

is in the chair conformation. The conformation of the seven-membered ring is restricted to a twist-boat by the double bond at C(7)–C(8). The nearly flat (r.m.s. displacement 0.112 Å) five-membered ring is also attached to the seven-membered ring; O(13) in the seven-membered ring is in the bridging position between the two outer rings.

The bonding pattern within the six-membered ring is normal with an average C–C single-bond length of 1.542 (35) Å. The longest of these, C(4)–C(5), 1.599 (9) Å, can be ascribed to the high degree of substitution on these two C atoms. The C(2)–Br bond length, 1.968 (6) Å, is slightly longer than expected for a C–Br single bond, 1.938 Å (*Tables of Interatomic Distances and Configuration in Molecules and Ions*, 1965). The bond angles within the ring, except C(3)C(4)C(5), are slightly larger than the normal tetrahedral angle, as expected for a six-membered ring. The bonding within the seven-membered ring is also normal; C(7)C(8) is a normal double bond and the other bonds are all C–C or C–O single bonds. The bond angles are all larger than the ideal tetrahedral or trigonal angles, which is typical for this size ring. In the five-membered ring the effect of ring strain is evident in the bond distances and angles. The bond angle, C(8)C(9)O(10), at the carbonyl is much less, 109.1 (15)°, than expected for an sp^2 C and has probably resulted in the short carbonyl bond, 1.188 (9) Å, and long ester C–O bond, 1.348 (10) Å, found at this point. The other bonds within the ring are more nearly normal and the angles are all smaller than the ideal angles, as is typical for a five-membered ring.

All intermolecular contacts are at or greater than the sum of the van der Waals radii. Two contacts, C(1)···C(6), 3.757 Å and C(11)···C(15), 3.630 Å, between adjacent molecules seem to be less than the sum of the methyl-group radii, but inspection of the H···H contacts between these groups shows no abnormally short contacts. Evidently, the protons on these groups must intermesh. The only other contact of interest is between the Br atom and the C(9)–O(9) carbonyl on an adjacent molecule. The contact distances, 3.511 Å for Br···O(9) and 3.577 Å for Br···C(9), are at the sum of the van der Waals radii for these atoms.

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References

- AZAROFF, L. V. (1955). *Acta Cryst.* **8**, 701–704.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–101, 149–150. Birmingham: Kynoch Press.
 MEULENAER, J. DE & TOMPA, H. (1965). *Acta Cryst.* **19**, 1014–1018.
 PETTIT, G. R., HERALD, C. L., ALLEN, M. S., VON DREELE, R. B., VANELL, L. D., KAO, J. P. Y. & BLAKE, W. (1977). *J. Am. Chem. Soc.* **99**, 262–263.
 ROLLETT, J. S. & CARRUTHERS, R. S. (1974). Private communication.
Tables of Interatomic Distances and Configuration in Molecules and Ions (1965). Edited by L. E. SUTTON. Supplement, p. S14s. London: The Chemical Society.

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The Structure of the Cyclic Trimer of Poly(ethylene terephthalate)*

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Abstract

$C_{30}H_{24}O_{12}$, $M_r = 576.5$, is monoclinic, space group Cc , with $a = 17.101$ (4), $b = 19.695$ (5), $c = 8.360$ (2) Å, $\beta = 96.47$ (2)°, $V = 2798$ (1) Å³, $D_m = 1.375$ (2), $D_c = 1.365$ Mg m⁻³, $Z = 4$, $\mu(Cu K\alpha) = 0.77$ mm⁻¹,

* 3.8.11.16.19.24-Hexaoxotribenzol fgh.pqr.za₁b₁|-11.4.11.14.21.24|hexaoxacyclotriacontane.

$F(000) = 1200$. The structure was solved by direct methods and refined by block-diagonal least-squares calculations to the final $R = 0.034$. The absolute configuration was not determined. The double bonds of the ester groups cross one another and form parallel rows. Two carbonyl groups (all are in the *cis* position) show large deviations from the benzene plane, thus explaining irregularities found in the IR spectra.

