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# 9,10-Dihydroindeno[1,2-*a*]indene

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**Abstract.** Monoclinic, $P2_1/c$, $a=5{\cdot}295\ (6)$, $b=9{\cdot}972\ (11)$, $c=20{\cdot}56\ (2)$ Å, $\beta=97{\cdot}0\ (2)°$, $C_{16}H_{12}$, $Z=4$, $D_x=1{\cdot}259$ g cm$^{-3}$. The molecule has approximate *mm*2 symmetry. The geometries of the five-membered rings are similar to that of cyclopentadiene except for the bonds involved in the benzene rings.

**Introduction.** The intensity data were recorded on equi-inclination Weissenberg photographs (Cu $K\alpha$) and measured with a Rigaku microdensitometer. The weaker spots were estimated visually. The systematic absences were $h0l$ with $l$ odd and $0k0$ with $k$ odd. The structure was solved by the symbolic addition method and refined by a block-matrix least-squares method; the weighting system was: $\sqrt{w}=32/|F_o|$ when $|F_o|>32$, $\sqrt{w}=1{\cdot}0$ when $32\geq|F_o|\geq 8$, $\sqrt{w}=0{\cdot}77$ when $8>|F_o|$. The final $R$ value was 0·11 for 1806 measured reflexions (2480 accessible reflexions with $2\theta\leq 160°$).

The final atomic parameters are listed in Table 1.

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† A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30452 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final atomic parameters and their standard deviations* ($\times 10^4$)

Temperature factors are of the form: $T=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$.

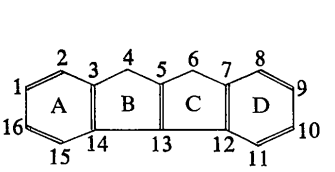
| | $x$ | $y$ | $z$ | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| C(1) | 11693 (9) | 4033 (5) | 4495 (2) | 494 (22) | 130 (6) | 25 (1) | −32 (9) | −10 (4) | −2 (2) |
| C(2) | 9758 (9) | 3113 (5) | 4408 (2) | 483 (20) | 131 (5) | 19 (1) | 8 (8) | 12 (3) | 5 (2) |
| C(3) | 8497 (7) | 2912 (4) | 3792 (2) | 288 (15) | 94 (4) | 21 (1) | 1 (7) | 14 (3) | 4 (2) |
| C(4) | 6258 (8) | 2005 (4) | 3565 (2) | 351 (17) | 112 (5) | 23 (1) | −23 (7) | 19 (3) | 6 (2) |
| C(5) | 5851 (7) | 2329 (4) | 2851 (2) | 266 (15) | 91 (4) | 27 (1) | −8 (7) | 10 (3) | −4 (2) |
| C(6) | 3951 (7) | 1930 (4) | 2281 (2) | 271 (16) | 125 (5) | 31 (1) | −26 (7) | 6 (3) | −10 (2) |
| C(7) | 4900 (7) | 2748 (4) | 1740 (2) | 301 (16) | 95 (4) | 23 (1) | 42 (7) | −4 (3) | −8 (2) |
| C(8) | 3982 (8) | 2827 (5) | 1088 (2) | 392 (19) | 132 (6) | 28 (1) | 75 (8) | −17 (4) | −16 (2) |
| C(9) | 5242 (9) | 3598 (5) | 683 (2) | 536 (23) | 157 (6) | 22 (1) | 119 (10) | −12 (4) | −4 (2) |
| C(10) | 7384 (9) | 4321 (5) | 913 (2) | 521 (23) | 152 (6) | 23 (1) | 92 (10) | 23 (4) | 12 (2) |
| C(11) | 8295 (8) | 4289 (4) | 1578 (2) | 315 (17) | 130 (5) | 23 (1) | 30 (8) | 10 (3) | 11 (2) |
| C(12) | 7048 (7) | 3510 (4) | 1990 (2) | 262 (15) | 91 (4) | 22 (1) | 34 (6) | 7 (3) | −1 (2) |
| C(13) | 7517 (7) | 3217 (4) | 2681 (2) | 223 (14) | 85 (4) | 21 (1) | 7 (6) | 9 (3) | 0 (1) |
| C(14) | 9229 (7) | 3638 (4) | 3253 (2) | 252 (14) | 84 (4) | 19 (1) | 10 (6) | 5 (3) | 3 (1) |
| C(15) | 11168 (8) | 4552 (4) | 3346 (2) | 333 (17) | 104 (5) | 26 (1) | −31 (7) | 0 (3) | 9 (2) |
| C(16) | 12406 (9) | 4734 (5) | 3972 (2) | 440 (21) | 123 (5) | 27 (1) | −61 (8) | −26 (4) | 3 (2) |

