Table 3. *Bond distances* (Å) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·506 (8)	C(13)—C(14)	1·588 (7)
C(1)—C(10)	1·535 (8)	C(13)—C(18)	1·556 (7)
C(2)—C(3)	1·437 (8)	C(13)—C(27)	1·546 (7)
C(2)—O(C2)	1·239 (8)	C(14)—C(15)	1·540 (8)
C(3)—C(4)	1·329 (9)	C(14)—C(26)	1·544 (8)
C(3)—O(C3)	1·358 (7)	C(15)—C(16)	1·535 (9)
C(4)—C(5)	1·536 (8)	C(16)—C(17)	1·563 (8)
C(4)—C(23)	1·502 (8)	C(17)—C(18)	1·594 (7)
C(5)—C(6)	1·534 (8)	C(17)—C(22)	1·539 (8)
C(5)—C(10)	1·563 (7)	C(17)—C(28)	1·536 (9)
C(5)—C(24)	1·536 (8)	C(18)—C(19)	1·584 (7)
C(6)—C(7)	1·518 (8)	C(19)—C(20)	1·553 (8)
C(7)—C(8)	1·526 (7)	C(20)—C(21)	1·536 (9)
C(8)—C(9)	1·564 (7)	C(20)—C(29)	1·572 (8)
C(8)—C(14)	1·571 (7)	C(20)—C(30)	1·525 (9)
C(9)—C(10)	1·563 (7)	C(21)—C(22)	1·523 (8)
C(9)—C(11)	1·540 (7)	C(30)—O(C30)	1·340 (9)
C(9)—C(25)	1·546 (8)	C(30)—O'(C30)	1·204 (9)
C(11)—C(12)	1·528 (8)	C(31)—O(C30)	1·433 (9)
C(12)—C(13)	1·557 (8)		

Atomic coordinates are listed in Table 2* and bond lengths in Table 3. Fig. 1 is an *ORTEP* perspective drawing of the molecule showing the atom labelling.

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* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52165 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

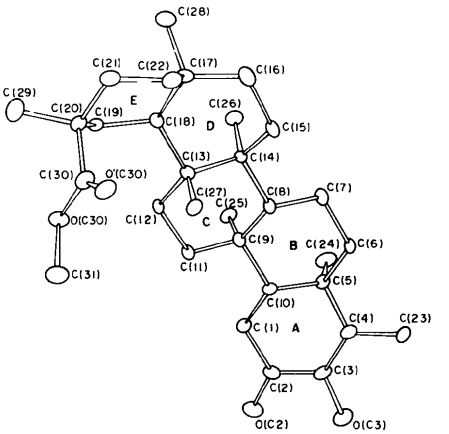

Fig. 1. Perspective view of the molecule showing the atom labelling.

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Structure of Methyl 3-Hydroxy-3′,3′-dimethylspiro-[1,2-benzo-1-cyclohexene-4,1′-cyclopropane]-2′-carboxylate*

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Abstract. $C_{16}H_{20}O_3$, $M_r = 260{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}739$ (1), $b = 22{\cdot}750$ (3), $c = 11{\cdot}631$ (1) Å, $\beta = 97{\cdot}99$ (1)°, $V = 2814{\cdot}0$ (4) Å^3, $Z = 8$, D_m (flotation in KI solution) = 1·24, $D_x = 1{\cdot}23$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}901$ cm^{-1}, $F(000) = 1120$, room temperature, $R = 0{\cdot}066$ for 1850 observed reflections. The cyclohexene moiety has a 'half-chair' conformation with the hydroxy O atom in the β conformation. The molecules are held together by bifurcated hydrogen bonds [O(1)···O(3′) $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 3{\cdot}014$ (8); O(1)···O(1′) $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 2{\cdot}858$ (8) Å].

