by expanding it and terminating the series at the quadratic term.

Hence the stationary neighbour assumption is equivalent to the assumption of uncorrelated motion and implies an incorrect value of the energy at the starting point of the motion.

References

ABRAHAMS, S. C., ROBERTSON, J. M. & WHITE, J. G. (1949). *Acta Cryst.* **2**, 233–244.

BAILEY, M. F. & DAHL, L. F. (1965). *Inorg. Chem.* **4**, 1306–1314.

BERNSTEIN, J. & TRUEBLOOD, K. N. (1971). *Acta Cryst.* B**27**, 2078–2089.

COIRO, V. M., GIACOMELLO, P. & GIGLIO, E. (1971). *Acta Cryst.* B**27**, 2112–2119.

CRAIG, D. P., MASON, R., PAULING, P. & SANTRY, D. P. (1965). *Proc. Roy. Soc.* A**286**, 98–116.

FILIPPAKIS, S. E., LEISEROWITZ, L. & SCHMIDT, G. M. J. (1967). *J. Chem. Soc.* (*B*), pp. 305–311.

GIGLIO, E. (1969). *Nature, Lond.* **222**, 339–341.

GOLDBERG, I. & SHMUELI, U. (1973*a*). *Acta Cryst.* B**29**, 421–431.*

GOLDBERG, I. & SHMUELI, U. (1973*b*). *Acta Cryst.* B**29**, 432–440.

GOLDBERG, I. & SHMUELI, U. (1973*c*). *Acta Cryst.* B**29**, 440–448.

GOLDBERG, I. & SHMUELI, U. (1973*d*). *Cryst. Struct. Commun.* **2**, 175–179.

HERBSTEIN, F. H. (1971). In *Perspectives in Structural Chemistry*. Vol. IV, pp. 166–395. New York: John Wiley.

KUAN, T., WARSHEL, A. & SCHNEPP, O. (1970). *J. Chem. Phys.* **52**, 3012–3020.

KUMAKURA, S., IWASAKI, F. & SAITO, Y. (1967). *Bull. Chem. Soc. Japan*, **40**, 1826–1833.

LYUBARSKII, G. YA. (1960). *The Application of Group Theory in Physics*. Oxford: Pergamon Press.

MASON, R. (1964). *Acta Cryst.* **17**, 547–555.

PAWLEY, G. S. (1967). *Phys. Stat. Sol.* **20**, 347–360.

RIETVELD, H. M., MASLEN, E. N. & CLEWS, C. J. B. (1970). *Acta Cryst.* B**26**, 693–706.

SCHERINGER, C. (1972). *Acta Cryst.* A**28**, 512–515.

TRUEBLOOD, K. N. (1969). In *Molecular Dynamics and Structure of Solids*. pp. 355–379. Edited by R. S. CARTER & J. J. RUSH. N.B.S. Special Publ. No. 301.

TSUCHIYA, H., MARUMO, F. & SAITO, Y. (1972). *Acta Cryst.* B**28**, 1935–1941.

WILLIAMS, D. E. (1966). *J. Chem. Phys.* **45**, 3770–3778.

WILLIAMS, D. E. (1967). *J. Chem. Phys.* **47**, 4680–4684.

WILLIAMS, D. E. (1972). *Acta Cryst.* A**28**, 84–88.

WILLIAMS, D. E. (1973). *Acta Cryst.* B**29**, 96–102.

WILLIAMS, R. M. & WALLWORK, S. C. (1967). *Acta Cryst.* **22**, 899–906.

WILLIAMS, R. M. & WALLWORK, S. C. (1968). *Acta Cryst.* B**24**, 168–174.

* In Goldberg & Shmueli (1973*a*) there is an error in the legend to Table 3. Instead of *Fractional atomic coordinates* ($\times 10^4$) the legend should read *Fractional atomic coordinates of the heavy atoms* ($\times 10^4$) *and of the hydrogen atoms* ($\times 10^3$).

Acta Cryst. (1973). B**29**, 2471

The Structure of *anti*-Tricyclo[4,2,2,0^{2,5}]deca-3,9-diene-7,8-*endo*-dicarboxylic Anhydride

BY GIUSEPPE FILIPPINI, GIANMARCO INDUNI AND MASSIMO SIMONETTA

Istituto di Chimica Fisica e Centro C.N.R., Universitá, via Saldini 50, 20133 *Milano, Italy*

(*Received* 21 *June* 1973; *accepted* 22 *June* 1973)

