Cuthbertson, Glidewell, Holden & Liles, 1979), and almost identical to those in Hector's base (II) itself (Butler, Glidewell & Liles, 1978*a*).

The S···S distance 2·822 (5) Å is substantially longer than those found in compounds containing a hypervalent S atom in the centre of an SSN chain, where the range of S–S is 2·435–2·497 Å (see Glidewell, Holden & Liles, 1980).

The intermolecular distance between H(1) and $N(1^i)$* is 2·53 Å, and may represent a weak hydrogen bond, with N(3)–H(1)···$N(1^i)$ of 140°. No other significant short inter- or intramolecular contacts occur.

Hector's base itself (II) and its adducts with carbon disulphide (III) and methyl isothiocyanate (I) all exhibit the same structure in the solid state as in solution, as determined by ^{13}C and ^{15}N NMR (Butler, Glidewell, Hussain & Maw, 1980): no prototropic shifts occur.

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* The superscript i refers to the symmetry position $x, \frac{1}{2} - y, \frac{1}{2} + z$.

References

Akiba, K., Tsuchiya, T., Inamoto, N., Onuma, K., Nagashima, N. & Nakamura, A. (1976). *Chem. Lett.* pp. 723–726.

Butler, A. R., Glidewell, C., Hussain, I. & Maw, P. R. (1980). *J. Chem. Res.* (*S*), pp. 114–115; (*M*), pp. 1843–1863.

Butler, A. R., Glidewell, C. & Liles, D. C. (1978*a*). *Acta Cryst.* **B34**, 3241–3245.

Butler, A. R., Glidewell, C. & Liles, D. C. (1978*b*). *Acta Cryst.* **B34**, 2570–2574.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Cuthbertson, A. F., Glidewell, C., Holden, H. D. & Liles, D. C. (1979). *J. Chem. Res.* (*S*), p. 316; (*M*), pp. 3714–3733.

Glidewell, C., Holden, H. D. & Liles, D. C. (1980). *Acta Cryst.* **B36**, 1244–1247.

Glidewell, C. & Liles, D. C. (1981). *Acta Cryst.* **B37**. In the press.

Sheldrick, G. M. (1976). *SHELX* 76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1981). **B37**, 1421–1424

1,16-Dihydrodiindeno[5,4-*c*:4′,5′-*g*]phenanthrene

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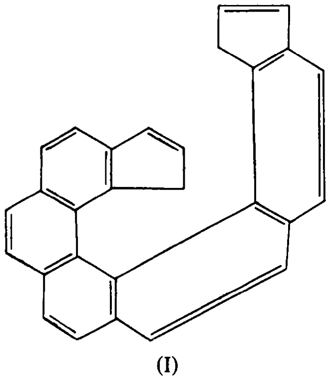
(*Received* 11 *November* 1980; *accepted* 28 *January* 1981)

Abstract. $C_{28}H_{18}$, orthorhombic, *Pbcn*, $a = 8{\cdot}851$ (3), $b = 15{\cdot}722$ (5), $c = 13{\cdot}307$ (4) Å, $M_r = 354{\cdot}46$, $V = 1851{\cdot}7$ Å^3, $Z = 4$, $D_m = 1{\cdot}27$ (1) (neutral buoyancy in aqueous ZnI_2), $D_c = 1{\cdot}271$ Mg m^{-3}, $F(000) = 744$; Mo $K\alpha$ radiation ($\lambda_{\bar{\alpha}} = 0{\cdot}71073$ Å, $\mu = 0{\cdot}065$ mm^{-1}). The structure was solved by direct methods and refined by full-matrix least squares to $R = 0{\cdot}068$ using 715 diffractometer reflections with $F_o > 4\sigma(F_o)$. The two arms of the helix are splayed further apart than in similar hexahelicenes and heptahelicenes, the dihedral angle between terminal-ring planes being 69·1°. Each molecule possesses a crystallographically required twofold axis.

Introduction. The structure determination reported here was undertaken so as to characterize unambiguously the title compound, (I), synthesized by Pesti & Katz (1980, unpublished work). The crystal structures of various helicenes have been reported (Lightner, Hefelfinger, Powers, Frank & Trueblood, 1972; Frank, Hefelfinger & Lightner, 1973; de Rango, Tsoucaris, Declercq, Germain & Putzeys, 1973; van den Hark & Noordik, 1973; van den Hark, Noordik & Beurskens, 1974; Beurskens, Beurskens & van den Hark, 1976; van den Hark & Beurskens, 1976; Le Bas, Navaza, Mauguen & de Rango, 1976; Le Bas, Navaza, Knossow & de Rango, 1976; Lindner & Kitschke, 1979; Marsh & Dunitz, 1979; Konno, Saito, Yamada & Kawazura, 1980) and a theoretical study has appeared (Navaza, Tsoucaris, Le Bas & de Rango, 1979).



