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Structure of (10*RS*,5*RS*,9*SR*)-10β-Ethyl-5,6,7,8,9,10-hexahydro-5α,9α-methanobenzocyclooctene-10α-carboxamide, C₁₆H₂₁NO

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Abstract

The title compound crystallizes in $P2_1/c$ with $a = 6.314$ (2), $b = 29.815$ (5), $c = 15.158$ (3) Å, $\beta = 109.55$ (2)°, $Z = 8$. The structure was refined to an R of 0.055 for 1935 observed reflexions $[I > 2\sigma(I)]$. The conformations of both symmetrically independent molecules are the same: the cyclohexene ring exhibits a half-chair and the cyclohexane moiety a chair conformation. The interatomic distances and angles are in agreement with the given atom type and hybridization. The symmetrically independent molecules are hydrogen bonded by N—H···O contacts of 2.870 and 2.857 Å between carboxamide groups, forming dimers.

Introduction

α,α-Disubstituted-1-benzylcyclohexenes as well as their intramolecular cycloalkylation products were synthesized (Mutak, 1979). The main products, 10,10-disubstituted ethyl-5,6,7,8,9,10-hexahydro-5,9-methanocyclooctenes, contain a basic skeleton similar to that of benzomorphan, typical of strong analgesics. Some derivatives of the title compound, *e.g.* 10α-, 10β-amines, acids and lactones, possess marked analgesic properties; amines also show antihistaminic, antiserotoninic and anticholinergic properties (Mutak, 1979).

The crystal structure investigation of these compounds has been undertaken to establish their configuration and conformation and also to correlate their biological activities with some structural parameters.

Experimental

The space group was determined from Weissenberg photographs recorded with Cu Kα radiation. Table 1 lists crystallographic and physical data.

Table 1. *Crystallographic and physical data*

Numbers in parentheses here and throughout this paper are the e.s.d.'s in the least significant digits.

C₁₆H₂₁NO

M_r	243.35	U	2689.02 Å³
Space group	$P2_1/c$	Z	8
a	6.314 (2) Å	D_c	1.202 Mg m⁻³
b	29.815 (5)	μ(Mo Kα)	0.168 mm⁻¹
c	15.158 (3)	Crystal shape	Prismatic
β	109.55 (2)°		

The intensities were collected on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation. 6441 independent reflexions were recorded in the ω–2θ mode in the range $3 < 2\theta < 56°$ but only 1935 of these had $I > 2\sigma(I)$. The data were corrected for background, Lorentz and polarization effects but not for absorption.

Structure determination and refinement

Overall temperature ($B = 3.73$ Å^2) and scale factors were determined (Wilson, 1942) and used to compute normalized structure amplitudes by the *NORMAL* routine included in *MULTAN* 77. In these calculations all reflexions (6441) were used. The structure was solved with *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The solution was based on 320 reflexions with $|E| > 2.06$. The E map corresponding to the solution with the best figure of merit (ABS FOM = 1.013, PSI ZERO = 249.5, RESID = 17.89) revealed 32 non-hydrogen atoms out of 36 in

both molecules. The remaining four C atoms belonging to the ethyl groups were located from the resulting Fourier synthesis. Refinement was by full-matrix least squares minimizing $\sum w\|F_o| - |F_c\|^2$. A weighting scheme of type 3 from XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) was used. The weights were assigned as: $w = w_1 w_2$, where $w_1 = 1$ for $F_o \leq 30$ and $w_1 = 30/|F_o|$ for $|F_o| > 30$; $w_2 = 1$ for $\sin\theta \geq 0.4$ and $w_2 = (\sin\theta)/0.4$ for $\sin\theta < 0.4$. Anisotropic refinement and a subsequent weighted difference synthesis located the H atoms. A scale factor, heavy-atom coordinates and anisotropic thermal parameters (325 variables in all) were refined. The H atoms were included in the structure factor calculation only. For H atoms the isotropic thermal parameters were those of the bonded atoms. Anisotropic thermal parameters (of the non-hydrogen atoms) are in the usual range: maximum values of U_{33} for C(2) and C(3) of 0.139 (6) and 0.145 (6) Å^2 were obtained, respectively. The final $R = 0.055$ and $R_w = 0.059$ for 1935 reflexions having $I > 2\sigma(I)$.

