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# The Crystal and Molecular Structure of *cis*-1,2-Dimethoxycarbonyl-1,2-bis(4-nitrophenyl)cyclobutane


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*cis*-1,2-Dimethoxycarbonyl-1,2-bis(4-nitrophenyl)cyclobutane, $C_{20}H_{18}O_8N_2$, crystallizes in space group $P2_1/c$ with $Z = 4$ and cell parameters $a = 10{\cdot}158\,(1)$, $b = 25{\cdot}598\,(3)$, $c = 8{\cdot}206\,(1)$ Å, $\beta = 110{\cdot}169\,(4)°$. Data were collected on an automatic four-circle diffractometer with Cu $K\alpha$ radiation, and the structure has been refined by full-matrix least squares to a residual $R = 0{\cdot}045$ for 2516 independent significant reflexions. The cyclobutane ring is puckered with a dihedral angle of 21·4° and contains a C—C bond of length 1·606 (3) Å between the two *cis*-substituted atoms.


## Introduction

Schauble, Freed & Swerdloff (1971) have shown that upon irradiation crystalline methyl α-(4-nitrophenyl)-acrylate (I) dimerizes to give dimethoxycarbonylbis(4-nitrophenyl)cyclobutane.

On the basis of NMR and chemical evidence, these authors proposed that the photodimerization had occurred in a head-to-head manner to give the *cis*-1,2 disubstituted isomer (II). We report here the results of an X-ray diffraction study of this compound.

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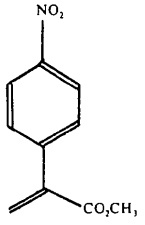
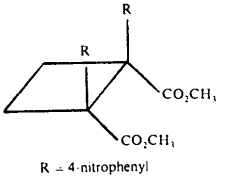



(I)




(II)

## Experimental

A sample of the photodimer was supplied by Professor Schauble of the Villanova University, Pennsylvania. Slow cooling of a saturated methanol solution yielded colourless hexagonal plates perpendicular to [010]. A prismatic cleavage fragment measuring 0·2 × 0·2 ×

0·4 mm parallel to **a**, **b** and **c** respectively was mounted along the $c$ axis for use in the analysis. Intensity data were collected on a Hilger & Watts Y290 automatic four-circle diffractometer with the $\omega/2\theta$ scan mode. All measurements were made at room temperature with nickel-filtered Cu $K\alpha$ radiation. Gradual variations in the experimental conditions were accommodated by scaling the data to the intensities of three reference reflections measured after every 50th reflection. 2516 $hkl$ with $I > 3\sigma(I)$ were collected over the range $0 \leq \theta \leq 70°$. The intensities were corrected for Lorentz and polarization effects. No correction was made for absorption; the linear absorption coefficient $\mu$(Cu $K\alpha$) is 9·28 cm$^{-1}$.

*Crystal data*

$C_{20}H_{18}O_8N_2$, FW 414·4. Monoclinic, $a$ = 10·158(1), $b$ = 25·598(3), $c$ = 8·206(1) Å, $\beta$ = 110·169(4)°, $V$ = 2002·9(4) Å$^3$, $D_o$ = 1·37 g cm$^{-3}$, $Z$ = 4, $D_c$ = 1·375 g cm$^{-3}$, $F(000)$ = 864. The conditions limiting possible reflection, $0k0$ $k = 2n$ and $h0l$ $l = 2n$, uniquely determine the space group as $P2_1/c$ ($C_{2h}^5$, No. 14).

### Structure determination and refinement

Normalized structure factors were obtained by the $K$-curve method (Karle & Karle, 1965) and 246 reflec-

Table 1. *Final positional and thermal parameters*

Estimated standard deviations are given, where appropriate, in parentheses. Anisotropic thermal parameters are $\times 10^3$. The expression used was: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{32}lkc^*b^* + 2U_{31}lhc^*a^* + 2U_{21}khb^*a^*)]$. The H atoms were given an isotropic thermal parameter, $U$ = 0·06 Å$^2$. The expression used was $\exp(-8\pi^2 U \sin^2\theta/\lambda^2)$.

| | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $2U_{32}$ | $2U_{31}$ | $2U_{21}$ |
|---|---|---|---|---|---|---|---|---|---|
| C(1) | −0·2258 (2) | 0·1570 (1) | −0·0469 (3) | 47 (1) | 42 (1) | 54 (1) | −5 (2) | 48 (2) | 8 (2) |
| C(2) | −0·2478 (2) | 0·1025 (1) | 0·0373 (3) | 45 (1) | 45 (1) | 52 (1) | −6 (2) | 49 (2) | −2 (2) |
| C(3) | −0·3821 (3) | 0·1263 (1) | 0·0586 (3) | 47 (1) | 61 (2) | 64 (2) | −1 (2) | 63 (2) | 6 (2) |
| C(4) | −0·3351 (3) | 0·1813 (1) | 0·0274 (4) | 55 (1) | 57 (1) | 67 (2) | −6 (2) | 64 (3) | 22 (2) |
| C(11) | −0·0806 (2) | 0·1804 (1) | 0·0103 (3) | 52 (1) | 43 (1) | 52 (1) | −2 (2) | 49 (2) | 2 (2) |
| C(12) | −0·0571 (3) | 0·2306 (1) | 0·0753 (4) | 63 (2) | 49 (1) | 78 (2) | −24 (3) | 56 (3) | 1 (3) |
| C(13) | 0·0757 (3) | 0·2526 (1) | 0·1245 (4) | 74 (2) | 57 (2) | 85 (2) | −30 (3) | 50 (3) | −28 (3) |
| C(14) | 0·1832 (3) | 0·2237 (1) | 0·1075 (4) | 57 (2) | 75 (2) | 63 (2) | −6 (3) | 38 (3) | −31 (3) |
| C(15) | 0·1646 (3) | 0·1736 (1) | 0·0421 (4) | 54 (2) | 70 (2) | 67 (2) | 4 (3) | 56 (3) | 1 (3) |
| C(16) | 0·0311 (3) | 0·1524 (1) | −0·0075 (3) | 52 (1) | 50 (1) | 65 (2) | −7 (2) | 58 (2) | 0 (2) |
| N(1) | 0·3238 (3) | 0·2470 (1) | 0·1590 (4) | 69 (2) | 109 (2) | 99 (2) | −34 (4) | 46 (3) | −67 (3) |
| O(11) | 0·3337 (4) | 0·2930 (2) | 0·2011 (6) | 104 (2) | 131 (3) | 256 (5) | −112 (6) | 76 (5) | −125 (4) |
| O(12) | 0·4223 (3) | 0·2203 (2) | 0·1619 (5) | 65 (2) | 175 (3) | 163 (3) | −75 (5) | 100 (4) | −58 (4) |
| C(17) | −0·2864 (3) | 0·1558 (1) | −0·2444 (3) | 55 (1) | 47 (1) | 57 (2) | 4 (2) | 48 (2) | 8 (2) |
| O(13) | −0·4132 (2) | 0·1340 (1) | −0·3011 (2) | 54 (1) | 86 (1) | 54 (1) | 0 (2) | 36 (2) | −13 (2) |
| O(14) | −0·2290 (2) | 0·1725 (1) | −0·3395 (3) | 80 (1) | 75 (1) | 64 (1) | 14 (2) | 71 (2) | −28 (2) |
| C(18) | −0·4807 (4) | 0·1301 (2) | −0·4856 (4) | 80 (2) | 141 (3) | 57 (2) | −3 (4) | 27 (3) | −28 (4) |
| C(21) | −0·1306 (2) | 0·0895 (1) | 0·2067 (3) | 50 (1) | 43 (1) | 51 (1) | −4 (2) | 50 (2) | −3 (2) |
| C(22) | −0·1214 (3) | 0·1147 (1) | 0·3603 (4) | 64 (2) | 54 (1) | 56 (2) | −7 (2) | 49 (3) | 21 (2) |
| C(23) | −0·0146 (3) | 0·1041 (1) | 0·5134 (4) | 73 (2) | 63 (2) | 53 (2) | −14 (3) | 39 (3) | 18 (3) |
| C(24) | 0·0865 (3) | 0·0684 (1) | 0·5126 (3) | 59 (2) | 60 (2) | 54 (2) | 8 (2) | 36 (2) | 11 (2) |
| C(25) | 0·0806 (3) | 0·0418 (1) | 0·3648 (4) | 62 (2) | 62 (2) | 64 (2) | 4 (3) | 51 (3) | 27 (3) |
| C(26) | −0·0285 (3) | 0·0525 (1) | 0·2131 (3) | 61 (2) | 55 (1) | 55 (2) | −10 (2) | 53 (3) | 14 (2) |
| N(2) | 0·2029 (3) | 0·0578 (1) | 0·6741 (4) | 76 (2) | 89 (2) | 68 (2) | 1 (3) | 28 (3) | 29 (3) |
| O(21) | 0·1979 (3) | 0·0757 (1) | 0·8077 (3) | 134 (2) | 150 (3) | 62 (2) | −42 (3) | 1 (3) | 112 (4) |
| O(22) | 0·2975 (3) | 0·0305 (1) | 0·6675 (4) | 93 (2) | 187 (3) | 92 (2) | 5 (4) | 25 (3) | 132 (4) |
| C(27) | −0·2699 (2) | 0·0565 (1) | −0·0854 (3) | 50 (1) | 45 (1) | 52 (1) | 3 (2) | 40 (2) | 1 (2) |
| O(23) | −0·3669 (2) | 0·0245 (1) | −0·0702 (2) | 65 (1) | 56 (1) | 67 (1) | −15 (2) | 58 (2) | −31 (2) |
| O(24) | −0·2047 (2) | 0·0496 (1) | −0·1813 (2) | 75 (1) | 60 (1) | 72 (1) | −33 (2) | 88 (2) | −18 (2) |
| C(28) | −0·3963 (3) | −0·0211 (1) | −0·1795 (4) | 79 (2) | 57 (2) | 85 (2) | −30 (3) | 49 (3) | −40 (3) |