Introduction

Poly(ethylene terephthalate) (PET) contains, under room conditions, 1.3–1.7% of the cyclic trimer of PET (Goodman & Nesbitt, 1960). This substance was extracted with methanol and crystallized by slow evaporation from ethyl acetate solution. The crystals obtained, described as form *A*, are stable up to ~423 K (Binns, Frost, Smith & Yeadon, 1966), when changes appear in IR spectra in the range 1050 to 1450 cm⁻¹. When the temperature is raised above 473 K the crystals irreversibly change to form *B*. The transition to form *B* is accompanied by a large thermal absorption (Binns *et al.*, 1966). The transformation of form *B* to form *A* occurs only by dissolution and subsequent crystallization.

Table 1. Final positional parameters, their e.s.d.'s ($\times 10^4$) and B_{eq} values (\AA^2) of the non-H atoms

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
C(11)	3864 (2)	7463 (2)	5882 (4)	4.4
C(12)	4251 (2)	8079 (2)	6270 (4)	3.9
C(13)	4659 (2)	8154 (2)	7857 (4)	4.1
C(14)	4618 (3)	7624 (2)	8889 (5)	5.3
C(15)	4228 (3)	7034 (2)	8520 (5)	5.1
C(16)	3839 (2)	6965 (2)	6995 (5)	4.4
C(17)	3434 (2)	6296 (2)	6542 (5)	4.8
O(171)	3088 (2)	6280 (1)	5086 (3)	5.7
O(172)	3439 (2)	5819 (2)	7469 (4)	7.3
C(18)	5069 (2)	8790 (2)	8260 (4)	4.1
O(181)	5071 (2)	9233 (1)	7139 (3)	4.5
O(182)	5381 (2)	8888 (1)	9660 (3)	5.6
C(19)	2667 (2)	5678 (2)	4561 (5)	5.3
C(10)	5380 (2)	9887 (2)	7553 (4)	3.9
C(21)	2878 (2)	327 (2)	4341 (4)	4.4
C(22)	2328 (2)	355 (2)	2991 (4)	3.9
C(23)	2046 (2)	948 (2)	2280 (4)	3.9
C(24)	2343 (2)	1564 (2)	2946 (5)	4.8
C(25)	2867 (2)	1561 (2)	4330 (5)	4.7
C(26)	3127 (2)	957 (2)	5021 (4)	4.2
C(27)	3746 (2)	971 (2)	6512 (4)	4.8
O(271)	4133 (2)	388 (1)	6717 (3)	5.0
O(272)	3807 (2)	1418 (2)	7425 (4)	8.5
C(28)	1486 (2)	979 (2)	845 (5)	4.6
O(281)	1068 (1)	405 (1)	620 (3)	4.4
O(282)	1323 (2)	1490 (2)	8 (4)	7.2
C(29)	4742 (2)	350 (2)	8034 (5)	4.6
C(20)	437 (2)	366 (2)	-756 (4)	4.9
C(31)	960 (2)	8048 (2)	1011 (4)	4.6
C(32)	1365 (2)	7470 (2)	1443 (4)	4.4
C(33)	1346 (2)	6929 (2)	323 (4)	4.2
C(34)	951 (2)	7025 (2)	-1218 (5)	5.0
C(35)	546 (3)	7621 (2)	-1648 (4)	4.9
C(36)	566 (2)	8140 (2)	-463 (4)	3.8
C(37)	112 (2)	8770 (2)	-1098 (4)	4.2
O(371)	146 (2)	9240 (1)	169 (3)	5.1
O(372)	-209 (2)	8876 (2)	-2381 (3)	6.7
C(38)	1750 (2)	6277 (2)	685 (5)	4.7
O(381)	2068 (2)	6259 (1)	2281 (3)	5.5
O(382)	1781 (2)	5817 (1)	-222 (4)	6.5
C(39)	-172 (3)	9918 (2)	-239 (5)	5.7
C(30)	2552 (3)	5665 (2)	2784 (6)	6.2