(I)

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Preliminary oscillation and Weissenberg photographs showed that the light-yellow crystal (0·23 × 0·23 × 0·40 mm) used in the diffraction study belonged to space group *Pbcn* ($0kl$, $k \neq 2n$; $h0l$, $l \neq 2n$; $hk0$, $h + k \neq 2n$). Intensity data ($3 \leq 2\theta \leq 45°$, T = 299 K) were collected on an Enraf–Nonius CAD-4F κ-geometry diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the ω–2θ scan technique. Scan rates varied from 1·34 to 20·12° min^{-1} in ω. Three standard reflections, measured every 3600 s of X-ray exposure time, showed no decay. Of the 1211 unique reflections collected, the 715 with $F_o > 4\sigma(F_o)$ were used in the structure refinement. Unit-cell dimensions were obtained from a least-squares fit to the setting angles of 25 reflections with $2\theta > 30°$. Data were corrected for Lorentz and polarization effects but not for absorption. Further details of the data-reduction procedure can be found in Silverman, Dewan, Giandomenico & Lippard (1980).

The structure was solved using the multisolution Σ_2 sign-expansion direct methods program of *SHELX* 76 (Sheldrick, 1978) which revealed the positions of all C atoms. H atoms were placed in calculated positions and constrained to ride 0·95 Å from their respective C atoms. A common isotropic temperature factor for all H atoms converged at U = 0·116 (8) Å^2. All C atoms were refined anisotropically.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq} (Å^2)
C(1)	0·1388 (8)	0·3571 (3)	0·0621 (4)	0·076 (4)
C(2)	0·0036 (8)	0·3295 (4)	0·0331 (4)	0·082 (4)
C(3)	−0·1114 (7)	0·3857 (4)	0·0696 (4)	0·065 (4)
C(4)	−0·2670 (9)	0·3844 (4)	0·0530 (5)	0·088 (5)
C(5)	−0·3510 (7)	0·4498 (5)	0·0861 (5)	0·086 (5)
C(6)	−0·2867 (7)	0·5197 (4)	0·1381 (4)	0·074 (4)
C(7)	−0·3717 (8)	0·5956 (6)	0·1563 (5)	0·097 (5)
C(8)	−0·3065 (9)	0·6665 (5)	0·1906 (5)	0·096 (6)
C(9)	−0·1532 (8)	0·6665 (4)	0·2195 (4)	0·076 (4)
C(10)	−0·0750 (7)	0·7442 (3)	0·2443 (5)	0·101 (5)
C(11)	−0·0712 (5)	0·5895 (3)	0·2242 (3)	0·058 (3)
C(12)	−0·1317 (6)	0·5183 (3)	0·1666 (3)	0·053 (3)
C(13)	−0·0453 (6)	0·4501 (3)	0·1273 (3)	0·052 (3)
C(14)	0·1225 (6)	0·4345 (3)	0·1261 (4)	0·061 (3)
H(1)	0·2322	0·3313	0·0446	*
H(2)	−0·0141	0·2799	−0·0060	*
H(4)	−0·3128	0·3381	0·0188	*
H(5)	−0·4568	0·4493	0·0744	*
H(7)	−0·4773	0·5956	0·1435	*
H(8)	−0·3640	0·7173	0·1957	*
H(10)	−0·1303	0·7955	0·2526	*
H(141)	0·1595	0·4243	0·1920	*
H(142)	0·1747	0·4814	0·0975	*

* A common isotropic temperature factor for the H atoms converged at U = 0·116 (8) Å^2.

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Values have not been corrected for thermal motion.

C(1)–C(2)	1·330 (7)	C(11)–C(12)	1·457 (7)
C(1)–C(14)	1·491 (7)	C(12)–C(13)	1·418 (6)
C(2)–C(3)	1·433 (7)	C(13)–C(14)	1·506 (7)
C(3)–C(4)	1·395 (8)	C(3)···H(141^{i})	3·257 (5)
C(3)–C(13)	1·399 (7)	C(4)···H(141^{i})	3·580 (7)
C(4)–C(5)	1·343 (8)	C(5)···H(141^{i})	3·428 (6)
C(5)–C(6)	1·418 (8)	C(6)···H(141^{i})	2·937 (6)
C(6)–C(7)	1·431 (8)	C(11)···H(141^{i})	2·932 (5)
C(6)–C(12)	1·423 (7)	C(11)···H(142^{i})	3·059 (5)
C(7)–C(8)	1·335 (8)	C(12)···H(141^{i})	2·405 (5)
C(8)–C(9)	1·411 (8)	C(13)···H(141^{i})	2·639 (5)
C(9)–C(10)	1·441 (8)	C(14)···C(11^{i})	3·180 (7)
C(9)–C(11)	1·413 (7)	C(14)···C(12^{i})	3·058 (7)
C(10)–C(10^{i})	1·337 (12)	C(14)···C(13^{i})	3·362 (7)
C(11)–C(11^{i})	1·436 (9)	C(7)···C(7^{ii})	3·37 (1)