Experimental. The title compound belongs to the class of synthetic agricultural pyrethroids. Crystals were grown from petroleum ether solution. Crystal approx. 0·55 × 0·5 × 0·22 mm, Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta <$ 23·5°, h 0 to 12, k 0 to 25, l −13 to 13, 4125 unique reflections collected but less than 50% (1850) judged

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significant ($|F_o| > 3\sigma|F_o|$) due to weak diffraction. Lattice parameters from 21 reflections ($18 < 2\theta < 38°$). Three standard reflections (004, $\bar{5}\bar{2}2$, $\bar{3}\bar{7}2$) every 2000 s, 4% variation in intensity. No correction for absorption, structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (*LALS*; Gantzel, Sparks & Trueblood, 1961) of scale factor, positional and anisotropic thermal parameters for non-H atoms (isotropic thermal parameters for H atoms held fixed at the values of the non-H atoms to which they are attached; positional parameters for H atoms located from a difference Fourier map, also held fixed), converged to $R = 0{\cdot}066$, $wR = 0{\cdot}065$, $S = 2{\cdot}2$, $w(|F_o| - |F_c|)^2$ minimized, $w = (8{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}01|F_o|^2)^{-1}$, $(\Delta/\sigma)_{max} = 0{\cdot}1$, final $\Delta\rho$ excursions $< |0{\cdot}2|$ e Å^{-3}. No corrections for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Fig. 1 gives a perspective view of the molecule (*PLUTO*; Motherwell & Clegg, 1978) with crystallographic numbering of atoms. The atomic parameters along with their e.s.d.'s and equivalent isotropic thermal parameters, for non-H atoms, are given in Table 1* and Table 2 gives the bond lengths and angles.

Related literature. There are several examples of structures having spiro six–three ring systems [Zelnik, Lavie, Levy, Wang & Paul (1977); Bradshaw, Hanson & Hitchcock (1982); Chexal, Tamm, Clardy & Hirotsu (1979); Paul, Fenical, Raffii & Clardy (1982); Ruedi, Schmid, Prewo, Bieri & Eugster (1983); Zelnik, McMillan, Paul, Lavie, Toscano & Dasilva (1977); Stalhandske, Svensson & Sarnstrand (1977); Hursthouse & Neidle (1973);

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52241 (17 pp.). Copies may be obtained through Thc Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