The crystal and molecular structure of the title compound, $C_{12}H_{10}O_3$, has been determined by direct methods and refined by full matrix least-squares calculations. The crystals are monoclinic, space group $P2_1/c$, with $a=6{\cdot}385$ (1), $b=21{\cdot}922$ (2), $c=6{\cdot}622$ (1) Å, $\beta=91{\cdot}76$ (2)°, $Z=4$. Intensities for 1487 independent reflexions were collected visually from Weissenberg photographs. The final R for 1010 observed reflexions is 0·088. Within experimental error the molecule has symmetry m. The geometry is in close agreement with the results found for bicyclo[2,2,2]octene-2,3-*endo*-dicarboxylic anhydride. The anhydride group is significantly non-planar as in other derivatives. The anisotropic temperature factors of the heavier atoms can be accounted for by a Schomaker–Trueblood rigid-body treatment; on this basis, corrections to the observed bond distances are about 0·006 Å.

Introduction

The structure of *anti*-tricyclo[4,2,2,0^{2,5}]deca-3,9-diene-7,8-*endo*-dicarboxylic anhydride (TRIC) has been determined as part of a systematic study of a series of chemically related compounds (Destro, Filippini, Gramaccioli & Simonetta, 1969, 1971; Filippini, Gramaccioli, Rovere & Simonetta, 1972).

The knowledge of an accurate experimental conformation of these molecules was considered necessary in view of theoretical work we are carrying out on an extensive group of rigid molecules with internal strain (Gramaccioli, Simonetta & Suffritti, 1973; Filippini, Gramaccioli, Simonetta & Suffritti, 1973).

Experimental

Crystals of TRIC were obtained from reaction of cyclooctatetraene with maleic anhydride and recrystallized from benzene/light petroleum (Reppe, Schichting, Klager & Toeppel, 1948).

Weissenberg photographs indicated the crystals to

be monoclinic, space group $P2_1/c$ (the systematic absences are $0k0$ and $h0l$ with k or l odd).

The unit-cell dimensions (Table 1) were obtained from a least-squares fit to measurements of $\sin^2\theta$ on zero-level Weissenberg photographs (taken at 21 °C with Cu $K\alpha$ radiation, the film being held in the asymmetric position, following the Straumanis technique) and of 2θ values on a diffractometer. Eccentricity coefficients were included as parameters in the least-squares calculations and weights were assigned as inversely proportional to $\sin^2 2\theta$.

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

For the determination and the refinement of the structure, intensities were collected from multiple film equi-inclination Weissenberg photographs and measured visually. The layers 0–5 along **a** were obtained from one crystal, almost square in section, whose diameter was about 0·2 mm. A total of 1487 reflexions were collected, of which 450 were too weak to be observed ('less than'). The intensities were corrected for Lorentz and polarization effects but not for absorption ($\mu R = 0{\cdot}17$) or extinction.

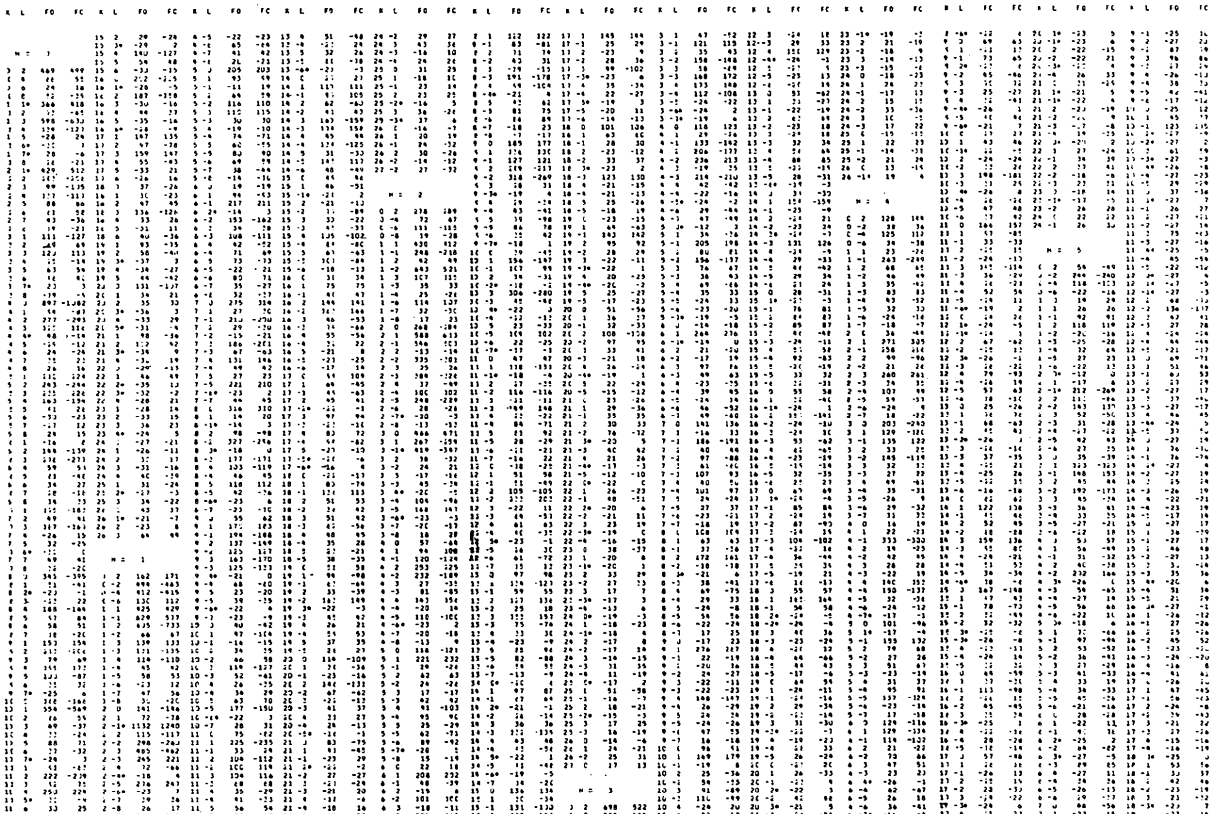
Previously (Filippini & Simonetta, 1970) data (layers 0–6 along **a**) had been obtained from the same crystal

