corresponding bond in 3,4-disubstituted sydnone derivatives may also be attributed to the steric effect (Ueng, Wang & Yeh, 1987a,b).

The bond lengths (S—C), bond angles (CSC') and the angles between the CSC' plane and the sydnone ring of the title compounds and other diaryl sulfides are comparable (Von Deuten & Klar, 1981). There is no apparent trend in such bond lengths. A $\pi$ interaction between the S atom and the sydnone ring would be expected if the CSC plane and the aryl ring were coplanar. However, this is not obvious; as an example 4-(dimethylamino)phenyl 4-nitrophenyl sulfide (Von Deuten & Klar, 1981) does have one of the aryl rings which is coplanar with the CSC plane, but both S—C lengths are about the same. The shortening of the S—C bonds of the title compounds relative to those of other diaryl sulfides and the average bond lengths of 1,3,5-trithiane [1·818 (5) Å (Fleming & Lynton, 1967)] may be attributable to orbital eletronegativity effects.

The C(7)—S(1)—C(7') bond angles of the title compounds [97·4 (1), 101·0 (1) and 100·3 (1)°] are comparable with corresponding angles in the cyclic 1,3,5-trithane [99·2 (6) and 100·7 (5)°]. The dihedral angles ($\omega$) between the sydnone ring and the phenyl ring are listed in Table 4 for 3,4-disubstituted compounds. It is certainly clear that all such compounds have angles greater than 50°.

Compound (3) has exact $C_2$ molecular symmetry bisecting the C—S—C' angle which coincides with the crystallographic twofold axis along the $b$ axis. Compound (1) has a pseudo twofold axis; the two sydnone-aryl parts of the compound are essentially the

same. However, compound (2) adopts a quite different conformation from those of the other two compounds. The difference can be seen clearly in Fig. 1. The packing in the crystal is also quite different (Fig. 2).

Instead of the 'morino' conformation (Von Deuten & Klar, 1981) with one ring in the C—S—C' plane and the other perpendicular, as found in most other diaryl sulfides, the three title compounds have the butterfly conformation [61·7 (1), 59·3 (1); 88·1 (1), 73·6 (1); 72·9 (1), 72·9 (1)°, respectively].

**References**

Enraf–Nonius (1979). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.
FLEMING, J. E. & LYNTON, H. (1967). *Can. J. Chem.* **45**, 353–357.
GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* A37, S339.
HAŠEK, J., OBRDA, J., KUML, K., NEŠPŮREK, S., CHOJNACKI, H. & ŠORM, M. (1978). *Acta Cryst.* B34, 2756–2759.
HAŠEK, J., OBRDA, J., KUML, K., NEŠPŮREK, S. & ŠORM, M. (1979). *Acta Cryst.* B35, 2449–2451.
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
UENG, C.-H., WANG, Y. & YEH, M.-Y. (1987a). *Acta Cryst.* C43, 1122–1125.
UENG, C.-H., WANG, Y. & YEH, M.-Y. (1987b). *J. Chin. Chem. Soc.* **34**, 105–110.
VON DEUTEN, K. & KLAR, G. (1981). *Cryst. Struct. Commun.* **10**, 231–239.

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# Structures of two Psychoactive 1,4-Benzodiazepines

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**Abstract.** (I) 8-Chloro-6-(2-chlorophenyl)-2-methyl-4*H*-imidazo[1,2-*a*][1,4]benzodiazepine, $C_{18}H_{13}Cl_2N_3$, $M_r = 342\cdot2$, monoclinic, $P2_1/c$, $a = 13\cdot168$ (3), $b = 14\cdot852$ (3), $c = 8\cdot286$ (2) Å, $\beta = 94\cdot45$ (2)°, $V = 1615\cdot6$ Å$^3$, $Z = 4$, $D_x = 1\cdot407$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = $0\cdot71069$ Å, $\mu = 3\cdot95$ cm$^{-1}$, $F(000) = 704$, $T = 293$ K, $R = 0\cdot042$ for 796 observed reflections. (II) 8-Chloro-6-(2-chlorophenyl)-1-(4-pyridyl)-1,2,4-triazolo[4,3-*a*]-[1,4]benzodiazepine, $C_{21}H_{13}Cl_2N_5$, $M_r = 406\cdot3$, orthorhombic, *Pbca*, $a = 21\cdot560$ (3), $b = 8\cdot790$ (1), $c = 19\cdot866$ (5) Å, $V = 3764\cdot9$ Å$^3$, $Z = 8$, $D_x = $ 1·434 g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = $0\cdot71069$ Å, $\mu = 3\cdot5$ cm$^{-1}$, $F(000) = 1664$, $T = 293$ K, $R = 0\cdot042$ for 2196 observed reflections. The angle between the mean planes of the chlorophenyl ring and the fused benzo moiety is 77 (1)° in (I) and 78 (1)° in (II). The seven-membered heterocyclic ring adopts a cycloheptatriene-like boat conformation with bow and stern angles of 55 (1) and 33 (1)° in (I), and 53·4 (7) and 34·3 (7)° in (II). In both compounds the five-membered heterocyclic ring and the two aromatic rings are each planar to within ±0·02 Å. Bond lengths and angles are normal.

