yielding the *exo* (4) and *endo* (2) sulfides respectively. The cyclohexane residue is in the chair conformation with a *cis* junction to the furanone ring. The furan ring has an envelope conformation with four of the atoms being coplanar [max. deviation 0·015 (5) Å] and the fifth atom [C(5*A*), C(5*B*)] 0·47 (1) Å out of the plane.

A number of hexahydrobenzofuranone derivatives have been studied by X-ray diffraction. $C_{10}H_{16}O_3$* (Burnett & Rossmann, 1971) shows a chair cyclohexane with *trans* ring junction and also shows the disparate C—O distances in the furanone rings observed in the present case. The C—O bond adjacent to the carbonyl group is very much shorter than the other C—O bond. A chair conformation with a *cis* junction is found in $C_{19}H_{26}O_3$† (Andriamialisoa, Fetizon, Hanna, Pascard & Prange, 1984).

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* Hexahydro-6-hydroxy-6,7a-dimethyl-2(3*H*)-benzo[*b*]furanone.

† Hexahydro-3-(6,6-dimethyl-3-oxocyclohex-1-en-5-yl)-4-methyl-4-vinyl-2(3*H*)-benzo[*b*]furanone.

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2-Chlorobiphenyl-4-carbonitrile

By H. H. Sutherland, Z. Ali-Adib and S. Basu

Physics Department, The University of Hull, Hull HU6 7RX, England

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Abstract. $C_{13}H_8ClN$, $M_r = 213·7$, monoclinic, $P2_1/c$, $a = 14·31$ (2), $b = 6·96$ (3), $c = 11·67$ (2) Å, $\beta = 110·1$ (2)°, $V = 1091·5$ Å^3, $Z = 4$, $D_m = 1·33$, $D_x = 1·30$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 2·7$ mm^{-1}, $F(000) = 440$, $T = 293$ K, $R = 0·066$ for 1012 observed densitometer-measured equi-inclination Weissenberg data. The average C—C bond in the phenyl rings is 1·396 Å. The molecule is non-planar; the angle between the phenyl rings is 51·9 (1)°; the C—Cl bond is 1·743 (7) Å; the C—C≡N bonds are 1·439 (13) and 1·149 (15) Å, the C—C bond making an angle of 3·4 (1)° with the phenyl plane.

Introduction. The structure determination of the title compound forms part of an investigation into liquid-crystal compounds and their chemical precursors.

Experimental. D_m measured by flotation in aqueous cadmium *n*-dodecatungstoborate. Pale-yellow opaque crystals used in data collection, dimensions 0·09 × 0·08 × 0·28 and 0·17 × 0·07 × 0·15 mm, respectively, for *c*- and *b*-axis Weissenberg data. 1070 reflections measured by the SERC Microdensitometer Service, Daresbury Laboratory, from multiple-film photographs using Cu $K\alpha$ radiation. $-15 \le h \le 15$; $0 \le k \le 8$; $0 \le l \le 9$; 1012 unique observed reflections, $R_{\text{int}} = 0·05$. Structure solved by Patterson synthesis and refined (on *F*) by blocked-matrix least squares with anisotropic thermal parameters for the non-H atoms; H-atom positions, initially obtained from a difference synthesis and placed at geometrically reasonable positions, refined with constrained C—H bond distances and isotropic thermal parameters; final $R = 0·066$, unit weights, $wR = 0·066$. $(\Delta/\sigma)_{\text{max}}$ in final refinement cycle 0·004 for positional and 0·013 for thermal parameters. Max. and min. heights in final $\Delta\rho$ map +0·6 and −0·3 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs used: *SHELX76* (Sheldrick, 1976) and local programs supplied by HHS and Drs C. Morgan and M. J. Mottram.

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Discussion. Table 1* gives atomic parameters and Table 2 bond lengths and angles. The atomic numbering is shown in Fig. 1. The phenyl rings are planar to within 0·017 Å, with an average C—C bond of 1·396 Å. The Cl—C(2) bond of 1·743 (7) Å is in good agreement with the values of 1·741 (6) and 1·745 (6) Å found in 2-chlorobiphenyl-4′-carbonitrile (Sutherland & Ali-Adib, 1986) and 4-acetyl-3′-chlorobiphenyl (Sutherland, Rawas & Mottram, 1985), respectively, and larger than the 1·723 (10) and 1·725 (10) Å in 3-chlorobiphenyl-4-carbonitrile (Sutherland & Rawas, 1984) and 2-chlorobiphenyl-4-carboxylic acid (Sutherland & Hoy, 1969). The Cl atom is displaced by 0·08 (1) Å from the phenyl ring, C(7) is displaced by 0·009 (8) Å, C(13) by 0·09 (1) Å and N by 0·14 (1) Å with the C(4)—C(13) bond being inclined at 3·4 (1)° to the phenyl ring. The bond lengths of 1·439 (13) Å for C(4)—C(13) and 1·149 (15) Å for C(13)—N are in good agreement with the values of 1·441 (10), 1·131 (10), 1·438 (14) and 1·140 (16) Å for the corresponding bonds found in 2-chlorobiphenyl-4′-carbonitrile and in 3-chlorobiphenyl-4-carbonitrile, respectively.

