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A Second Modification of Hexaethyl Cyclopropanehexacarboxylate

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Abstract. $C_{21}H_{30}O_{12}$, $M_r = 474 \cdot 48$, monoclinic, $P2_1$, $a = 8 \cdot 902$ (1), $b = 9 \cdot 496$ (1), $c = 14 \cdot 736$ (1) Å, $\beta = 92 \cdot 46$ (1)°, $V = 1244 \cdot 5$ Å^3, $Z = 2$, $D_x = 1 \cdot 27$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1 \cdot 5418$ Å, $\mu = 8$ cm^{-1}, $F(000) = 504$, $T = 293$ K. The structure was refined to $R = 0 \cdot 064$ for 3521 unique observed reflections. The terminal ethyl groups display very high thermal motion, which makes the bond-length precision unreliable. Compared with the orthorhombic modification [Schrumpf, Jones & Sheldrick (1987). *Acta Cryst.* C43, 1758–1760], the following differences are observed: (i) the density of the monoclinic form is $0 \cdot 04$ g cm^{-3} higher; (ii) the mean ring bond length is $1 \cdot 507$ Å monoclinic, $1 \cdot 516$ Å orthorhombic (bond-length e.s.d.'s $0 \cdot 005$, $0 \cdot 004$ Å respectively); (iii) the torsion angle C(11)–O(12)–C(12)–C(13) is $-104°$, the absolute value being much lower than the other chemically equivalent torsion angles in the monoclinic (137–170°) or orthorhombic (146–162°) form (torsion angle e.s.d.'s $<1°$).

Experimental. We recently reported the crystal structure of an orthorhombic (*Pccn*) form of the title compound, and the existence of a second, monoclinic form (Schrumpf, Jones & Sheldrick, 1987). At the time, we were unable to obtain good single crystals of the monoclinic form. We have now obtained good crystals (from acetic acid/water) as a by-product of a reaction intended to produce diethyl 2,2,3,3-tetramethylcyclopropane-1,1-dicarboxylate. We are currently studying this unexpected reaction further.
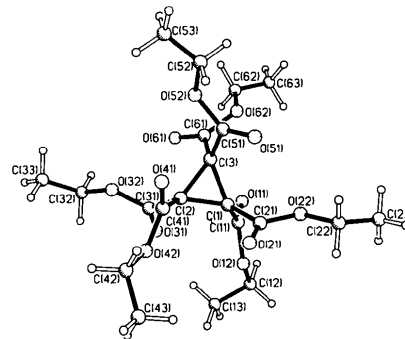
Fig. 1. The title molecule in the crystal, showing the atom-numbering scheme. Radii are arbitrary.

A colourless prism $0 \cdot 75 \times 0 \cdot 45 \times 0 \cdot 3$ mm was mounted on a glass fibre and used to record 4516 profile-fitted intensities (Clegg, 1981) on a Stoe–Siemens four-circle diffractometer (monochromated Cu $K\alpha$ radiation, $2\theta_{max}$ 140°). Merging equivalents gave 3895 unique reflections (R_{int} $0 \cdot 021$), of which 3521 with $F > 4\sigma(F)$ were used for all calculations (programs *SHELX*76 and *SHELXS*84; Sheldrick, 1976, 1984). Index ranges after merging were $|h|, |k| \geq 10, |l| \leq 17$. Cell constants were refined from 2θ values of 88 reflections in the range 60–70°. Three check reflections showed no significant intensity variation.