Scattering factors given by Cromer & Mann (1968) and (for H) Stewart, Davidson & Simpson (1965) were used.

The calculations were carried out on a Univac 1110 computer at the University Computing Centre in Zagreb with XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Coordinates are listed in Tables 2 and 3.*

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34844 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final atomic coordinates for non-hydrogen atoms* ($\times 10^4$)

Atoms marked by a prime correspond to molecule *B*.

	x	y	z
C(1)	3246 (7)	3473 (2)	2681 (4)
C(2)	2021 (9)	3107 (2)	2257 (6)
C(3)	2015 (10)	2978 (2)	1385 (7)
C(4)	3223 (10)	3221 (2)	940 (4)
C(5)	5684 (8)	3857 (2)	834 (3)
C(6)	3975 (9)	4171 (2)	140 (3)
C(7)	3024 (8)	4498 (2)	677 (3)
C(8)	4842 (8)	4735 (2)	1445 (3)
C(9)	6641 (7)	4424 (1)	2082 (3)
C(10)	5891 (6)	4126 (1)	2754 (3)
C(11)	7583 (8)	4132 (2)	1484 (3)
C(12)	4505 (6)	3726 (1)	2247 (3)
C(13)	4477 (7)	3594 (1)	1361 (3)
C(14)	8089 (7)	3960 (1)	3494 (3)
C(15)	4565 (8)	4396 (2)	3278 (3)
C(16)	5592 (11)	4825 (2)	3759 (4)
C(1')	8598 (7)	919 (2)	1535 (3)
C(2')	9682 (8)	549 (2)	2048 (4)
C(3')	9505 (9)	461 (2)	2907 (4)
C(4')	8259 (8)	740 (2)	3260 (3)
C(5')	5966 (8)	1422 (2)	3233 (3)
C(6')	7699 (8)	1734 (2)	3912 (3)
C(7')	8903 (8)	2013 (2)	3384 (3)
C(8')	7231 (8)	2254 (2)	2549 (3)
C(9')	5368 (7)	1952 (1)	1924 (3)
C(10')	6064 (6)	1607 (1)	1311 (3)
C(11')	4234 (7)	1706 (2)	2516 (3)
C(12')	7316 (6)	1208 (1)	1888 (3)
C(13')	7151 (7)	1115 (1)	2763 (3)
C(14')	3844 (7)	1450 (1)	566 (3)
C(15')	7532 (8)	1819 (2)	778 (3)
C(16')	6651 (9)	2238 (2)	204 (4)
N	8400 (6)	3523 (1)	3658 (3)
N'	3503 (6)	1014 (1)	412 (3)
O	9526 (6)	4229 (1)	3915 (3)
O'	2474 (5)	1726 (1)	114 (2)

Table 3. *Positional parameters* ($\times 10^3$) *for the hydrogen atoms*

	x	y	z		x	y	z
H(1)	300	366	330	H(1')	900	106	92
H(2)	125	287	258	H(2')	1050	33	171
H(3)	100	267	118	H(3')	1010	18	330
H(4)	300	320	18	H(4')	850	67	395
H(5)	640	371	37	H(5')	500	122	358
H(6)1	500	439	10	H(6')1	680	190	428
H(6)2	275	394	−40	H(6')2	900	153	442
H(7)1	210	472	26	H(7')1	975	225	389
H(7)2	200	430	100	H(7')2	1000	183	313
H(8)1	410	490	185	H(8')1	810	244	219
H(8)2	550	500	118	H(8')2	640	248	289
H(9)	770	467	250	H(9')	425	211	145
H(11)1	825	433	118	H(11')1	285	156	205
H(11)2	833	394	198	H(11')2	350	189	284
H(15)1	300	442	276	H(15')1	775	161	34
H(15)2	420	417	376	H(15')2	920	189	132
H(16)1	700	478	432	H(16')1	650	251	66
H(16)2	500	500	421	H(16')2	500	217	−26
H(16)3	525	511	329	H(16')3	775	238	−20
H(N)1	1000	344	416	H(N')1	215	92	0
H(N)2	725	333	329	H(N')2	450	83	66

