Refinement

Refinement on F
$R = 0.064$
$wR = 0.088$
$S = 0.7885$
1839 reflections
266 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.84$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
N(1*a*)	0.3322 (4)	0.2882 (6)	0.4444 (4)	0.071 (3)
C(1)	0.2810 (5)	0.2674 (8)	0.3517 (6)	0.074 (4)
O(1)	0.2167 (5)	0.2072 (8)	0.3402 (5)	0.112 (5)
C(2)	0.3049 (6)	0.3184 (9)	0.2591 (6)	0.082 (4)
N(2)	0.3693 (4)	0.3937 (6)	0.2670 (4)	0.067 (3)
C(3)	0.2622 (8)	0.291 (1)	0.1579 (7)	0.126 (8)
S(3)	0.3069 (2)	0.3631	0.0711 (2)	0.140 (2)
C(4)	0.3789 (5)	0.4252 (8)	0.1749 (5)	0.072 (4)
C(5)	0.4435 (5)	0.5077 (7)	0.1573 (5)	0.074 (4)
C(6)	0.4158 (6)	0.6246 (9)	0.1778 (6)	0.093 (5)
C(7)	0.328 (1)	0.649 (1)	0.105 (1)	0.15 (1)
C(8)	0.488 (1)	0.706 (1)	0.167 (1)	0.14 (1)
N(2*a*)	0.5273 (4)	0.4808 (7)	0.2268 (4)	0.070 (3)
C(9)	0.6007 (5)	0.4805 (7)	0.1958 (5)	0.075 (4)
O(9)	0.6087 (4)	0.5015 (8)	0.1046 (4)	0.106 (4)
C(10)	0.6829 (5)	0.4610 (7)	0.2792 (5)	0.073 (4)
N(10)	0.6629 (4)	0.4105 (6)	0.3745 (4)	0.065 (3)
C(11)	0.7450 (5)	0.383 (1)	0.2435 (6)	0.094 (5)
O(11)	0.7419 (4)	0.2888 (7)	0.3095 (4)	0.093 (3)
C(12)†	0.839 (1)	0.431 (2)	0.275 (2)	0.12 (1)
C(13)	0.6938 (4)	0.3191 (7)	0.3820 (5)	0.062 (3)
C(14)	0.6874 (5)	0.2352 (7)	0.4635 (5)	0.071 (4)
C(15)	0.6214 (6)	0.1470 (8)	0.4189 (8)	0.090 (5)
C(16)	0.6581 (9)	0.064 (1)	0.361 (2)	0.14 (1)
C(17)	0.604 (1)	−0.039 (2)	0.344 (3)	0.19 (2)
C(18)	0.5332 (7)	0.193 (1)	0.362 (1)	0.105 (6)
O(1)*W*	0.5000	0.4327 (8)	0.5000	0.085 (5)
C(1)Me	0.5000	0.6162 (9)	0.5000	0.104 (7)
O(1*a*)Me‡	0.593 (1)	0.617 (2)	0.515 (2)	0.10 (1)
O(1*b*)Me‡	0.552 (3)	0.718 (2)	0.494 (4)	0.20 (3)

† Occupancy 0.5.
‡ Occupancy 0.25.

Table 2. *Selected torsion angles* (°)

Thiazole	
N(1*a*)—C(1)—C(2)—N(2)	−7.9 (7)
C(14′)—N(1*a*)—C(1)—C(2)	−177.2 (7)
Valine	
C(9)—N(2*a*)—C(5)—C(4)	134.6 (8)
N(2*a*)—C(5)—C(4)—N(2)	47.0 (6)
N(2*a*)—C(5)—C(6)—C(7)	−179.0 (1)
N(2*a*)—C(5)—C(6)—C(8)	55.0 (9)
Oxazoline	
N(10)—C(10)—C(9)—N(2*a*)	18.8 (6)
C(10)—C(9)—N(2*a*)—C(5)	174.5 (9)
Isoleucine	
C(1′)—N(1′*a*)—C(14)—C(13)	−127.1 (7)
N(1′*a*)—C(14)—C(13)—N(10)	−21.2 (6)
N(1′*a*)—C(14)—C(15)—C(16)	−154.0 (1)
N(1′*a*)—C(14)—C(15)—C(18)	72.9 (8)
C(14)—C(15)—C(16)—C(17)	163.0 (2)

Crystals were obtained from aqueous methanol solution by slow evaporation at room temperature. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). Refinement was by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). During the last stage of the refinement, all H atoms, except for those of the solvents, were placed in assumed positions and included only for the calculation of structure factors. The y coordinate of S(3) was fixed to define the origin during refinement. The molecular conformation was depicted using *ORTEP* (Johnson, 1971).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry and a stereoscopic drawing of crystal packing, viewed down the b axis, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71549 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1063]

References

In, Y., Doi, M., Inoue, M., Ishida, T., Hamada, Y. & Shioiri, T. (1993). *Chem. Pharm. Bull.* **41**, 1686–1690.

