

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 ΔF 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.

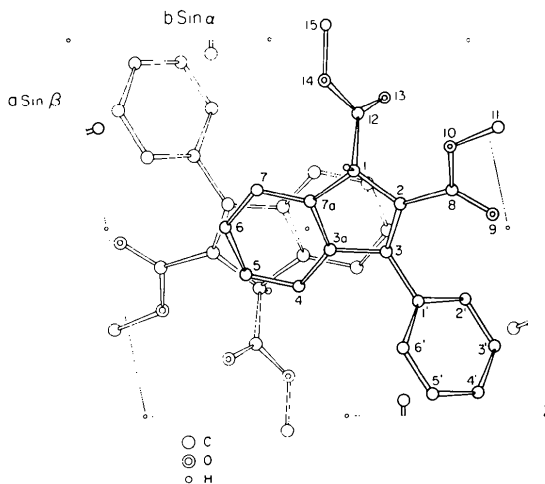


Fig. 1. The structure projected along *c*. H atoms other than H(1) are not shown.

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° ; C(3)C(2)–C(8)O(9) 12.3° ; C(2)C(1)–C(12)O(13) 24.8° . 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, *Pna*2₁ (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),

we could find no report of the crystal structure of this common substance. Crystals of phthalic anhydride obtained by sublimation were suitable for X-ray analysis. A crystal, $0.3 \times 0.4 \times 1.0$ mm, mounted along *c*, was used for recording crystal data and intensities. Cell parameters in satisfactory agreement with those reported were determined by a least-squares fit to the settings for four angles of eight reflections on a Picker FACS-I diffractometer.

Intensity data were collected on a Picker FACS-I system ($\text{Cu } K\alpha$, $\lambda = 1.54178 \text{ \AA}$, graphite monochromator) using a scintillation counter with pulse-height analyzer, $\theta-2\theta$ scan, $2.0^\circ \text{ min}^{-1}$ rate, 10s background measured at the two extremes of each scan, attenuators when the count rate exceeded 10 000 counts s^{-1} , and 2.0° scan range with dispersion factor allowing for α_1, α_2 splitting at large 2θ values. One independent set of data was measured, consisting of 532 reflections of which 527 were considered to be observed by the criterion $I > 3\sigma(I)$. Three standard reflections were monitored every 50 measurements to check the crystal alignment and stability; no decrease in intensity of the standards was observed. Lorentz and polarization corrections were applied, but no correction was made for absorption.

The crystal structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). The *E* map revealed all nonhydrogen atoms. Full-matrix least-squares refinement in which positional and isotropic thermal parameters were varied reduced *R* to 0.102. A difference map at this stage revealed all the H atoms. The structure was further refined with anisotropic temperature factors for nonhydrogen atoms and isotropic temperature factors (of the nonhydrogen atoms to which the H atoms are attached) for H atoms, and *R* reduced to 0.037. Refinement was terminated at this stage since the ratios of shifts in parameters to

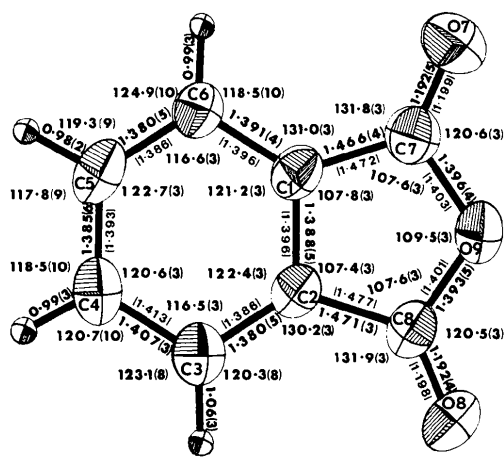


Fig. 1. Bond lengths (\AA) and angles ($^\circ$) in the molecule with standard deviations in parentheses. Bond lengths after thermal corrections are in square brackets (Schomaker & Trueblood, 1968).

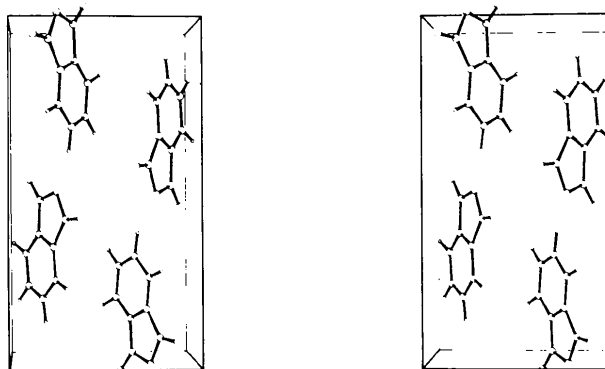


Fig. 2. Stereoscopic view of a unit cell, *c* axis projection, with *b* axis vertical and *a* axis horizontal.