The C(1)—C(7) bond, 1·498 (11) Å, is in good agreement with the value of 1·486 (12) Å in 3-chlorobiphenyl-4-carbonitrile, but slightly larger than 1·466 (9) Å found for the corresponding bond length in 2-chlorobiphenyl-4′-carbonitrile. The angles C(3)—C(4)—C(5), C(3)—C(4)—C(13), C(5)—C(4)—C(13), and C(4)—C(13)—N (Table 2) compare with the corresponding angles of 118·5 (9), 120·5 (9), 121·0 (10) and 178·9 (13)° found in 3-chlorobiphenyl-4-carbonitrile, and with 119·2 (10), 119·9 (9), 120·9 (9) and 178·9 (14)° found in molecule *B* of 4′-bromobiphenyl-4-carbonitrile (Kronebusch, Gleason & Britton, 1976).

The angle between the phenyl rings about the central C—C bond, φ_1, of 51·9 (1)° is similar to the value of 52·3 (1)° found in 2-chlorobiphenyl-4′-carbonitrile, but slightly larger than the 49·2° found in 4-acetyl-2′-chlorobiphenyl (Sutherland & Hoy, 1968), and, as would be expected, considerably larger than the 31·0 (3)° in 3-chlorobiphenyl-4-carbonitrile. The axis of the molecule, defined by C(1), C(4), C(7), C(10), deviates from collinearity. Not only is there a rotation φ_1 about the C(1)—C(7) bond but also $\varphi_2 = 0·3$ (3)° of the ring C(1)—C(6) about an axis in its plane through C(1) perpendicular to C(1)—C(7), and $\varphi_3 = 2·4$ (3)°, the corresponding angle of rotation for ring C(7)—C(12). The shortest intermolecular contacts are 3·387 (14) Å between C(13) and N(1) at (−*x*, −*y*, 1 − *z*), 3·449 (16) Å between C(3) and N(1) at (−*x*, 1 − *y*, 1 − *z*) and 3·476 (13) Å between N(1) and N(1) at (−*x*, −*y*, 1 − *z*).

Table 1. *Fractional coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10⁴) *with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
Cl(1)	2838 (2)	6292 (3)	3491 (2)	700 (13)
C(1)	2290 (5)	2684 (9)	2594 (6)	340 (40)
C(2)	2125 (5)	4209 (9)	3293 (7)	457 (42)
C(3)	1433 (5)	4110 (9)	3879 (6)	483 (44)
C(4)	863 (5)	2443 (10)	3794 (6)	438 (44)
C(5)	1022 (5)	886 (8)	3122 (6)	472 (44)
C(6)	1719 (5)	1028 (9)	2548 (6)	457 (43)
C(7)	3039 (5)	2719 (10)	1958 (6)	353 (43)
C(8)	3727 (5)	1222 (10)	2106 (6)	547 (46)
C(9)	4413 (5)	1208 (11)	1504 (7)	621 (54)
C(10)	4406 (6)	2639 (13)	671 (7)	593 (60)
C(11)	3714 (6)	4128 (11)	523 (6)	602 (54)
C(12)	3045 (5)	4182 (9)	1138 (6)	495 (47)
C(13)	101 (6)	2352 (10)	4337 (8)	549 (52)
N(1)	−502 (6)	2301 (10)	4781 (8)	905 (59)

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

Cl—C(2)	1·743 (7)	C(7)—C(8)	1·403 (10)
C(1)—C(2)	1·408 (10)	C(8)—C(9)	1·389 (12)
C(2)—C(3)	1·384 (12)	C(9)—C(10)	1·390 (12)
C(3)—C(4)	1·402 (9)	C(10)—C(11)	1·403 (12)
C(4)—C(5)	1·401 (10)	C(11)—C(12)	1·380 (12)
C(5)—C(6)	1·383 (11)	C(7)—C(12)	1·400 (10)
C(1)—C(6)	1·403 (9)	C(4)—C(13)	1·439 (13)
C(1)—C(7)	1·498 (11)	C(13)—N	1·149 (15)
Cl—C(2)—C(1)	119·7 (6)	C(6)—C(1)—C(7)	120·1 (6)
Cl—C(2)—C(3)	117·5 (5)	C(2)—C(1)—C(7)	124·5 (6)
C(2)—C(3)—C(4)	120·2 (6)	C(1)—C(7)—C(8)	121·2 (6)
C(3)—C(4)—C(5)	118·5 (7)	C(1)—C(7)—C(12)	121·5 (6)
C(4)—C(5)—C(6)	119·9 (6)	C(7)—C(8)—C(9)	122·1 (7)
C(5)—C(6)—C(1)	123·2 (6)	C(8)—C(9)—C(10)	120·9 (7)
C(2)—C(1)—C(6)	115·4 (7)	C(9)—C(10)—C(11)	116·7 (8)
C(1)—C(2)—C(3)	122·7 (6)	C(10)—C(11)—C(12)	122·9 (7)
C(3)—C(4)—C(13)	121·0 (7)	C(11)—C(12)—C(7)	120·2 (6)
C(5)—C(4)—C(13)	120·4 (6)	C(12)—C(7)—C(8)	117·1 (7)
N—C(13)—C(4)	179·0 (8)		