| | $x$ | $y$ | $z$ | | $x$ | $y$ | $z$ |
|---|---|---|---|---|---|---|---|
| H(31) | −0·388 | 0·120 | 0·177 | H(182) | −0·495 | 0·166 | −0·541 |
| H(32) | −0·471 | 0·114 | −0·032 | H(183) | −0·424 | 0·108 | −0·539 |
| H(41) | −0·411 | 0·203 | −0·058 | H(22) | −0·194 | 0·141 | 0·361 |
| H(42) | −0·293 | 0·203 | 0·137 | H(23) | −0·010 | 0·123 | 0·623 |
| H(12) | −0·136 | 0·251 | 0·092 | H(25) | 0·156 | 0·158 | 0·366 |
| H(13) | 0·091 | 0·290 | 0·165 | H(26) | −0·035 | 0·033 | 0·104 |
| H(15) | 0·246 | 0·153 | 0·030 | H(281) | −0·469 | −0·044 | −0·160 |
| H(16) | 0·014 | 0·116 | −0·057 | H(282) | −0·426 | −0·012 | −0·305 |
| H(181) | −0·576 | 0·113 | −0·514 | H(283) | −0·306 | −0·043 | −0·152 |

tions with $|E| \geq 1{\cdot}8$ were phased with *MULTAN* (Germain, Main & Woolfson, 1971). All non-hydrogen atoms were successfully located from an $E$ map based on phases corresponding to the solution with the highest figure of merit, 1·08. A structure factor calculation with these coordinates gave a residual $R = 0{\cdot}36$.

Previous attempts at solving the structure by various direct methods and Patterson search techniques using a more limited data set of 1210 reflections obtained by one of us (GTD) had proved unsuccessful. Comparison of the two sets of data gave an inter-set residual of 0·048 indicating that these failures were not due to the quality of the earlier data set but could be wholly ascribed to the limited extent of the data obtainable from the very small crystals initially available.

The structure was refined by a full-matrix least-squares technique. After initial isotropic refinement the positions of the H atoms were calculated assuming a C—H bond length of 1·0 Å. These coordinates, together with an isotropic temperature factor $U_H$ = 0·06 Å$^2$, were included in the subsequent anisotropic refinement but no attempt was made to refine the parameters of these atoms. In the final cycle all parameter shifts were less than one-tenth of the corresponding estimated standard deviations. During the refinement relative weights were assigned to the unscaled structure factors according to the function $w^{-1} = (a + |F_o| + b|F_o|^2 + c|F_o|^3)$. In the final cycle values of $a = 100{\cdot}0$, $b = 0{\cdot}00276$ and $c = 3{\cdot}0 \times 10^{-5}$ were used and an analysis of the weighting scheme showed that the mean value of $w(|F_o| - |F_c|)^2$ was approximately constant over ranges of both $|F_o|$ and $\sin\theta/\lambda$. The final value of $R$ was 0·045 and that of the weighted residual $R'$ $(= [\Sigma\, w(|F_o| - |F_c|)^2/\Sigma\, w|F_o|^2]^{1/2})$ was 0·061.

