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.** $\mathrm{C_{25}H_{44}N_2O}$, $M_r = 388{\cdot}6$, 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 $2\theta$ values of 22 reflections, $6{\cdot}5 < \theta < 36{\cdot}9°$, on a CAD-4 diffractometer. Intensity data were collected with Ni-filtered Cu $K\alpha$ radiation using a crystal of dimensions $0{\cdot}3 \times 0{\cdot}2 \times 1{\cdot}6$ mm. Max. $(\sin\theta)/\lambda = 0{\cdot}627$ Å$^{-1}$, $\omega$–$2\theta$ 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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revealed the positions of 22 out of 28 non-H atoms. A weighted Fourier synthesis and Karle recycling gave the positions of the rest of the non-H atoms. Successive difference Fourier syntheses revealed the positions of 40 H atoms; those of the remaining four were fixed stereochemically. After the final cycle of refinement with anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms (for nine H atoms, the temperature factors were fixed because they assumed non-positive values when refined), $R = 0{\cdot}038$ and $wR = 0{\cdot}037$. The function minimized was $\sum w(|F_o| - F_c|)^2$, where $w = 1/\sigma^2(F_o)$. The residual electron density in the final difference Fourier map was between 0·14 and −0·14 e Å$^{-3}$. The max. shift/e.s.d. for non-H atoms was 0·29. All calculations were performed with the Enraf–Nonius (1979) *Structure Determination Package* on a PDP 11/44 computer. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The numbering scheme and the packing diagram are given in Fig. 1. The final coordinates and equivalent temperature factors for non-H atoms are given in Table 1.* The bond lengths and bond angles are given in Table 2 and endocyclic torsion angles in Table 3.

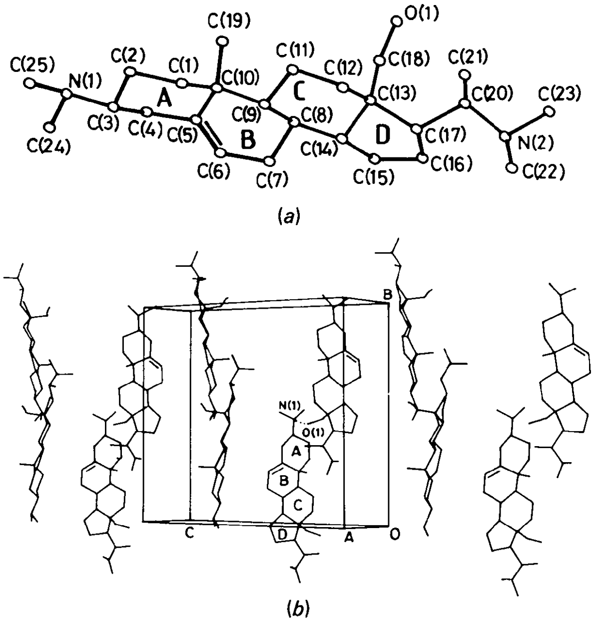

Fig. 1. (*a*) The numbering scheme and (*b*) the packing diagram for the compound 3*β*,20α-bis(dimethylamino)pregn-5-en-18-ol.

Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| O(1) | 0·1995 (5) | 1·0438 (2) | 0·7596 (1) | 4·57 (6) |
| N(1) | 0·0435 (6) | 0·5201 (2) | 0·8535 (1) | 3·66 (7) |
| N(2) | 0·3374 (6) | 1·2076 (2) | 0·9179 (2) | 3·65 (7) |
| C(1) | 0·3247 (6) | 0·7082 (2) | 0·8418 (2) | 3·36 (7) |
| C(2) | 0·2677 (7) | 0·6283 (2) | 0·8255 (2) | 3·58 (8) |
| C(3) | 0·1159 (7) | 0·5947 (2) | 0·8747 (2) | 3·42 (8) |
| C(4) | −0·0831 (7) | 0·6445 (2) | 0·8840 (2) | 3·54 (8) |
| C(5) | −0·0259 (6) | 0·7252 (2) | 0·8954 (2) | 2·95 (7) |
| C(6) | −0·1190 (6) | 0·7619 (2) | 0·9427 (2) | 3·33 (8) |
| C(7) | −0·0884 (6) | 0·8422 (2) | 0·9559 (2) | 3·05 (7) |
| C(8) | 0·0209 (6) | 0·8827 (2) | 0·9003 (2) | 2·62 (7) |
| C(9) | 0·2084 (6) | 0·8362 (2) | 0·8739 (2) | 2·52 (6) |
| C(10) | 0·1290 (6) | 0·7603 (2) | 0·8479 (2) | 2·67 (7) |
| C(11) | 0·3460 (6) | 0·8792 (2) | 0·8249 (2) | 3·11 (7) |
| C(12) | 0·4200 (6) | 0·9553 (2) | 0·8494 (2) | 2·96 (7) |
| C(13) | 0·2270 (6) | 1·0024 (2) | 0·8703 (2) | 2·55 (6) |
| C(14) | 0·1078 (6) | 0·9572 (2) | 0·9226 (2) | 2·51 (7) |
| C(15) | −0·0485 (6) | 1·0121 (2) | 0·9531 (2) | 3·25 (7) |
| C(16) | 0·0750 (7) | 1·0854 (2) | 0·9517 (2) | 3·21 (7) |
| C(17) | 0·2792 (6) | 1·0738 (2) | 0·9094 (2) | 2·64 (7) |
| C(18) | 0·0763 (6) | 1·0186 (2) | 0·8131 (2) | 3·07 (7) |
| C(19) | 0·0146 (7) | 0·7679 (2) | 0·7824 (2) | 3·76 (8) |
| C(20) | 0·3373 (6) | 1·1444 (2) | 0·8726 (2) | 3·17 (7) |
| C(21) | 0·5459 (8) | 1·1379 (2) | 0·8345 (2) | 4·37 (9) |
| C(22) | 0·5174 (8) | 1·2081 (3) | 0·9622 (2) | 5·7 (1) |
| C(23) | 0·319 (1) | 1·2782 (2) | 0·8859 (2) | 6·3 (1) |
| C(24) | −0·0975 (9) | 0·4850 (2) | 0·9009 (2) | 5·7 (1) |
| C(25) | 0·2281 (9) | 0·4715 (2) | 0·8395 (2) | 5·6 (1) |

The mean value of the C($sp^3$)–C($sp^3$) bonds [1·538 (5) Å] is nearly the same as that commonly observed (1·533 Å) (Sutton, 1965), although the bond lengths C(2)–C(3), C(9)–C(10), C(13)–C(17) and C(16)–C(17) show some significant deviations. The bond angles C(8)–C(14)–C(15), C(8)–C(14)–C(13), C(13)–C(17)–C(20), C(14)–C(13)–C(17) and C(12)–C(13)–C(17) show significant deviations from the ideal tetrahedral value of 109·4° (Table 2). These deviations are common in steroids as a result of strain induced by ring junctions, side chains and bond unsaturations. The bond length C(5)–C(6) is 1·322 (5) Å, indicating double-bond character.

