Die Koppelung sollte beim entsprechenden Mangan-Komplex, falls er isotyp ist, wesentlich stärker sein. Entsprechende Versuche sind bereits im Gange.

Literatur

BERGERHOFF, G. & KASPER, H. (1968). *Acta Cryst.* B 24, 388.
BROWN, R. D. & COATES, G. E. (1962). *J. Chem. Soc. (London)*, p. 4723.
BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* 10, 180.
DUNITZ, J. D. (1957). *Acta Cryst.* 10, 307.
GOUBEAU, J. (1961). *Angew. Chem.* 73, 305.

GRAMMACCIOLI, C. M. & MARSH, R. E. (1966). *Acta Cryst.* 21, 594.
HARKER, D. (1936). *Z. Kristallogr.* 93, 136.
HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* 3, 210.
International Tables for X-ray Crystallography (1959). Vol. I. Birmingham: Kynoch Press.
JARVIS, J. A. J. (1962). *Acta Cryst.* 15, 964.
LINGAFELTER, E. C., SIMMONS, G. L., MOROSIN, B., SCHERINGER, C. & FREIBURG, C. (1961). *Acta Cryst.* 14, 1222.
MACGILLAVRY, C. H. & BIJVOET, J. M. (1936). *Z. Kristallogr.* 94, 231.
PETERSON, S. W. & LEVY, H. A., (1957). *J. Chem. Phys.* 26, 220.
RADEMACHER, P., STØLEVIK, R. & LÜTTKE, W. (1968). *Angew. Chem.* 80, 842.
SCHMIDPETER, A. (1963). *Chem. Ber.* 96, 3275.
TEMPLETON, D. H. (1962). University of California, Bericht UCRL–9146.
WEHE, D. J., BUSING, W. R. & LEVY, H. A. (1962). Oak Ridge National Laboratory, Bericht ORNL–229.

The Structure of 5-Norbornene-2,3-*endo*-dicarboxylic Anhydride

By RICCARDO DESTRO, GIUSEPPE FILIPPINI, CARLO MARIA GRAMACCIOLI AND MASSIMO SIMONETTA

Istituto di Chimica Fisica, Università di Milano, Milano, Italy

The crystal and molecular structure of 5-norbornene-2,3-*endo*-dicarboxylic anhydride* has been determined and refined by least-squares methods. The crystals are orthorhombic, space group $P2_12_12_1$, with $a = 13.539$, $b = 9.565$, $c = 5.972$ Å and four molecules per unit cell. Intensity data were collected visually from Weissenberg photographs about two crystal axes. The final R index is 0.071; the average standard deviation in the atomic positions is about 0.004 Å. The anisotropic temperature factors of the heavier atoms in the molecule can be accounted for by a Schomaker–Trueblood rigid-body treatment; on this basis, corrections to the observed bond distances are in the range 0.007–0.010 Å.

In the norbornene nucleus, the bridgehead angle C(1)–C(7)–C(4) is 93.5°. The geometry of the anhydride group is closely similar to that found in succinic anhydride. The two C–C single bonds which connect the anhydride ring with the cyclopentene ring are found to be significantly longer (1.57 and 1.58 Å) than normal. A similar feature observed in cyclopentadiene dimer probably accounts for its chemical reactivity in the Cope rearrangement.

Introduction

The conformation of molecules with internal strain is a subject of considerable recent interest. Among these molecules, the derivatives of norbornane and other related polycyclic hydrocarbons have been the object of several methods of structural investigation, including electron diffraction in the gas phase (Hamilton, 1954; Morino, Kuchitsu & Yokozechi, 1967) and X-ray diffraction of crystals (Baenziger, Doyle & Carpenter, 1961; Ferguson, Fritchie, Robertson & Sim, 1961; Bruechner, Hamer, Robertson & Sim, 1962; Cesur &

Grant, 1965; Baenziger, Richards & Doyle, 1965; MacDonald & Trotter, 1965a, b; Destro, Gramaccioli & Simonetta, 1968; Bellobono, Destro, Gramaccioli & Simonetta, 1969). At the same time, several techniques of semi-empirical calculations for the determination *a priori* of molecular geometry have been developed and applied to these substances (Wilcox, 1960; Gleicher & Schleyer, 1967; Allinger, Hirsch, Miller, Tyminski & Van Catledge, 1968; Simonetta Favini, Mariani & Gramaccioni, 1968; for a review of these methods see Williams, Stang & Schleyer, 1968). For further applications of these methods, precise structural data on some additional norbornene derivatives were considered desirable. For 5-norbornene-2,3-*endo*-dicarboxylic anhydride(I), a particular point of interest arises from

