Cyclic Trimer of Poly(ethylene terephthalate).* Structure Recalculation

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Abstract. $C_{30}H_{24}O_{12}$, $M_r = 576.5$, monoclinic, $a = 17.101$ (4), $b = 19.695$ (5), $c = 8.360$ (2) Å, $\beta = 96.47$ (2)°, $V = 2798$ (1) Å^3, $D_m = 1.375$ (2), $D_c = 1.369$ Mg m^{-3}, $Z = 4$, originally refined in space group Cc, recalculated in space group $C2/c$ assuming that the molecule lies on the crystallographic twofold axis. $R = 0.0395$ for 1485 observed reflections.

Introduction. The title compound was originally refined in space group Cc using block-diagonal least squares (Hašek, Ječný, Langer, Huml & Sedláček, 1980). A slight indication of the non-centrosymmetric space group by statistical tests, the failure of direct methods to solve the phase problem in $C2/c$ and a smooth structure determination in Cc with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) led the authors to the preference of the non-centrosymmetric space group. Further study of this compound indicated the presence of a centre of symmetry and the necessity to recalculate the structure in space group $C2/c$. Full-matrix least-squares refinement with *SHELX* (Sheldrick, 1976) was used on the same intensity data. The final agreement factors $R_1 = \sum ||F_o| - |F_c||/\sum |F_o| = 0.0395$ and $R_{w_2} = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.0496$, where $w = 1/[\sigma^2(F) + 0.0009F^2]$, are higher in $C2/c$, but the resulting structure parameters are in better agreement with the expected molecular geometry.†

Discussion. The final positional and thermal parameters are given in Tables 1 and 2. Bond lengths, bond angles, selected torsion angles and interplanar angles are in Tables 3–6, respectively. The numbering of the

atoms is the same as in the original paper. A molecule of $C_{30}H_{24}O_{12}$ consists of two parts, symmetrically related by the crystallographic twofold axis going

Table 1. *Fractional coordinates* ($\times 10^4$) *and* B_{eq} *values* (Å^2) (*Hamilton*, 1959) *of the non-*H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}
C(11)	6251 (1)	7467 (1)	4722 (3)	4·27 (6)
C(12)	6645 (1)	8065 (1)	5129 (3)	4·06 (6)
C(13)	7047 (1)	8145 (1)	6663 (2)	3·85 (6)
C(14)	7036 (2)	7622 (1)	7771 (3)	4·86 (7)
C(15)	6639 (2)	7032 (1)	7371 (3)	4·88 (7)
C(16)	6247 (1)	6946 (1)	5836 (3)	4·17 (6)
C(17)	5843 (1)	6288 (1)	5430 (3)	4·64 (6)
O(171)	5510 (1)	6270 (1)	3904 (2)	5·38 (5)
O(172)	5829 (1)	5817 (1)	6345 (2)	6·70 (6)
C(18)	7477 (1)	8779 (1)	7175 (3)	4·04 (6)
O(181)	7463 (1)	9235 (1)	5985 (2)	4·55 (4)
O(182)	7796 (1)	8882 (1)	8519 (2)	5·77 (5)
C(19)	5059 (2)	5673 (1)	3392 (3)	5·47 (7)
C(10)	7777 (1)	9900 (1)	6401 (3)	4·41 (6)
C(21)	5275 (1)	10342 (1)	3172 (3)	4·03 (6)
C(25)	5262 (1)	11560 (1)	3192 (3)	4·53 (6)
C(26)	5542 (1)	10951 (1)	3870 (2)	3·92 (6)
C(27)	6131 (1)	10975 (1)	5337 (3)	4·47 (6)
O(271)	6533 (1)	10395 (1)	5544 (2)	4·42 (4)
O(272)	6248 (1)	11459 (1)	6205 (2)	7·46 (6)
C(29)	7152 (1)	10358 (1)	6892 (3)	4·47 (7)

