

Fig. 1. The structure viewed along *a*. Molecules drawn in heavy outline lie at about  $x = \frac{3}{4}$ ; those in lighter outline at about  $x = \frac{1}{4}$ . H atoms not involved in hydrogen bonding have been omitted.

3. *A* and *B* share one side, and *B* and *C* two sides. Each of these rings is in the envelope conformation, with one atom lying significantly out of the plane of the remaining four. C(10), the apex of the shared flap of envelopes

*B* and *C*, is almost equidistant from the four-atom planes.

The orientation of the carboxamide group is stabilized by the intramolecular hydrogen bridge N(19)—H(19,2)···O(9). Molecules related by the *b* translation are joined by N—H···O and O—H···O bonds, as shown in Fig. 1. Contacts between the chains so formed appear to be consistent with van der Waals interactions.

The author is grateful to Dr A. W. McCulloch for suggesting the problem and for supplying specimen material.

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## Dimethyl 3-Phenyl-1*H*-indene-1,2-dicarboxylate

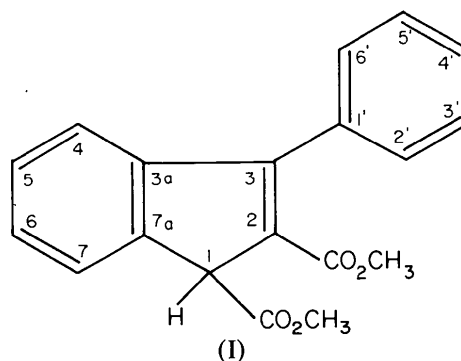
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**Abstract.** C<sub>19</sub>H<sub>16</sub>O<sub>4</sub>, F.W. 308.3; triclinic, *P* $\bar{1}$ , *a* = 10.470 (5), *b* = 12.141 (6), *c* = 8.098 (4) Å,  $\alpha$  = 122.7 (1),  $\beta$  = 113.7 (1),  $\gamma$  = 68.1 (1)° (20°C). *D<sub>m</sub>* = 1.322, *Z* = 2, *D<sub>x</sub>* = 1.317 g cm<sup>-3</sup>. The final *R* (observed reflexions only) is 0.040. Van der Waals forces govern the molecular packing, which is dominated by face-to-face stacking of the indene nuclei.

**Introduction.** The title compound (I) has been characterized by chemical and spectroscopic techniques (Matheson, McCulloch, McInnes & Smith, 1976). However, it was considered essential to have further proof of the proposed structure, and to establish the molecular conformation. The crystal-structure analysis was undertaken for this purpose.



The crystals supplied were clear and colourless, with well defined faces. The crystal class was established

from single-crystal photographs. The specimen used for measurement of intensities and cell parameters was a parallelepiped of dimensions 0.24 × 0.35 × 0.57 mm. This was mounted on a four-circle diffractometer with the longest axis roughly parallel to the  $\varphi$  axis. The intensities of 2642 independent reflexions within the limiting sphere  $2\theta = 130^\circ$  were measured with Ni-filtered Cu  $K\alpha$  radiation and a scintillation counter. The  $\theta$ - $2\theta$  scan mode was used, with scans of  $2^\circ$  for  $2\theta < 100^\circ$ , and  $3^\circ$  otherwise. Background counts were accumulated for 10 s at the end of each scan. 250 reflexions were considered to be unobserved, as their net counts were less than either 100, or 10% of the corresponding background count. No correction was made for absorption ( $\mu = 7.7 \text{ cm}^{-1}$ ).

The structure was readily determined by symbolic-addition procedures, with all non-hydrogen atoms appearing in the first  $E$  map. Refinement was by block-

Table 1. *Final atomic coordinates* ( $\times 10^4$  for C and O atoms,  $\times 10^3$  for H atoms) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	3445 (1)	6465 (1)	4185 (2)
C(2)	4371 (1)	7470 (1)	5160 (2)
C(3)	5598 (1)	6892 (1)	4567 (2)
C(3a)	5546 (1)	5492 (1)	3042 (2)
C(4)	6502 (2)	4512 (2)	1907 (3)
C(5)	6187 (2)	3256 (2)	564 (3)
C(6)	4943 (2)	2979 (2)	349 (3)
C(7)	3974 (2)	3958 (2)	1455 (2)
C(7a)	4278 (2)	5217 (1)	2793 (2)
C(1')	6896 (2)	7439 (2)	5388 (2)
C(2')	6881 (2)	8595 (2)	5411 (3)
C(3')	8125 (3)	9090 (2)	6297 (4)
C(4')	9368 (2)	8435 (3)	7138 (4)
C(5')	9397 (2)	7284 (3)	7090 (4)
C(6')	8173 (2)	6773 (2)	6203 (3)
C(8)	3991 (2)	8841 (1)	6719 (2)
O(9)	4611 (2)	9724 (1)	7490 (2)
O(10)	2822 (1)	8999 (1)	7177 (2)
C(11)	2340 (2)	10302 (2)	8707 (4)
C(12)	1937 (2)	6856 (1)	3079 (2)
O(13)	1538 (1)	7608 (2)	2367 (3)
O(14)	1085 (1)	6220 (1)	2966 (2)
C(15)	-402 (2)	6521 (3)	1988 (4)
H(1)	338 (2)	634 (2)	525 (3)
H(4)	736 (2)	469 (2)	207 (3)
H(5)	681 (3)	257 (2)	-27 (4)
H(6)	474 (2)	210 (2)	-58 (3)
H(7)	313 (2)	374 (2)	136 (3)
H(2')	600 (2)	904 (2)	483 (3)
H(3')	806 (3)	992 (3)	628 (4)
H(4')	1023 (4)	877 (4)	776 (6)
H(5')	1025 (3)	682 (3)	774 (4)
H(6')	820 (2)	594 (2)	618 (3)
H(11,1)	137 (4)	1043 (4)	846 (5)
H(11,2)	256 (5)	1102 (4)	874 (7)
H(11,3)	287 (6)	1027 (6)	999 (9)
H(15,1)	-52 (3)	644 (3)	66 (4)
H(15,2)	-71 (3)	740 (3)	276 (4)
H(15,3)	-85 (4)	585 (4)	177 (6)