---

* Bicyclo[2,2,1]hept-5-ene-2,3-*endo*-dicarboxylic (acid) anhydride.

the resemblance of its conformation to that of cyclo-pentadiene dimer(II), whose remarkable ease in giving the Cope rearrangement is probably connected with its molecular geometry (Destro, Gramaccioli & Simonetta 1968; Bellobono, Destro, Gramaccioli & Simonetta, 1969).
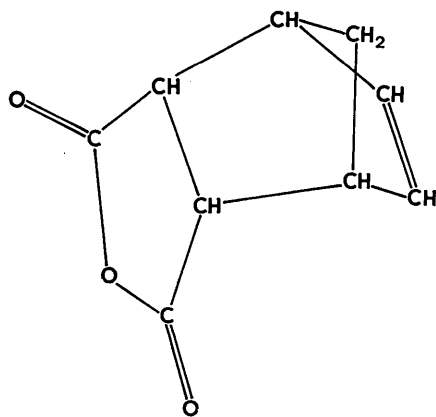
(I)
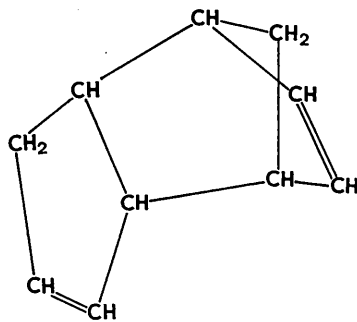
(II)

Experimental

Crystals of 5-norbornene 2,3-*endo*-dicarboxylic acid anhydride were obtained from the reaction of cyclo-pentadiene with maleic anhydride and recrystallized from benzene/light petroleum.* They are prismatic, elongated along **c**. Weissenberg photographs indicated them to be orthorhombic with space group $P2_12_12_1$ (the systematic absences are $h00$, $0k0$ and $00l$ with h, k or l odd).

The unit-cell dimensions (see Table 1) were obtained from a least-squares fit to measurements of $\sin^2 \theta$ for 40 $hk0$ and 48 $h0l$ reflexions on zero-level Weissen-berg photographs taken at 21 °C with Cu $K\alpha$ radiation, the film was held in the asymmetric position, essen-tially following the Straumanis technique. Eccentricity coefficients were included as parameters in the least-

---

squares calculations and weights were assigned as in-versely proportional to $\sin^2 2\theta$.

Table 1. *Crystal data for*
5-norbornene-2,3-endo-dicarboxylic anhydride

$a = 13\cdot5394 \pm 0\cdot0008$ Å	$C_9H_8O_3$
$b = 9\cdot5648 \pm 0\cdot0016$	M.W. 164·15
$c = 5\cdot9721 \pm 0\cdot0003$	$Z = 4$
	$F(000) = 344$
$D_m = 1\cdot417$ g.cm^{-3}	$D_x = 1\cdot409$ g.cm^{-3}

$$\lambda(\text{Cu } K\alpha_1) = 1\cdot54051 \text{ Å}$$
$$\lambda(\text{Cu } K\alpha_2) = 1\cdot54433$$
$$\lambda(\text{Cu } K\alpha) = 1\cdot5418$$

The density was measured by flotation in a dilute Thoulet (K_2HgI_4) solution.

For the determination of the structure, intensity data were collected from multiple-film equi-inclination Weis-senberg photographs. The layers 0–5 along **c** and 0–2 along **b** were obtained from two crystals, almost square in section, whose diameters were about 0·30 mm. Cop-per $K\alpha$ radiation was used. The intensities were esti-mated visually and corrected for Lorentz and polari-zation factors; no corrections either for absorption or for extinction were made.

The processing of the observed data was first carried on within the single layers, obtaining film and time factors by minimizing $\Sigma(\ln I_i - \ln KI_j)^2$, as indicated by Rae (1965). Standard deviations were assigned to single observations by means of an analysis of the residuals (Gramaccioli & Mariani, 1967). The evaluation of rela-tive scale factors for all the layers and subsequent aver-aging of the observed $|F|^2$ values were performed ac-cording to the method proposed by Rollett & Sparks, modified to account for the dependence of the weights upon the final scale factors (Hamilton, Rollett & Sparks, 1965; Duchamp, 1964). Of 1048 reflexions with-in the effective sphere of copper radiation, 950 were collected, of which 148 were too weak to be observed.