Ireland, C. M., Durso, A. R. Jr, Newman, R. A. & Hacker, M. P. (1982). *J. Org. Chem.* **47**, 1807–1811.

Ishida, T., In, Y., Doi, M., Inoue, M., Hamada, Y. & Shioiri, T. (1992). *Biopolymers*, **32**, 131–143.

Ishida, T., Tanaka, M., Nabae, M., Inoue, M., Kato, S., Hamada, Y. & Shioiri, T. (1988). *J. Org. Chem.* **53**, 107–112.

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3974, revised. Oak Ridge National Laboratory, Tennessee, USA.

Schmitz, F. J., Ksebati, M. B., Chang, J. S., Wang, J. L., Hossain, M. B., van der Helm, D., Engel, M. H., Serban, A. & Silfer, J. A. (1989). *J. Org. Chem.* **54**, 3463–3472.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Acta Cryst. (1994). **C50**, 434–438

A Steroid Derivative that Crystallizes with Three Molecules in the Asymmetric Unit

CAROLYN PRATT BROCK, IVAN STOILOV AND
DAVID S. WATT

Department of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA

(*Received* 13 *January* 1993; *accepted* 31 *August* 1993)

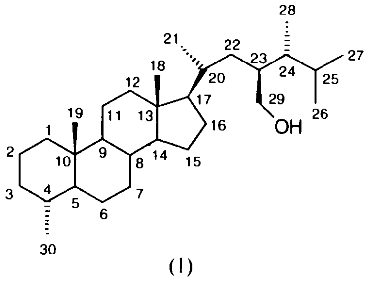
Abstract

Crystals of (20*R*,23*S*,24*R*)-5α-dinosteran-29-ol, $C_{30}H_{54}O$, grow as blades elongated in the direction of hydrogen-bonded chains of OH groups and thin in the direction of the 50 Å c axis. There are three independent molecules in the unit cell. Hydrogen-bonding requirements dictate that the OH group of each molecule be within a few ångströms

of two other OH groups, but the bulk and shape of the rest of the molecule preclude the three being related by crystallographic symmetry elements. The local symmetry of the hydrogen-bonded chains is approximately 3_1, but the only crystallographic symmetry parallel to the chains is translation. A survey of the Cambridge Structural Database shows that hydrocarbons with a single OH substituent commonly crystallize with more than one molecule in the asymmetric unit, or in space groups with three- or fourfold axes.

Comment

The structure was determined in order to confirm the stereochemistry at C20, C23 and C24 (see scheme below). What was expected to be a routine structure determination was complicated greatly by the presence of three independent molecules in the asymmetric unit and by the poor quality of the crystals that could be grown.



(I)

Bond lengths and angles are normal (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) to within the precision of the determination. The three independent molecules differ in the conformation of the alkyl chain (atoms C23–C29 and the OH group; see Fig. 1). The molecules are slightly bowed; the torsion angles C19—C10· · ·C13—C18 are 0.09 (11), 4.0 (12) and 2.6 (12)° for molecules (I), (II) and (III), respectively.

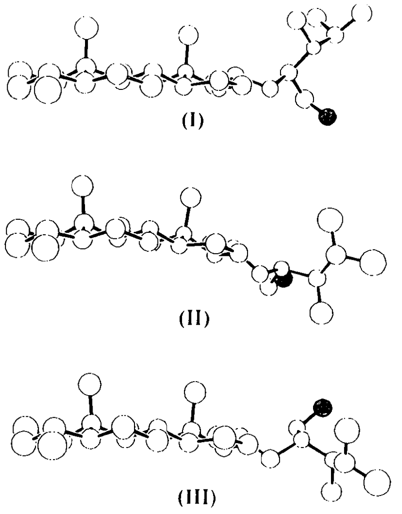


Fig. 1. Perspective drawing of the three independent molecules of (20*R*,23*S*,24*R*)-5α-dinosteran-29-ol. The shapes of the spheres correspond to 50% probability contours of atomic displacement; the H atoms have been omitted. Molecule (I) containing atoms C101–C130 is on top and the molecule (III) containing atoms C301–C330 is at the bottom. In this and the following figures, the O atoms have been shaded.