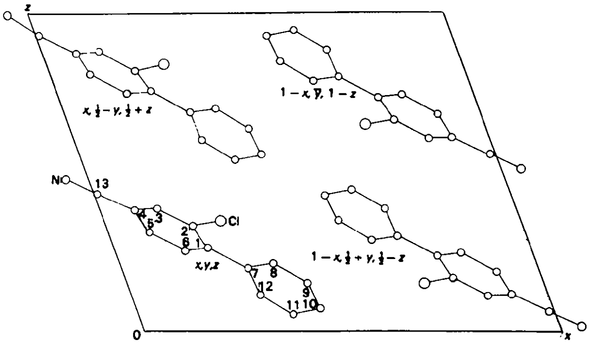

Fig. 1. The arrangement of the molecules in the unit cell viewed along **b**.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, intermolecular contact distances and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43123 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of an *ent*-Beyerene Diterpene

By William H. Watson* and Iraj Tavanaiepour

Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, *USA*

and Esther F. Lee and Tom J. Mabry

Department of Botany, The University of Texas, Austin, Texas 78713, *USA*

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Abstract. $C_{20}H_{32}O_3$, $M_r = 320{\cdot}48$, monoclinic, $P2_1$, $a = 17{\cdot}623\,(4)$, $b = 7{\cdot}429\,(1)$, $c = 7{\cdot}203\,(1)$ Å, $\beta = 100{\cdot}46\,(2)°$, $V = 927{\cdot}4\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}148$, D_m (flotation) $= 1{\cdot}142$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 5{\cdot}566$ cm^{-1}, $F(000) = 352$, $T = 295$ K, $R = 0{\cdot}048$ for 1301 reflections. The molecule is a beyerene-type tetracyclic diterpene [9,11,17-trihydroxy-*ent*-beyer-15(16)-ene; *Chemical Abstracts* name (5*R*,8*R*,9*R*,10*R*,11*S*,13*S*)-13-(hydroxymethyl)-17-norkaur-15-ene-9,11-diol]. The molecule contains three *trans*-fused six-membered rings in slightly distorted chair conformations, a bridging ethylene moiety forming a five-membered ring in an envelope conformation and a seven-membered ring in a boat conformation. There are three relatively long C—C bonds of 1·569 (6), 1·572 (5), and 1·595 (5) Å which agree well with the values 1·562, 1·573 and 1·595 Å calculated by molecular-mechanics techniques. There is one C—C bond of 1·488 (9) Å which is statistically equivalent to the value in reference compounds but is 0·041 (9) Å shorter than the calculated value.

Introduction. As part of a chemotaxonomic study of the genus *Helianthus*, the terpenoid constituents of *Helianthus laciniatus* A. Gray were reinvestigated. A previous study of this species reported five partially characterized tetracyclic diterpenes (Ortega, Ayala, Guerrero & Romo de Vivar, 1972). Comparison of the physical properties of the *ent*-beyerene (1) indicated that it was not one of the compounds previously isolated. In order to confirm the substitution pattern and relative configuration based upon ^{1}H and ^{13}C NMR the structure was determined by X-ray diffraction. Also, the *ent*-beyerenes provide another opportunity to evaluate the use of molecular mechanics (MM) in reproducing natural-product structures and conformations.

Experimental. A colorless crystal of dimensions 0·52 × 0·41 × 0·31 mm, Syntex $P2_1$ diffractometer, θ:2θ scan, variable scan rate, $5{\cdot}1 \le 2\theta \le 114{\cdot}7°$, graphite-monochromated Cu $K\alpha$ radiation; lattice parameters from a least-squares refinement of 15 reflections ($12{\cdot}6 \le 2\theta \le 65{\cdot}6°$), angles measured by a centering routine associated with the diffractometer, systematic absences ($0k0$, $k = 2n + 1$) consistent with space group $P2_1$, Laue symmetry 2, a monitored reflection, 200, showed no significant change in intensity; 1409 independent reflections measured ($0 \le h \le 19$, $0 \le k \le 8$, $-7 \le l \le 7$) with 1312 intensities greater than $3\sigma(I)$, slow scan measurement of 54 equivalent reflections ($R_{\rm int} = 0{\cdot}014$), Lorentz–polarization corrections applied, consideration of crystal shape, transmission factors and $R_{\rm int}$ resulted in neglect of absorption; direct methods (*MULTAN78*: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed the positions of all non-hydrogen atoms. Least-squares refinement followed by a difference Fourier synthesis

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* To whom correspondence should be addressed.

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