**Introduction.** The compounds (I) (Gall & Kamdar, 1981) and (II) (Szmuszkovicz, 1972) are related to the classical psychoactive 5-phenyl-1,4-benzodiazepin-2-ones such as diazepam,* but differ from these in containing a five-membered hetero-ring fused across the N1–C2 bond of the parent system. Compound (II) differs from the medicinally used triazolam† only in that the substituent at C1 is a pyridyl ring rather than a methyl group. Both compounds are potent anticonvulsants, being some two to three times more active than diazepam in the antipentylenetetrazole and thiosemicarbazide tests. As a muscle relaxant, (II) is approximately three times more potent than (I), and is comparable in potency to diazepam as measured by the 'chimney' test and also a number of other tests.



(I)          (II)
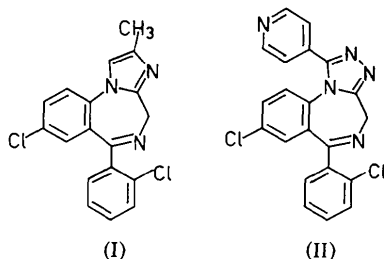
**Experimental.** Compound (I): crystals from amyl acetate, crystal size $0.05 \times 0.2 \times 0.4$ mm, Enraf–Nonius CAD-4 diffractometer, cell dimensions from setting angles of 23 reflections in range $8 < \theta < 13°$, graphite-monochromated Mo $K\alpha$ radiation, 3298 reflections scanned by $\omega/2\theta$ scans up to $\theta = 25°$, 3161 unique, 796 $[I > 2.5\sigma(I)]$ reflections considered observed and used in the analysis, index range $h$ −13 to 12, $k$ 0 to 16, $l$ 0 to 10. Two standard reflections measured every 2 h showed no significant variation in intensity. No absorption corrections applied. Structure determined by direct methods and refined by least squares on $F$ using anisotropic thermal parameters for the heavier atoms. H atoms placed in calculated positions (C–H = $1.08$ Å) 'riding' on their respective bonded atoms and two common $U_{iso}$ parameters were refined, one for the C(13) methyl H atoms and one for the other H atoms. Weights, $w = 1/\sigma^2(F)$, which resulted in a satisfactory weighting analysis, were used in the least-squares refinement. Refinement converged to $R = 0.042$, $wR = 0.037$ with maximum shift/e.s.d. ratio of $0.1$. Residual electron density in final difference map $\pm 0.2$ e Å$^{-3}$. No correction for secondary extinction.

Compound (II): crystals from butanone/ethanol; diffractometer measurements and structure determination as for compound (I) except: crystal size $0.3 \times$

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* 7-Chloro-1,3-dihydro-1-methyl-5-phenyl-2H-1,4-benzodiazepin-2-one. Marketed as Valium (Roche).
† Marketed as Halcion (Upjohn).

$0.4 \times 0.6$ mm, cell dimensions from 25 reflections in the range 8–24°, 3321 unique reflections measured, 2196 considered observed, index range $h$ 0 to 25, $k$ 0 to 10, $l$ 0 to 23. H atoms located from difference Fourier maps and refined with isotropic temperature factors (other species refined anisotropically). Final $R = 0.042$, $wR = 0.058$, $w = 1/[\sigma^2(F) + 0.005F^2]$, residual electron density $\pm 0.3$ e Å$^{-3}$.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Computations were carried out on the University of Birmingham Honeywell DPS 8/70 computer and on the CDC 7600 at the University of Manchester Regional Computer Centre with the *SHELX*76 (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg, 1978) programs.