Determination and refinement of the structure

A sharpened three-dimensional Patterson synthesis gave the orientation of the C–O and O–O vectors in the anhydride group. Starting from these vectors, a plau-sible model for the whole molecule was assumed and found to be consistent with the maxima observed around the origin. The position of the molecule in the unit cell was found by carrying out a series of structure factor calculations based on low-angle ($\sin^2 \theta/\lambda^2 \leq 0\cdot15$) $hk0$ and $h0l$ reflexions, each time assigning a dif-ferent set of coordinates to the centre of the molecule and recording on a map the discrepancy index $R(=\Sigma||F_o| - |F_c||/\Sigma|F_o|)$. A program written for this purpose by Albano, Bellon, Pompa & Scatturin (1963b) was employed, where the speed of calculation is sub-stantially increased by the use of Fourier transforms. A considerable amount of time-consuming calcula-tion was avoided by excluding all positions forbidden

Table 2. *Observed and alculated structure factors*

The columns contain values of k, l, $10|F|_{obs}$, $10|F|_{cal}$, $\alpha(°)$.

by packing considerations; for this purpose the molecule was regarded as a sphere of minimum radius = 3 Å.

The R index of the 'best' result from these calculations was 0·42 for all observed reflexions.* Refinement of the positions of the heavier atoms by block-diagonal least-squares was then initiated, minimizing the function $\Sigma w(|F|_{obs} - |F|_{cal})^2$ and using for this purpose a program written by Albano, Bellon, Pompa & Scatturin (1963a) for our IBM 1620 computer. The first cycles were based on a relative small number of reflexions (72), each of them characterized by a low Bragg angle ($\sin^2 \theta/\lambda^2 \leq 0·08$) and by a relatively large value of the structure factor ($|F| \geq 10$); later on, all the remaining reflexions were included by gradually extending these limits. Initially unit weights were assigned to all the reflexions included; later, weights of the forms $w = 1/\bar{f}^2$ were used, where $\bar{f}$ is the average scattering factor for all the atoms at the calculated value of $\sin \theta/\lambda$. At the beginning, isotropic temperature factors were held constant and equal to 4·5 Å^2. Later on, when there were a sufficient number of reflexions in the refinement temperature factors were allowed to vary.

After the first least-squares cycles a constant decrease of the R index was noticed. At the end of this stage of the refinement (20 cycles) the R index was 0·15 for all observed data. This is essentially the same line of procedure as that adopted by us for 2,4,6-trinitrophenetole (Gramaccioli, Destro & Simonetta, 1968); the number of least-squares cycles does not involve a great deal of computing time, because the initial number of reflexions is very small. A three-dimensional difference Fourier synthesis was then calculated as a check on our results; this indicated small shifts in some atomic coordinates and temperature factors. All the hydrogen atoms were represented by sharp peaks at the expected positions. After a new cycle of least-squares, with the

---

* In this work, atomic scattering factors for hydrogen were taken from Forsyth & Wells (1959) and for the heavier atoms from Cromer & Waber (1965).

hydrogen atoms included in the structure-factor calculations, the R index was 0·12 for all observed reflexions. At this stage, the least-square refinement was continued on an IBM 7040 computer, using the program $ORFLS$ written by Busing, Martin & Levy (1962). Anisotropic temperature factors were adopted for all atoms except hydrogen, which were included only in the structure-factor calculations. Weights were assigned according to Hughes's scheme. For eight of the strongest reflexions ($|F|_{obs} > 44$), which were suspected of suffering from systematic errors of measurements, the weight was taken as zero; the reflexions too weak to be measured ('less than') were also omitted from the least-squares.

After three cycles, convergence was virtually reached at an R index of 0·076. Introducing new weights equal to $1/\sigma^2$ (where σ^2 is the variance in $|F|_{obs}$, derived during our data reduction) led to a slight improvement in the R index, which fell to 0·071 after three cycles. The molecular geometry also showed a slight improvement, the symmetry of the molecule becoming closer to m.

At this stage the refinement was considered complete. Nearly all the parameter shifts were about 0·1 times the standard deviation; the largest shift, 0·3σ, occurred for B_{33} of C(2).