diagonal least squares, minimizing  $\Sigma w\Delta F^2$ . H atoms were located in a  $\Delta F$  synthesis, and were included in the refinement. The 62 most intense reflexions were corrected for extinction, the largest correction being 22% of  $F_o$ . The weighting scheme used in the final stages was  $w = w_1 w_2$ , where  $w_1 = 5/F_o$  for  $F_o > 5$ ,  $w_1 = 1$  otherwise, and  $w_2 = 2.5 \sin^2 \theta$  for  $\sin^2 \theta < 0.4$ ,  $w_2 = 1$  otherwise. With this scheme the weighted residual showed no obvious dependence on  $F_o$  or  $\theta$ . In the final refinement cycle the largest coordinate shift of a non-

Table 2. *Bond lengths* (Å) and *angles* ( $^\circ$ )

C(1)-C(2)	1.511 (2)	C(2)-C(1)-C(7a)	102.4 (2)
C(2)-C(3)	1.354 (3)	C(2)-C(1)-C(12)	115.1 (2)
C(3)-C(3a)	1.472 (2)	C(7a)-C(1)-C(12)	112.8 (2)
C(3a)-C(4)	1.390 (3)	C(1)-C(2)-C(3)	111.2 (2)
C(4)-C(5)	1.383 (3)	C(1)-C(2)-C(8)	121.4 (2)
C(5)-C(6)	1.386 (3)	C(3)-C(2)-C(8)	127.1 (2)
C(6)-C(7)	1.387 (3)	C(2)-C(3)-C(3a)	108.5 (2)
C(7)-C(7a)	1.382 (2)	C(2)-C(3)-C(1')	129.1 (2)
C(3a)-C(7a)	1.399 (3)	C(3a)-C(3)-C(1')	122.1 (2)
C(1)-C(7a)	1.512 (2)	C(3)-C(3a)-C(4)	130.6 (2)
C(3)-C(1')	1.484 (3)	C(3)-C(3a)-C(7a)	109.0 (2)
C(1')-C(2')	1.388 (3)	C(4)-C(3a)-C(7a)	120.4 (2)
C(2')-C(3')	1.394 (4)	C(3a)-C(4)-C(5)	118.6 (2)
C(3')-C(4')	1.375 (4)	C(4)-C(5)-C(6)	120.8 (2)
C(4')-C(5')	1.366 (5)	C(5)-C(6)-C(7)	120.9 (2)
C(5')-C(6')	1.384 (4)	C(6)-C(7)-C(7a)	118.6 (2)
C(6')-C(1')	1.391 (3)	C(7)-C(7a)-C(3a)	120.6 (2)
C(2)-C(8)	1.470 (2)	C(7)-C(7a)-C(1)	130.6 (2)
C(8)-O(9)	1.198 (3)	C(3a)-C(7a)-C(1)	108.8 (2)
C(8)-O(10)	1.335 (3)	C(3)-C(1')-C(2')	122.8 (2)
O(10)-C(11)	1.442 (3)	C(3)-C(1')-C(6')	118.2 (2)
C(1)-C(12)	1.511 (3)	C(2')-C(1')-C(6')	119.0 (2)
C(12)-O(13)	1.196 (3)	C(1')-C(2')-C(3')	119.8 (2)
C(12)-O(14)	1.331 (2)	C(2')-C(3')-C(4')	120.4 (3)
O(14)-C(15)	1.442 (3)	C(3')-C(4')-C(5')	120.2 (3)
C-H (mean)	0.97 (3)	C(4')-C(5')-C(6')	120.2 (3)
C-H (range)	0.91-1.00	C(5')-C(6')-C(1')	120.4 (2)
		C(2)-C(8)-O(9)	126.1 (2)
		C(2)-C(8)-O(10)	111.0 (2)
		O(9)-C(8)-O(10)	123.0 (2)
		C(8)-O(10)-C(11)	116.5 (2)
		C(1)-C(12)-O(13)	126.3 (2)
		C(1)-C(12)-O(14)	110.1 (2)
		O(13)-C(12)-O(14)	123.5 (2)
		C(12)-O(14)-C(15)	116.4 (2)

Table 3. *Distances of some atoms from certain planes*

Atoms given in bold type define the plane. Units of distance are 0.001 Å.