Table 2. *Fractional coordinates* ($\times 10^3$) *and* B_{iso} *values* (Å^2) *of the* H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{iso}
H(11)	599 (1)	741 (1)	368 (3)	5·1 (5)
H(12)	668 (1)	840 (1)	440 (3)	4·5 (5)
H(14)	729 (1)	771 (1)	878 (3)	6·2 (6)
H(15)	662 (1)	668 (1)	812 (3)	5·9 (6)
H(191)	454 (2)	573 (1)	391 (3)	5·5 (6)
H(192)	533 (2)	527 (1)	376 (3)	6·1 (6)
H(101)	819 (1)	986 (1)	726 (3)	5·3 (5)
H(102)	798 (1)	1006 (1)	542 (3)	4·5 (5)
H(21)	546 (1)	993 (1)	362 (3)	4·5 (5)
H(25)	545 (1)	1197 (1)	364 (3)	5·4 (6)
H(291)	691 (2)	1018 (1)	785 (3)	6·3 (6)
H(292)	738 (1)	1081 (1)	713 (3)	5·5 (5)

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* 1,4,11,14,21,24-Hexaoxatribenzo[*abc,klm,uvw*]cyclotriacontane-5,10,15,20,25,30-hexone. (Note that the systematic name given previously is incorrect.)

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36935 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å)

E.s.d.'s are in parentheses. Roman numerals mark the respective segments.

Bond	C(sp²)–C(sp²) 3–4	C(sp²)=O 4–7	C(sp²)–O 4–5	O–C(sp³) 5–6	C(sp³)–C(sp³) 6–6'	2–2''	1–1''	Aromatic bonds 2–3	1–3
I	1·487 (3)	1·209 (2)	1·338 (2)	1·443 (3)	—	—	—	1·387 (3)	1·393 (3)
II	1·498 (3)	1·200 (3)	1·334 (3)	1·458 (3)	1·491 (3)	1·382 (5)	1·381 (4)	1·388 (3)	1·389 (3)
VI	1·490 (3)	1·204 (3)	1·338 (3)	1·444 (3)	1·483 (6)	1·368 (3)	1·381 (3)	1·389 (3)	1·386 (3)

Table 4. *Bond angles* (°)

E.s.d.'s are in parentheses. Roman numerals mark the respective segments.

Angle	C(sp²) 1–3–4	2–3–4	1–3–2	3–4–5	5–4–7	7–4–3	C(sp²) 4–5–6	O(sp²) 5–6–6'	C(sp³) 2''–2–3	C(sp²) 1''–1–3
I	122·5 (2)	118·3 (2)	119·2 (2)	112·5 (2)	123·2 (2)	124·3 (2)	117·0 (2)	110·8 (2)	120·7 (2)	120·0 (2)
II	122·0 (2)	118·5 (2)	119·5 (2)	111·6 (2)	123·6 (2)	124·7 (2)	117·5 (2)	107·4 (2)	120·2 (2)	120·2 (2)
VI	121·8 (2)	118·7 (2)	119·5 (2)	112·2 (2)	123·2 (2)	124·5 (2)	117·4 (2)	107·8 (2)	120·3 (2)	120·3 (2)

Table 5. *Selected torsion angles* (°)

Roman numerals mark the respective segments. E.s.d.'s are <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	3·1	6·5	89·5	—	−175·8
II	−20·1	0·7	148·2	58·6	−161·8
VI	−0·8	−5·3	164·3	62·5	−179·1

Table 6. *Angles between weighted mean planes of the benzene and ester groups* (°)

E.s.d.'s of angles are <0·5°.

Plane	Atoms defining the plane	χ²
a	C(11), C(12), C(13), C(14), C(15), C(16)	21
b	C(21), C(22), C(23), C(24), C(25), C(26)	214
c	C(16), C(17), O(171), O(172)	29
d	C(13), C(18), O(181), O(182)	12
e	C(26), C(27), O(271), O(272)	33

Angles: benzene–benzene: a–b 26·8, a–aⁱ 45·0; benzene–ester: a–c 2·0, a–d 2·9, b–e 20·2; ester–ester: c–d 2·3, e–eⁱ 40·3. Symmetry code: (i)1 − x, y, ½ − z.

through the centre of the C(30)—C(19) bond and the centre of benzene 2. As expected, the refinement in the centrosymmetric space group resulted in a geometry which was close (within one e.s.d.) to the average geometry of the two related parts of the molecule in Cc. Although the newly refined structure parameters should be preferred, the discussion in the preceding paper remains valid.

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