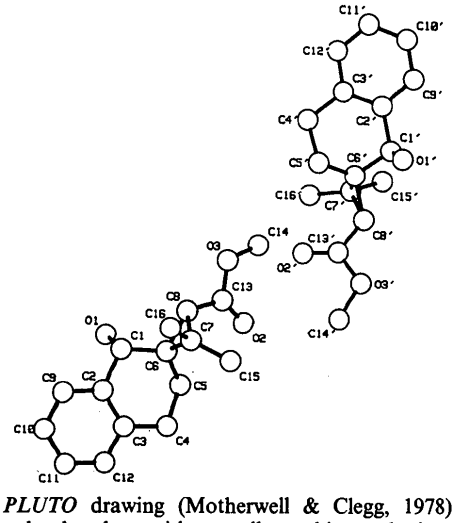

Fig. 1. *PLUTO* drawing (Motherwell & Clegg, 1978) of the molecules along with crystallographic numbering.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq} (Å^2)
O(1)	1548 (5)	374 (2)	9861 (4)	2·9 (2)
O(2)	−630 (5)	2145 (2)	9655 (5)	4·8 (2)
O(3)	−2208 (5)	1527 (2)	9080 (4)	3·6 (2)
C(1)	1271 (7)	501 (3)	11023 (5)	2·2 (2)
C(2)	2490 (7)	448 (3)	11857 (5)	2·3 (2)
C(3)	3183 (7)	941 (3)	12282 (6)	2·7 (2)
C(4)	2710 (8)	1558 (3)	12008 (7)	3·5 (3)
C(5)	1696 (8)	1586 (3)	10939 (6)	3·1 (3)
C(6)	708 (7)	1117 (3)	11048 (6)	2·4 (2)
C(7)	−344 (8)	1228 (3)	11780 (6)	3·1 (3)
C(8)	−634 (7)	1166 (3)	10445 (5)	2·5 (2)
C(9)	2942 (8)	−112 (3)	12161 (6)	2·9 (3)
C(10)	4077 (9)	−186 (4)	12852 (7)	3·8 (3)
C(11)	4776 (9)	303 (4)	13256 (7)	4·3 (3)
C(12)	4331 (8)	862 (4)	12964 (6)	3·9 (3)
C(13)	−1112 (8)	1674 (3)	9708 (6)	3·3 (3)
C(14)	−2719 (9)	1959 (3)	8221 (7)	4·7 (3)
C(15)	−456 (9)	1828 (4)	12326 (7)	4·8 (3)
C(16)	−826 (9)	738 (3)	12496 (7)	4·1 (3)
O(1′)	4958 (5)	4067 (2)	3004 (4)	3·3 (2)
O(2′)	9267 (5)	3427 (2)	3383 (5)	4·7 (2)
O(3′)	9077 (5)	4306 (2)	4198 (4)	3·9 (2)
C(1′)	5379 (8)	4027 (3)	1877 (5)	2·6 (3)
C(2′)	4364 (7)	3680 (3)	1118 (5)	2·6 (3)
C(3′)	4540 (8)	3092 (3)	852 (6)	3·0 (3)
C(4′)	5754 (9)	2768 (3)	1301 (7)	4·2 (3)
C(5′)	6554 (8)	3090 (3)	2301 (6)	3·4 (3)
C(6′)	6647 (8)	3734 (3)	1973 (5)	2·4 (3)
C(7′)	7592 (8)	3929 (3)	1194 (6)	3·0 (3)
C(8′)	7740 (6)	4120 (2)	2492 (4)	2·7 (2)
C(9′)	3232 (6)	3953 (2)	709 (4)	3·2 (2)
C(10′)	2288 (7)	3649 (3)	76 (5)	4·3 (2)
C(11′)	2453 (8)	3068 (3)	−205 (5)	4·7 (2)
C(12′)	3574 (8)	2795 (3)	173 (5)	4·1 (2)
C(13′)	8766 (6)	3895 (2)	3354 (4)	3·2 (2)
C(14′)	10150 (7)	4158 (3)	5050 (5)	4·9 (2)
C(15′)	7252 (6)	4421 (3)	322 (5)	4·0 (2)
C(16′)	8489 (7)	3487 (3)	790 (5)	4·5 (2)

Table 2. *Average bond distances* (Å) *and bond angles* (°) *for the two molecules with e.s.d.'s in parentheses*