The structure was solved by routine direct methods

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^3$)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3047 (3)	2031 (5)	2853 (2)	69 (1)
C(2)	2188 (3)	3312 (5)	2519 (2)	71 (1)
C(3)	2613 (3)	2168 (5)	1862 (2)	72 (1)
C(11)	2262 (4)	970 (5)	3426 (3)	80 (1)
O(11)	1526 (3)	0	3136 (2)	95 (1)
O(12)	2562 (3)	1256 (4)	4299 (2)	93 (1)
C(12)	1793 (6)	381 (7)	4972 (4)	123 (2)
C(13)	600 (7)	1164 (9)	5360 (4)	143 (3)
C(21)	4711 (3)	2219 (5)	3166 (3)	75 (1)
O(21)	5298 (3)	3306 (4)	3360 (2)	90 (1)
O(22)	5336 (2)	950 (4)	3180 (2)	88 (1)
C(22)	6980 (4)	923 (6)	3327 (4)	110 (2)
C(23)	7488 (6)	−459 (7)	3478 (7)	162 (4)
C(31)	559 (4)	3450 (5)	2745 (3)	78 (1)
O(31)	−29 (3)	2833 (5)	3351 (2)	93 (1)
O(32)	−105 (3)	4382 (4)	2193 (2)	95 (1)
C(32)	−1744 (4)	4515 (7)	2254 (4)	117 (2)
C(33)	−2279 (6)	5663 (10)	1796 (5)	167 (4)
C(41)	2978 (4)	4734 (5)	2486 (3)	78 (1)
O(41)	3665 (3)	5155 (4)	1869 (2)	90 (1)
O(42)	2715 (3)	5436 (4)	3236 (2)	96 (1)
C(42)	3429 (11)	6808 (6)	3336 (5)	147 (3)
C(43)	4031 (12)	7044 (10)	4171 (8)	204 (5)
C(51)	3822 (4)	2484 (5)	1192 (3)	82 (1)
O(51)	5149 (3)	2367 (5)	1373 (2)	100 (1)
O(52)	3194 (3)	2931 (5)	423 (2)	104 (1)
C(52)	4247 (7)	3425 (13)	−233 (4)	160 (4)
C(53)	3611 (13)	4188 (14)	−896 (8)	237 (6)
C(61)	1375 (4)	1228 (5)	1459 (3)	87 (1)
O(61)	63 (3)	1479 (5)	1467 (2)	104 (1)
O(62)	1988 (3)	100 (5)	1086 (2)	107 (1)
C(62)	919 (8)	−917 (8)	707 (6)	150 (3)
C(63)	1436 (14)	−2164 (10)	689 (10)	265 (8)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·508 (5)	C(1)—C(3)	1·500 (5)
C(1)—C(11)	1·506 (5)	C(1)—C(21)	1·543 (4)
C(2)—C(3)	1·514 (5)	C(2)—C(31)	1·508 (4)
C(2)—C(41)	1·524 (5)	C(3)—C(51)	1·521 (4)
C(3)—C(61)	1·519 (5)	C(11)—O(11)	1·199 (5)
C(11)—O(12)	1·330 (5)	O(12)—C(12)	1·485 (5)
C(12)—C(13)	1·434 (8)	C(21)—O(21)	1·186 (4)
C(21)—O(22)	1·326 (5)	O(22)—C(22)	1·470 (4)
C(22)—C(23)	1·402 (8)	C(31)—O(31)	1·207 (4)
C(31)—O(32)	1·324 (4)	O(32)—C(32)	1·471 (4)
C(32)—C(33)	1·358 (8)	C(41)—O(41)	1·186 (4)
C(41)—O(42)	1·320 (5)	O(42)—C(42)	1·454 (7)
C(42)—C(43)	1·341 (10)	C(51)—O(51)	1·205 (4)
C(51)—O(52)	1·312 (5)	O(52)—C(52)	1·454 (6)
C(52)—C(53)	1·323 (13)	C(61)—O(61)	1·192 (5)
C(61)—O(62)	1·331 (5)	O(62)·C(62)	1·451 (7)
C(62)—C(63)	1·271 (12)		

