well during the refinement.

The geometry of the guest molecules was optimized by fitting the initial model into the difference density map by using the graphics program *O* (Jones & Kjeldgaad, 1993) on a Silicon Graphics workstation and was not refined further.

Data collection: *CRYSTAL LOGIC* (Unpublished). Cell refinement: *CRYSTAL LOGIC*. Data reduction: *CRYSTAL LOGIC*. Program(s) used to solve structure: isomorphic method (Rontoyianni *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *O* (Jones & Kjeldgaad, 1993). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1218). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-(4-Chlorophenyl)-3a-methyl-5-phenyl-3a,4-dihydro-1,2,4-oxadiazolo[4,5-*a*][1,5]-benzodiazepine

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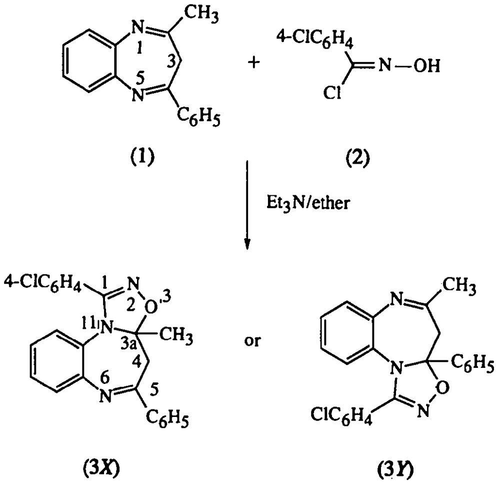
Abstract

We report here the peri- and regioselectivity experienced in the 1,3-dipolar cycloaddition of 1,5-benzodiazepine to a nitrile oxide. The crystal structure of the title cycloadduct, $C_{23}H_{18}ClN_3O$, shows that the condensation occurs at the N=C double bond at the 1 and 2 positions of the benzodiazepine.

Comment

Several benzodiazepine derivatives containing additional rings are of pharmacological interest (Sternbach, 1978). In connection with investigations on possible approaches to novel benzodiazepine derivatives with an additional fused heterocyclic ring (Aversa, Giannetto, Ferlazzo & Romeo, 1982), we have tested the C=N double bond of the 1,5-benzodiazepine system as a dipolarophile in the 1,3-dipolar cycloadditions of nitrile oxides.

The condensation of 2-methyl-4-phenyl-1,5-benzodiazepine [(1); Barltrop, Richards, Russel & Ryback, 1959] with a slight excess of 4-chlorobenzonitrile oxide, generated *in situ* from 4-chlorobenzohydroxamoyl chloride [(2); Grundmann & Dean, 1965; Liu, Shelton & Howe, 1980), gives the title 1,2,4-oxadiazolo[4,5-*a*][1,5]-benzodiazepine, (3) (*X* or *Y*), as indicated below.



The structure of cycloadduct (3) was assigned by X-ray crystallographic analysis which shows it to be adduct (3*X*), *i.e.* the N=C double bond at the 1 and 2 positions of the benzodiazepine is the site of addition (Fig. 1). The reaction is periselective as only one C=N double bond is affected. The O atom of the dipole is linked to the C atom of the C=N dipolarophile making the reaction regioselective.

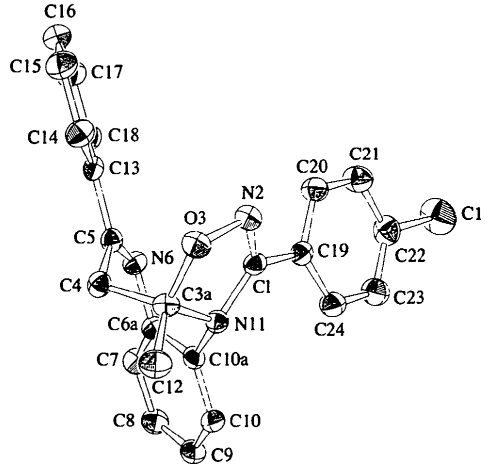


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule with displacement ellipsoids at the 50% probability level and the atomic numbering scheme. H atoms have been omitted for clarity.