Comparison of the observed and calculated structure factors for the insignificant reflections, 1200 in number, showed no marked discrepancies. The maximum deviation of the electron density from zero obtained in a final difference Fourier synthesis was less than 0·25 e Å$^{-3}$.

The atomic scattering factors listed by Hanson, Herman, Lea & Skillman (1964) were used throughout. All calculations were performed on the CDC-6600 computer at the University of London Computer Centre. The final positional and thermal parameters are listed in Table 1.†

## Discussion

The structure consists of discrete molecules of *cis*-1,2-dimethoxycarbonyl-1,2-bis(4-nitrophenyl)cyclobutane. Fig. 1 depicts a stereoscopically related pair of molecules viewed along **a***. The atomic nomenclature, bond lengths and angles are shown in Fig. 2. Table 2 contains details of molecular geometry.

The cyclobutane ring is distinctly puckered, with a dihedral angle of 21·4° typical of non-fused cyclobutanes (Shirrell & Williams, 1973). The most notable feature of the ring is the length of the C—C bond linking the two disubstituted atoms, 1·606 (3) Å. The average of the three remaining bond lengths in the ring, 1·553 (2) Å, while appreciably longer than the normal paraffinic value of 1·541 (3) Å (Kennard *et al.*, 1972) is close to the value of 1·550 (2) Å quoted by Cotton & Frenz (1974) as the average of cyclobutane-ring bond lengths observed in previous studies. The lengthening of the ring bonds in cyclobutane has been rationalized by Dunitz & Schomaker (1952) in terms of the close approaches of the transannular C atoms.

Cyclobutane-ring bond lengths of ≥1·6 Å have been observed in *o*-distyrylbenzene dimer [1·606 (8) Å, Van den Hark, Buerskens & Laarhoven (1974)],

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32147 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

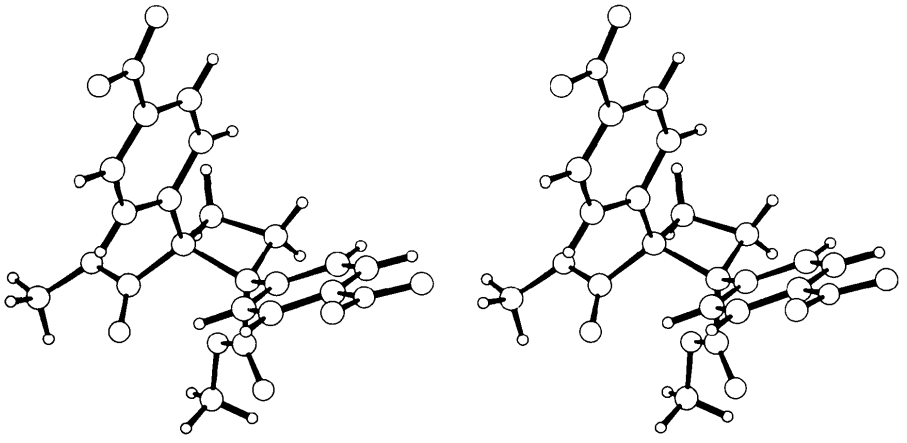


Fig. 1. A stereo drawing of the molecule of *cis*-1,2-dimethoxycarbonyl-1,2-bis(4-nitrophenyl)cyclobutane.

in 1-phenyl-3,3-biphenylene-allene dimer [1·606(6) Å, Dreissig, Luger & Rewicki (1974)] and in 3-(*E*)-benzylidene-1-*tert*-butyl-2-oxo-4-*trans*-phenylcyclobutanecarbonitrile [1·614(3) Å, McDonald (1975)] while Camerman & Camerman (1970) report a value of 1·66(3) Å in the *cis–syn* dimer of dimethylthymine. Since these examples also involve substituted C atoms, it would appear that steric hindrance between the substituent groups may be responsible for the additional lengthening. Indeed the intramolecular contacts C(11)···C(21) (2·97 Å) and C(17)···C(27) (2·84 Å) are appreciably shorter than the 3·4 Å expected for non-bonded contact between aromatic C atoms. However, if the transannular contacts C(1)···C(3) and C(2)···C(4) of 2·19 Å are responsible for the increase of 0·025 Å in the average of the four ring bond lengths from the paraffinic value to the observed value of 1·566 Å, it seems unlikely that the $C_\alpha \cdots C_\alpha$ contacts of 2·84 and 2·97 Å could alone cause an additional increase of 0·040 to 1·606 Å. Furthermore, *cis* substitution of adjacent C atoms frequently results in no significant lengthening of the corresponding ring bond, for example tetracyanocyclobutane (Harel & Hirshfeld, 1975) $C_\alpha \cdots C_\alpha$ = 2·82, C—C = 1·568(2) Å; tetramethoxycarbonylcyclobutane (Margulis, 1971) $C_\alpha \cdots C_\alpha$ = 2·89, C—C = 1·572(5) Å; cyclobutanedi-