Table 1. *Crystal data*

$C_{12}H_{10}O_3$	F.W. 202·16
Monoclinic	Space group $P2_1/c$
$a = 6{\cdot}385$ (1) Å	λ(Cu $K\alpha_1$) = 1·54051 Å
$b = 21{\cdot}922$ (2)	λ(Cu $K\alpha_2$) = 1·54433
$c = 6{\cdot}622$ (1)	λ(Cu $K\alpha$) = 1·5418
$\beta = 91{\cdot}76$ (2)°	μ(Cu $K\alpha$) = 8·7 cm^{-1}
$D_m = 1{\cdot}44$ g cm^{-3}	$F(000) = 424$
$D_x = 1{\cdot}448$ g cm^{-3}	$Z = 4$

Table 2. *Observed and calculated structure factors* (× 10)

'Less than' reflexions are indicated as negative F_o. Reflexions marked by an asterisk and 'less than' were omitted from the fina least-squares cycles. Form factors for heavier atoms were taken from Cromer & Waber (1965) and for H atoms from Stewart, Davidson & Simpson (1965).



with an equi-inclination Philips diffractometer and Mo $K\alpha$ radiation, monochromated by an Si[111] crystal. The abundance of weak reflexions rendered our measurements rather unsatisfactory. Consequently, these data were used only for interlayer scaling.

The processing of the observed intensities was first carried out within the single layers, obtaining film and time factors by minimizing $\sum(\ln I_i - \ln KI_j)^2$, as indicated by Rae (1965). Standard deviations were assigned to single observations by means of an analysis of the residuals (Gramaccioli & Mariani, 1967).

Solution and refinement of the structure

Approximate absolute scale and temperature factors were obtained by Wilson's method. For the solution of the phase problem the structure factors were converted to the normalized structure factors $|E_{hkl}|$ (Hauptman & Karle, 1953). The values of the statistical averages $\langle|E|\rangle$ and $\langle|E^2-1|\rangle$ were 0·781 and 1·031 respectively, confirming a centrosymmetric structure. The sign determination was undertaken by application of the Sayre relationship (Sayre, 1952) to 177 reflexions with $E > 1{\cdot}5$. The whole process was performed with a Fortran IV computer program (Long, 1965).

The E map (Karle, Hauptman, Karle & Wing, 1958) computed with the phases corresponding to the highest consistency index (0·81) revealed all the carbon and oxygen atoms. The structure-factor calculation including all observed data gave an R of 0·24.

Refinement was performed by a full-matrix least-squares process, with a modified version of the *ORFLS* program (Busing, Martin & Levy, 1962), minimizing the function $\sum w(|F_o|-|F_c|)^2$ and adopting unit weights. Of the 1037 observed reflexions 27 were omitted as being probably affected by extinction or other observational errors. Only position and isotropic temperature factors of the heavier atoms were allowed to vary. After four cycles R was 0·12. Subsequently anisotropic temperature factors were adopted for carbon and oxygen atoms; the hydrogen atoms were at first included only in the structure-factor calculations, with positional parameters derived from the presumed molecular geometry and isotropic temperature factors of 4·0 Å^2; subsequently, coordinates and isotropic temperature factors of the hydrogen atoms were included in the refinement. Weights were taken as $w = 1/(a+b|F_o|+cF_o^2)$ (Cruickshank, 1961), where a, b and c were derived from a least-squares fit of $(|F_o|-|F_c|)^2$ as a function of $|F_o|$. Owing to some irregularity in the distribution of $w\Delta F^2$ *versus* $|F_o|$, the ΔF's were fitted to two parabolae, one for $F_o \leq 10$ and the other for $F_o > 10$. The final R was 0·088. No parameter shifts exceeded 0·15 times the corresponding standard deviation.