Since the bond lengths and angles could not be determined with the usual precision, it is fortunate that the most interesting aspect of the structure is the crystal packing. Only about 0.5% of the structures in the Cambridge Structural Database (CSD; Allen, Kennard & Taylor, 1983) have three or more molecules in the asymmetric unit (Brock & Dunitz, 1992). Although the increased difficulty in solving large structures may lower this frequency artificially, it seems likely that structural motifs composed of several independent molecules are indeed unusual. In this case the large *Z* may be a consequence of the relative sizes of the single functional group and the rest of the rather rigid molecule. In the crystal the OH group is expected to be part of a chain or ring (Etter, 1991), but the bulkiness of the substituents on C23 and the overall molecular shape preclude the formation of simple aggregates around screw or rotation axes. The closely related molecule cholesterol, $C_{27}H_{25}OH$, crystallizes in space group *P*1 with 16 molecules in the asymmetric unit (Shieh, Hoard & Nordman, 1977, 1981). Even the various solvates of cholesterol have large ($\geq$ 4) values of Z' (Shieh, Hoard & Nordman, 1981; Craven, 1986). The prevalence of large Z' values in cholesterol structures has been rationalized by Craven (1986) who cites the conflict between the drive towards optimal O· · ·O distances (each hydroxyl O atom within 2.5–2.9 Å of two other O atoms) and the drive towards optimal stacking of the ring systems (resulting in O· · ·O distances of *ca* 6.1 Å).

A search of the July 1991 version of the CSD for molecules of the type C_nH_mOH having $R \leq 0.070$, *SIGF* = 1 or 2 and $Z' \geq 1$ yielded 45 structures. Of these, eight (18%) are in trigonal or tetragonal space groups; the corresponding percentage for all structures meeting the same *R*, *SIGF* and Z' criteria is only 1.4% (Brock & Dunitz, 1992). Of the 37 structures in triclinic, monoclinic and orthorhombic space groups, 21 (57%) have $Z' > 1$, while only 8% of all structures in these three systems have $Z' > 1$. If the *SIGF* criterion is dropped and structures with $R \leq 0.120$ are included, the total number of alcohol structures rises to 99, but the percentages (16 and 53%) change very little.

The three independent molecules form hydrogen-bonded chains parallel to *a*, which is both the shortest of the crystal axes and the direction of fastest crystal growth (see Figs. 2 and 3). The local symmetry in the vicinity of the OH chains is approximately 3_1, but that symmetry cannot be crystallographic because it is impossible to fill one third of a cylinder (arc length $2\pi/3$) with the rest of the molecule (or parts thereof).

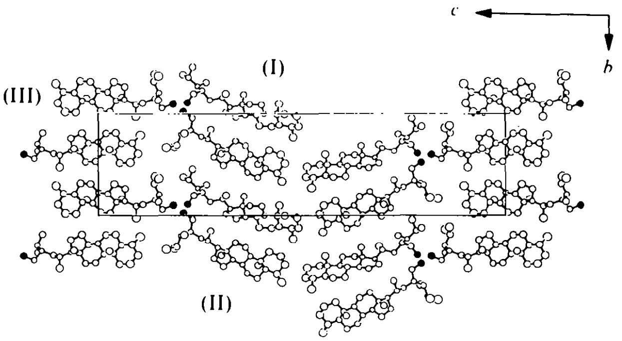


Fig. 2. Perspective drawing of the unit cell of (20*R*,23*S*,24*R*)-5α-dinosteran-29-ol. The hydrogen-bonded chains are located at approximately $y = \frac{1}{2}$, $z = \frac{1}{4}$ and $y = 0$, $z = \frac{3}{4}$. Molecules (I) and (II) alternate in the two columns on either side of the screw axes at $z = \frac{1}{2}$. The columns centered around the screw axes at $z = 0$ are composed exclusively of molecule (III).

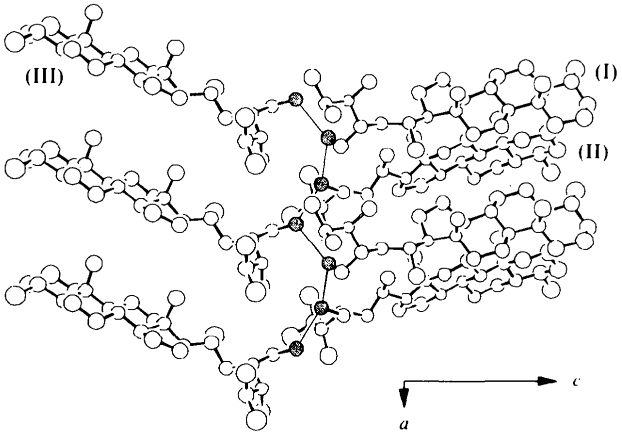


Fig. 3. Perspective drawing of one of the hydrogen-bonded chains in the unit cell of (20*R*,23*S*,24*R*)-5α-dinosteran-29-ol. The three molecules to the left of the chain of OH groups are molecule (III); to the right of the chain molecules (I) and (II) alternate.