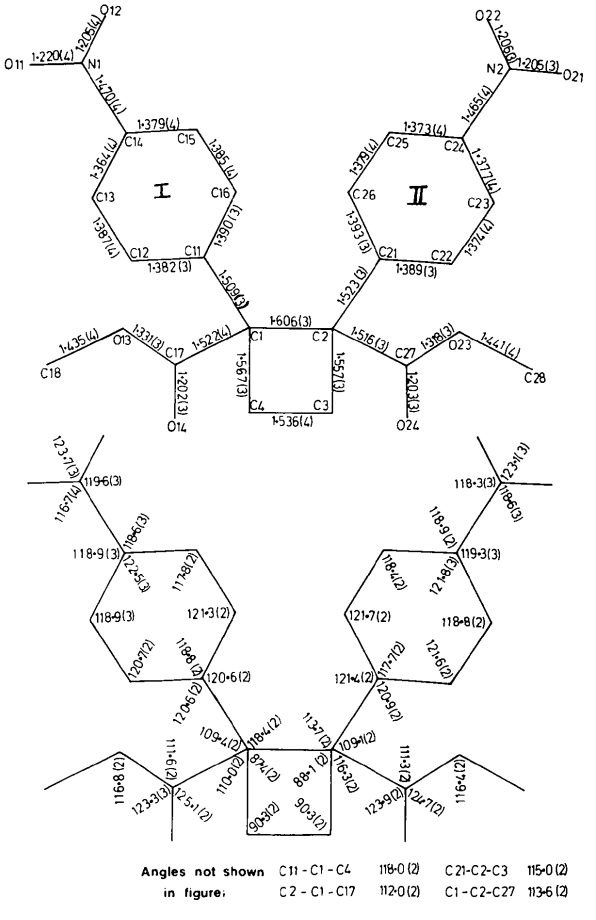



Fig. 2. A schematic drawing of the molecule showing the intramolecular bond lengths and angles.

Table 2. *Molecular geometry*

(*a*) Least-squares planes defined by atomic positions and distances of atoms (Å) from these planes. *X*, *Y* and *Z* refer to orthogonal coordinates obtained by the transformation:

$$\begin{pmatrix} X \\ Y \\ Z \end{pmatrix} = \begin{pmatrix} a\sin\beta & 0 & 0 \\ 0 & b & 0 \\ a\cos\beta & 0 & c \end{pmatrix} \begin{pmatrix} x \\ y \\ z \end{pmatrix}.$$

Plane *A* C(1), C(2) and C(4)
$0{\cdot}7403X + 0{\cdot}2356Y + 0{\cdot}6296Z + 0{\cdot}3919 = 0$
C(3) −0·399

Plane *B* C(2), C(3) and C(4)
$0{\cdot}4864X + 0{\cdot}1042Y + 0{\cdot}8676Z - 0{\cdot}1426 = 0$
C(1) −0·419

Plane *C* C(11), C(12), C(13), C(14), C(15) and C(16)
$0{\cdot}2036X - 0{\cdot}3613Y + 0{\cdot}9100Z + 1{\cdot}4974 = 0$
C(11) 0·007, C(12) −0·002, C(13) −0·003, C(14) 0·004, C(15) 0·002, C(16) −0·007, N(1) −0·003, O(11) −0·126, O(12) 0·142, C(1) −0·024

Plane *D* C(21), C(22), C(23), C(24), C(25) and C(26)
$0{\cdot}5487X + 0{\cdot}7202Y - 0{\cdot}4245Z - 0{\cdot}0436 = 0$
C(21) 0·010, C(22) 0·000, C(23) −0·011, C(24) 0·012, C(25) −0·002, C(26) −0·009, N(2) 0·036, O(21) −0·132, O(22) 0·192, C(2) 0·052

Plane *E* N(1), O(11) and O(12)
$0{\cdot}1354X - 0{\cdot}2631Y + 0{\cdot}9552Z + 1{\cdot}0829 = 0$
C(14) 0·043