Table 4. *Bond distances* (Å) *and angles* (°)

	Molecule A	Molecule B		Molecule A	Molecule B		Molecule A	Molecule B
C(1)—C(2)	1·368 (7)	1·392 (7)	C(6)—H(6)1	0·93	1·04	C(11)—H(11)1	0·93	1·02
C(1)—C(12)	1·408 (7)	1·405 (7)	C(6)—H(6)2	1·15	1·10	C(11)—H(11)2	0·94	0·95
C(1)—H(1)	1·15	1·13	C(7)—C(8)	1·508 (6)	1·529 (6)	C(12)—C(13)	1·393 (7)	1·392 (6)
C(2)—C(3)	1·375 (14)	1·368 (9)	C(7)—H(7)1	0·96	1·05	C(14)—O	1·220 (5)	1·224 (5)
C(2)—H(2)	1·06	1·06	C(7)—H(7)2	1·10	1·05	C(14)—N	1·329 (6)	1·325 (6)
C(3)—C(4)	1·382 (11)	1·372 (8)	C(8)—C(9)	1·531 (6)	1·532 (6)	C(15)—C(16)	1·507 (8)	1·518 (7)
C(3)—H(3)	1·10	1·02	C(8)—H(8)1	1·01	1·05	C(15)—H(15)1	1·04	0·95
C(4)—C(13)	1·390 (7)	1·398 (6)	C(8)—H(8)2	1·04	1·08	C(15)—H(15)2	1·07	1·12
C(4)—H(4)	1·11	1·03	C(9)—C(10)	1·542 (6)	1·545 (6)	C(16)—H(16)1	1·01	1·09
C(5)—C(6)	1·545 (7)	1·539 (6)	C(9)—C(11)	1·515 (7)	1·512 (7)	C(15)—H(16)2	1·03	1·06
C(5)—C(11)	1·513 (6)	1·516 (6)	C(9)—H(9)	1·05	0·95	C(16)—H(16)3	1·08	1·15
C(5)—C(13)	1·497 (7)	1·504 (7)	C(10)—C(12)	1·526 (5)	1·532 (5)	N—H(N)1	1·07	0·92
C(5)—H(5)	1·05	1·11	C(10)—C(14)	1·546 (5)	1·549 (5)	N—H(N)2	0·95	0·83
C(6)—C(7)	1·517 (8)	1·522 (8)	C(10)—C(15)	1·558 (7)	1·553 (7)			
C(2)—C(1)—C(12)	121·7 (6)	120·8 (5)	C(6)—C(7)—C(8)	112·3 (4)	111·3 (4)	C(5)—C(11)—H(11)1	114	120
H(1)—C(1)—C(2)	124	124	C(6)—C(7)—H(7)1	111	102	C(5)—C(11)—H(11)2	106	108
H(1)—C(1)—C(12)	112	114	C(6)—C(7)—H(7)2	107	115	C(9)—C(11)—H(11)1	105	105
C(1)—C(2)—C(3)	119·6 (7)	120·2 (5)	C(8)—C(7)—H(7)1	109	109	C(9)—C(11)—H(11)2	94	116
C(1)—C(2)—H(2)	126	116	C(8)—C(7)—H(7)2	108	107	C(1)—C(12)—C(10)	120·0 (4)	120·8 (4)
H(2)—C(2)—C(3)	114	123	C(7)—C(8)—C(9)	114·6 (4)	114·2 (4)	C(1)—C(12)—C(13)	118·2 (4)	118·5 (4)
C(2)—C(3)—C(4)	119·9 (6)	119·6 (5)	C(7)—C(8)—H(8)1	108	110	C(10)—C(12)—C(13)	121·7 (4)	120·6 (4)