C(2)–C(1)–C(14)	110·2 (5)	C(8)–C(9)–C(11)	120·4 (6)
C(1)–C(2)–C(3)	109·8 (5)	C(10)–C(9)–C(11)	118·0 (6)
C(2)–C(3)–C(4)	129·7 (6)	C(9)–C(10)–C(10^{i})	120·2 (4)
C(2)–C(3)–C(13)	109·6 (5)	C(9)–C(11)–C(11^{i})	118·1 (3)
C(4)–C(3)–C(13)	120·7 (6)	C(9)–C(11)–C(12)	116·5 (5)
C(3)–C(4)–C(5)	118·9 (6)	C(11^{i})–C(11)–C(12)	125·0 (3)
C(4)–C(5)–C(6)	122·1 (6)	C(6)–C(12)–C(11)	118·8 (5)
C(5)–C(6)–C(7)	121·2 (7)	C(6)–C(12)–C(13)	115·7 (5)
C(5)–C(6)–C(12)	120·3 (6)	C(11)–C(12)–C(13)	125·2 (5)
C(7)–C(6)–C(12)	118·3 (6)	C(3)–C(13)–C(12)	121·6 (5)
C(6)–C(7)–C(8)	121·8 (7)	C(3)–C(13)–C(14)	106·8 (4)
C(7)–C(8)–C(9)	120·6 (7)	C(12)–C(13)–C(14)	131·3 (5)
C(8)–C(9)–C(10)	121·6 (6)	C(1)–C(14)–C(13)	103·5 (4)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$; (ii) $-1 - x, y, \frac{1}{2} - z$.

In the final cycles of refinement, using *SHELX* 76, the weighting scheme $w = 1{\cdot}8887/[\sigma^2(F_o) + 0{\cdot}001800F_o^2]$ was employed and gave a satisfactory analysis of the variation of $w\Delta^2$ for groups of data sectioned according to parity group, $|F_o|$, $(\sin\theta)/\lambda$, $|h|$, $|k|$, or $|l|$. At convergence, no parameter shift was >0·001 of its estimated standard deviation, and a final difference-Fourier map showed no peak with electron density >0·20 e Å^{-3}. The final R was 0·068 and R_w $\{=[\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}\}$ was 0·081. Neutral-atom scattering factors for C were taken from *International Tables for X-ray Crystallography* (1974) while those for H were taken from Stewart, Davidson & Simpson (1965). Final atomic coordinates are given in Table 1, bond distances and angles in Table 2.* *ORTEP* II (Johnson, 1976) diagrams of the molecule and unit-cell contents appear in Figs. 1 and 2 respectively.

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* Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35945 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

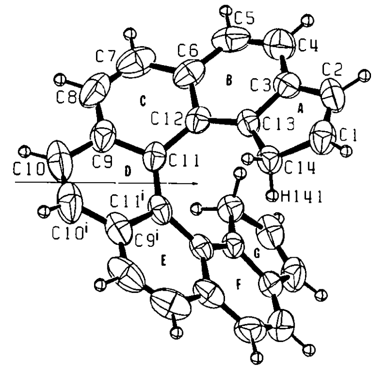

Fig. 1. Diagram of the title compound showing the atom numbering scheme. This does not correspond to the *Chemical Abstracts* numbering. A crystallographically required twofold axis passes through the mid-points of the bonds between C(10)–C(10^{i}) and C(11)–C(11^{i}). The symmetry code appears in Table 2. H atoms are labelled according to the C atom to which they are attached. The 40% probability thermal ellipsoids are depicted for the C atoms while H atoms have been assigned as arbitrary spheres with $B = 1 \cdot 0$ Å^2.