Plane *F* N(2), O(21) and O(22)
$0{\cdot}4946X + 0{\cdot}8108Y - 0{\cdot}3130Z - 0{\cdot}6468 = 0$
C(24) −0·042

Plane *G* C(1), C(17), O(13) and O(14)
$0{\cdot}4178X - 0{\cdot}8955Y - 0{\cdot}1534Z + 4{\cdot}5609 = 0$
C(1) −0·001, C(17) 0·002, O(13) −0·001, O(14) −0·001, C(18) 0·016, C(11) 0·050, C(2) 1·043, C(4) −1·144

Plane *H* C(2), C(27), O(23) and O(24)
$0{\cdot}6954X - 0{\cdot}5129Y + 0{\cdot}5034Z + 2{\cdot}4000 = 0$
C(2) 0·002, C(27) −0·007, O(23) 0·002, O(24) 0·003, C(28) 0·007, C(21) 1·443, C(1) −0·955, C(3) −0·875

(*b*) Dihedral angles

| | | | |
|---|---|---|---|
| Plane 1 | *A* | *C* | *D* |
| Plane 2 | *B* | *E* | *F* |
| Angle (°) | 21·4 | 7·3 | 8·8 |

(*c*) Torsion angles (°)

| | |
|---|---|
| C(1)—C(2)—C(3)—C(4) | −15·1 |
| C(2)—C(3)—C(4)—C(1) | 15·5 |
| C(3)—C(4)—C(1)—C(2) | −15·0 |
| C(4)—C(1)—C(2)—C(3) | 14·9 |
| O(14)—C(17)—C(1)—C(11) | −2·3 |
| O(24)—C(27)—C(2)—C(21) | −86·4 |
| O(24)—C(27)—C(2)—C(1) | 41·6 |
| O(24)—C(27)—C(2)—C(3) | 141·7 |

carboxylic acid $C_\alpha \cdots C_\alpha = 3{\cdot}05$, C—C = 1·546 (3) Å (van der Helm, Hsu & Sims, 1972).

Alternatively, the long bond in the present compound may originate in the electron-withdrawing properties of the substituent groups which, by lowering the electron density in the C(1)—C(2) bonding region, facilitate bond expansion. Again this does not appear to apply to the examples quoted above which also contain electron-withdrawing substituents. Additionally, in octahydroxycyclobutane (Bock, 1968) the ring bond lengths are only 1·562 (4) and 1·564 (4) Å, despite the strong electron-withdrawing effects of the hydroxyl groups and the close approaches (2·61–2·70 Å) of adjacent O atoms, none of which are involved in intramolecular hydrogen bonding.

Both phenyl groups are planar within experimental error; the nitro groups are twisted out of the planes of the benzene rings by 7·3 and 8·8°. The angular distortions within the six-membered ring may be correlated with the electron-withdrawing and electron-releasing effects of the substituent nitrophenyl and cyclobutyl groups respectively (Domenicano, Vaciago & Coulson, 1975). The lengths of the bonds connecting the 4-nitrophenyl groups to the cyclobutane ring are significantly unequal [C(1)—C(11), 1·509 (3); C(2)—C(21), 1·523 (3) Å] and correlate with the greater steric hindrance experienced by the pseudoaxial nitrophenyl group II over its pseudoequatorial counterpart. Steric effects also account for the relative orientations of these two groups.

As expected, both carbonyl C atoms lie within the plane defined by the three neighbouring atoms. As often found for methyl ester groups (Dunitz & Strickler, 1968) the methyl C atoms also lie within this plane, being synperiplanar with respect to the carbonyl C atom. Again the axial bond C(1)—C(17) [1·522 (4) Å] is slightly longer than its equatorial counterpart [C(2)—C(27) = 1·516 (3) Å]. The torsion angle O(14)—C(17)—C(1)—C(11) is −2·3° indicating that the carbonyl O atom is synperiplanar with a phenyl C atom. No such relation is observed for the second methoxycarbonyl group with respect either to atoms in the phenyl group II or to the neighbouring cyclobutane ring atoms C(1) and C(3).

The molecules form close-packed layers parallel to (010); within each layer every molecule is in contact with six others related to itself by *a* and *c* lattice translations. There are four such layers per unit cell corresponding to the four general positions. Each layer is sandwiched between a *c* glide and a $\bar{1}$-related layer. Four molecules in the *c*-glide-related layer and two in the $\bar{1}$-related layer bring the total number of contacts to 12. No unusually short intermolecular contacts are found.

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