1. **C(1)** 9; **C(2)** 58; **C(3)** -10; **C(3a)** -5; **C(4)** 9; **C(5)** 6; **C(6)** -9; **C(7)** -4; **C(7a)** 5; **C(8)** 23; **O(9)** 133; **C(12)** 1136; **C(1')** -216; **C(2')** 649
2. **C(1')** -12; **C(2')** 5; **C(3')** 4; **C(4')** -6; **C(5')** 0; **C(6')** 10; **C(3)** -81
3. **C(2)** -9; **C(8)** -5; **O(9)** 3; **O(10)** 4; **C(11)** -2
4. **C(1)** -29; **C(12)** -10; **O(13)** 5; **O(14)** 8; **C(15)** -4

Dihedral angles: 1-2 58.6; 1-3 7.3; 1-4 69.8°.

hydrogen atom was 30% of the corresponding e.s.d. of 0.0025 Å. The final  $R$  index (for observed reflexions only) was 0.040. The final  $\Delta F$  synthesis showed no electron density outside the limits  $\pm 0.2 \text{ e \AA}^{-3}$ . The final atomic positions are given in Table 1.\*

The computer programs used were those of Ahmed, Hall, Pippy & Huber (1966). Scattering factors were

\* A list of atomic thermal-motion parameters (anisotropic for the non-hydrogen atoms) and structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32247 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

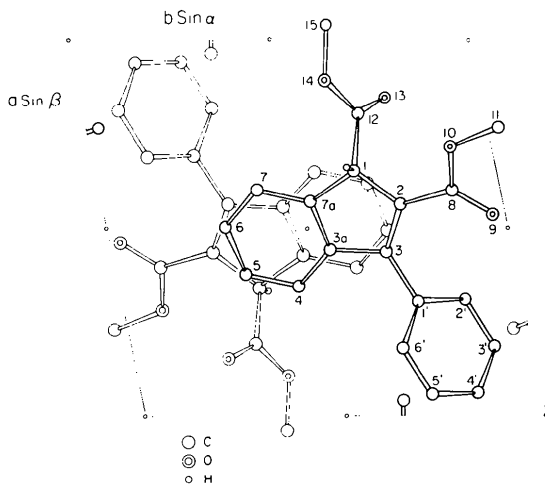


Fig. 1. The structure projected along  $c$ . H atoms other than H(1) are not shown.

taken from Stewart, Davidson & Simpson (1965) for H, and from Hanson, Herman, Lea & Skillman (1964).

**Discussion.** The analysis confirms the proposed formulation (I). Bond lengths and angles are given in Table 2, and details of some of the mean planes in Table 3. The molecular conformation and packing are illustrated in Fig. 1. The indene nucleus is planar except for C(2), which lies 0.058 Å from the mean plane of the remaining eight atoms, and on the same side of this plane as C(12). Each methoxycarbonyl group is planar, with the attached atom of the indene nucleus lying close to the plane. The methoxycarbonyl group at C(2) is nearly coplanar with the indene nucleus. Some torsion angles of interest are: C(3a)C(3)–C(1')C(6')  $-53.4^\circ$ ; C(3)C(2)–C(8)O(9)  $12.3^\circ$ ; C(2)C(1)–C(12)O(13)  $24.8^\circ$ . The molecular packing does not appear to involve forces other than van der Waals, and is dominated by face-to-face stacking of the indene nuclei, with a mean interplanar spacing of 3.52 Å.

The author is grateful to Dr A. W. McCulloch for suggesting the problem, and for supplying specimen material.

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## Phthalic Anhydride

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**Abstract.**  $\text{C}_8\text{H}_4\text{O}_3$ ,  $M_r = 148.12$ , orthorhombic,  $Pna2_1$  (systematic absences  $0kl$ ,  $k + l$  odd;  $h0l$ ,  $h$  odd),  $a = 7.859(5)$ ,  $b = 14.173(5)$ ,  $c = 5.911(3)$  Å,  $Z = 4$ ,  $D_c = 1.494$ ,  $D_m = 1.492 \text{ g cm}^{-3}$  (floatation in  $\text{CCl}_4/\text{hexane}$ ). The structure was refined to a final  $R$  of 0.037 for 527 reflections. The molecule is virtually planar and only

small differences in bond lengths between chemically equivalent atoms are noticeable.

**Introduction.** Although cell parameters have been reported for phthalic anhydride at least twice (Williams, Van Meter & McCrone, 1952; Pandey, 1954),