The observed and calculated structure factors are listed in Table 2 and the final parameters of the atoms in Tables 3 and 4. The standard deviations in the coordinates, derived from the residuals and the diagonal elements of the inverse matrix of the final least-squares cycle, correspond to positional uncertainties of the order of 0·006 Å for the C and O atoms and of 0·06 Å for the H atoms.

Table 4. *Parameters for the hydrogen atoms*

The atoms are numbered according to the C atom to which they are bonded.

	x	y	z	$10B$(Å^2)
H(1)	0·639 (7)	0·272 (2)	0·249 (7)	43 (9)
H(2)	0·653 (10)	0·329 (3)	−0·046 (10)	73 (15)
H(3)	0·235 (11)	0·315 (3)	−0·070 (10)	76 (16)
H(4)	0·191 (9)	0·434 (3)	−0·035 (8)	55 (12)
H(5)	0·615 (10)	0·440 (3)	−0·014 (9)	73 (14)
H(6)	0·541 (9)	0·476 (2)	0·314 (8)	59 (12)
H(7)	0·906 (9)	0·442 (2)	0·271 (8)	53 (11)
H(8)	0·923 (11)	0·333 (3)	0·233 (9)	66 (14)
H(9)	0·380 (8)	0·308 (2)	0·495 (8)	55 (11)
H(10)	0·336 (7)	0·418 (2)	0·514 (7)	44 (10)

Correction for thermal libration

The molecule of TRIC is expected to behave as a rigid body when undergoing thermal libration. Accordingly the tensors **T**, **L** and **S** (Schomaker & Trueblood, 1968) were derived from a least-squares treatment, with a program written by one of us (G.F.). In these calcula-

Table 3. *Heavy atom parameters and their standard deviations* ($\times 10^4$)

The temperature factor is in the form $T_l = \exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{13}hl+2b_{23}kl)]$.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	6109 (8)	3175 (2)	2551 (8)	274 (17)	20 (1)	235 (12)	−3 (3)	10 (11)	1 (3)
C(2)	5503 (9)	3456 (2)	480 (8)	309 (20)	24 (1)	252 (14)	1 (3)	16 (13)	−10 (3)
C(3)	3238 (11)	3470 (3)	−288 (9)	396 (24)	27 (1)	254 (15)	−13 (4)	−2 (14)	−4 (3)
C(4)	3052 (10)	4070 (3)	−91 (8)	345 (22)	30 (1)	267 (15)	13 (4)	−26 (14)	5 (4)
C(5)	5254 (10)	4163 (2)	708 (8)	353 (20)	22 (1)	229 (13)	−8 (3)	11 (13)	7 (3)
C(6)	5668 (8)	4340 (2)	2942 (7)	292 (18)	18 (1)	229 (12)	0 (3)	22 (11)	−6 (3)
C(7)	7958 (8)	4159 (2)	3436 (8)	251 (17)	21 (1)	238 (13)	−15 (3)	21 (11)	−3 (3)
C(8)	8245 (8)	3472 (2)	3148 (8)	231 (17)	21 (1)	272 (13)	4 (3)	39 (12)	2 (3)
C(9)	4583 (8)	3369 (2)	4055 (8)	242 (16)	24 (1)	260 (13)	−12 (3)	32 (11)	8 (3)
C(10)	4334 (8)	3962 (2)	4263 (7)	215 (16)	27 (1)	217 (12)	1 (3)	26 (11)	−6 (3)
C(11)	8547 (8)	4264 (2)	5633 (8)	255 (17)	25 (1)	265 (14)	−13 (3)	14 (11)	−8 (3)
C(12)	9076 (9)	3246 (2)	5137 (9)	237 (17)	25 (1)	339 (16)	−1 (3)	41 (12)	9 (3)
O(1)	9216 (6)	3725 (2)	6517 (6)	320 (13)	30 (1)	243 (10)	−4 (3)	−48 (8)	4 (2)
O(2)	8490 (7)	4716 (2)	6613 (6)	385 (14)	30 (1)	322 (11)	−15 (3)	8 (10)	−26 (3)
O(3)	9603 (7)	2752 (2)	5678 (7)	395 (15)	28 (1)	400 (13)	7 (3)	−23 (11)	27 (3)

tions, equal weights were assigned to the thermal parameters of the heavy atoms; the results are shown in Table 5. The agreement between observed and calculated values of B_{ij} for each atom is good, no difference exceeding 3σ. The mean square rotational displacements of the molecule amount to 20, 11, 10 $(°)^2$ about the principal axes of the tensor **L**. The thermal ellipsoids are shown in Fig. 1.