Table 2. Final positional parameters, their e.s.d.'s ($\times 10^3$) and B_{iso} values (\AA^2) of the H atoms

	<i>x</i>	<i>y</i>	<i>z</i>	B_{iso}
H(11)	368 (2)	737 (1)	488 (3)	3.3 (7)
H(12)	425 (2)	844 (2)	551 (3)	3.4 (7)
H(14)	485 (2)	770 (2)	982 (4)	4.3 (8)
H(15)	427 (2)	675 (2)	931 (3)	3.5 (7)
H(191)	215 (2)	569 (2)	508 (4)	4.7 (8)
H(192)	291 (2)	531 (1)	492 (4)	3.2 (6)
H(101)	581 (2)	988 (2)	856 (4)	5.0 (8)
H(102)	555 (2)	10 (2)	665 (5)	6.7 (10)
H(21)	312 (2)	933 (2)	470 (4)	3.8 (7)
H(22)	222 (2)	991 (2)	247 (4)	4.9 (8)
H(24)	216 (2)	196 (2)	246 (4)	3.7 (7)
H(25)	309 (3)	205 (2)	476 (5)	8.2 (12)
H(291)	451 (2)	24 (2)	914 (5)	6.7 (10)
H(292)	492 (2)	78 (2)	839 (4)	3.8 (7)
H(201)	16 (2)	84 (2)	-81 (4)	4.3 (8)
H(202)	73 (2)	15 (2)	-162 (4)	4.6 (8)
H(31)	90 (2)	835 (2)	170 (4)	4.2 (7)
H(32)	171 (2)	747 (2)	249 (4)	4.8 (8)
H(34)	103 (2)	665 (2)	-189 (5)	6.7 (10)
H(35)	29 (3)	768 (2)	-274 (5)	8.0 (12)
H(391)	-40 (2)	5 (1)	71 (3)	2.7 (6)
H(392)	-54 (2)	985 (2)	-90 (4)	3.9 (7)
H(301)	221 (3)	522 (3)	244 (6)	11.4 (16)
H(302)	309 (2)	577 (2)	231 (4)	5.3 (9)

During the preparation of form *B* the single crystals changed to a white polycrystalline material. Therefore the subsequent investigation of the crystal and molecular structure concerns only crystal form *A* of the cyclic trimer of PET. A preliminary structure investigation was published by Farrow, McIntosh & Ward (1960), in which the *gauche* conformation of the $-\text{O}-\text{CH}_2-\text{CH}_2-\text{O}-$ groups and the *cis* configuration of the *p*-phenylene groups were predicted from X-ray and optical work.

Structure determination and refinement

The phase problem was solved by the program *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974). The structure was refined by the least-squares method in the block-diagonal approximation (Ahmed, Hall, Pippy & Huber, 1966). The function minimized was $\sum w\Delta^2$ where $w = [\sigma_F^2 + (0.03F_o)^2]^{-1}$ and $\Delta = |F_o| - |F_c|$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974); the H atoms were considered as a spherical approximation to the bonded atoms. Unobserved reflexions were eliminated from the refinement.

The non-hydrogen atoms were refined with anisotropic and H atoms with isotropic temperature factors until all parameter shifts were less than 0.3 of the corresponding e.s.d.'s. The final agreement factors are: $R_1 = \sum \Delta / \sum |F_o| = 0.034$, $R_2 = [\sum \Delta^2 / \sum |F_o|^2]^{1/2} = 0.057$, $Rw_2 = [\sum w\Delta^2 / \sum w|F_o|^2]^{1/2} = 0.040$, $S =$

$[\sum w\Delta^2/(m-n)]^{1/2} = 1.18$, where m is the number of reflexions used in the refinement (1485) and n the number of refined parameters (475).