**Discussion.** Atomic parameters for molecules (I) and (II) are listed in Table 1,* and bond lengths and selected bond and torsion angles are in Table 2. The atomic numbering scheme is shown in Fig. 1.

Bond lengths and angles generally agree well in the two structures and also with those found previously in analogous structures (Hamor & Martin, 1983). The N(1)–C(3) bond is shortened from the normal single-bond value of ca $1.47$ Å to $1.38$ Å, due to electron delocalization between N(1) and N(3). Consistent with this the N(3)–C(3) formal double bond is slightly longer than a normal C=N double bond [cf. N(5)= C(6)]. There is a near-planar disposition of bonds about N(1) and C(3), so that the geometry of this bond resembles that of a double bond [more nearly in (I) than in (II), where, presumably because of the large substituent at C(1), a significant distortion occurs, torsion angle C(11)–N(1)–C(3)–C(4) −9.3°, compared with −4.2° in (I)]. The seven-membered ring adopts a cycloheptatriene-like boat conformation, as is commonly found in this class of compounds, the other two double bonds being N(5)–C(6) and the shared aromatic bond C(11)–C(12) (see Table 2). The 'boat' can be described in terms of the angles between the central plane, consisting of atoms N(1), C(3), N(5), C(6) and the 'bow' and 'stern' planes, consisting of atoms C(3), C(4), N(5) and N(1), C(6), C(11), C(12), respectively. The bow and stern angles, 54.9 (12) and 33.0 (12)° in (I), and 53.4 (7) and 34.3 (7)° in (II), compare with values of 56.5 and 35.3° respectively in 8-chloro-1-[(dimethylamino)methyl]-6-phenyl-4H-imidazo[1,2-a][1,4]benzodiazepine (Butcher & Hamor, 1984) and 55.8 and 36.2° in 1-methyl-6-phenyl-8-(trifluoromethyl)-4H-1,2,4-triazolo[4,3-a][1,4]benzodiazepine (Kemmish & Hamor, 1988). The corre-

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51507 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$)