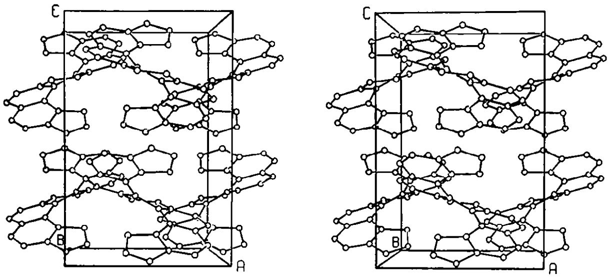

Fig. 2. Stereoscopic unit-cell packing diagram. H atoms have been omitted for clarity.

Discussion. The structure consists of discrete molecules of the title compound (Fig. 1) and the space group, uniquely defined as *Pbcn*, requires equal numbers of both enantiomers to be present in each crystal (Fig. 2). Each molecule possesses a crystallographically required twofold axis passing through the mid-points of the bonds between atoms C(10)–C(10^{i}) and C(11)–C(11^{i}). The C(1)–C(2) distance of 1·330 (7) Å indicates that the second double bond of each cyclopentadiene ring is located between these two atoms. The C(1)–C(14) and C(13)–C(14) distances of 1·491 (7) and 1·506 (7) Å both indicate single bonds. These observations were taken into consideration when calculating the number and position of the H atoms. The closest intermolecular non-H atom contact of the structure is 3·37 (1) Å between C(7)···C(7^{ii}). In common with the structures of most helicenes reported to date, the present compound shows the usual pattern of shortened bond lengths along the periphery of the helix and lengthened bonds around the inner core of the molecule.

The present compound can be compared with various other hexahelicenes and heptahelicenes that have been structurally characterized. One feature that all these compounds appear to have in common is the near constancy of the shortest intramolecular C···H contact. In 2-methylhexahelicene (Frank *et al.*, 1973) and 2-bromohexahelicene (Lightner *et al.*, 1972) these distances are on the order of 2·5 Å, in heptahelicene (Beurskens *et al.*, 1976; van den Hark & Beurskens, 1976) this distance averages 2·49 Å and in tribenzo[*f,l,r*]heptahelicene (van den Hark *et al.*, 1974) it averages 2·41 Å. In the present compound this distance is similar, being 2·41 Å between H(141) and C(12^{i}). One notable consequence of this limiting C···H distance on the present compound arises as a result of H(141) being projected below the plane of the terminal ring to which it is attached (ring *A*, Fig. 1). This has the effect of forcing the two arms of the helix further apart than is observed in similar helicenes, a fact which is mirrored in the large dihedral angle of 69·1° between the terminal ring planes, *A* and *G*. In other helicenes, where the H atoms lie in the plane of the terminal rings, the helix arms are not splayed as far apart and the dihedral angle between these rings is much less, being 58·5° in hexahelicene (de Rango *et al.*, 1973), 54·8° in 2-methylhexahelicene, 33·2° in tribenzo[*f,l,r*]heptahelicene and averaging 32·3° in heptahelicene. One prediction suggested by these observations is that the isomer of the present compound, with the second double bond of the cyclopentadiene ring located between C(1) and C(14), should be much less strained since the two methylene protons would now be located on C(2) and projected away from the inner portion of the molecule. Also, upon deprotonation of both cyclopentadiene rings of the present compound to produce cyclopentadienyl moieties, the helix strain should again be relieved.

The increased strain in the present compound, as a result of the increased repulsion experienced between the two arms of the helix, can be seen in the value of the C(12)–C(11)–C(11^{i})–C(12^{i}) torsion angle which is 40·8°. In heptahelicene the equivalent angle averages 25·0° and similar angles in 2-methylhexahelicene are 26 and 30°, while in 2-bromohexahelicene they are 26·6 and 27·9°. As in heptahelicene, 2-methylhexahelicene and 2-bromohexahelicene, the strain in the present compound occurs mainly in the central portion of the helix, in the vicinity of C(11)–C(11^{i}). This fact is indicated by the change in the values of the torsion angles around the inner core of the molecule on going from terminus to terminus [C(14)–C(13)–C(12)–C(11), 4·8 (8); C(13)–C(12)–C(11)–C(11^{i}), 20·2 (8); C(12)–C(11)–C(11^{i})–C(12^{i}), 40·8 (10)°].The localization of strain in these compounds is in contrast to the situation in 1,16-dimethylhexahelicene (van den Hark & Noordik, 1973) and tribenzo[*f,l,r*]heptahelicene where the strain appears to be spread over the entire helix, resulting in more nearly equal torsion angles around the inner cores of the molecules. The least-squares planes of rings *A* to *G*, and the dihedral angles between them, also attest to the localization of strain at the center of the molecule. The largest deviation from the least-squares plane through the atoms of ring *A* is 0·013 Å. The largest deviation from ring *B* is 0·050 Å and increases to 0·124 Å for ring *C* and 0·142 Å for ring *D*. The dihedral angles between the planes of consecutive rings *A*, *B*, *C*, *D* are 6·8, 14·1, and 17·4° respectively.

