approximately about the axis of least moment-of-inertia.

From the diffraction data it is possible to obtain a rough estimate of an average librational frequency assuming a single independent frequency for the librations about each principal axis of inertia (Cruickshank, 1958):

$$\bar{\theta}_i^2 = (h/8\pi^2\nu_iI_i)\coth(h\nu_i/2k_BT)$$

where $\bar{\theta}_i^2$ is the mean-square librational displacement about the principal axis i, h and k_B are Planck's and Boltzmann's constants, I_i is the corresponding inertia moment and T is the absolute temperature. The resulting frequencies are 2·3, 2·4 and 2·1 THz respectively, showing that, on average, the largest librational movement about the least-inertia axis is due to modes of lower frequency.

Crystal packing. Crystal-energy packing calculations have been carried out with empirical atom–atom potential functions which account for both van der Waals and electrostatic interactions. The results of an energy minimization process show that the molecules tend to readjust within the unit cell in order to increase the short contact distances, giving additional evidence for the presence of hydrogen bonds for two short contacts N(3)—H···O(1)(x, y, $z-1$) = 3·065 (6) and N(3)—H···N(1)($-x+2$, $-y+1$, $-z+1$) = 3·049 (4) Å.

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5β-(1-Pentyl)-2,3,3aβ,4,5,7aα-hexahydroindene-4β-carboxanilide

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Abstract. $C_{21}H_{29}NO$, $M_r = 311·47$, monoclinic, $P2_1/c$, $a = 9·678$ (7), $b = 19·883$ (11), $c = 9·901$ (4) Å, $\beta = 97·26$ (5)°, $V = 1890·0$ (2) Å^3, $Z = 4$, $D_x = 1·094$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0·06$ mm^{-1}, $F(000) = 680$, $T = 298$ K, final $R = 0·061$ for 1447 unique observed reflections. The ring fusion is *trans* and the pentyl and carboxanilide substituents are oriented in *cis* positions. The five-membered ring exhibits a twist conformation and the six-membered ring has a half-chair conformation. The molecules are held together by hydrogen bonds.

Introduction. The intramolecular Diels–Alder (IMDA) reaction is a highly useful tool in synthetic organic chemistry, which has become increasingly important in recent years (Ciganek, 1984; Fallis, 1984). Stereochemical aspects play a central role in these synthetic applications, as well as in many theoretical studies dealing with IMDA reactions.

The title compound was prepared in connection with an NMR study of the stereochemistry involved in the IMDA reaction in non-activated systems (Kaltia, Matikainen & Hase, 1988; Matikainen, Kaltia & Hase,

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and* U_{eq} ($\text{Å}^2 \times 10^2$) *for the non*-H *atoms*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	$U_{\rm eq}$
O	3988 (3)	7532 (2)	3834 (3)	5·7 (2)
N	4638 (4)	7244 (2)	1784 (4)	4·6 (2)
C(1)	1465 (5)	8123 (3)	2602 (5)	4·9 (3)
C(2)	656 (6)	7523 (3)	3078 (6)	6·2 (4)
C(3)	−695 (7)	7842 (4)	3414 (8)	7·9 (5)
C(4)	−839 (7)	8510 (4)	2671 (7)	7·4 (4)
C(5)	302 (6)	8502 (3)	1747 (6)	5·9 (4)
C(6)	817 (7)	9124 (3)	1160 (6)	6·4 (4)
C(7)	2130 (7)	9198 (3)	996 (6)	6·6 (4)
C(8)	3293 (6)	8698 (3)	1425 (5)	5·5 (4)
C(9)	4357 (7)	9013 (3)	2532 (6)	6·5 (4)
C(10)	5149 (7)	9610 (4)	2114 (8)	7·6 (4)
C(11)	6201 (8)	9891 (4)	3179 (8)	7·9 (5)
C(12)	7138 (10)	10423 (4)	2810 (10)	10·2 (6)
C(13)	8215 (12)	10699 (6)	3863 (12)	13·5 (9)
C(14)	2700 (5)	8010 (3)	1817 (5)	4·5 (3)
C(15)	3818 (5)	7573 (2)	2583 (5)	4·2 (3)
C(16)	5803 (5)	6825 (2)	2163 (5)	4·5 (3)
C(17)	6205 (6)	6598 (3)	3472 (5)	5·6 (4)
C(18)	7371 (6)	6191 (3)	3733 (6)	6·5 (4)
C(19)	8124 (7)	5999 (3)	2730 (7)	7·3 (4)
C(20)	7730 (7)	6225 (3)	1430 (7)	7·2 (4)
C(21)	6587 (6)	6639 (3)	1154 (6)	6·3 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