Table 5. *Rigid-body tensors for anti-tricyclo*[4,2,2,$0^{2,5}$]-*deca*-3,9-*diene*-7,8-*endo-dicarboxylic anhydride*

The tensors are referred to a Cartesian coordinate system defined by unit vectors **a***, **b**, **a*** × **b**. All values have been multiplied by 10^4.

T(Å^2)	3789 (400)	−1390 (164)	−1149 (314)
		1188 (74)	−212 (113)
			3922 (417)
L(rad^2)	45 (5)	3 (2)	15 (4)
		32 (3)	2 (3)
			47 (6)
S(Å. rad)	117 (35)	22 (12)	−369 (45)
	−68 (23)	−6 (11)	95 (26)
	382 (46)	−169 (20)	−112 (36)

The correction of the distances was derived, under the rigid-body assumption, from the tensor **L**. This procedure resulted in a moderate increase (around 0·006 Å) in all of them; a negligible correction for the bond angles (less than 0·1°) was found.

Results and discussion

The molecule as viewed along the direction corresponding to the maximum moment of inertia is shown in Fig. 2, with bond distances and angles involving the C and O atoms: values in parentheses include a correction for thermal motion. Distances and angles involving the hydrogen atoms are given in Table 6. The standard deviations in the bond distances involving only the heavy atoms are about 0·0075 Å and in the angles 0·5°; in the C–H bond distances they are about 0·055 Å and in the C–C–H angles about 3–4°.

Table 6. *Bond distances and angles involving the hydrogen atoms*

C(1)–H(1)	1·01 Å	C(6)—H(6)	0·95 Å
C(2)–H(2)	0·99	C(7)—H(7)	1·03
C(3)–H(3)	0·93	C(8)—H(8)	0·90
C(4)–H(4)	0·95	C(9)—H(9)	1·01
C(5)–H(5)	0·96	C(10)–H(10)	0·99
C(2)—C(1)–H(1)	114°	C(5)—C(6)—H(6)	111°
C(8)—C(1)–H(1)	106	C(7)—C(6)—H(6)	113
C(9)—C(1)–H(1)	115	C(10)–C(6)—H(6)	111
C(2)—C(3)–H(3)	130	C(5)—C(4)—H(4)	133
C(4)—C(3)–H(3)	135	C(3)—C(4)—H(4)	132
C(1)—C(2)–H(2)	105	C(6)—C(5)—H(5)	110
C(3)—C(2)–H(2)	116	C(4)—C(5)—H(5)	116
C(5)—C(2)–H(2)	120	C(2)—C(5)—H(5)	114
C(6)—C(7)–H(7)	115	C(1)—C(8)—H(8)	109
C(11)–C(7)–H(7)	102	C(12)–C(8)—H(8)	100
C(8)—C(7)–H(7)	113	C(7)—C(8)—H(8)	120
C(1)—C(9)–H(9)	125	C(6)—C(10)–H(10)	117
C(10)–C(9)–H(9)	119	C(9)—C(10)–H(10)	129

The molecular symmetry is very close to *m*, there being no difference between 'chemically equivalent' bond distances and angles exceeding 3σ: this seems to indicate

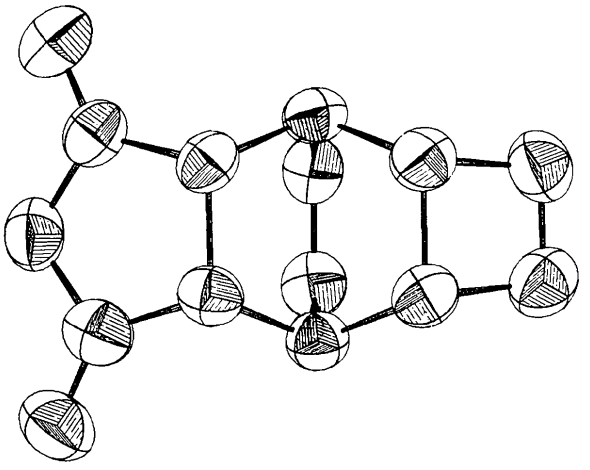


Fig. 1. The molecule viewed along the direction corresponding to the maximum moment of inertia. The thermal ellipsoids are drawn at 50% probability (Johnson, 1965).