C(2)—C(3)—H(3)	109	126	C(7)—C(8)—H(8)2	111	102	C(4)—C(13)—C(5)	119·2 (5)	118·0 (4)
H(3)—C(3)—C(4)	132	115	H(8)1—C(8)—C(9)	108	115	C(4)—C(13)—C(12)	119·3 (5)	119·3 (4)
C(3)—C(4)—C(13)	121·2 (6)	121·6 (5)	H(8)2—C(8)—C(9)	113	105	C(5)—C(13)—C(12)	121·4 (4)	122·5 (4)
C(3)—C(4)—H(4)	125	111	C(8)—C(9)—C(10)	116·0 (4)	116·7 (4)	O—C(14)—C(10)	120·0 (4)	120·1 (4)
H(4)—C(4)—C(13)	112	126	C(8)—C(9)—C(11)	109·1 (4)	109·7 (4)	O—C(14)—N	120·7 (3)	121·1 (3)
C(6)—C(5)—C(11)	109·7 (4)	108·9 (4)	C(8)—C(9)—H(9)	98	114	C(10)—C(14)—N	119·4 (3)	118·7 (3)
C(6)—C(5)—C(13)	108·4 (4)	109·2 (4)	C(10)—C(9)—C(11)	109·4 (4)	108·7 (4)	H(N)1—N—C(14)	114	119
C(11)—C(5)—C(13)	111·7 (4)	110·7 (4)	H(9)—C(9)—C(10)	106	99	H(N)2—N—C(14)	117	121
H(5)—C(5)—C(6)	99	113	H(9)—C(9)—C(11)	118	108	C(10)—C(15)—C(16)	117·9 (5)	117·7 (4)
H(5)—C(5)—C(11)	104	105	C(9)—C(10)—C(12)	111·7 (3)	111·7 (3)	C(10)—C(15)—H(15)1	101	110
H(5)—C(5)—C(13)	123	110	C(9)—C(10)—C(15)	112·0 (3)	112·5 (3)	C(10)—C(15)—H(15)2	107	106
C(5)—C(6)—C(7)	109·6 (4)	110·3 (4)	C(9)—C(10)—C(14)	105·3 (3)	105·7 (3)	H(15)1—C(15)—C(16)	117	105
C(5)—C(6)—H(6)1	97	105	C(12)—C(10)—C(14)	109·8 (3)	110·4 (3)	H(15)2—C(15)—C(16)	111	110
C(5)—C(6)—H(6)2	106	109	C(12)—C(10)—C(15)	110·2 (4)	109·2 (3)	C(15)—C(16)—H(16)1	114	110
H(6)1—C(6)—C(7)	90	119	C(14)—C(10)—C(15)	107·5 (3)	107·2 (3)	C(15)—C(16)—H(16)2	124	108
H(6)2—C(6)—C(7)	118	106	C(5)—C(11)—C(9)	108·4 (4)	108·9 (4)	C(15)—C(16)—H(16)3	113	116
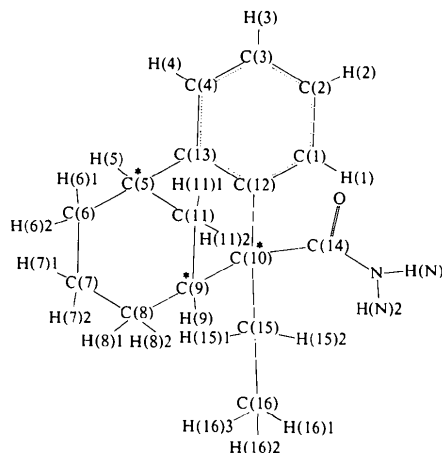
Fig. 1. Structural formula and atom numbering; the chiral atoms are marked by an asterisk.

Table 5. *Displacements from least-squares planes* (Å)

Atoms included in the calculation of the planes are denoted by an asterisk.