Experimental

The compound was obtained as described in the literature (Stoilov, Kolaczkowska, St Pyrek, Brock, Watt, Carlson & Moldowan, 1992). Crystals of (20*R*,23*S*,24*R*)-5α-dinosteran-29-ol grow as very thin plates; the best crystals obtained (< 0.1 mm thick) were grown by slow evaporation from CH_2Cl_2/MeOH solutions.

Crystal data

$C_{30}H_{54}O$
$M_r = 430.76$
Monoclinic
$P2_1$
$a = 6.6289\ (5)$ Å
$b = 12.5448\ (14)$ Å
$c = 49.810\ (4)$ Å
$\beta = 90.068\ (6)^\circ$
$V = 4142.1\ (7)$ Å^3
$Z = 6$
$D_x = 1.036$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 22 reflections
$\theta = 8.2$–10.3°
$\mu = 0.056$ mm^{-1}
$T = 294\ (1)$ K
Very thin blades; the plate normal is c^* and the long direction is **a**
0.5 × 0.5 × 0.1 mm
Colorless

Data collection

Enraf-Nonius CAD-4-VAX diffractometer
ω scans; stationary background measurements
Absorption correction: none
7618 measured reflections
7618 independent reflections

2377 observed reflections $[I > 3\sigma(I)]$
$\theta_{\max} = 25.0^\circ$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 14$
$l = -59 \rightarrow 59$
3 standard reflections frequency: 60 min intensity variation: 1.9%

Refinement

Refinement on F
$R = 0.075$
$wR = 0.068$
$S = 2.62$
2377 reflections
372 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.4.6A)
Absolute configuration: chosen to correspond with that expected from the synthetic pathway

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

	x	y	z	B_{iso}
O101	0.3319 (11)	−0.1090	0.21766 (14)	4.3 (2)
O201	0.6914 (12)	−0.0224 (9)	0.20817 (16)	4.7 (2)
O301	0.0146 (12)	−0.0977 (9)	0.18306 (16)	4.9 (2)
C101	0.212 (2)	0.0019 (14)	0.4570 (3)	6.3 (4)
C102	0.156 (2)	0.0205 (16)	0.4873 (3)	7.5 (5)
C103	0.039 (2)	0.1191 (16)	0.4908 (3)	6.9 (4)
C104	−0.150 (2)	0.1246 (14)	0.4736 (3)	5.5 (4)
C105	−0.086 (2)	0.1072 (14)	0.4434 (3)	5.2 (4)
C106	−0.266 (2)	0.1237 (13)	0.4250 (3)	5.7 (4)
C107	−0.197 (2)	0.1154 (14)	0.3949 (3)	6.1 (4)
C108	−0.086 (2)	0.0150 (13)	0.3889 (2)	4.2 (3)
C109	0.095 (2)	0.0004 (13)	0.4077 (2)	3.9 (3)
C110	0.027 (2)	0.0033 (14)	0.4390 (3)	5.0 (4)
C111	0.233 (2)	−0.0874 (13)	0.4013 (2)	4.9 (4)
C112	0.295 (2)	−0.0915 (13)	0.3712 (2)	4.8 (3)
C113	0.110 (2)	−0.0881 (13)	0.3530 (2)	3.4 (3)
C114	−0.003 (2)	0.0146 (13)	0.3599 (2)	3.9 (3)
C115	−0.159 (2)	0.0304 (13)	0.3375 (3)	4.9 (4)
C116	−0.047 (2)	−0.0150 (13)	0.3128 (3)	4.9 (3)
C117	0.153 (2)	−0.0662 (12)	0.3225 (2)	3.1 (3)
C118	−0.019 (2)	−0.1847 (14)	0.3559 (2)	5.1 (4)
C119	−0.106 (2)	−0.0963 (16)	0.4439 (3)	6.8 (4)
C120	0.216 (2)	−0.1605 (12)	0.3047 (2)	3.9 (3)
C121	0.433 (2)	−0.1971 (15)	0.3125 (3)	6.5 (4)
C122	0.214 (2)	−0.1331 (12)	0.2743 (2)	3.8 (3)
C123	0.247 (2)	−0.2270 (12)	0.2550 (2)	4.2 (3)
C124	0.049 (2)	−0.2677 (12)	0.2427 (2)	3.1 (3)
C125	0.080 (2)	−0.3520 (12)	0.2204 (2)	4.2 (3)
C126	0.177 (2)	−0.4559 (14)	0.2305 (3)	5.8 (4)
C127	−0.124 (2)	−0.3793 (14)	0.2072 (3)	6.1 (4)
C128	−0.098 (2)	−0.3034 (13)	0.2649 (2)	5.0 (4)
C129	0.402 (2)	−0.1954 (13)	0.2334 (3)	5.2 (4)
C130	−0.264 (2)	0.2288 (17)	0.4777 (3)	8.3 (5)
C201	0.300 (2)	0.3605 (14)	0.4198 (3)	6.6 (4)
C202	0.233 (2)	0.4049 (15)	0.4478 (3)	7.0 (4)
C203	0.376 (2)	0.4886 (16)	0.4577 (3)	7.2 (4)
C204	0.400 (2)	0.5834 (15)	0.4384 (3)	6.2 (4)
C205	0.468 (2)	0.5374 (13)	0.4109 (3)	4.4 (3)
C206	0.512 (2)	0.6268 (13)	0.3906 (3)	4.8 (4)
C207	0.603 (2)	0.5776 (13)	0.3650 (2)	4.3 (3)
C208	0.476 (2)	0.4919 (13)	0.3529 (2)	3.6 (3)