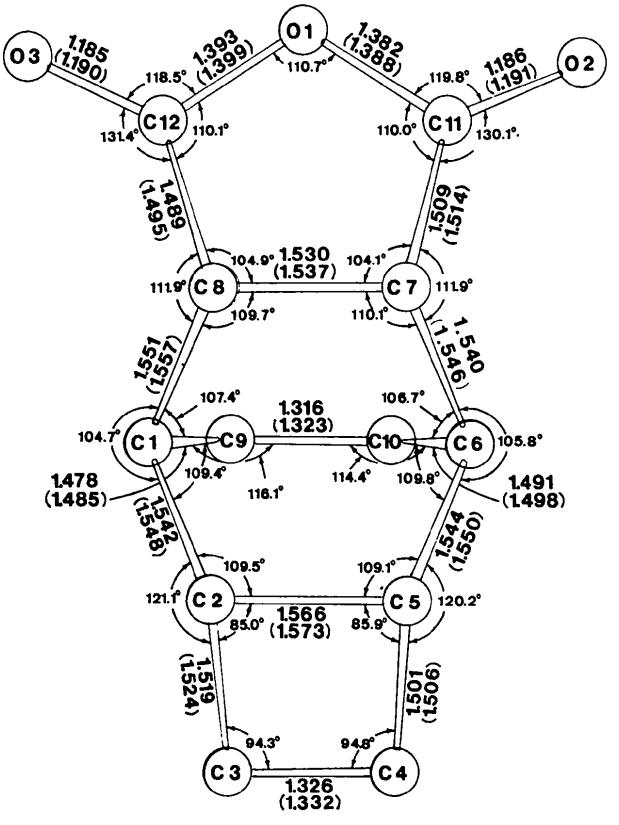


Fig. 2. Bond distances and angles involving the heavy atoms. Values in parentheses include corrections for thermal libration. Standard deviations are about 0·0075 Å in the bond distances and 0·5° in the bond angles.

that the reported precision is not overestimated, and strongly supports the view that the conformation found here is close to that of the isolated molecule. A similar observation was made for the *endo* and *exo* isomers of bicyclo[2,2,1]hept-5-ene-2,3-dicarboxylic anhydride (Destro *et al.*, 1969; Filippini *et al.*, 1972) and for bicyclo[2,2,2]octene-2,3-*endo*-dicarboxylic anhydride (BIC) (Destro *et al.*, 1971).

The geometries of TRIC and BIC are very similar, the only significant difference being the distance C(2)–C(5), which is part of a cyclobutene ring in TRIC and therefore some 0·03 Å longer. This allows the intra-annular angles at C(3) and C(4) to exceed 90°, slightly reducing the distortion from 120°. Examples of this kind can be found in strained molecules, such as tricyclo[5,2,1,$0^{2,6}$]deca-4,8-dienyl *p*-bromobenzoate (Bellobono, Destro, Gramaccioli & Simonetta, 1969), three dihalobenzocyclobutenes (Hardgrove, Templeton & Templeton, 1968), *cis*-benzocyclobutene-1,2-diol dinitrate (Allen & Trotter, 1970*a*) and benzocyclobutene-1,2-dione (Allen & Trotter, 1970*b*). In our case the lengthening is due only to relief of angular strain, suggesting that the presence of substituents at C(2) and C(5) affects the C(2)–C(5) bond distance in a minor way.

The dihedral angles and the distances of the relevant atoms from the planes are summarized in Table 7; the torsion angles are given in Table 8.

The four-membered ring (*A*) is planar: the same is true for the planes defined by the atoms C(1), C(2), C(5) and C(6) (*B*), C(1), C(6), C(10) and C(9) (*C*), C(1), C(6), C(7) and C(8) (*D*).

The geometry of the anhydride group is close to those of the bicyclo derivatives already mentioned (Destro *et al.*, 1969, 1971; Filippini *et al.*, 1972) and to succinic anhydride (Ehrenberg, 1965; Biagini & Cannas, 1965).

Plane *C* is slightly bent towards plane *D* and the anhydride group.

The atoms C(7), C(8), C(11), C(12) and O(1) define a plane (*E*) at the limits of significance ($\Delta \simeq 3\sigma$) at the expense of the other two oxygen atoms O(2) and O(3), as can be seen from plane (*E'*). An alternative analysis of the anhydride group shows that it can be considered as being formed of two parts defined by the atoms C(8), C(12), O(1) and O(3) (*F*) and C(7), C(11), O(1) and O(2) (*F'*), which are themselves planar and include an angle of 2·3°.