Table 1. Final positional ($\times 10^4$; for H $\times 10^3$) and thermal parameters of phthalic anhydride with estimated standard deviations in parentheses

The temperature factors ($\times 10^4$) for the nonhydrogen atoms are of the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	7679 (2)	6579 (2)	6134 (7)	147 (3)	36 (1)	257 (8)	11 (2)	-19 (6)	-4 (3)
C(2)	8462 (3)	6236 (2)	4197 (7)	140 (4)	35 (1)	239 (8)	4 (1)	-17 (5)	-7 (3)
C(3)	9158 (3)	6823 (2)	2582 (6)	169 (4)	45 (1)	299 (11)	1 (2)	-7 (6)	11 (3)
C(4)	9019 (3)	7799 (2)	2981 (8)	187 (4)	40 (1)	333 (10)	-4 (2)	-19 (6)	27 (3)
C(5)	8249 (3)	8133 (2)	4931 (7)	193 (5)	33 (1)	366 (11)	10 (2)	-40 (6)	-8 (3)
C(6)	7564 (3)	7543 (2)	6549 (7)	187 (4)	38 (1)	284 (9)	16 (2)	19 (5)	7 (3)
C(7)	7047 (3)	5769 (2)	7427 (7)	171 (4)	46 (2)	292 (11)	8 (2)	7 (7)	9 (3)
C(8)	8393 (3)	5200 (2)	4300 (6)	165 (4)	36 (1)	271 (9)	-1 (2)	-22 (6)	-8 (3)
O(7)	6275 (3)	5719 (2)	9159 (7)	289 (5)	69 (1)	363 (9)	4 (2)	112 (6)	22 (3)
O(8)	8949 (2)	4605 (1)	3081 (7)	254 (4)	41 (1)	377 (7)	5 (2)	22 (5)	-32 (3)
O(9)	7493 (2)	4953 (1)	6243 (7)	202 (3)	35 (1)	350 (7)	-7 (1)	24 (4)	10 (2)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>		<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(C3)	983 (3)	654 (2)	120 (7)	5.4 (6)	H(C5)	810 (3)	882 (2)	509 (5)	4.4 (5)
H(C4)	953 (3)	826 (2)	192 (6)	5.6 (6)	H(C6)	699 (3)	776 (1)	796 (6)	4.9 (5)

estimated standard deviations were all less than 0.3. The refinement was based on F_o , the quantity minimized being $\Sigma w(F_o - F_c)^2$. The weighting scheme used was based on counter statistics as defined by Corfield, Doedens & Ibers (1967); the value of p was 0.04. The scattering factors used were those of Hanson, Herman, Lea & Skillman (1964).*

Discussion. The final atomic coordinates and thermal parameters are given in Table 1. The standard deviations for atomic positions were taken from the least-squares matrix. Bond lengths and angles including those after thermal corrections (in square brackets) and estimated standard deviations (in parentheses) are given on the thermal ellipsoid plot of the molecule in Fig. 1. The molecule is virtually planar, with a maximum deviation of 0.04 Å from the least-squares plane. Fig. 2 shows the crystal packing where stacking of the benzene rings is observed. Though chemical equivalency allows for a mirror plane through O(9) bisecting bonds C(1)–C(2) and C(5)–C(4), no such plane is used in the crystal. Unlike tetrachlorophthalic anhydride (Rudman, 1971) and the perylene complex of pyromellitic dianhydride (Boeyens & Herbstein, 1965), where large discrepancies between the pairs of C=O and C–O

bonds in each molecule were reported, only small differences in bond lengths between chemically equivalent atoms in phthalic anhydride are noticeable. In this respect, phthalic anhydride is like the pyrene–pyromellitic dianhydride complex at 110K (Herbstein & Snyman, 1969).

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32258 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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(25*R*)-Cholest-5-ene-3β,26-diol

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Abstract. C₂₇H₄₆O₂, orthorhombic, $P2_12_12_1$, $a = 19.182(3)$, $b = 22.062(5)$, $c = 11.605(3)$ Å, $V = 4911(2)$ Å³, $Z = 8$, $D_x = 1.089$ g cm⁻³. The analysis establishes the configuration at C(25) as (25*R*). The crystal is built up of molecules connected by hydrogen bonds, forming infinite chains running along a .

Introduction. The specimen was a clear, thin needle cut to 0.1 × 0.1 × 0.1 mm. Systematic absences were $h00$, h odd; $0k0$, k odd and $00l$, l odd. Intensities were measured in the θ – 2θ mode on an automatic Syntex $P2_1$ four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å) monochromatized by a graphite crystal.