The resulting positional parameters and B_{eq} values (Hamilton, 1959) of all the non-H atoms are summarized in Table 1 and positional parameters and B_{iso} values of the H atoms in Table 2. The six highest residual peaks in the final difference map calculated without unobserved reflexions are in the range 0.28–0.35 e Å⁻³, mostly near the carbonyl groups.*

Discussion

The numbering scheme of the atoms and the division of the molecule into six segments are shown in Fig. 1. Fig. 2 shows the numbering of one segment.

A comparison of the equivalent distances and angles in the separate segments is given in Tables 3 and 4. E.s.d.'s of the mean values show that the least-squares e.s.d.'s are underestimated. A complete summary of the bond distances, the interbond angles and their e.s.d.'s has been deposited.*

The average C(sp³)–C(sp³) distance, 1.48 (2) Å, is significantly shorter than that for a C–C single bond. This effect has been systematically observed in other structures with the –O–C(sp³)–C(sp³)–O– group (Hašek, Hlavatá & Huml, 1977; Hašek & Huml, 1978)

* Lists of structure factors, anisotropic thermal parameters and additional molecular-geometry data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35446 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

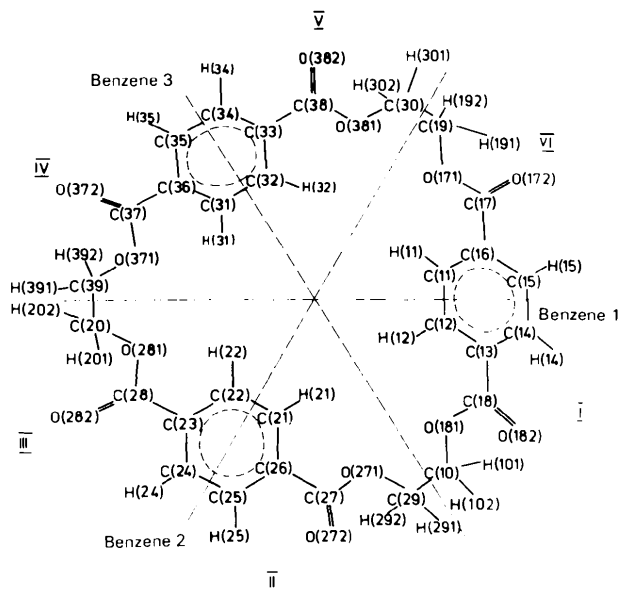


Fig. 1. The numbering scheme and division of the molecule of the cyclic trimer of PET into six segments.

and it has been shown that it is not an effect of the thermal motion or errors in experimental data (Goldberg, 1975, 1976; Dunitz, Dobler, Seiler & Phizackerley, 1974).

The sums of the interbond angles around atom 4 are different from 360° in segments II and III. The torsions of both ester groups of benzene 2 are probably connected with the different role of this benzene in intermolecular interactions (Fig. 3) and may be correlated with the high χ^2 values (Table 6).

Important differences are also seen in torsion angles 5–4–3–1 and 6'–6–5–4 (Table 5). These differences are not predicted in the paper by Farrow, McIntosh & Ward (1960).

We have studied the vibrational spectra of some aromatic esters in the liquid and solid states (Sedláček, Štokr & Schneider, 1980). Rotamers of these compounds were determined from the disappearance of some absorption bands in the crystalline state as compared to solution. It was shown that the differences in torsion angles of these compounds led to changes in the IR spectra in the regions of about 500 cm⁻¹ and 1460 cm⁻¹.

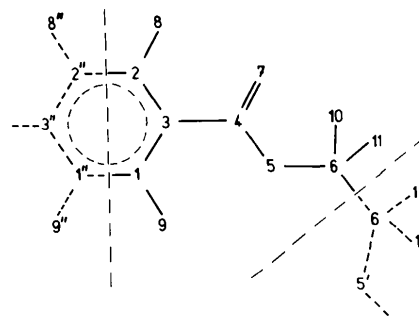


Fig. 2. The numbering of a segment. H atoms numbered as 10 and 11 correspond to those whose last digit is 1 or 2 in Fig. 1.