Molecular packing

If we assume as van der Waals radii for carbon, oxygen and hydrogen the values of 1·8, 1·4 and 1·2 Å respectively (Kitaigorodskii, 1961), in no case do two atoms belonging to different molecules lie at a smaller distance than the sum of the corresponding van der Waals radii. The molecular packing seen along **c** is represented in Fig. 3.

References

ALLEN, F. H. & TROTTER, J. (1970*a*), *J. Chem. Soc.* (*B*), pp. 916–920.

ALLEN, F. H. & TROTTER, J. (1970*b*). *J. Chem. Soc.* (*B*), pp. 1551–1555.

Table 7. *Some planes of interest*

The coefficients q_i are the direction cosines relative to the crystallographic axes *a*, *b* and *c*. In the least-squares treatment, the method described by Schomaker, Waser, Marsh & Bergman (1959) was adopted, with weights inversely proportional to the positional standard deviation along the normal to the plane. Atoms marked with an asterisk were omitted from least-squares calculations.

Plane *A*	Δ	Plane *B*	Δ	Plane *C*	Δ	Plane *D*	Δ
C(2)	0·001	C(1)	0·002	C(1)	−0·001	C(1)	−0·009
C(3)	−0·001	C(2)	−0·005	C(6)	0·001	C(6)	0·007
C(4)	0·001	C(5)	0·005	C(9)	0·003	C(7)	−0·015
C(5)	−0·001	C(6)	−0·002	C(10)	−0·003	C(8)	0·017

Plane *E*	Δ	Plane *E'*	Δ	Plane *F*	Δ	Plane *F'*	Δ
C(7)	0·019	C(7)	0·032	C(8)	0·000	C(7)	−0·001
C(8)	−0·016	C(8)	−0·031	C(12)	−0·001	C(11)	0·004
C(11)	−0·018	C(11)	0·010	O(1)	0·000	O(1)	−0·001
C(12)	0·007	C(12)	−0·007	O(3)	0·000	O(2)	−0·001
O(1)	0·004	O(1)	0·016				
O(2)*	−0·071	O(2)	−0·021				
O(3)*	0·028	O(3)	−0·002				

		q_1	q_2	q_3	D	Dihedral angles	
Plane	*A*	0·3359	0·1254	−0·9434	1·8292 Å	*A* ∧ *B*	120·9°
Plane	*B*	0·9749	0·1271	−0·2127	−4·3258	*B* ∧ *C*	123·8
Plane	*C*	0·7033	0·0087	0·6889	−3·9695	*C* ∧ *D*	121·1
Plane	*D*	0·2374	0·1307	−0·9694	−0·2065	*B* ∧ *D*	115·1
Plane	*E*	0·9467	0·1763	−0·2980	−5·7211	*D* ∧ *E*	120·4
Plane	*E'*	0·9457	0·1934	−0·2901	−5·8768	*F* ∧ *F'*	177·7
Plane	*F*	0·9411	0·1895	−0·3088	−5·7522		
Plane	*F'*	0·9464	0·2097	−0·2747	−6·0956		

Table 8. *Torsion angles*

The convention proposed by Klyne & Prelog (1960) is adopted.

C(1)—C(2)—C(3)—C(4)	109·9°	C(3)—C(4)—C(5)—C(6)	−109·5°
C(2)—C(3)—C(4)—C(5)	−0·2		
C(3)—C(4)—C(5)—C(2)	0·2	C(4)—C(5)—C(2)—C(3)	−0·1
C(4)—C(5)—C(6)—C(7)	158·9	C(8)—C(1)—C(2)—C(3)	−157·6
C(5)—C(6)—C(7)—C(11)	−175·9	C(12)-C(8)—C(1)—C(2)	179·7
C(6)—C(7)—C(11)-O(1)	122·0	O(1)—C(12)-C(8)—C(1)	−116·9
C(7)—C(11)-O(1)—C(12)	−2·0	C(11)-O(1)—C(12)-C(8)	0·0
C(1)—C(2)—C(5)—C(6)	−0·8	C(6)—C(10)-C(9)—C(1)	−0·6
C(2)—C(5)—C(6)—C(10)	−52·4	C(9)—C(1)—C(2)—C(5)	53·2
C(5)—C(6)—C(10)-C(9)	56·7	C(10)-C(9)—C(1)—C(2)	−55·9
C(2)—C(5)—C(6)—C(7)	62·4	C(8)—C(1)—C(2)—C(5)	−61·6°
C(5)—C(6)—C(7)—C(8)	−60·6	C(7)—C(8)—C(1)—C(2)	64·2
C(9)—C(10)-C(6)—C(7)	−57·5	C(8)—C(1)—C(9)—C(10)	57·1
C(10)-C(6)—C(7)—C(8)	56·2	C(7)—C(8)—C(1)—C(9)	−51·9
C(7)—C(8)—C(12)-O(1)	2·0	O(1)—C(11)-C(7)—C(8)	3·2
C(11)-C(7)—C(8)—C(12)	−3·0		