C(3)—C(1)—C(2)	60·4 (2)	C(11)—C(1)—C(2)	118·8 (2)
C(11)—C(1)—C(3)	119·9 (3)	C(21)—C(1)—C(2)	118·1 (3)
C(21)—C(1)—C(3)	119·1 (3)	C(21)—C(1)—C(11)	111·8 (3)
C(3)—C(2)—C(1)	59·5 (2)	C(31)—C(2)—C(1)	118·5 (3)
C(31)—C(2)—C(3)	118·2 (3)	C(41)—C(2)—C(1)	119·8 (3)
C(41)—C(2)—C(3)	119·1 (3)	C(41)—C(2)—C(31)	112·3 (3)
C(2)—C(3)—C(1)	60·0 (2)	C(51)—C(3)—C(1)	119·2 (3)
C(51)—C(3)—C(2)	118·5 (3)	C(61)—C(3)—C(1)	118·8 (3)
C(61)—C(3)—C(2)	118·4 (3)	C(61)—C(3)—C(51)	112·6 (3)
O(11)—C(11)—C(1)	125·0 (4)	O(12)—C(11)—C(1)	109·1 (3)
O(12)—C(11)—O(11)	125·9 (4)	C(12)—O(12)—C(11)	117·0 (4)
C(13)—C(12)—O(12)	110·3 (5)	O(21)—C(21)—C(1)	125·4 (3)
O(22)—C(21)—C(1)	107·2 (3)	O(22)—C(21)—O(21)	127·4 (3)
C(22)—O(22)—C(21)	115·6 (3)	C(23)—C(22)—O(22)	110·6 (4)
O(31)—C(31)—C(2)	125·0 (3)	O(32)—C(31)—C(2)	109·3 (3)
O(32)—C(31)—O(31)	125·7 (3)	C(32)—O(32)—C(31)	115·9 (3)
C(33)—C(32)—O(32)	111·5 (4)	O(41)—C(41)—C(2)	125·2 (4)
O(42)—C(41)—C(2)	108·8 (3)	O(42)—C(41)—O(41)	126·0 (4)
C(42)—O(42)—C(41)	116·4 (4)	C(43)—C(42)—O(42)	113·5 (6)
O(51)—C(51)—C(3)	123·5 (4)	O(52)—C(51)—C(3)	109·7 (3)
O(52)—C(51)—O(51)	126·7 (3)	C(52)—O(52)—C(51)	114·6 (3)
O(52)—C(52)—C(51)	113·6 (7)	O(61)—C(61)—C(3)	125·0 (4)
O(62)—C(61)—C(3)	109·3 (3)	O(62)—C(61)—O(61)	125·7 (4)
C(62)—O(62)—C(61)	114·9 (4)	C(63)—C(62)—O(62)	113·4 (7)

and refined on F to $R = 0\cdot064$, $wR = 0\cdot087$. Non-H atoms were anisotropic, and H atoms were included using a riding model with C—H 0·96 Å, H—C—H 109·5°, fixed isotropic temperature factors. An extinction correction of the form $F_{\text{corr}} = F_c/(1 + xF_c^2/\sin 2\theta)^{0\cdot25}$ was applied; x refined to $1\cdot3$ (3) $\times 10^{-5}$. The weighting scheme was $w^{-1} = \sigma^2(F) + 0\cdot0005F^2$. 298 parameters; $S = 2\cdot8$; max. $(\Delta/\sigma) = 0\cdot2$; max. features in final $\Delta\rho$ map $+0\cdot4$, $-0\cdot3$ e Å^{-3}. Atomic scattering factors those of *SHELX*.

Final atomic coordinates are given in Table 1, with derived bond lengths and angles in Table 2.* The molecule is shown in Fig. 1, with a packing plot in Fig. 2. We had hoped to use the ring bond lengths in our studies of structural correlations in cyclopropane derivatives, but the extremely high thermal motion of the ethyl groups [especially at C(63)] makes these distances unreliable. The C—C bond lengths of these groups suffer from severe librational shortening.

Related literature. See Schrumpf, Jones & Sheldrick (1987) and references therein.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44120 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
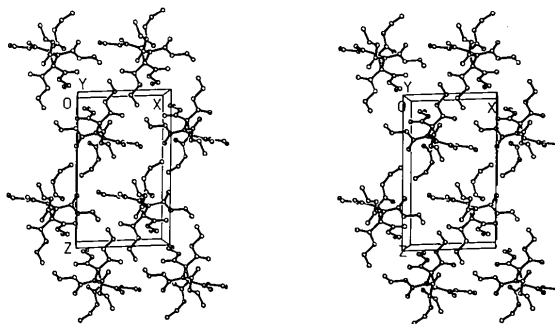

Fig. 2. Stereographic packing diagram of the title compound (H atoms omitted).

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