$$U_{eq} = (U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)/3.$$

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| Compound (I) | | | | |
| Cl(8) | -1633 (2) | -4262 (1) | 97 (3) | 90 |
| Cl(2') | -2395 (2) | -5232 (1) | -6087 (2) | 62 |
| N(1) | -4558 (6) | -6547 (5) | -3776 (8) | 53 |
| N(3) | -5287 (6) | -7771 (4) | -4929 (9) | 57 |
| N(5) | -2704 (6) | -7707 (4) | -4065 (7) | 50 |
| C(1) | -5453 (8) | -6266 (6) | -4571 (12) | 64 |
| C(2) | -5893 (7) | -7006 (7) | -5303 (11) | 62 |
| C(3) | -4514 (7) | -7465 (6) | -4014 (11) | 50 |
| C(4) | -3626 (7) | -7980 (5) | -3299 (9) | 53 |
| C(6) | -2382 (6) | -6902 (6) | -3819 (8) | 47 |
| C(7) | -2135 (7) | -5644 (6) | -1872 (9) | 53 |
| C(8) | -2500 (9) | -4952 (6) | -974 (10) | 64 |
| C(9) | -3528 (9) | -4793 (6) | -1010 (10) | 71 |
| C(10) | -4218 (7) | -5329 (6) | -1898 (11) | 64 |
| C(11) | -3850 (9) | -6028 (6) | -2804 (10) | 55 |
| C(12) | -2826 (8) | -6195 (5) | -2814 (9) | 51 |
| C(13) | -6840 (7) | -7106 (6) | -6362 (11) | 78 |
| C(1') | -1422 (7) | -6669 (6) | -4608 (10) | 52 |
| C(2') | -1353 (7) | -5921 (5) | -5633 (9) | 49 |
| C(3') | -472 (8) | -5739 (6) | -6360 (10) | 58 |
| C(4') | 381 (8) | -6270 (7) | -6011 (11) | 75 |
| C(5') | 341 (8) | -6985 (7) | -4993 (12) | 84 |
| C(6') | -561 (9) | -7188 (6) | -4336 (11) | 73 |
| | | | | |
| Compound (II) | | | | |
| Cl(8) | -4307 (1) | -3663 (1) | 5546 (1) | 43 |
| Cl(2') | -4136 (1) | 2423 (1) | 4532 (1) | 64 |
| N(1) | -4063 (1) | -460 (3) | 2944 (1) | 32 |
| N(2) | -4244 (2) | 401 (4) | 1928 (2) | 45 |
| N(3) | -3608 (2) | 274 (4) | 2007 (2) | 45 |
| N(5) | -2805 (2) | 373 (4) | 3530 (2) | 39 |
| N(4") | -6468 (2) | 214 (5) | 2707 (2) | 74 |
| C(1) | -4508 (2) | -42 (4) | 2486 (2) | 35 |
| C(3) | -3511 (2) | -248 (4) | 2611 (2) | 37 |
| C(4) | -2911 (2) | -596 (5) | 2933 (2) | 43 |
| C(6) | -3160 (1) | 172 (4) | 4033 (2) | 31 |
| C(7) | -3756 (2) | -1711 (4) | 4692 (2) | 32 |
| C(8) | -4238 (2) | -2710 (4) | 4781 (2) | 34 |
| C(9) | -4666 (2) | -2978 (4) | 4279 (2) | 40 |
| C(10) | -4600 (2) | -2229 (4) | 3675 (2) | 36 |
| C(11) | -4118 (1) | -1216 (4) | 3573 (2) | 31 |
| C(12) | -3688 (1) | -937 (3) | 4086 (1) | 28 |
| C(1') | -3031 (1) | 1005 (4) | 4674 (2) | 31 |
| C(2') | -3464 (2) | 1947 (4) | 4976 (2) | 37 |
| C(3') | -3383 (2) | 2543 (5) | 5612 (2) | 44 |
| C(4') | -2854 (2) | 2186 (5) | 5962 (2) | 48 |
| C(5') | -2409 (2) | 1278 (5) | 5675 (2) | 48 |
| C(6') | -2494 (2) | 704 (5) | 5030 (2) | 41 |
| C(1") | -5181 (2) | -6 (4) | 2592 (2) | 37 |
| C(2") | -5569 (2) | -568 (5) | 2099 (2) | 51 |
| C(3") | -6198 (2) | -437 (7) | 2180 (2) | 70 |
| C(5") | -6092 (2) | 738 (6) | 3181 (2) | 65 |
| C(6") | -5457 (2) | 648 (5) | 3149 (2) | 50 |

Table 2. *Selected molecular dimensions for compounds* (I) *and* (II)

Values in parentheses are e.s.d.'s. $X(2)$ represents C(2) in (I) and N(2) in (II).