C209	0.426 (2)	0.4054 (13)	0.3733 (2)	3.9 (3)
C210	0.321 (2)	0.4540 (13)	0.3989 (3)	4.8 (4)
C211	0.304 (2)	0.3160 (13)	0.3606 (3)	5.2 (4)
C212	0.417 (2)	0.2650 (13)	0.3360 (3)	4.4 (3)
C213	0.467 (2)	0.3505 (12)	0.3147 (2)	3.4 (3)
C214	0.584 (2)	0.4366 (12)	0.3298 (2)	3.4 (3)
C215	0.670 (2)	0.5057 (14)	0.3070 (3)	5.3 (4)
C216	0.715 (2)	0.4267 (14)	0.2840 (2)	4.7 (4)
C217	0.625 (2)	0.3169 (12)	0.2932 (2)	3.4 (3)
C218	0.276 (2)	0.3935 (14)	0.3016 (3)	5.5 (4)
C219	0.115 (2)	0.4965 (14)	0.3927 (3)	5.9 (4)
C220	0.567 (2)	0.2466 (12)	0.2686 (2)	3.7 (3)
C221	0.446 (2)	0.1498 (13)	0.2760 (3)	5.3 (4)
C222	0.760 (2)	0.2230 (12)	0.2524 (2)	4.1 (3)
C223	0.726 (2)	0.1578 (12)	0.2258 (2)	3.5 (3)
C224	0.866 (2)	0.1970 (13)	0.2032 (2)	4.5 (4)
C225	0.813 (2)	0.3091 (14)	0.1920 (3)	5.5 (4)
C226	0.935 (2)	0.3343 (16)	0.1670 (3)	9.5 (5)
C227	0.593 (3)	0.3255 (16)	0.1869 (3)	8.8 (5)
C228	1.086 (2)	0.1902 (15)	0.2104 (3)	7.4 (5)
C229	0.756 (2)	0.0406 (14)	0.2306 (3)	4.5 (3)
C230	0.548 (2)	0.6639 (16)	0.4498 (3)	8.2 (5)
C301	−0.452 (2)	−0.0156 (14)	−0.0533 (3)	6.2 (4)
C302	−0.587 (2)	−0.0207 (16)	−0.0790 (3)	8.1 (5)
C303	−0.505 (2)	−0.1069 (17)	−0.0978 (3)	7.4 (4)
C304	−0.505 (2)	−0.2142 (15)	−0.0838 (3)	5.9 (4)
C305	−0.374 (2)	−0.2074 (14)	−0.0573 (3)	4.8 (4)
C306	−0.354 (2)	−0.3149 (15)	−0.0431 (3)	6.5 (4)
C307	−0.209 (2)	−0.3093 (14)	−0.0191 (3)	6.1 (4)
C308	−0.277 (2)	−0.2235 (13)	0.0004 (2)	4.1 (3)
C309	−0.305 (2)	−0.1164 (14)	−0.0138 (2)	4.4 (3)
C310	−0.455 (2)	−0.1236 (13)	−0.0382 (3)	4.7 (3)
C311	−0.355 (2)	−0.0291 (13)	0.0063 (3)	5.2 (4)
C312	−0.210 (2)	−0.0225 (14)	0.0299 (3)	5.1 (4)
C313	−0.188 (2)	−0.1276 (13)	0.0446 (2)	4.3 (3)
C314	−0.128 (2)	−0.2101 (13)	0.0235 (3)	4.7 (4)
C315	−0.067 (2)	−0.3090 (15)	0.0392 (3)	6.7 (4)
C316	0.025 (2)	−0.2620 (14)	0.0656 (3)	5.6 (4)
C317	−0.013 (2)	−0.1384 (13)	0.0648 (2)	3.8 (3)
C318	−0.389 (2)	−0.1572 (14)	0.0581 (3)	6.1 (4)
C319	−0.671 (2)	−0.1475 (14)	−0.0284 (3)	6.2 (4)
C320	−0.033 (2)	−0.0917 (14)	0.0930 (2)	4.5 (3)
C321	−0.088 (2)	0.0241 (16)	0.0930 (3)	7.8 (5)
C322	0.166 (2)	−0.1072 (13)	0.1090 (2)	4.9 (3)
C323	0.138 (2)	−0.1505 (12)	0.1383 (2)	3.7 (3)
C324	0.331 (2)	−0.2033 (13)	0.1492 (2)	3.8 (3)
C325	0.368 (2)	−0.3149 (15)	0.1364 (3)	5.7 (4)
C326	0.195 (3)	−0.3873 (16)	0.1381 (3)	8.4 (5)
C327	0.559 (3)	−0.3648 (17)	0.1480 (3)	9.4 (5)
C328	0.513 (2)	−0.1320 (14)	0.1476 (3)	5.7 (4)
C329	0.064 (2)	−0.0565 (12)	0.1569 (2)	4.1 (3)
C330	−0.415 (2)	−0.2931 (17)	−0.1030 (3)	9.2 (5)