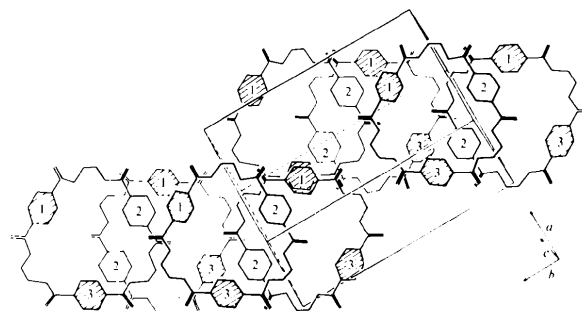


Fig. 3. Projection of the cyclic trimer of PET perpendicular to the plane defined by the centres of the benzene rings. Molecules in the bottom row from right to left are in equivalent positions x, y, z ; $x, 2-y, z + \frac{1}{2}$; $x + \frac{1}{2}, y + \frac{1}{2}, z$; $x + \frac{1}{2}, \frac{3}{2}-y, z + \frac{1}{2}$ and corresponding distances of benzene centres from the projection plane are –3.77, –6.71, 2.95 and 0.00 Å. Molecules in the upper row are translated by 0, –1, 0. Because b is parallel to the projection plane, the distances of the atoms from this plane are not changed.

Table 3. Comparison of bond lengths (Å)

Roman numerals mark the respective segments. Least-squares e.s.d.'s of distances between non-H atoms are 0.004–0.006 Å and those involving H atoms 0.03–0.05 Å.

Bond	C(sp ²) C(sp ²)	C(sp ²) O	C(sp ²)– O	O– C(sp ³)	C(sp ³)– C(sp ³)	Aromatic bonds				C(sp ²) H	C(sp ³)–H		
	3–4	4–7	4–5	5–6	6–6'	2–2''	1–1''	2 3	1 3	2–8	1–9	6 10	6–11
I	1.456	1.244	1.280	1.421	1.513	1.358	1.403	1.362	1.436	0.87	0.96	1.03	0.92
II	1.543	1.161	1.327	1.430	–	–	–	1.374	1.412	1.06	0.93	1.09	0.94
III	1.449	1.241	1.339	1.486	1.464	1.381	1.388	1.405	1.373	0.94	0.98	1.04	1.01
IV	1.526	1.168	1.401	1.466	–	–	–	1.421	1.350	0.97	0.85	0.96	0.82
V	1.474	1.186	1.383	1.467	1.476	1.388	1.360	1.397	1.416	0.94	0.98	1.07	1.07
VI	1.516	1.217	1.293	1.431	–	–	–	1.378	1.355	0.86	0.90	1.02	0.88
Mean value	1.49 (2)	1.20 (2)	1.34 (2)	1.45 (1)	1.48 (2)	1.38 (1)	1.38 (1)	1.39 (1)	1.39 (1)	0.94 (3)	0.93 (2)	1.04 (2)	0.94 (4)

Table 4. Comparison of the interbond angles (°)

Roman numerals mark the respective segments. E.s.d.'s of interbond angles are 0.3–0.4°, or 2–3° if the bonds involve H atoms.

Angle	C(sp ²)		C(sp ²)		O(sp ²)	C(sp ³)	C(sp ²)			
	1–3–4	2–3–4	1–3–2	3–4–5	5–4–7	7–4–3	4–5–6	5–6–6'	2''–2–3	1''–1–3
I	118.3	124.7	117.0	117.1	119.8	123.1	117.0	108.1	124.7	118.1
II	119.2	119.0	121.7	112.0	124.3	123.3	118.8	106.3	120.4	115.7
III	124.1	117.9	117.9	112.0	121.5	125.9	116.3	111.2	119.8	124.3
IV	127.5	112.0	120.6	107.5	123.5	129.0	116.5	106.5	117.2	122.8
V	123.0	118.3	118.6	110.2	123.9	125.9	118.0	109.1	121.9	118.9
VI	120.4	118.4	121.0	113.8	122.9	123.3	118.1	110.5	118.0	121.1
Mean value	122 (2)	118.4 (2)	120 (1)	112 (1)	123 (1)	125 (1)	117 (1)	109 (1)	120 (1)	120 (1)