I thank Professor S. J. Lippard for the use of his X-ray diffraction facilities.

References

BEURSKENS, P. T., BEURSKENS, G. & VAN DEN HARK, TH. E. M. (1976). *Cryst. Struct. Commun.* **5**, 241–246.

FRANK, G. W., HEFELFINGER, D. T. & LIGHTNER, D. A. (1973). *Acta Cryst.* B**29**, 223–230.

HARK, TH. E. M. VAN DEN & BEURSKENS, P. T. (1976). *Cryst. Struct. Commun.* **5**, 247–252.

HARK, TH. E. M. VAN DEN & NOORDIK, J. H. (1973). *Cryst. Struct. Commun.* **2**, 643–646.

HARK, TH. E. M. VAN DEN, NOORDIK, J. H. & BEURSKENS, P. T. (1974). *Cryst. Struct. Commun.* **3**, 443–446.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 100. Birmingham: Kynoch Press.

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

KONNO, M., SAITO, Y., YAMADA, K. & KAWAZURA, H. (1980). *Acta Cryst.* B**36**, 1680–1683.

LE BAS, G., NAVAZA, A., KNOSSOW, M. & DE RANGO, C. (1976). *Cryst. Struct. Commun.* **5**, 713–718.

LE BAS, G., NAVAZA, A., MAUGUEN, Y. & DE RANGO, C. (1976). *Cryst. Struct. Commun.* **5**, 357–361.

LIGHTNER, D. A., HEFELFINGER, D. T., POWERS, T. W., FRANK, G. W. & TRUEBLOOD, K. N. (1972). *J. Am. Chem. Soc.* **94**, 3492–3497.

LINDNER, H. J. & KITSCHKE, B. (1979). *Bull. Soc. Chim. Belg.* **88**, 831–839.

MARSH, W. & DUNITZ, J. D. (1979). *Bull. Soc. Chim. Belg.* **88**, 847–852.

NAVAZA, J., TSOUCARIS, G., LE BAS, G. & DE RANGO, C. (1979). *Bull. Soc. Chim. Belg.* **88**, 863–870.

RANGO, C. DE, TSOUCARIS, G., DECLERCQ, J.-P., GERMAIN, G. & PUTZEYS, J. P. (1973). *Cryst. Struct. Commun.* **2**, 189–192.

SHELDRICK, G. M. (1978). *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 34–42. Delft Univ. Press.

SILVERMAN, L. D., DEWAN, J. C., GIANDOMENICO, C. M. & LIPPARD, S. J. (1980). *Inorg. Chem.* **19**, 3379–3383.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1981). B**37**, 1424–1426

Dibenzepin Hydrochloride: 10-[2-(Dimethylamino)ethyl]-5,10-dihydro-5-methyl-11*H*-dibenzo[*b*,*e*][1,4]diazepin-11-one Hydrochloride

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(*Received* 22 *December* 1980; *accepted* 26 *January* 1981)

Abstract. $C_{18}H_{22}N_3O^+.Cl^-$, $M_r = 331·85$, triclinic, $P\bar{1}$, $a = 11·516\ (1)$, $b = 10·668\ (1)$, $c = 7·441\ (1)$ Å, $\alpha = 106·34\ (1)$, $\beta = 91·62\ (1)$, $\gamma = 96·06\ (1)°$, $U = 870·6\ (3)$ Å^3, $Z = 2$, $D_x = 1·27$ Mg m^{-3}, μ(Cu $K\alpha$) = 2·013 mm^{-1}. Final $R = 0·047$ for 2335 independent reflexions. The molecules are held together through salt bridges of type $N^+—H\cdots Cl^-$.

Introduction. From this study of the crystal structure of dibenzepin hydrochloride and studies of other antidepressant drugs in progress, we have accumulated information about their conformations and charge distributions from which conclusions may be drawn about structure–activity relations.

A crystal sample of dibenzepin hydrochloride was kindly provided by Sandoz Laboratories (Madrid). A crystal ~0·3 × 0·3 × 0·4 mm was selected for X-ray investigation. The intensities of all 2930 unique reflexions with $2 < \theta < 65°$ were measured at 295 K with monochromatic Cu $K\alpha$ radiation on a Philips PW1100 diffractometer. The ω–2θ scan technique was used. There was no appreciable change in periodically

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