		Molecule A	Molecule B
Plane I	C(5)*	0·009 (7)	0·014 (7)
	C(10)*	−0·008 (6)	−0·013 (6)
	C(12)*	0·018 (6)	0·029 (6)
	C(13)*	−0·018 (6)	−0·029 (6)
	C(9)	0·313 (6)	0·346 (6)
	C(11)	−0·496 (7)	−0·467 (6)
Plane II	C(5)*	−0·026 (9)	−0·032 (9)
	C(6)*	0·026 (10)	0·031 (9)
	C(8)*	−0·026 (9)	−0·031 (9)
	C(9)*	0·027 (8)	0·032 (8)
	C(7)	−0·599 (9)	−0·615 (10)
	C(11)	0·742 (9)	0·737 (8)

Description and discussion of the structure

The structural formula with the atom numbering is given in Fig. 1. Bond distances and angles are listed in Table 4. The conformation of the molecules is shown in

Figs. 2, 3 and Tables 5, 6. The molecular packing is illustrated in Fig. 4. The C—C lengths between $C(sp^3)$ atoms range from 1·507 (8) to 1·558 (7) Å for molecule A and from 1·516 (6) to 1·553 (7) Å for B. The C(5)—C(13) and C(10)—C(12) bonds are equivalent in their hybridization type $[C(sp^3)—C(sp^2)]$, but
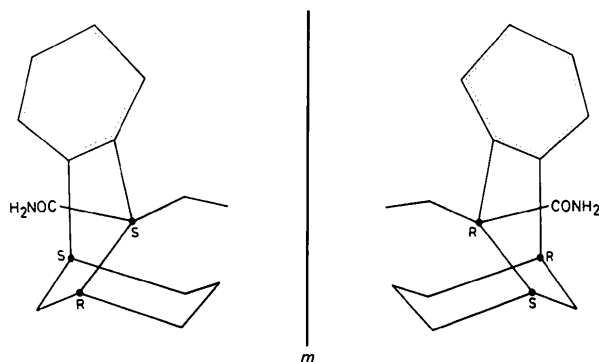
Fig. 2. Schematic drawing of the molecules showing their conformation; both enantiomers appear in the crystal as a consequence of the symmetry operations of the $P2_1/c$ space group.
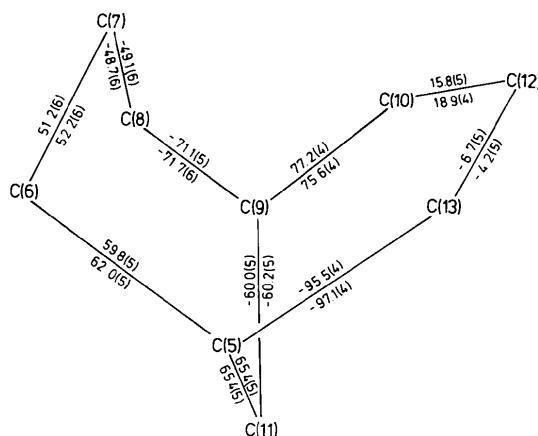

Fig. 3. The conformation of the molecules illustrated as the bridged cyclooctene ring; numerical values of the torsion angles (°) marked outside the ring correspond to molecule A and those inside the ring to molecule B.

Table 6. *Torsion angles* (°)