Ring *A* is in a chair conformation with the best rotational axis bisecting the C(1)–C(2) and C(4)–C(5) bonds and with the asymmetry parameter $\Delta C_2$[C(1)–C(2)] = 0·3° (Duax & Norton, 1975). The best mirror plane for this ring passes through C(1) and C(4) with asymmetry parameter $\Delta C_s$[C(1)–C(4)] = 3·2°. Ring *C* is also in a chair conformation with the best rotational axis bisecting the C(9)–C(11) and C(13)–C(14) bonds, with $\Delta C_2$[C(9)–C(11)] = 4·5°. The best mirror passes through C(9) and C(13) with $\Delta C_s$[C(9)–C(13)] = 2·4°. Ring *B* is in an 8*β*,9α-half-chair conformation with C(8) and C(9) +0·335 (3) and −0·410 (3) Å from the plane formed by C(5), C(6), C(7) and C(10). The asymmetry parameter $\Delta C_s$[C(5)–C(6)] = 2·4°.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51101 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| O(1)—C(18) | 1·426 (4) | C(8)—C(9) | 1·536 (5) |
| N(1)—C(3) | 1·490 (5) | C(8)—C(14) | 1·526 (5) |
| N(1)—C(24) | 1·465 (6) | C(9)—C(10) | 1·559 (5) |
| N(1)—C(25) | 1·472 (6) | C(9)—C(11) | 1·541 (5) |
| N(2)—C(20) | 1·483 (4) | C(10)—C(19) | 1·544 (5) |
| N(2)—C(22) | 1·448 (6) | C(11)—C(12) | 1·541 (5) |
| N(2)—C(23) | 1·446 (6) | C(12)—C(13) | 1·530 (5) |
| C(1)—C(2) | 1·529 (6) | C(13)—C(14) | 1·552 (5) |
| C(1)—C(10) | 1·540 (6) | C(13)—C(17) | 1·562 (5) |
| C(2)—C(3) | 1·518 (5) | C(13)—C(18) | 1·543 (5) |
| C(3)—C(4) | 1·539 (6) | C(14)—C(15) | 1·526 (5) |
| C(4)—C(5) | 1·523 (5) | C(15)—C(16) | 1·533 (5) |
| C(5)—C(6) | 1·322 (5) | C(16)—C(17) | 1·556 (5) |
| C(5)—C(10) | 1·519 (5) | C(17)—C(20) | 1·536 (5) |
| C(6)—C(7) | 1·493 (5) | C(20)—C(21) | 1·520 (6) |
| C(7)—C(8) | 1·531 (5) | | |

| | | | |
|---|---|---|---|
| C(3)—N(1)—C(24) | 112·0 (3) | C(1)—C(10)—C(19) | 110·0 (3) |
| C(3)—N(1)—C(25) | 111·7 (3) | C(5)—C(10)—C(9) | 110·0 (3) |
| C(24)—N(1)—C(25) | 109·6 (3) | C(5)—C(10)—C(19) | 109·0 (3) |
| C(20)—N(2)—C(22) | 114·3 (3) | C(9)—C(10)—C(19) | 111·9 (3) |
| C(20)—N(2)—C(23) | 112·9 (4) | C(9)—C(11)—C(12) | 113·4 (3) |
| C(22)—N(2)—C(23) | 110·4 (3) | C(11)—C(12)—C(13) | 111·2 (3) |
| C(2)—C(1)—C(10) | 114·7 (4) | C(12)—C(13)—C(14) | 106·0 (3) |
| C(1)—C(2)—C(3) | 111·8 (3) | C(12)—C(13)—C(17) | 116·6 (3) |
| N(1)—C(3)—C(2) | 110·5 (3) | C(12)—C(13)—C(18) | 111·0 (3) |
| N(1)—C(3)—C(4) | 109·2 (3) | C(14)—C(13)—C(17) | 99·7 (2) |
| C(2)—C(3)—C(4) | 110·2 (3) | C(14)—C(13)—C(18) | 110·9 (3) |
| C(3)—C(4)—C(5) | 113·4 (3) | C(17)—C(13)—C(18) | 111·8 (3) |
| C(4)—C(5)—C(6) | 119·9 (3) | C(8)—C(14)—C(13) | 114·9 (3) |
| C(4)—O(5)—C(10) | 116·5 (3) | C(8)—C(14)—C(15) | 118·7 (3) |
| C(6)—C(5)—C(10) | 123·5 (4) | C(13)—C(14)—C(15) | 104·6 (3) |
| C(5)—C(6)—C(7) | 125·0 (3) | C(14)—C(15)—C(16) | 104·0 (3) |
| C(6)—C(7)—C(8) | 112·5 (3) | C(15)—C(16)—C(17) | 107·4 (3) |
| C(7)—C(8)—C(9) | 110·1 (3) | C(13)—C(17)—C(16) | 104·0 (3) |
| C(7)—C(8)—C(14) | 110·4 (3) | C(13)—C(17)—C(20) | 118·6 (3) |
| C(9)—C(8)—C(14) | 109·1 (3) | C(16)—C(17)—C(20) | 111·2 (3) |
| C(8)—C(9)—C(10) | 111·7 (3) | O(1)—C(18)—C(13) | 110·0 (3) |
| C(8)—C(9)—C(11) | 112·2 (3) | N(2)—C(20)—C(17) | 109·0 (3) |
| C(10)—C(9)—C(11) | 112·9 (3) | N(2)—C(20)—C(21) | 113·1 (4) |
| C(1)—C(10)—C(5) | 107·0 (3) | C(17)—C(20)—C(21) | 113·3 (3) |
| C(1)—C(10)—C(9) | 108·8 (3) | | |