Table 1 (*cont.*)

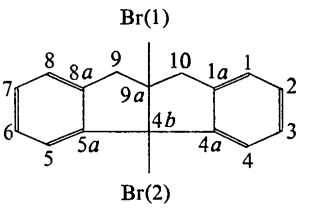
$T=\exp[-B(\sin\theta/\lambda)^2]$

| | $x$ | $y$ | $z$ | $B$ |
|---|---|---|---|---|
| H(1) | 1·2553 (102) | 0·4290 (55) | 0·4947 (26) | 5·8 (14) |
| H(2) | 0·9175 (76) | 0·2621 (41) | 0·4770 (19) | 2·4 (9) |
| H(3) | 0·6807 (73) | 0·0973 (40) | 0·3640 (18) | 2·3 (9) |
| H(4) | 0·4648 (65) | 0·2153 (36) | 0·3781 (17) | 1·3 (7) |
| H(5) | 0·3952 (78) | 0·0873 (42) | 0·2165 (19) | 2·8 (9) |
| H(6) | 0·1986 (68) | 0·2095 (37) | 0·2331 (17) | 1·7 (8) |
| H(7) | 0·2449 (83) | 0·2292 (47) | 0·0917 (22) | 3·7 (11) |
| H(8) | 0·4682 (91) | 0·3675 (50) | 0·0238 (23) | 4·1 (12) |
| H(9) | 0·8387 (110) | 0·4885 (61) | 0·0643 (28) | 6·5 (16) |
| H(10) | 0·9831 (74) | 0·4808 (41) | 0·1760 (19) | 2·2 (9) |
| H(11) | 1·1713 (74) | 0·5031 (40) | 0·2948 (19) | 2·0 (9) |
| H(12) | 1·3799 (89) | 0·5424 (48) | 0·4045 (23) | 4·3 (11) |

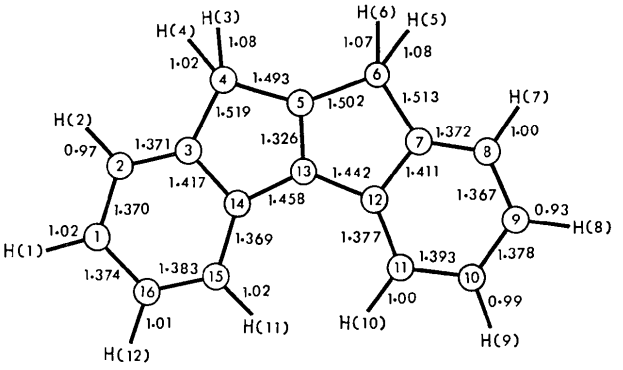
**Discussion.** The material (I) was synthesized by Haga (1968). Otaka, Marumo & Saito (1971) reported the crystal structure of its derivative, 4b,9a-dibromo-9,10-dihydroindeno[1,2-*a*]indene (II) and established the chemical structure.



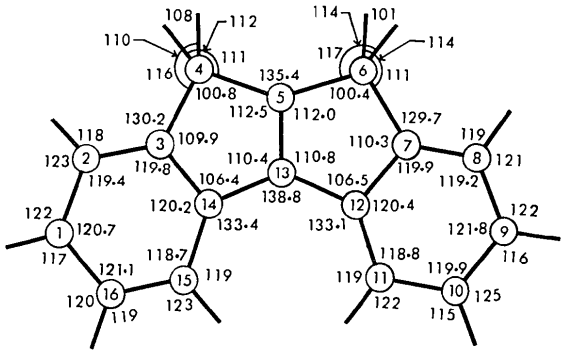

(I)




(II)




(*a*)



(*b*)

Fig. 1. (*a*) Bond lengths (Å). E.s.d.'s are ±0·007 Å except for those involving hydrogen atoms, where the e.s.d.'s are ±0·06 Å. (*b*) Bond angles (°). E.s.d.'s are ±0·4° except for those involving hydrogen atoms, where the e.s.d.'s are ±3°.