Angle	C(sp ²)–C(sp ²)–H			O–C(sp ³)–H		C(sp ³)–C(sp ³)–H		H–C(sp ³)–H	
	2''–2–8	3–2–8	1''–1–9	3–1–9	5–6–10	5–6–11	6'–6–10	6'–6–11	10–6–11
I	122	113	121	120	113	108	106	104	113
II	116	123	123	121	111	113	116	114	95
III	122	118	113	122	106	102	105	113	125
IV	121	116	120	123	104	104	117	114	106
V	126	112	117	124	109	103	108	110	120
VI	114	128	121	117	105	112	115	110	106
Mean value	120 (2)	118 (3)	119 (1)	121 (1)	108 (1)	107 (2)	111 (2)	111 (2)	111 (4)

Table 5. Comparison of some torsion angles (°)

Roman numerals mark the respective segments. E.s.d.'s are smaller than 0.5°.

Torsion angle	5 4–3 1	6–5 4–7	6'–6 5–4	5' 6'–6–5	7–4–3–1
	I	3.3	6.0	89.2	–
II	17.6	9.1	147.2	58.2	155.4
III	–22.3	–5.2	148.4	–	166.4
IV	2.7	6.8	88.9	59.9	183.2
V	5.7	–8.0	167.2	–	176.8
VI	4.5	–2.2	161.1	62.5	184.7

Every benzene 1 overlaps with benzene 3 of a symmetrically equivalent molecule (Figs. 3 and 4). The planes of benzenes 1 and 3 are approximately parallel (dihedral angle 1.4°) and the distance between their centres is 3.786 Å. The overlapping benzene rings form zones alternating with zones which contain only

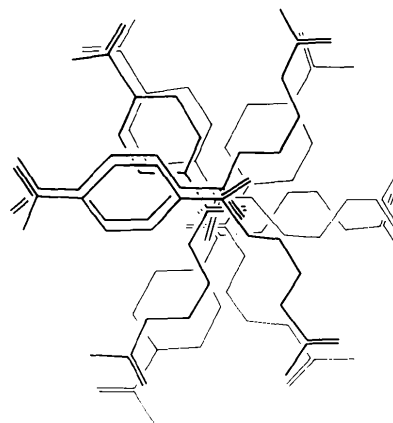


Fig. 4. Ester double-bond crossing and overlap of benzenes 1 and 3.

Table 6. *Angles between some weighted mean planes*
(°)

Deviations of atoms from the planes are $\times 10^3$ (Å). The asterisks denote atoms excluded from the plane definition. E.s.d.'s of deviations are smaller than 0.004 Å.

Plane	Atoms defining the plane	χ^2
<i>a</i>	C(11), C(12), C(13), C(14), C(15), C(16) 17 -12 3 3 1 -11	40
<i>b</i>	C(21), C(22), C(23), C(24), C(25), C(26) 22 4 -14 24 -6 -17	114
<i>c</i>	C(31), C(32), C(33), C(34), C(35), C(36) -5 15 -17 11 1 -3	47
<i>d</i>	C(16), C(17), O(171), O(172), C(11)*, C(15)*, C(19)* 0 -1 0 0 95 3 -44	0.1
<i>e</i>	C(13), C(18), O(181), O(182), C(12)*, C(14)*, C(10)* -3 10 2 -2 -100 43 -160	9
<i>f</i>	C(26), C(27), O(271), O(272), C(21)*, C(25)*, C(29)* 9 39 6 -13 458 464 79	127
<i>g</i>	C(23), C(28), O(281), O(282), C(22)*, C(24)*, C(20)* 12 48 -6 -13 322 -385 -25	188
<i>h</i>	C(36), C(37), O(371), O(372), C(31)*, C(35)*, C(39)* 1 -3 0 1 57 -46 166	0.8
<i>i</i>	C(33), C(38), O(381), O(382), C(32)*, C(34)*, C(30)* 4 14 2 3 85 -62 138	15
<i>j</i>	Plane through benzene centres	0