| | (I) | (II) |
|---|---|---|
| (a) Bond lengths (Å) | | |
| Cl(8)-C(8) | 1.727 (9) | 1.742 (3) |
| Cl(2')-C(2') | 1.730 (8) | 1.747 (3) |
| N(1)-C(1) | 1.370 (9) | 1.372 (4) |
| N(1)-C(3) | 1.379 (9) | 1.374 (4) |
| N(1)-C(11) | 1.413 (10) | 1.420 (4) |
| X(2)-N(3) | 1.409 (9) | 1.385 (4) |
| X(2)-C(1) | 1.363 (11) | 1.306 (4) |
| X(2)-C(13) | 1.476 (11) | |
| N(3)-C(3) | 1.303 (9) | 1.302 (4) |
| N(5)-C(4) | 1.470 (8) | 1.478 (5) |
| N(5)-C(6) | 1.280 (8) | 1.271 (4) |
| C(3)-C(4) | 1.482 (10) | 1.475 (5) |
| C(6)-C(12) | 1.488 (10) | 1.502 (4) |
| C(6)-C(1') | 1.508 (10) | 1.495 (4) |
| C(7)-C(8) | 1.377 (10) | 1.372 (5) |
| C(7)-C(12) | 1.413 (10) | 1.391 (4) |
| C(8)-C(9) | 1.372 (11) | 1.379 (5) |
| C(9)-C(10) | 1.377 (11) | 1.376 (5) |
| C(10)-C(11) | 1.390 (10) | 1.383 (4) |
| C(11)-C(12) | 1.372 (10) | 1.399 (4) |
| C(1')-C(2') | 1.406 (9) | 1.385 (5) |
| C(1')-C(6') | 1.375 (10) | 1.382 (5) |
| C(2')-C(3') | 1.375 (10) | 1.379 (5) |
| C(3')-C(4') | 1.384 (10) | 1.372 (5) |
| C(4')-C(5') | 1.360 (10) | 1.372 (6) |
| C(5')-C(6') | 1.378 (11) | 1.389 (5) |
| C(1)-C(1") | | 1.467 (5) |
| C(1")-C(2") | | 1.380 (5) |
| C(1")-C(6") | | 1.382 (5) |
| C(2")-C(3") | | 1.370 (6) |
| C(3")-N(4") | | 1.328 (6) |
| N(4")-C(5") | | 1.325 (6) |
| C(5")-C(6") | | 1.373 (6) |
| | | |
| (b) Bond angles (°) | | |
| C(11)-N(1)-C(3) | 126.0 (9) | 124.0 (3) |
| N(1)-C(3)-C(4) | 119.6 (10) | 121.5 (3) |
| C(3)-C(4)-N(5) | 109.7 (6) | 111.4 (3) |
| C(4)-N(5)-C(6) | 117.6 (7) | 117.2 (3) |
| N(5)-C(6)-C(12) | 127.5 (8) | 127.0 (3) |
| C(6)-C(12)-C(11) | 124.1 (9) | 124.4 (3) |
| C(12)-C(11)-N(1) | 120.2 (9) | 120.2 (3) |
| C(12)-C(11)-C(10) | 121.5 (9) | 120.3 (3) |
| C(12)-C(7)-C(8) | 119.6 (9) | 120.3 (3) |
| C(7)-C(8)-C(9) | 120.1 (9) | 121.5 (3) |
| C(8)-C(9)-C(10) | 121.4 (9) | 118.7 (3) |
| C(9)-C(10)-C(11) | 118.5 (9) | 120.9 (3) |
| C(3)-N(1)-C(1) | 106.0 (8) | 104.5 (3) |
| N(1)-C(1)-X(2) | 106.8 (8) | 109.8 (3) |
| C(1)-X(2)-N(3) | 109.8 (8) | 108.1 (2) |
| X(2)-N(3)-C(3) | 104.5 (7) | 107.0 (3) |
| N(3)-C(3)-N(1) | 112.9 (8) | 110.6 (3) |
| | | |
| (c) Torsion angles (°); e.s.d.'s ca 1.5 (I), 0.5° (II) | | |
| C(11)-N(1)-C(3)-C(4) | -4.2 | -9.3 |
| N(1)-C(3)-C(4)-N(5) | -66.1 | -62.7 |
| C(3)-C(4)-N(5)-C(6) | 66.4 | 66.9 |
| C(4)-N(5)-C(6)-C(12) | 0.9 | -1.0 |
| N(5)-C(6)-C(12)-C(11) | -42.4 | -42.1 |
| C(6)-C(12)-C(11)-N(1) | 0.6 | 1.8 |
| C(12)-C(11)-N(1)-C(3) | 41.0 | 42.5 |
| C(12)-C(6)-C(1')-C(2') | -55.8 | -61.7 |
| N(5)-C(6)-C(1')-C(2') | 126.1 | 123.0 |
| | | |
| Interplanar angles (°) (see text) | | |
| Benzo/phenyl | 77.0 (12) | 77.9 (7) |
| Bow angle | 54.9 (12) | 53.4 (7) |
| Stern angle | 33.0 (12) | 34.3 (7) |

sponding 'classical' benzodiazepine, 7-chloro-5-(2-chlorophenyl)-1,3-dihydro-1-methyl-2*H*-1,4-benzodiazepin-2-one, has a somewhat steeper boat-shaped ring with bow and stern angles of 57.5 and 38.8°, typical for this class of compound (Chananont, Hamor & Martin, 1981). The deviation of the seven-membered ring from an ideal cycloheptatriene boat with mirror symmetry and zero torsion angles about the three double bonds in terms of the 'deviation parameter', $\Delta$, defined in Hamor & Martin (1983), is 2.1° for (I) and 4.6° for (II). The corresponding values for the three benzodiazepines cited above are similar, 2.2, 3.2 and 8.2°, respectively.