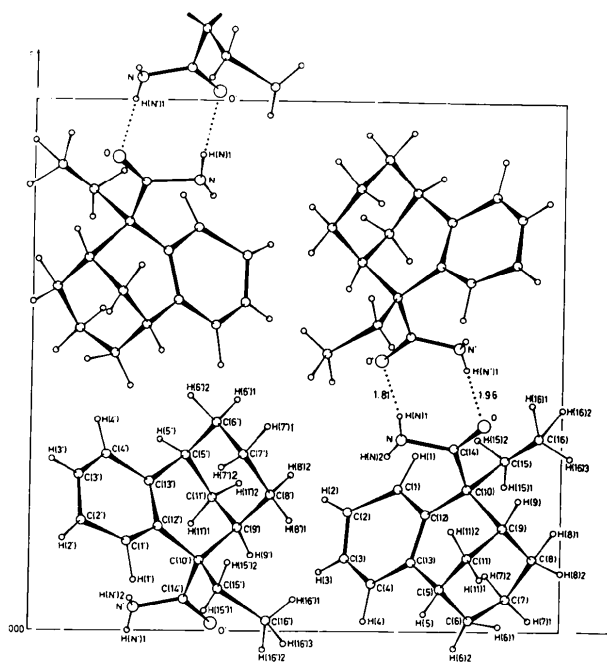|	Molecule A	Molecule B
C(12)—C(13)—C(5)—C(6)	−97·1 (4)	−95·5 (4)
C(12)—C(13)—C(5)—C(11)	23·8 (6)	24·3 (6)
C(11)—C(5)—C(6)—C(7)	−60·2 (6)	−61·3 (6)
C(13)—C(5)—C(6)—C(7)	62·0 (5)	59·8 (5)
C(5)—C(6)—C(7)—C(8)	51·2 (6)	52·2 (6)
C(6)—C(7)—C(8)—C(9)	−49·1 (6)	−48·7 (6)
C(7)—C(8)—C(9)—C(10)	−71·1 (5)	−71·7 (5)
C(7)—C(8)—C(9)—C(11)	53·0 (6)	52·4 (6)
C(8)—C(9)—C(10)—C(12)	77·2 (4)	75·6 (4)
C(11)—C(9)—C(10)—C(12)	−46·8 (4)	−48·9 (4)
C(8)—C(9)—C(10)—C(14)	−163·6 (3)	−164·2 (4)
C(8)—C(9)—C(10)—C(15)	−47·0 (4)	−47·6 (4)
C(11)—C(9)—C(10)—C(14)	72·4 (4)	71·3 (4)
C(11)—C(9)—C(10)—C(15)	−171·0 (3)	−172·2 (3)
C(9)—C(10)—C(12)—C(13)	15·8 (5)	18·9 (4)
C(14)—C(10)—C(12)—C(13)	−100·7 (4)	−98·4 (4)
C(15)—C(10)—C(12)—C(13)	141·0 (4)	143·9 (4)
C(9)—C(10)—C(14)—O	52·8 (6)	49·9 (6)
C(9)—C(10)—C(14)—N	−126·5 (5)	−132·2 (4)
C(9)—C(10)—C(15)—C(16)	−50·3 (5)	−52·0 (5)
C(9)—C(11)—C(5)—C(6)	65·4 (5)	65·4 (5)
C(5)—C(11)—C(9)—C(8)	−60·2 (5)	−60·0 (5)
C(10)—C(12)—C(13)—C(5)	−4·2 (5)	−6·7 (5)
H(5)—C(5)—C(6)—C(7)	−168	−178
H(6)1—C(6)—C(7)—C(8)	−46	−68
H(6)2—C(6)—C(7)—C(8)	173	171
H(7)1—C(7)—C(8)—C(9)	−172	−161
H(7)2—C(7)—C(8)—C(9)	69	77
H(8)1—C(8)—C(9)—C(10)	49	56
H(8)2—C(8)—C(9)—C(10)	160	177
H(9)—C(9)—C(10)—C(15)	61	75
H(N)1—N—C(14)—C(10)	177	176
H(N)2—N—C(14)—C(10)	2	−7
H(16)1—C(15)—C(16)—C(10)	68	−63
H(16)2—C(15)—C(16)—C(10)	170	53
H(16)3—C(15)—C(16)—C(10)	−84	178

Fig. 4. Projection of the crystal structure along **a**. The symmetrically independent molecules (A and B, the latter marked by primes) are hydrogen bonded by N—H···O' of 2·870 (4) and N'—H···O of 2·857 (4) Å between carboxamide groups, forming dimers.