A check of the final weighting functions by plotting the average value of $w(|F|_{obs} - |F|_{cal})^2$ for various intervals of $|F|$, as suggested by Cruickshank et al. (1961), showed this value to be approximately constant (within 16% of the average), whereas Hughes's weights were worse in this respect (variations of the order 1 to 2). However, the final goodness of fit $(\Sigma w(|F|_{obs} - |F|_{cal})^2/(n-p)^{1/2}$ is 4·97. Thus, these weights appear to be valid on a relative scale, but we were overly optimistic in assigning standard deviations to individual observations.

Precision of the results

The observed and final calculated structure factors are listed in Table 2; the final parameters of the heavier atoms are given in Table 3 and of the hydrogen atoms in Table 4.

Table 3. *The heavy atom parameters and their standard deviations*

All the values in this Table have been multiplied by 10^4. The temperature factor is in the form
$$\exp\left[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)\right].$$

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	366 (3)	2248 (5)	1800 (7)	46 (2)	132 (6)	267 (11)	−2 (3)	10 (3)	−1 (7)
C(2)	1331 (3)	1368 (4)	1987 (6)	54 (2)	96 (5)	187 (11)	−1 (3)	−1 (3)	10 (5)
C(3)	2012 (3)	2301 (5)	3392 (6)	50 (2)	109 (5)	188 (9)	7 (3)	3 (3)	3 (5)
C(4)	1341 (3)	3601 (5)	3949 (7)	50 (2)	109 (6)	271 (12)	3 (3)	10 (3)	−51 (6)
C(5)	1174 (3)	4358 (5)	1770 (8)	65 (3)	106 (5)	377 (16)	19 (4)	65 (5)	21 (7)
C(6)	597 (3)	3549 (6)	489 (7)	67 (3)	145 (7)	245 (13)	40 (4)	12 (4)	31 (7)
C(7)	353 (3)	2830 (6)	4233 (6)	49 (2)	148 (7)	266 (13)	6 (3)	32 (3)	−1 (7)
C(8)	1872 (3)	1173 (5)	−169 (6)	72 (3)	91 (5)	220 (11)	24 (3)	1 (4)	0 (6)
C(9)	2864 (3)	2636 (5)	1885 (6)	49 (2)	113 (5)	261 (12)	13 (3)	11 (3)	7 (6)
O(1)	2731 (2)	1971 (4)	−162 (5)	60 (2)	131 (4)	265 (9)	12 (2)	45 (3)	12 (5)
O(2)	1671 (3)	488 (4)	−1766 (5)	110 (3)	152 (5)	260 (10)	23 (3)	−27 (4)	−62 (6)
O(3)	3563 (2)	3367 (5)	2180 (6)	49 (2)	185 (6)	479 (14)	−12 (3)	14 (3)	9 (7)

Table 4. *Parameters for the hydrogen atoms*

	x	y	z	B
H(1)	−0·023	0·158	0·158	4·5 Å^2
H(2)	0·121	0·058	0·317	4·5
H(3)	0·218	0·170	0·500	4·0
H(4)	0·172	0·410	0·512	4·5
H(5)	0·148	0·512	0·145	5·5
H(6)	0·053	0·397	−0·132	5·5
H(7)	0·034	0·212	0·554	4·0
H(8)	−0·025	0·362	0·425	5·0

The standard deviations in the coordinates of the heavier atoms, derived from the residuals and the diagonal elements of the final least-squares cycle, correspond to positional uncertainties of the order of 0·004 Å. The corresponding standard deviations in the bond distances are around 0·007 Å and in the angles around 0·5°. No discussion of the precision of the C–H bonds and angles is thought to be appropriate.
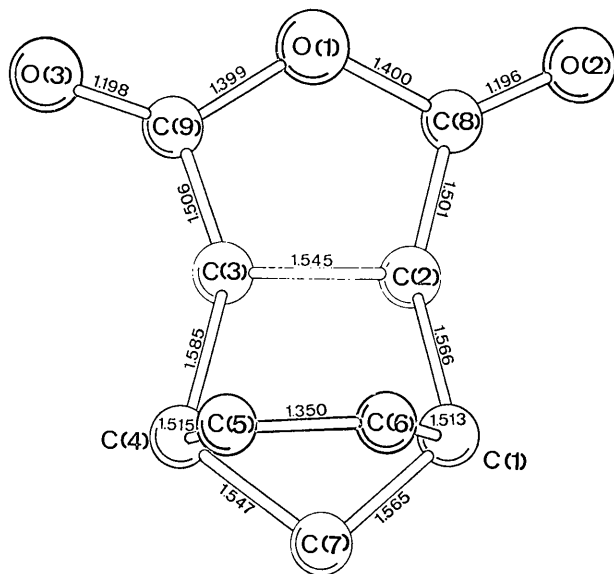
Fig. 1. The molecule of 5-norbornene-2,3-*endo*-dicarboxylic anhydride, viewed along the direction corresponding to the maximum moment of inertia. Bond distances are corrected for thermal libration.