Ring *D* is in between 13$\beta$,14$\alpha$-half-chair and 13$\beta$-envelope conformations with the phase angle of pseudorotation $\Delta = 10{\cdot}4°$ and maximum angle of torsion $\varphi = 45{\cdot}4°$ (Altona, Geise & Romers, 1968). The asymmetry parameter $\Delta C_s$[C(13)] which gives the distortion from the ideal mirror symmetry bisecting the C(15)—C(16) bond is 12·7°. The atoms C(13) and C(14) are +0·474 (3) and −0·250 (3) Å from the plane formed by C(15), C(16) and C(17).

Fig. 2 shows the Newman projections along the bonds involved in the fusion of the rings. As can be seen, the atoms H(8) and H(9) as well as C(18) and H(14) have antiperiplanar configuration. Table 3 compares the observed ring torsion angles with those calculated for appropriately substituted cyclohexane rings (Geise, Altona & Romers, 1967). The table also gives the average torsion angles observed for the 8$\beta$,9$\alpha$-half-chair conformation in a few steroid structures along with those observed for an ideal 13$\beta$,14$\alpha$-half-chair (Duax & Norton, 1975). The differences observed in ring *A* torsion angles are attributed to the quasi-*trans A*/*B* ring fusion in the present structure, whereas the calculations are based on *trans*-fused rings. The variations in the torsion angles C(12)—C(11)—C(9)—C(8) and C(11)—C(9)—C(8)—C(14) in ring *C* are due to the transmission effect from ring *B* which is in an 8$\beta$,9$\alpha$-half-chair conformation unlike the chair conformation used in the calculations. The deviations in the torsion angles of ring *D* may be due to the presence of the substituents in the present structure (Fig. 1).

In the extended structure there is a hydrogen bond between N(1) and the phenolic O(1) atom [O(1)—H(O)···N(1) = 2·825 Å, Fig. 1(*b*)]. The torsion angle C(1)—C(10)—C(13)—C(18) which gives the twist of the steroid molecule about the line joining C(10) and C(13) is 53·3 (3)°.

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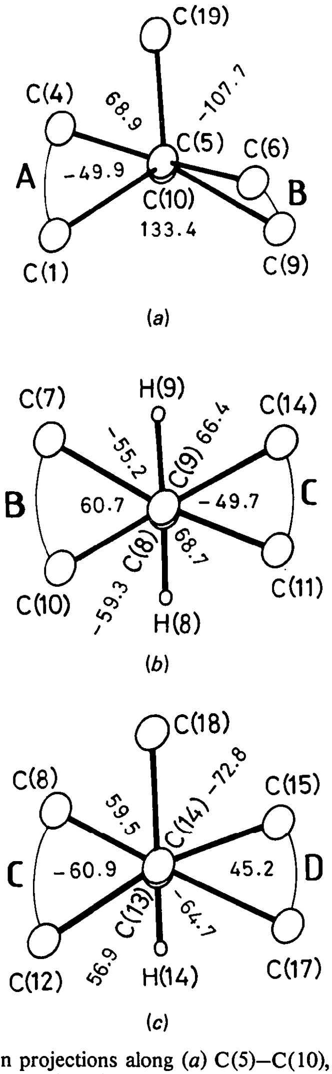

Fig. 2. Newman projections along (*a*) C(5)—C(10), (*b*) C(8)—C(9) and (*c*) C(13)—C(14) (angles in °).