their lengths of 1·497 (7) and 1·526 (5) Å for A and 1·504 (7) and 1·532 (5) Å for B show significant differences ($>3\sigma$) which can be attributed to steric influences. Bond lengths in phenyl rings are in the range 1·367 (7) to 1·408 (7) Å (A) and 1·368 (9) to 1·405 (7) Å (B). Interatomic distances in the carbox-

amide group, C(14)—O of 1·220 (5) (A) and 1·224 (5) Å (B) and C(14)—N of 1·329 (6) (A) and 1·325 (6) Å (B), are in the usual range. Differences in the interatomic distances between the two independent molecules (A and B) are about 1σ; the exceptions are C(1)—C(2) and C(7)—C(8) where they are $>3\sigma$.

The valence angles involving only the $C(sp^3)$ atoms range from 108·4 (4) to 116·0 (4)° (A) and 108·9 (4) to 116·7 (4)° (B). The bond angles for the $C(sp^2)$

atoms range from 119·6 (7) to 121·7 (6)° (A) and from 119·6 (5) to 121·6 (5)° (B).

There is no difference in the conformations of the symmetrically independent molecules. The geometry of the present molecules can be considered in two ways: (1) by dividing the ring part into two moieties, cyclohexane and cyclohexene, or (2) as a cyclooctene ring. C(5), C(6), C(7), C(8), C(9), C(11) form a cyclohexane ring in a chair conformation. C(5), C(11), C(9), C(10), C(12), C(13) are in a cyclohexene ring with a typical half-chair conformation. C(5), C(10), C(12), C(13) form plane I (Table 5) and atoms C(9) and C(11) are displaced from this plane by 0·313 and −0·496 Å in A and 0·346 and −0·467 Å in B. C(5), C(6), C(8), C(9) lie in plane II (Table 5) and C(7) and C(11) are displaced from it by −0·599 and 0·742 Å in A and −0·615 and 0·737 Å in B. Thus the conformation can be described by means of cis-fused six-membered rings. Dihedral angles of 74·9 (2) (A) and 75·4 (2)° (B) define the relative orientation of fused cyclohexane and cyclohexene rings and also the puckering of a cyclooctene ring. The number of possible cyclooctene ring conformations is limited due to the bridging C(11) atom. The conformation of the cyclooctene ring with corresponding torsion angles is shown in Fig. 3.

The symmetrically independent molecules (A and B) are hydrogen bonded by N—H···O contacts of 2·870 (4) and 2·857 (4) Å between carboxamide groups, forming dimers.

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The First Example of a Cyclophosphazene 'Anticlathrate Structure': Geometry of 2,2,4,4,6,6-Hexa(1-aziridinyl)cyclotri(phosphazene), $N_3P_3az_6$, from an X-ray Investigation of the Complex $N_3P_3az_6 . 3CCl_4$

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Abstract

The molecular structure of hexaaziridinylcyclotri(phosphazene), $N_3P_3(NC_2H_4)_6$ (i.e. $N_3P_3az_6$), a powerful antitumor agent, was determined from an X-ray single-crystal analysis of the complex $N_3P_3az_6.3CCl_4$, $M_r = 849$. This complex crystallizes in the monoclinic system, space group $C2/c$, with the parameters $a =$ 15·609 (5), $b = 18·748$ (4), $c = 12·208$ (6) Å, $\beta = 106·40$ (3)°, and $Z = 4$, $V = 3428$ Å³, $d_m = 1·5$ (2), $d_x = 1·645$ Mg m⁻³, $\mu(Mo\ K\alpha) = 1·13$ mm⁻¹. Each $N_3P_3az_6$ is inserted in a monocapped icosahedron formed by 13 CCl_4 molecules and directly connected to six of them by a 'bent halogen bond' (N···Cl = 3·144 Å; ∠N···Cl—C = 157°); so far as is known, such a bond is described here for the first time. The crystal network is a 'bee's nest net' where the host lattice is built up from CCl_4 molecules, and the $N_3P_3az_6$ guest

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