Selected angles between planes: *j*-*a* 29.4, *j*-*b* 3.4, *j*-*c* 28.0, *j*-*d* 31.8, *j*-*e* 22.8, *j*-*f* 18.4, *j*-*g* 30.1, *j*-*h* 30.6, *j*-*i* 26.7.

Benzene-benzene: *a*-*b* 27.1, *b*-*c* 26.5, *a*-*c* 45.0; benzene-ester: *a*-*d* 3.1, *a*-*e* 3.3, *b*-*f* 22.9, *b*-*g* 18.2, *c*-*h* 2.6, *c*-*i* 3.6; ester-ester: *d*-*e* 5.1, *e*-*f* 49.3, *f*-*g* 41.0, *g*-*h* 45.4, *h*-*i* 1.1, *i*-*d* 42.3.

benzenes 2 which are not in a position to overlap each other (Fig. 3).

A projection of the structure along the line between the centres of benzenes 1 and 2 and all angles between the benzene weighted mean planes have been deposited.* Some of the shortest intermolecular distances are C(11)···C(36)ⁱ 3.445 (5), O(271)···H(291)ⁱⁱ 2.59 (4), C(15)···H(201)ⁱⁱⁱ 2.87 (4) Å, where (i) is $x + \frac{1}{2}, \frac{3}{2} - y, z + \frac{1}{2}$, (ii) is $x, 2 - y, z + \frac{1}{2}$, (iii) is $x + \frac{1}{2}, y - \frac{1}{2}, 1 + z$.

* See previous footnote.

Conclusion

Molecules of the cyclic trimer of PET in crystal form *A* are arranged so that the double bonds of the ester groups cross one another and form parallel rows. The stability of this arrangement is supported by parallel benzene rings of neighbouring molecules.

The conformations around the C(*sp*³)-C(*sp*³) bonds are *gauche* and both stable conformations around O-C(*sp*³) bonds are present. All the carbonyl groups are in the *cis* position but two of them have $\sim 20^\circ$ deviations from the benzene plane. These results provide new data useful to a rigorous interpretation of the IR spectra in the region of conformationally sensitive bands.

References

- AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). NRC Crystallographic Programs for the IBM/360 System, *World List of Crystallographic Computer Programs*, 2nd ed. Appendix, p. 52. Utrecht: Oosthoek.
- BINNS, G. L., FROST, I. S., SMITH, F. S. & YEADON, E. C. (1966). *Polymer*, **7**, 583-585.
- DUNITZ, J. D., DOBLER, M., SEILER, P. & PHIZACKERLEY, R. P. (1974). *Acta Cryst.* **B30**, 2733-2750.
- FARROW, G., MCINTOSH, I. & WARD, I. M. (1960). *Macromol. Chem.* **38**, 147-158.
- GOLDBERG, I. (1975). *Acta Cryst.* **B31**, 754-763.
- GOLDBERG, I. (1976). *Acta Cryst.* **B32**, 41-46.
- GOODMAN, I. & NESBITT, B. F. (1960). *Polymer*, **1**, 384-396.
- HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609-610.
- HAŠEK, J., HLAVATÁ, D. & HUML, K. (1977). *Acta Cryst.* **B33**, 3372-3376.
- HAŠEK, J. & HUML, K. (1978). *Acta Cryst.* **B34**, 1812-1814.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain-la-Neuve, Belgium.
- SEDLÁČEK, B., ŠTOKR, J. & SCHNEIDER, B. (1980). *Coll. Czech. Chem. Commun.* To be published.