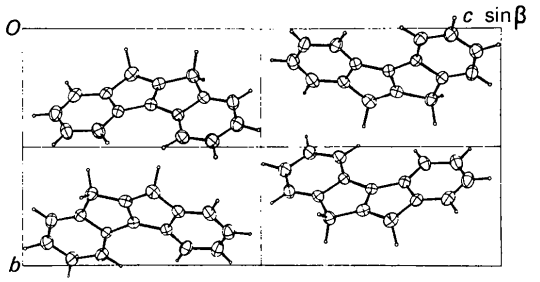



Fig. 2. Projection of the crystal structure along the *a* axis.

The interest of the present study is in the planarity of the molecule. The equations of the best planes are given in Table 2. The molecule may be represented by a single plane with equation $0{\cdot}624X-0{\cdot}742Y-0{\cdot}245Z=-1{\cdot}181$. The largest carbon atom deviation from this plane is 0·089 Å. The bond lengths and angles are shown in Fig. 1. The C(3)–C(14) and C(7)–C(12) bonds are longer than the other bonds in the benzene rings. A similar phenomenon was observed in [1]-benzothieno[2,3-*b*][1]benzothiophene (Goldberg & Shmueli, 1971).

Table 2. *Best planes and deviations of atoms from the planes*

The equations of the planes are of the form $AX+BY+CZ=D$ where $X$, $Y$ and $Z$ are coordinates (in Å) referred to the orthogonal axes $X\|\mathbf{a}^*$, $Y\|\mathbf{b}$ and $Z\|\mathbf{c}$ respectively, and $D$ is the origin-to-plane distance.

| | | | | | |
|---|---|---|---|---|---|
| *A* ring | $0{\cdot}653X-0{\cdot}719Y-0{\cdot}238Z=-0{\cdot}893$ | | | | |
| C(1) | −0·004 Å | C(2) | 0·004 Å | C(3) | −0·003 Å |
| C(14) | 0·003 | C(15) | −0·003 | C(16) | 0·004 |
| H(1) | −0·102 | H(2) | −0·030 | H(11) | 0·044 |
| H(12) | −0·026 | | | | |
| *B* ring | $0{\cdot}629X-0{\cdot}736Y-0{\cdot}250Z=-1{\cdot}139$ | | | | |
| C(3) | −0·001 Å | C(4) | 0·005 Å | C(5) | −0·008 Å |
| C(13) | 0·007 | C(14) | −0·003 | | |
| *C* ring | $0{\cdot}600X-0{\cdot}752Y-0{\cdot}273Z=-1{\cdot}408$ | | | | |
| C(5) | 0·008 Å | C(6) | −0·005 Å | C(7) | 0·000 Å |
| C(12) | 0·004 | C(13) | −0·008 | | |
| *D* ring | $0{\cdot}581X-0{\cdot}777Y-0{\cdot}243Z=-1{\cdot}444$ | | | | |
| C(7) | 0·017 Å | C(8) | −0·012 Å | C(9) | −0·002 Å |
| C(10) | 0·010 | C(11) | −0·005 | C(12) | −0·008 |
| H(7) | −0·004 | H(8) | −0·019 | H(9) | 0·031 |
| H(10) | −0·006 | | | | |

The hydrogen atoms are not included in the least-squares calculation.

The geometries of the five-membered rings are close to that of cyclopentadiene (Liebling & Marsh, 1965), where the average length of the two double bonds is 1·32 Å and the length of the single bond between the double bonds is 1·44 Å and the average of the remaining two single bonds is 1·50 Å. No significant amount of overlap between the molecules is found in the crystal structure (Fig. 2). The shortest intermolecular distance is 3·487 Å between C(1) atoms related by a centre of symmetry.

## References

GOLDBERG, I. & SHMUELI, U. (1971). *Acta Cryst.* B**27**, 2164–2173.

HAGA, T. (1968). Japanese patent 6800509.

LIEBLING, G. & MARSH, R. E. (1965). *Acta Cryst.* **19**, 202–205.

OTAKA, Y., MARUMO, F. & SAITO, Y. (1971). *Acta Cryst.* B**27**, 2195–2199.