Corrections for thermal libration

A 'rigid' molecule such as this anhydride was expected to behave as a rigid body in undergoing thermal librations. Accordingly the tensors **T**, **L**, and **S** (Schomaker & Trueblood, 1968) were derived from a least-squares treatment. A series of three programs for our IBM 1620 computer was written for this purpose (Filippini & Gramaccioli, 1969). In these calculations equal weights were assigned to all thermal factors; the results are shown in Table 5. The agreement between observed and calculated values of U_{ij} for each atom is fairly good, the differences rarely exceeding the standard deviation. The mean-square rotational displacements of the molecule amount to 29·20 and 11 (°)^2 about the principal axes of the tensor **L**.

The corrections for distances have been derived under the rigid-body assumption from the tensor **L**, according to Schomaker & Trueblood (1968). This procedure resulted in a substantial increase for all of them (0·007 to 0·010 Å). A negligible correction of the bond angles (0·1° to 0·2°) was found.* Values for all bond distances and angles before and after corrections for thermal libration are given in Table 6.

Discussion

The molecular structure

The molecule as viewed along the direction corresponding to the maximum moment of inertia is shown in Fig. 1. The conformation of the norbornene nucleus is in essential agreement with the results of previous crystallographic work, such as the structure of *anti*-7-norbornenyl *p*-bromobenzoate (Macdonald & Trotter, 1965*b*), where most of the bond lengths and angles are nearly identical with ours. A useful comparison can be made also with the tricyclo[3,2,1,0² ⁴]octane nucleus (Macdonald & Trotter, 1965*a*) and especially with cyclopentadiene dimer (Destro, Gramaccioli & Simonetta, 1968; Bellobono, Destro, Graccioli & Simonetta,

* The correction for bond angles was made under the assumption that triangulation is valid for deducing intramolecular angles from corrected distances when dealing with a rigid body. It is clear that in a general case this cannot be done (Busing & Levy, 1964).

Table 5. *Rigid-body tensors for 5-norbornene-2,3-endo-dicarboxylic anhydride, referred to a Cartesian coordinate system, whose origin and directions of axes coincide with the crystallographic axes*

Standard deviations are given in brackets.

T (Å^2)	943 (39)	−305 (44)	−191 (43)	
(× 10⁴)		941 (45)	−256 (54)	('unreduced')
			969 (86)	
L (rad²)	68 (7)	−18 (6)	−18 (5)	
		47 (6)	−1 (4)	
			67 (6)	
S (Å.rad)	14 (12)	112 (14)	−171 (22)	
(× 10⁴)	−75 (12)	−37 (13)	139 (19)	[trace (**S**) = 0]
	166 (16)	−177 (16)	23 (17)	

1969), whose conformation is, on the whole, not very different from that of our molecule, the anhydride group being replaced by another five-membered ring. In cyclopentadiene dimer we found a single bond considerably longer than usual ($1\cdot59 \pm 0\cdot013$ Å, uncorrected for thermal libration). This fact could account, at least in part, for the ease with which the substance undergoes certain chemical reactions, such as the Cope rearrangement, where this bond is broken; the lengthening of this bond is probably the result of repulsion between non-bonded atoms in the same molecule.

A similar situation might be expected to occur in the anhydride. Owing to the m symmetry of the isolated molecule, there are two bonds corresponding to the 'stretched' bond in cyclopentadiene dimer, C(1)–C(2) and C(3)–C(4). Both are found to be longer than usual.