Table 2. *Selected geometric parameters* (Å, °)

For molecules (I), (II) and (III), numbers in atom labels should be preceded by 1, 2 and 3, respectively.

	(I)	(II)	(III)
O01—C29	1.418 (14)	1.433 (14)	1.439 (12)
C01—C02	1.572 (18)	1.564 (17)	1.565 (19)
C01—C10	1.523 (16)	1.575 (18)	1.550 (18)
C02—C03	1.471 (20)	1.497 (19)	1.532 (20)
C03—C04	1.519 (17)	1.536 (19)	1.516 (20)
C04—C05	1.580 (16)	1.553 (17)	1.582 (17)
C04—C30	1.524 (20)	1.514 (19)	1.501 (19)
C05—C06	1.519 (16)	1.540 (17)	1.529 (19)
C05—C10	1.518 (17)	1.547 (17)	1.517 (16)
C06—C07	1.571 (16)	1.540 (15)	1.534 (16)
C07—C08	1.490 (17)	1.491 (16)	1.516 (17)
C08—C09	1.529 (15)	1.525 (16)	1.529 (17)
C08—C14	1.544 (15)	1.523 (15)	1.529 (17)
C09—C10	1.624 (15)	1.576 (16)	1.570 (15)
C09—C11	1.466 (16)	1.519 (17)	1.520 (17)
C10—C19	1.549 (18)	1.495 (16)	1.541 (16)
C11—C12	1.553 (15)	1.568 (16)	1.519 (17)
C12—C13	1.523 (15)	1.548 (16)	1.515 (17)
C13—C14	1.530 (16)	1.526 (15)	1.529 (16)
C13—C17	1.571 (14)	1.552 (14)	1.543 (15)
C13—C18	1.491 (16)	1.522 (15)	1.538 (15)
C14—C15	1.536 (15)	1.537 (16)	1.521 (18)
C15—C16	1.546 (15)	1.544 (17)	1.561 (17)
C16—C17	1.550 (15)	1.568 (16)	1.572 (17)
C17—C20	1.537 (15)	1.557 (15)	1.526 (15)
C20—C21	1.557 (16)	1.499 (16)	1.498 (20)
C20—C22	1.550 (14)	1.544 (14)	1.554 (15)
C22—C23	1.540 (16)	1.575 (15)	1.571 (15)
C23—C24	1.536 (15)	1.541 (15)	1.538 (15)
C23—C29	1.539 (15)	1.504 (17)	1.578 (16)
C24—C28	1.541 (15)	1.506 (17)	1.506 (16)
C24—C25	1.549 (16)	1.553 (18)	1.560 (17)
C25—C26	1.538 (18)	1.519 (18)	1.469 (19)
C25—C27	1.541 (16)	1.495 (17)	1.521 (19)
C02—C01—C10	111.8 (12)	110.3 (13)	110.7 (13)
C01—C02—C03	111.4 (14)	111.2 (13)	108.9 (14)
C02—C03—C04	113.9 (14)	113.9 (14)	110.3 (13)
C03—C04—C05	108.1 (12)	107.1 (13)	109.6 (13)
C03—C04—C30	111.8 (14)	110.6 (13)	107.0 (13)
C05—C04—C30	112.4 (13)	113.0 (13)	110.3 (13)
C04—C05—C06	110.1 (12)	111.4 (12)	112.7 (13)
C04—C05—C10	112.9 (12)	114.2 (12)	111.6 (12)
C06—C05—C10	114.6 (13)	111.1 (11)	110.6 (11)
C05—C06—C07	109.6 (11)	109.2 (12)	112.0 (13)
C06—C07—C08	113.1 (13)	113.7 (11)	110.2 (13)
C07—C08—C09	111.4 (12)	111.6 (11)	111.4 (11)
C07—C08—C14	111.5 (12)	111.6 (11)	111.6 (12)
C09—C08—C14	107.0 (10)	106.5 (11)	109.3 (12)
C08—C09—C10	111.5 (11)	111.2 (12)	112.6 (12)
C08—C09—C11	116.3 (11)	111.2 (10)	110.7 (11)
C10—C09—C11	113.7 (12)	113.0 (11)	114.6 (13)
C01—C10—C05	108.8 (13)	107.7 (11)	107.2 (11)