O—C(15)	1·231 (5)	C(8)—C(14)	1·552 (7)
N—C(15)	1·358 (6)	C(9)—C(10)	1·499 (9)
N—C(16)	1·415 (6)	C(10)—C(11)	1·480 (10)
C(1)—C(2)	1·534 (8)	C(11)—C(12)	1·468 (10)
C(1)—C(5)	1·520 (7)	C(12)—C(13)	1·483 (13)
C(1)—C(14)	1·523 (7)	C(14)—C(15)	1·515 (7)
C(2)—C(3)	1·528 (9)	C(16)—C(17)	1·381 (7)
C(3)—C(4)	1·516 (10)	C(16)—C(21)	1·379 (7)
C(4)—C(5)	1·521 (9)	C(17)—C(18)	1·385 (8)
C(5)—C(6)	1·478 (8)	C(18)—C(19)	1·359 (8)
C(6)—C(7)	1·310 (8)	C(19)—C(20)	1·371 (8)
C(7)—C(8)	1·522 (8)	C(20)—C(21)	1·378 (8)
C(8)—C(9)	1·538 (8)		

C(15)—N—C(16)	129·4 (4)	C(10)—C(11)—C(12)	119·1 (7)
C(2)—C(1)—C(5)	100·7 (4)	C(11)—C(12)—C(13)	119·5 (8)
C(2)—C(1)—C(14)	120·4 (5)	C(1)—C(14)—C(8)	109·7 (4)
C(5)—C(1)—C(14)	111·2 (4)	C(1)—C(14)—C(15)	112·7 (4)
C(1)—C(2)—C(3)	103·1 (5)	C(8)—C(14)—C(15)	111·5 (4)
C(2)—C(3)—C(4)	106·7 (5)	O—C(15)—N	122·7 (4)
C(3)—C(4)—C(5)	104·7 (5)	O—C(15)—C(14)	122·6 (4)
C(1)—C(5)—C(4)	102·3 (5)	N—C(15)—C(14)	114·7 (4)
C(1)—C(5)—C(6)	111·8 (5)	N—C(16)—C(17)	124·2 (5)
C(4)—C(5)—C(6)	122·3 (6)	N—C(16)—C(21)	117·3 (4)
C(5)—C(6)—C(7)	121·6 (6)	C(17)—C(16)—C(21)	118·5 (5)
C(6)—C(7)—C(8)	125·9 (6)	C(16)—C(17)—C(18)	119·5 (5)
C(7)—C(8)—C(9)	109·7 (5)	C(17)—C(18)—C(19)	121·8 (6)
C(7)—C(8)—C(14)	111·2 (5)	C(18)—C(19)—C(20)	118·9 (6)
C(9)—C(8)—C(14)	114·6 (4)	C(19)—C(20)—C(21)	120·2 (6)
C(8)—C(9)—C(10)	116·4 (5)	C(16)—C(21)—C(20)	121·2 (5)
C(9)—C(10)—C(11)	115·6 (6)		

1988). The X-ray structure determination was undertaken to ascertain the stereochemistry of the molecule.

Experimental. Plate-like crystals from water–ethanol mixture, crystal dimensions $0{\cdot}24 \times 0{\cdot}52 \times 0{\cdot}36$ mm, Nicolet $P3$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Unit cell from 18 reflections in range $18 < 2\theta < 24°$. Data collection: 2641 ($-10 \le h \le 10$, $0 \le k \le 21$, $0 \le l \le 10$) independent reflections with $2\theta < 45°$, 1447 with $I > 2\sigma(I)$ used in refinement. Two standard reflections checked every 50 reflections, no significant deviations. No correction for absorption. Structure solved with *SHELXS*86 (Sheldrick, 1985), H atoms from difference synthesis. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F, H isotropic with common temperature factor ($U = 0{\cdot}06$ Å^2), $w = (\sigma^2 + 0{\cdot}0093F^2)^{-1}$. $R = 0{\cdot}061$, $wR = 0{\cdot}074$ for 1447 reflections. Final $(\Delta/\sigma)_{\rm max} = 0{\cdot}45$, $S = 2{\cdot}74$, max. and min. heights in final difference synthesis 0·19 and $-0{\cdot}16$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *PLUTO*78 (Motherwell & Clegg, 1978) used for diagrams.

Discussion. The atomic parameters are given in Table 1 and the bond lengths and angles in Table 2.[*] A perspective view of the molecule with numbering scheme is shown in Fig. 1 and the crystal packing in Fig. 2.