Another point of interest is the value of the bridgehead angle C(1)–C(7)–C(4), which is very far from the tetrahedral value, as in all these compounds. Our result ($93\cdot5°$) is close to the values $92\cdot7$ ($\pm0\cdot8°$) found in cyclopentadiene dimer and $93\cdot2°$ ($\pm1\cdot5$) and $92\cdot0°$ ($\pm0\cdot8°$) obtained for norbornane and norbornadiene respectively, by electron diffraction in the gas phase (Morino, Kuchitsu & Yokozechi, 1967). The values reported by Macdonald & Trotter (1965a, b) are slightly higher ($96°$ and $97° \pm1\cdot2°$ in the tricyclooctane and norbornene nuclei respectively). The difference between these two and the others is at the limit of signi-

Table 6. *Bond distances and angles in the anhydride*

The two values relate to uncorrected and corrected values respectively for thermal motion. No corrections have been applied to data involving hydrogen atoms.

C(1)–C(2)	1·558 Å	1·566 Å	C(1)–H(1)	1·04 Å
C(1)–C(6)	1·504	1·513	C(2)–H(2)	1·05
C(1)–C(7)	1·556	1·565	C(3)–H(3)	1·14
C(2)–C(3)	1·534	1·545	C(4)–H(4)	0·99
C(2)–C(8)	1·493	1·501	C(5)–H(5)	0·86
C(3)–C(4)	1·576	1·585	C(6)–H(6)	1·16
C(3)–C(9)	1·498	1·506	C(7)–H(7)	1·03
C(4)–C(5)	1·506	1·515	C(7)–H(8)	1·11
C(4)–C(7)	1·537	1·547		
C(5)–C(6)	1·340	1·350		
C(8)–O(1)	1·390	1·400		
C(8)–O(2)	1·188	1·196		
C(9)–O(1)	1·390	1·399		
C(9)–O(3)	1·190	1·198		

C(2)–C(1)–C(6)	108·1°	108·1°
C(2)–C(1)–C(7)	97·8	97·7
C(6)–C(1)–C(7)	101·1	101·0
C(1)–C(2)–C(3)	103·3	103·3
C(1)–C(2)–C(8)	114·7	114·6
C(3)–C(2)–C(8)	104·4	104·4
C(2)–C(3)–C(4)	103·1	103·2
C(2)–C(3)–C(9)	105·0	105·1
C(4)–C(3)–C(9)	113·7	113·6
C(3)–C(4)–C(5)	106·5	106·5
C(3)–C(4)–C(7)	98·4	98·3
C(5)–C(4)–C(7)	101·3	101·3
C(4)–C(5)–C(6)	107·7	107·6
C(5)–C(6)–C(1)	107·6	107·6
C(1)–C(7)–C(4)	93·4	93·5
C(2)–C(8)–O(1)	109·8	109·8
C(2)–C(8)–O(2)	130·4	130·3
O(1)–C(8)–O(2)	119·8	119·9
C(3)–C(9)–O(1)	109·3	109·2
C(3)–C(9)–O(3)	130·5	130·5
O(1)–C(9)–O(3)	120·2	120·3
C(8)–O(1)–C(9)	111·3	111·4

H(1)–C(1)–C(2)	109°	H(5)–C(5)–C(4)	122°
H(1)–C(1)–C(6)	127	H(5)–C(5)–C(6)	130
H(1)–C(1)–C(7)	109	H(6)–C(6)–C(1)	139
H(2)–C(2)–C(1)	108	H(6)–C(6)–C(5)	112
H(2)–C(2)–C(3)	98	H(7)–C(7)–C(1)	118
H(2)–C(2)–C(8)	125	H(7)–C(7)–C(4)	114
H(3)–C(3)–C(2)	107	H(8)–C(7)–C(1)	105
H(3)–C(3)–C(4)	110	H(8)–C(7)–C(4)	108
H(3)–C(3)–C(9)	117	H(8)–C(7)–H(7)	115
H(4)–C(4)–C(3)	103		
H(4)–C(4)–C(5)	117		
H(4)–C(4)–C(7)	127		

ficance; it may well be connected with the fact that in both compounds a p-bromobenzoyl group is attached to the 'apical' position of the bridge.

Equations of the planes through the regions of the molecule which are of particular interest are given in Table 7, together with interplanar angles and the de-

Table 7. *Some planes of interest*

The coefficients q_i are the direction cosines relative to the crystallographic axes a, b and c. Atoms were given equal weight in the least-squares treatment, which follows the method described by Schomaker, Waser, Marsh & Bergman (1959).