C01—C10—C09	109.9 (11)	106.6 (12)	108.4 (13)
C01—C10—C19	111.0 (13)	108.9 (12)	109.8 (12)
C05—C10—C09	107.1 (12)	107.2 (11)	107.6 (12)
C05—C10—C19	112.9 (11)	114.3 (12)	113.1 (12)
C09—C10—C19	107.0 (11)	111.9 (11)	110.6 (11)
C09—C11—C12	113.8 (11)	112.1 (11)	114.5 (13)
C11—C12—C13	111.1 (10)	110.9 (11)	112.6 (13)
C12—C13—C14	106.4 (11)	105.2 (10)	106.3 (11)
C12—C13—C17	116.0 (10)	115.6 (11)	117.4 (12)
C12—C13—C18	112.3 (12)	111.0 (11)	109.9 (12)
C14—C13—C17	99.3 (11)	101.0 (10)	100.9 (11)
C14—C13—C18	112.4 (10)	112.4 (11)	111.4 (12)
C17—C13—C18	109.8 (11)	111.2 (10)	110.5 (11)
C08—C14—C13	112.9 (11)	117.2 (10)	115.0 (12)
C08—C14—C15	116.0 (11)	118.4 (11)	118.0 (13)
C13—C14—C15	105.9 (11)	102.9 (10)	105.6 (11)
C14—C15—C16	101.9 (11)	104.8 (12)	103.1 (13)
C15—C16—C17	108.6 (11)	105.9 (10)	106.9 (12)
C13—C17—C16	102.6 (10)	102.6 (10)	102.7 (11)
C13—C17—C20	118.2 (11)	122.0 (10)	119.7 (11)
C16—C17—C20	112.0 (10)	111.2 (10)	111.8 (12)
C17—C20—C21	109.5 (11)	113.5 (10)	113.2 (12)
C17—C20—C22	112.8 (11)	108.5 (10)	110.2 (11)
C21—C20—C22	108.6 (10)	114.5 (11)	109.1 (13)
C20—C22—C23	116.1 (11)	115.0 (10)	114.8 (10)
C22—C23—C24	112.2 (10)	111.3 (11)	112.2 (10)
C22—C23—C29	109.7 (11)	110.7 (11)	109.0 (10)
C24—C23—C29	112.4 (10)	110.5 (11)	111.8 (10)
C23—C24—C25	113.4 (10)	114.4 (12)	112.0 (11)
C23—C24—C28	110.7 (10)	113.1 (12)	113.2 (12)
C25—C24—C28	113.6 (11)	110.9 (13)	112.4 (12)
C24—C25—C26	113.6 (11)	111.2 (13)	113.9 (13)
C24—C25—C27	110.0 (11)	114.0 (14)	110.3 (14)
C26—C25—C27	108.6 (12)	110.5 (13)	111.9 (15)
O01—C29—C23	111.3 (11)	112.0 (11)	109.5 (10)

Table 3. *Contact distances* (Å)

O101···O201	2.661 (11)	O201···O301$^{\rm i}$	2.657 (11)
O101···O301	2.721 (11)		

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

The crystal mounted for data collection was covered with epoxy to prevent sublimation. The Laue symmetry of the crystal was clearly $2/m$ even though the value of β is very near 90°. The fraction of reflections having $I > 3\sigma(I)$ (2377/7618 = 0.31) was low, even though reflection intensities were measured with 100 s scans (scan width 0.8° ω plus allowance for $K\alpha_1-K\alpha_2$ split), with an additional 10 s at each end of the scan for a background measurement. Only 299 reflections had $2\sigma(I) < I < 3\sigma(I)$. Even though one of the axes was 50 Å long, there were no obvious problems with reflection overlap.