Table 3. *Observed values for the ring torsion angles* (°) *compared with the calculated values of Geise, Altona & Romers* (1967) *for rings A and C, and with the average for a selection of other steroids* (*Duax & Norton,* 1975) *for rings B and D*

| | Observed | Calculated | |
|---|---|---|---|
| Ring *A* | | | |
| 10–1–2–3 | −57·7 (4) | −57 | |
| 1–2–3–4 | 53·1 (4) | 55 | |
| 2–3–4–5 | −49·6 (4) | −56 | |
| 3–4–5–10 | 50·7 (3) | 58 | |
| 1–10–5–4 | −49·9 (4) | −58 | |
| 5–10–1–2 | 53·2 (4) | 58 | |
| Ring *C* | | With *D* | Without *D* |
| 13–12–11–9 | −56·0 (4) | −56 | −56 |
| 12–11–9–8 | 51·1 (4) | 57·3 | 56 |
| 11–9–8–14 | −49·7 (3) | −58 | −58 |
| 9–8–14–13 | 57·4 (3) | 58 | 58 |
| 8–14–13–12 | −60·9 (3) | −58 | −59 |
| 14–13–12–11 | 57·4 (3) | 58 | 58 |
| Ring *B* | | | |
| 6–5–10–9 | 15·4 (5) | 13·5 | |
| 10–5–6–7 | 1·2 (5) | 2·3 | |
| 5–6–7–8 | 12·8 (5) | 14·2 | |
| 6–7–8–9 | −42·6 (4) | −44·7 | |
| 7–8–9–10 | 60·7 (3) | 63·1 | |
| 8–9–10–5 | −45·9 (3) | −45·8 | |
| Ring *D* | | | |
| 17–13–14–15 | 45·2 (3) | 47·7 | |
| 13–14–15–16 | −34·6 (3) | −38·2 | |
| 14–15–16–17 | 9·7 (3) | 13·4 | |
| 15–16–17–13 | 18·2 (3) | 16·2 | |
| 16–17–13–14 | −38·1 (3) | −38·3 | |

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# Structure of an Analogue of the Triazolobenzodiazepine Alprazolam*

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**Abstract.** 1-Methyl-6-phenyl-8-(trifluoromethyl)-4*H*-*s*-triazolo[4,3-*a*][1,4]benzodiazepine sesquihydrate, $C_{18}H_{13}F_3N_4.1{\cdot}5H_2O$, $M_r = 369{\cdot}3$, monoclinic, $C2/c$, $a = 13{\cdot}115\ (4)$, $b = 9{\cdot}673\ (3)$, $c = 27{\cdot}007\ (7)$ Å, $\beta = 96{\cdot}79\ (3)°$, $V = 3402{\cdot}1$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}442$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}16$ cm$^{-1}$, $F(000) = 1528$, $T = 294$ K, $R = 0{\cdot}049$ for 1544 observed reflections. The seven-membered ring is in a cycloheptatriene-like boat conformation with bow and stern angles of 55·5 (8) and 36·2 (8)°, respectively. The angle between the 6-phenyl ring and the fused benzo moiety is 63·5 (8)°. The triazolo ring and the two aromatic rings are each planar to within the limits of experimental error.

**Introduction.** The title compound (Hester, Rudzik & Kamdar, 1971) belongs to the class of triazolobenzodiazepines, exemplified by alprazolam† and triazolam,‡ which have found use in clinical practice as anxiolytics and hypnotics. It differs from alprazolam only in that the substituent at C8 is a trifluoromethyl group, rather than a chlorine atom. However, its affinity for the benzodiazepine receptor *in vitro* is about 12 times less strong.

**Experimental.** Crystals were grown from amyl acetate. A crystal of size 0·15 × 0·4 × 0·4 mm was mounted on

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* Contribution from the Crystallography Unit, Universities of Aston and Birmingham.

† 8-Chloro-1-methyl-6-phenyl-4*H*-*s*-triazolo[4,3-*a*][1,4]-benzodiazepine. Marketed as Xanax (Upjohn).

‡ 8-Chloro-6-(2-chlorophenyl)-1-methyl-4*H*-*s*-triazolo[4,3-*a*]-[1,4]benzodiazepine. Marketed as Halcion (Upjohn).

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