Plane A		Plane B		Plane C		Plane D	
	Δ		Δ		Δ		Δ
C(1)	−0·010 Å	C(1)	0·001 Å	C(1)	(0·000 Å)	C(2)	0·035 Å
C(2)	0·014	C(4)	−0·001	C(4)	(0·000)	C(3)	−0·029
C(3)	−0·014	C(5)	0·002	C(7)	(0·000)	C(9)	−0·013
C(4)	0·010	C(6)	−0·002			C(8)	0·002
						O(1)	0·027
						O(2)	−0·026
						O(3)	0·004

Plane A:	$q_1 = -0\cdot3430$	$q_2 = -0\cdot4517$	$q_3 = 0\cdot8237$	$D = -0\cdot246$ Å
Plane B:	−0·8099	0·4428	0·3846	0·963
Plane C:	−0·4888	0·8127	−0·3171	1·164
Plane D:	−0·4941	0·7869	−0·3696	−0·334

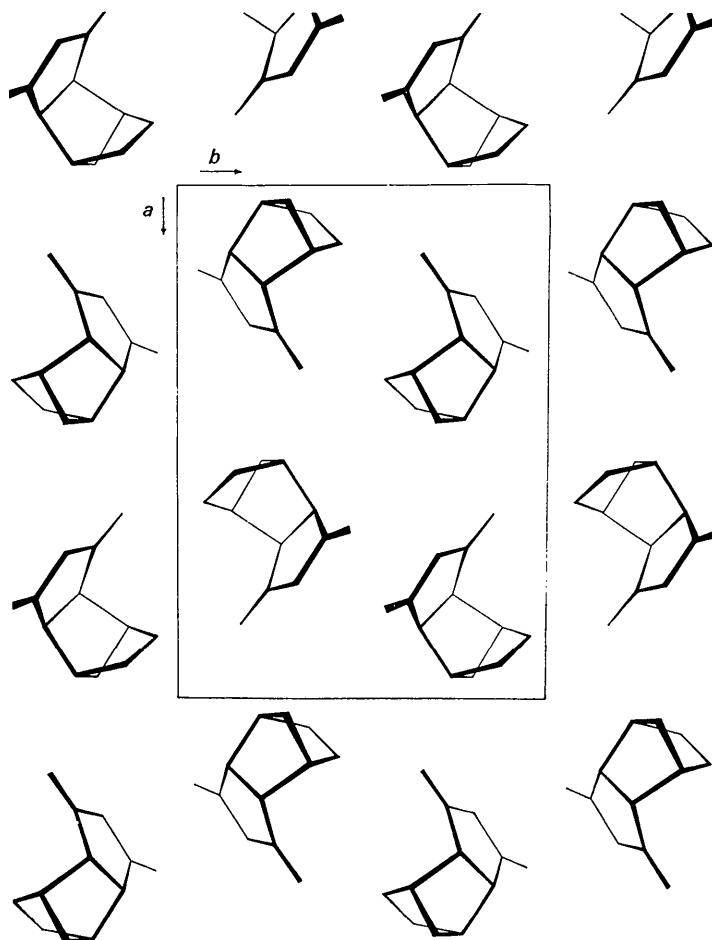| Dihedral angle		
	$A \wedge B$	113·2°
	$A \wedge C$	117·4
	$B \wedge C$	129·3
	$A \wedge D$	119·3

Fig. 2. Molecular packing viewed along c.

A C 25B – 4

viations of the individual atoms from the planes. These deviations appear to be significant in the anhydride group (up to 0·035 Å); the group is, however, still virtually planar and, on the whole, its geometry is in close agreement with the results obtained for succinic anhydride by Ehrenberg (1965) and by Biagini & Cannas (1965).

Molecular packing

If we assume as van der Waals radii for carbon, oxygen and hydrogen the values 1·8, 1·4 and 1·2 Å respectively (Kitaigorodskii, 1961), we see that in only six cases do two atoms belonging to different molecules lie at a distance smaller than the sum of the corresponding van der Waals radii. A summary of these distances is given in Table 8. The molecular packing seen along **c** is represented in Fig.2.

Table 8. *Intermolecular contact distances*

(Uncorrected for thermal libration.)