The five-membered cyclopentane ring exhibits a twist conformation with puckering parameters $Q = 0{\cdot}462$ Å and $\varphi = 349{\cdot}1°$ (Cremer & Pople, 1975; Boeyens,

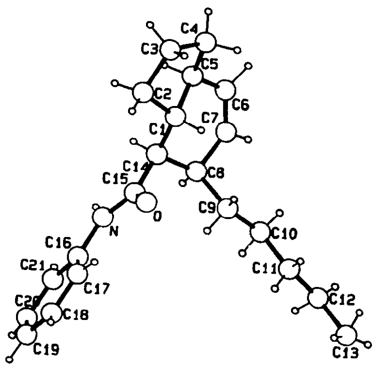

Fig. 1. Perspective view of the molecule.

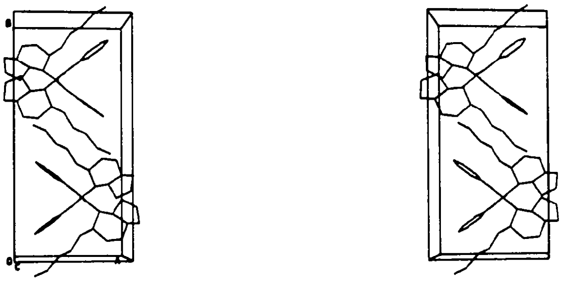

Fig. 2. Stereoscopic view of the unit cell.

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51098 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1978). The puckering parameters for the six-membered ring are $Q = 0{\cdot}488$ Å, $\theta = 47{\cdot}8°$ and $\varphi = 340{\cdot}8°$; the resulting conformation is near to the $^1H_6$ half-chair form. The ring fusion is *trans* and the pentyl and the carboxanilide substituents are oriented in *cis* positions.

The molecules are held together by hydrogen bonds between amide N and amide O atoms. The $\mathrm{N}\cdots\mathrm{O}^{\mathrm{i}}$ and $\mathrm{H}\cdots\mathrm{O}^{\mathrm{i}}$ distances are 2·943 (8) and 2·12 (9) Å, while the $\mathrm{N{-}H}\cdots\mathrm{O}^{\mathrm{i}}$ angle is 172 (5)° [symmetry code: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$]. Thus, the molecules are bonded into chains along the *c* axis.

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The Structure of 3β,20α-Bis(dimethylamino)pregn-5-en-18-ol

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Abstract. $C_{25}H_{44}N_2O$, $M_r = 388{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}185$ (2), $b = 18{\cdot}123$ (2), $c = 20{\cdot}852$ (2) Å, $U = 2337{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}104$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}47$ mm^{-1}, $F(000) = 864$, $T = 293$ K. Final $R = 0{\cdot}038$ for 1791 reflections with $I \geq 3\sigma(I)$. Rings *A* and *C* are in chair conformation. Ring *B* is in an 8β,9α-half-chair conformation. Ring *D* adopts a conformation in between 13β,14α-half-chair and 13β-envelope. There is a quasi-*trans* fusion of rings *A* and *B*, whilst ring systems *B*/*C* and *C*/*D* are *trans* fused about the bonds C(8)—C(9) and C(13)—C(14).

Introduction. As part of our crystallographic studies on steroidal alkaloids we report here the crystal structure of the compound 3β,20α-bis(dimethylamino)pregn-5-en-18-ol. Earlier we reported the crystal structures of the compounds 11α-hydroxycon-1,4-dienine-3-one monohydrate and 11α-hydroxycon-1,4,18-trienine-3-one (Radhakrishnan, Viswamitra, Bhutani & Ali, 1988). In steroidal alkaloids N atoms are incorporated in the rings or the side chains of the steroidal skeleton. The title compound has two N atoms in the side chains (Fig. 1). The other two compounds we reported earlier have N atoms in a ring. The compounds were isolated from the bark of the plant *Holarrena antidysentrica* (Bhutani, Ali, Sharma, Vaid & Gupta, 1988). These are important in herbal medicine as antidysentrics and anthelmintics.

Experimental. The needle-shaped crystals were grown from methanol solution by slow evaporation. The cell parameters were refined from accurately determined 2θ values of 22 reflections, $6{\cdot}5 < \theta < 36{\cdot}9°$, on a CAD-4 diffractometer. Intensity data were collected with Ni-filtered Cu *K*α radiation using a crystal of dimensions 0·3 × 0·2 × 1·6 mm. Max. $(\sin\theta)/\lambda = 0{\cdot}627$ Å^{-1}, ω–2θ scans. Absorption correction (North, Phillips & Mathews, 1968) was applied; transmission factors varied from 94·3 to 99·7%. Lorentz and polarization corrections were also applied. 2867 reflections were measured for $0 \leq h \leq 7$, $0 \leq k \leq 21$, $0 \leq l \leq 24$; 1791 reflections have $I \geq 3\sigma(I)$. Three strong reflections monitored periodically showed that the crystal was stable to X-rays.

The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982). An *E* map computed with the best set of phases (CFOM = 2·998)

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