O(1)—C(1)	1·451 (8)	C(5)—C(6)	1·522 (11)
O(2)—C(13)	1·194 (9)	C(6)—C(7)	1·522 (11)
O(3)—C(13)	1·357 (9)	C(6)—C(8)	1·520 (11)
O(3)—C(14)	1·453 (9)	C(7)—C(8)	1·553 (9)
C(1)—C(2)	1·523 (10)	C(7)—C(15)	1·520 (10)
C(1)—C(6)	1·517 (11)	C(7)—C(16)	1·518 (11)
C(2)—C(3)	1·396 (10)	C(8)—C(13)	1·481 (9)
C(2)—C(9)	1·390 (10)	C(9)—C(10)	1·366 (11)
C(3)—C(4)	1·518 (11)	C(10)—C(11)	1·383 (12)
C(3)—C(12)	1·386 (11)	C(11)—C(12)	1·379 (12)
C(4)—C(5)	1·534 (12)		
C(13)—O(3)—C(14)	115·2 (6)	C(7)—C(6)—C(8)	61·3 (5)
O(1)—C(1)—C(2)	106·7 (5)	C(6)—C(7)—C(8)	59·3 (5)
O(1)—C(1)—C(6)	110·1 (5)	C(6)—C(7)—C(15)	119·5 (6)
C(2)—C(1)—C(6)	112·0 (6)	C(6)—C(7)—C(16)	120·6 (6)
C(1)—C(2)—C(3)	121·6 (6)	C(8)—C(7)—C(15)	117·0 (6)
C(1)—C(2)—C(9)	118·8 (6)	C(8)—C(7)—C(16)	118·4 (6)
C(3)—C(2)—C(9)	119·6 (7)	C(15)—C(7)—C(16)	112·4 (6)
C(2)—C(3)—C(4)	121·6 (7)	C(6)—C(8)—C(7)	59·6 (5)
C(2)—C(3)—C(12)	118·9 (7)	C(6)—C(8)—C(13)	122·9 (6)
C(4)—C(3)—C(12)	119·5 (7)	C(7)—C(8)—C(13)	121·3 (6)
C(3)—C(4)—C(5)	113·0 (7)	C(2)—C(9)—C(10)	120·7 (7)
C(4)—C(5)—C(6)	108·9 (6)	C(9)—C(10)—C(11)	120·1 (8)
C(1)—C(6)—C(5)	111·0 (6)	C(10)—C(11)—C(12)	119·9 (8)
C(1)—C(6)—C(7)	119·6 (6)	C(3)—C(12)—C(11)	120·9 (8)
C(1)—C(6)—C(8)	114·4 (6)	O(2)—C(13)—O(3)	122·7 (7)
C(5)—C(6)—C(7)	120·4 (6)	O(2)—C(13)—C(8)	128·0 (7)
C(5)—C(6)—C(8)	122·4 (6)	O(3)—C(13)—C(8)	109·4 (6)

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2-Ethynyl-1,3-dimethoxybenzene

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Abstract. $C_{10}H_{10}O_2$, $M_r = 162{\cdot}2$, orthorhombic, $Cmc2_1$, $a = 11{\cdot}587\ (2)$, $b = 9{\cdot}262\ (2)$, $c = 8{\cdot}603\ (3)$ Å, $V = 923{\cdot}3\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}17$ g cm^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}54184$ Å, $\mu = 6{\cdot}2$ cm^{-1}, $F(000) = 344$, $T = 295\ (1)$ K, 1000 unique data measured, final $R = 0{\cdot}034$ for 929 reflections with $I > 3{\cdot}0\sigma(I)$. The molecule lies across a crystallographic mirror. The six-membered ring is nearly planar, with maximum deviation 0·002 (3) Å. The two methoxy substituents are nearly coplanar with the benzenoid ring with C—C—O—C torsion angles ± 1·4 (2)°, and are oriented *anti* to the ethynyl substituent. The triple-bond distance is 1·163 (2) Å.

Experimental. A white crystal of (1) was isolated by slow sublimation under reduced pressure and elevated temperature from the crude reaction product of 2-(1-chlorovinyl)-1,3-dimethoxybenzene and lithium diisopropylamide in tetrahydrofuran at room temperature. Crystal size 0·55 × 0·50 × 0·28 mm, cell dimensions from setting angles of 25 reflections having $20 < \theta < 30°$. Data collection on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0·33–3·59° min^{-1}. Two octants of data having $4 < 2\theta < 150°$, $0 \le h \le 14$, $0 \le k \le 11$, $-10 \le l \le 10$ measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 200, 040, 004 decreased by 5·4%; thus, a linear decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0·948 to 0·999. 1059 total data were collected, of which 1000 were unique in point group $mm2$ and not systematically absent; 929 observed with $I > 3\sigma(I)$. When hkl and $hk\bar{l}$ were averaged R_{int} was 0·03. Systematic absences hkl with $h + k$ odd and $h0l$ with l odd indicated space groups $Cmcm$ or $Cmc2_1$, or with re-indexing $Ama2$. $Cmc2_1$ was shown to be correct by successful refinement, while no successful model was found in the other two. Structure solved by direct methods, using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Two weak reflections which had F_{obs} much greater than F_{calc} and F_{obs} much greater than F_{obs} for the Friedel-related reflection were given zero weight in the refinement. Non-H atoms refined

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