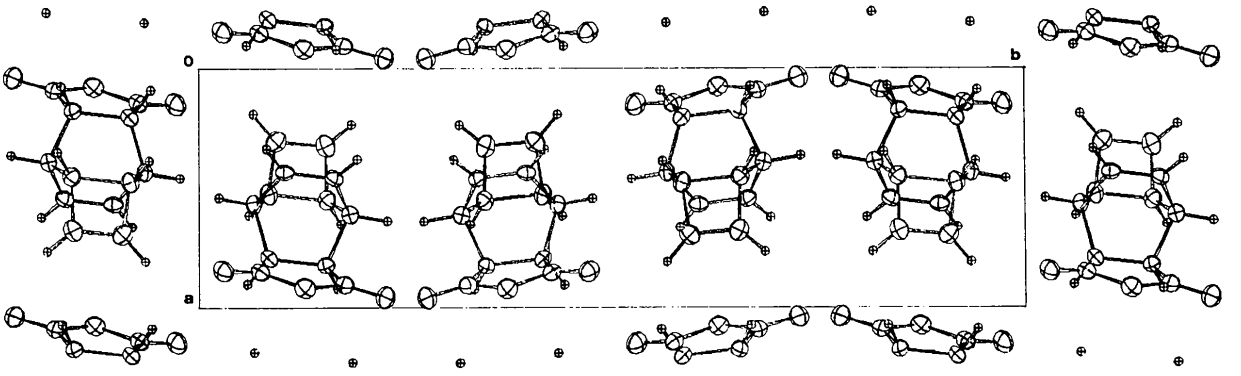


Fig. 3. Molecular packing seen along **c**.

BELLOBONO, I. R., DESTRO, R., GRAMACCIOLI, C. M. & SIMONETTA, M. (1969). *J. Chem. Soc.* (*B*), pp. 710–716.

BIAGINI, S. & CANNAS, M. (1965). *Ric. Sci.* **35** (II A), 1518–1525.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

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

DESTRO, R., FILIPPINI, G., GRAMACCIOLI, C. M., SIMONETTA, M. (1969). *Acta Cryst.* B**25**, 2465–2472.

DESTRO, R., FILIPPINI, G., GRAMACCIOLI, C. M. & SIMONETTA, M. (1971). *Acta Cryst.* B**27**, 2023–2028.

EHRENBERG, M. (1965). *Acta Cryst.* **19**, 698–703.

FILIPPINI, G., GRAMACCIOLI, C. M., ROVERE, C. & SIMONETTA, M. (1972). *Acta Cryst.* B**28**, 2869–2874.

FILIPPINI, G., GRAMACCIOLI, C. M., SIMONETTA, M. & SUFFRITTI, G. B. (1973). *J. Chem. Phys.* In the press.

FILIPPINI, G. & SIMONETTA, M. (1970). *Atti Accad. Nazl. Lincei, Rend. Classe Sci. Fis. Mat. Nat.* **49**, 389–391.

GRAMACCIOLI, C. M. & MARIANI, C. (1967). *Rend. Ist. Lombardo Sci. Lett.* A**101**, 704–710.

GRAMACCIOLI, C. M., SIMONETTA, M. & SUFFRITTI, G. B. (1973). *Chem. Phys. Lett.* **20**, 23–28.

HARDGROVE, G. L., TEMPLETON, L. K. & TEMPLETON, D. H. (1968). *J. Phys. Chem.* **72**, 668–676.

HAUPTMAN, H. & KARLE, J. (1953). *Solution of the Phase Problem. I. The Centrosymmetric Crystal.* ACA Monograph No. 3. Pittsburgh: Polycrystal Book Service.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL 3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

KARLE, I. L., HAUPTMAN, H., KARLE, J. & WING, A. B. (1958). *Acta Cryst.* **11**, 257–263.

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

KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.

LONG, R. E. (1965). Doctoral Thesis, Univ. of California, Los Angeles.

RAE, A. D. (1965). *Acta Cryst.* **19**, 683–684.

REPPE, W., SCHICHTING, O., KLAGER, K. & TOEPPEL, T. (1948). *Liebigs Ann.* **560**, 1–92.

SAYRE, D. (1952). *Acta Cryst.* **5**, 60–65.

SCHOEMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

SCHOEMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600–604.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.