C(1)–C(5)	$-x, y-\frac{1}{2}, \frac{1}{2}-z$	3·565 Å
C(5)–H(1)	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	2·67
C(7)–H(6)	$x, y, z+1$	2·88
C(8)–H(3)	$x, y, z-1$	2·96
C(9)–O(2)	$\frac{1}{2}-x, -y, \frac{1}{2}+z$	3·159
O(2)–H(3)	$x, y, z-1$	2·36

References

ALBANO, V., BELLON, P. L., POMPA, F. & SCATTURIN, V. (1963a). *Ric. Sci.* **33** (II-A), 1067.

ALBANO, V., BELLON, P. L., POMPA, F. & SCATTURIN, V. (1963b). *Ric. Sci.* **33** (II-A), 291.

ALLINGER, N. L., HIRSCH, J. A., MILLER, M. A., TYMINSKI, I. & VAN CATLEDGE, F. A. (1968). *J. Amer. Chem. Soc.* **90**, 1199.

BAENZIGER, N. C., DOYLE, J. R. & CARPENTER, C. L. (1961). *Acta Cryst.* **14**, 303.

BAENZIGER, N. C., RICHARDS, G. F. & DOYLE, J. R. (1965). *Acta Cryst.* **18**, 924.

BELLOBONO, I. R., DESTRO, R., GRAMACCIOLI, C. M. & SIMONETTA, M. (1969). *J. Chem. Soc.* In the press.

BIAGINI, S. & CANNAS, M. (1965). *Ric. Sci.* **8** (II-A), 1518.

BRUECHNER, D. A., HAMER, T. A., ROBERTSON, J. M. & SIM, G. A. (1962). *J. Chem. Soc.* p. 799.

BUSING, W. D. & LEVY, H. A. (1964). *Acta Cryst.* **17**, 142.

BUSING, W. D., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS.* U.S. Atomic Energy Commission Publication ORNL-TM 305.

CESUR, F. & GRANT, D. F. (1965). *Acta Cryst.* **18**, 55.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104.

CRUICKSHANK, D. W. J. *et al.* (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis,* Vol. 6. Oxford: Pergamon Press.

DESTRO, R., GRAMACCIOLI, C. M. & SIMONETTA, M. (1968). *Chem. Comm.* 568.

DUCHAMP, D. J. (1964). A.C.A. Meeting, Bozeman, Montana, paper B-14, p. 29.

EHRENBERG, M. (1965). *Acta Cryst.* **19**, 698.

FERGUSON, G., FRITCHIE, C. J., ROBERTSON, J. M. & SIM, G. A. (1961). *J. Chem. Soc.* p. 1976.

FILIPPINI, G. & GRAMACCIOLI, C. M. (1969). To be published.

FORSYTH, J. B. & WELLS, M. (1959). *Acta Cryst.* **12**, 412.

GLEICHER, G. J. & SCHLEYER, P. VON R. (1967). *J. Amer. Chem. Soc.* **89**, 582.

GRAMACCIOLI, C. M. & MARIANI, C. (1967). *R.C. Ist. Lombardo,* A **101**, 704.

GRAMACCIOLI, C. M., DESTRO, R. & SIMONETTA, M. (1968). *Acta Cryst.* B **24**, 129.

HAMILTON, W. C. (1954). Ph. D. Thesis, California Institute of Technology.

HAMILTON, W. C., ROLLETT, J. S. & SPARKS, R. A. (1965). *Acta Cryst.* **18**, 249.

KITAIGORODSKII, A. I. (1961). *Organic Chemical Crystallography,* English ed. p. 7. New York: Consultants Bureau.

MACDONALD, A. C. & TROTTER, J. (1965a). *Acta Cryst.* **18**, 243.

MACDONALD, A. C. & TROTTER, J. (1965b). *Acta Cryst.* **19**, 456.

MORINO, Y., KUCHITSU, K. & YOKOZECHI, A. (1967). *Bull. Chem. Soc. Japan,* **40** (6), 1552.

RAE, A. D. (1965). *Acta Cryst.* **19**, 683.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B **24**, 63.

SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600.

SIMONETTA, M., FAVINI, G., MARIANI, C. & GRAMACCIONI, P. (1968). *J. Amer. Chem. Soc.* p. **90**, 1280.

WILCOX, C. F. JR (1960). *J. Amer. Chem. Soc.* **82**, 414.

WILLIAMS, J. E., STANG, P. J., SCHLEYER, P. VON R. (1968) *Ann. Rev. Phys. Chem.* Vol. 10, p. 531.