The structure proved difficult to solve even though the cell dimensions put clear limits on the possible positions and orientations of the three independent molecules. The solution was eventually found with the 1983 version of the program package *DIRDIF* (Beurskens *et al.*, 1983) after numerous possible orientations and shift vectors had been examined. Later experiments, however, showed that the newest version of *DIRDIF* finds the solution nearly automatically (Beurskens, 1992). In each case the structure solution started from a 21-atom fragment [C(01)-C(20), C(30)] of a similar molecule found by searching the CSD. The absolute configuration was chosen to correspond with that expected from the synthetic pathway. After isotropic refinement, coordinates for all H atoms connected to C atoms were calculated on the basis of the expected stereochemistry ($R_{\rm C-H}$ = 1.00 Å) and the H atoms were included in the refinement as fixed contributions (B = 1 Å^2 larger than that of the attached C atom). The H atoms of the hydroxyl groups could not be located in difference Fourier syntheses with sufficient certainty to justify their inclusion. The relatively low number of reflections with measurable intensity precluded anisotropic refinement. Before the final cycles, coordinates for all H atoms were re-idealized. After refinement was complete, $(I_{\rm obs}-I_{\rm calc})/\sigma(I)$ was calculated for the 5241 reflections having $I < 3\sigma(I)$, which therefore were omitted from the refinement. The average value of -0.4 suggests a minor overlap problem; for the 55 reflections that had $|(I_{\rm obs}-I_{\rm calc})/\sigma(I)| > 3$, all had $I_{\rm obs} < I_{\rm calc}$. Only 3 reflections had $|(I_{\rm obs}-I_{\rm calc})/\sigma(I)| > 5$; an additional 11 had $|(I_{\rm obs}-I_{\rm calc})/\sigma(I)| > 4$.

Data collection: *CAD*-4-*VAX* (Enraf-Nonius, 1988). Cell refinement: *CAD*-4-*VAX*. Data reduction: local program (Brock). Program(s) used to solve structure: *DIRDIF*. Program(s) used to refine structure: local program [based on Ibers' least-squares program, which is in turn based on *ORFLS* (Busing, Martin & Levy, 1962)]. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *ORFFE* (Busing, Martin & Levy, 1964); local program (Brock).

We acknowledge the support of the University of Kentucky Major Research Instrumentation Bond Program in the purchase of equipment used in this study (bond ID No. 7E-8E48-25).

Lists of structure factors, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71604 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1061]

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146-153.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1-S19.

Beurskens, P. T. (1992). Personal communication.

Beurskens, P. T., Bosman, W. P., Doesburg, H. M., Gould, R. O., van den Hark, Th. E. M., Prick, P. A., Noordik, J. H., Beurskens, G., Parthasarthi, V., Bruins Slot, H. J. & Haltiwanger, R. C. (1983). *DIRDIF*. Technical Report 1983/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Brock, C. P. & Dunitz, J. D. (1992). Unpublished results.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

Craven, B. M. (1986). *The Physical Chemistry of Lipids*, edited by D. M. Small, pp. 149-182. New York: Plenum.

Enraf-Nonius (1988). *CAD-4 Manual*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Etter, M. C. (1991). *J. Phys. Chem.* **95**, 4601-4610.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Shieh, H.-S., Hoard, L. G. & Nordman, C. E. (1977). *Nature (London)*, **267**, 287-289.

Shieh, H.-S., Hoard, L. G. & Nordman, C. E. (1981). *Acta Cryst.* B**37**, 1538-1543.

Stoilov, I., Kolaczkowska, E., St Pyrek, J., Brock, C. P., Watt, D. S., Carlson, R. M. K. & Moldowan, J. M. (1992). *Tetrahedron Lett.* **33**, 7689-7692.

Acta Cryst. (1994). C**50**, 438–442

meso- and (±)-1,2-Dicyano-1,2-diphenylethane

L. L. Koh, Yan Xu, K. Y. Sim, E. Liang and
H. H. Huang*

Department of Chemistry, National University of Singapore, Lower Kent Ridge Road, Singapore 0511

(*Received* 4 *March* 1993; *accepted* 2 *August* 1993)

Abstract

Of the title compounds, (I) *meso*- and (II) (±)-2,3-diphenylbutanedinitrile, $C_{16}H_{12}N_2$, (I) adopts the *trans* conformation with disorder in the inner part of the molecule, whereas in (II), the two CN groups are *gauche* and the phenyl groups *trans*.

Comment

We have reported previously the crystal structures of *meso*- and (±)-2,3-dicyano-2,3-diphenylbutane and *meso*- and (±)-3,4-dicyano-3,4-diphenylhexane (Lam, Koh & Huang, 1991). This paper is concerned with the structures of *meso*-1,2-dicyano-1,2-diphenylethane, (I), and its (±) isomer, (II).

Acta Crystallographica Section C
ISSN 0108-2701 © 1994