In both (I) and (II) the five-membered heterocyclic ring and the two aromatic rings are each planar to within the limits of experimental error. The phenyl ring is steeply inclined to the plane of the fused benzo moiety; the interplanar angles (77 and 78° respectively) are in the expected range (73–86°) for 1,4-benzodiazepines with 2'-chloro-substituted phenyl rings. The C(6)—C(1') bond lengths (see Table 2)
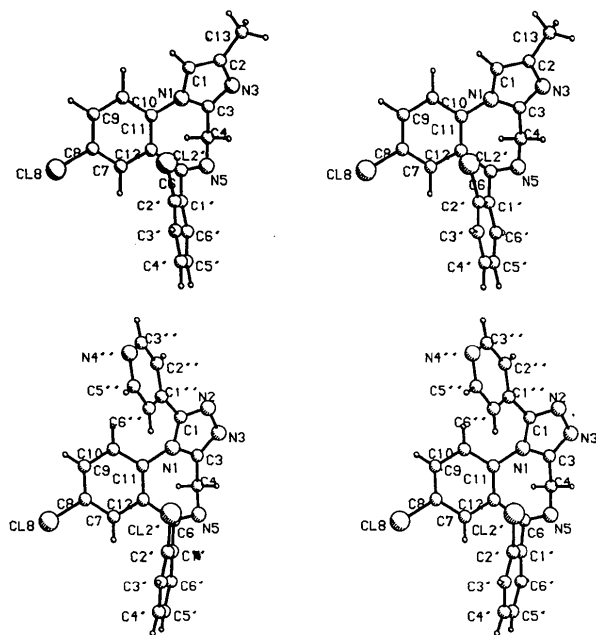
non-bonded intramolecular interactions, torsion angle N(1)—C(1)—C(1″)—C(2″) −136·4 (6)°. The C(1)—C(1″) bond, although apparently slightly shorter than C(6)—C(1′), also corresponds in length to a $Csp^2$—$Csp^2$ single bond to within the limits of experimental accuracy.

In both structures intermolecular contacts are of the normal van der Waals type.

Fig. 1. Stereoscopic view of molecule (I) (upper diagram) and (II) (lower diagram) in a direction perpendicular to the mean plane of atoms C(7)–C(12).

correspond to single bonds between $sp^2$-hybridized C atoms, thus excluding the possibility of any significant conjugation between the phenyl ring and the N(5)—C(6) double bond.

In compound (II) the pyridyl ring is oriented about the C(1)—C(1″) bond in such a way as to minimize

**References**

BUTCHER, H. J. & HAMOR, T. A. (1984). *Acta Cryst.* C40, 848–850.
CHANANONT, P., HAMOR, T. A. & MARTIN, I. L. (1981). *Acta Cryst.* B37, 1371–1375.
GALL, M. & KAMDAR, B. V. (1981). *J. Org. Chem.* 46, 1575–1585.
HAMOR, T. A. & MARTIN, I. L. (1983). *Progress in Medicinal Chemistry*, Vol. 20, edited by G. P. ELLIS & G. B. WEST, pp. 157–223. Amsterdam: Elsevier.
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
KEMMISH, H. J. & HAMOR, T. A. (1988). *Acta Cryst.* C44, 1823–1825.
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.
SZMUSZKOVICZ, J. (1972). German patent 2 222 068; *Chem. Abstr.* (1973). 78, 72240m.

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# Structure of 17α-Hydroxyprogesterone Caproate

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**Abstract.** 3,20-Dioxo-4-pregnen-17-yl hexanoate, $C_{27}H_{40}O_4$, $M_r = 428·61$, orthorhombic, $P2_12_12_1$, $a = 14·155$ (2), $b = 23·517$ (7), $c = 7·466$ (1) Å, $V = 2485·1$ (8) Å$^3$, $Z = 4$, $D_m = 1·14$, $D_x = 1·146$ Mg m$^{-3}$, $\lambda(Cu\ K\alpha) = 1·54178$ Å, $\mu = 5·59$ cm$^{-1}$, $F(000) = 936$, $R = 0·062$ for the 1263 observed $[I > 3\sigma(I)]$ reflections. Both rings $B$ and $C$ are in the chair conformation. Ring $D$ assumes the form between half-chair

   