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Structures of Two $C_{20}H_{24}O_4$* Dimers of 3-Acetyl-6,6-dimethylfulvene Epoxide

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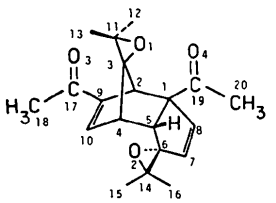
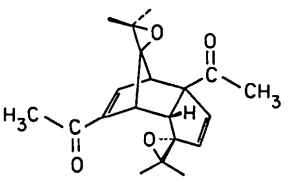
Abstract. Passage of the hydroperoxide (III) over silica gel leads to the formation of two *endo,syn* diepoxide dimers [(I), (II)] [Thomas, Perret & Bernardinelli (1985). *Chimia*, **39**, 228–229] of 3-acetyl-6,6-dimethylfulvene epoxide (IV), the crystal structures of which are reported here. (I): $C_{20}H_{24}O_4$, m.p. 411 K, $M_r = 328{\cdot}4$, monoclinic, $P2_1/n$, $a = 13{\cdot}100\ (3)$, $b = 8{\cdot}6517\ (11)$, $c = 15{\cdot}805\ (3)$ Å, $\beta = 93{\cdot}69\ (2)°$, $V = 1787{\cdot}6\ (6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}220$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}0784$ mm^{-1}, room temperature, $R = 0{\cdot}044$ for 1193 observed reflections [$|F_o| \geq 3\sigma(F_o)$ and $|F_o| \geq 7{\cdot}0$]. (II): $C_{20}H_{24}O_4$, m.p. 425 K, $M_r = 328{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}9764\ (17)$, $b = 14{\cdot}892\ (2)$, $c = 17{\cdot}043\ (5)$ Å, $V = 1770{\cdot}6\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}232$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}0791$ mm^{-1}, room temperature, $R = 0{\cdot}055$ for 1134 observed reflections [$|F_o| \geq 4\sigma(F_o)$]. Apart from the position of the acetyl group on the double bond C(9)–C(10), the two isomers reveal no quantifiable differences in their individual conformation. There are no unusual distances or angles.



(I)



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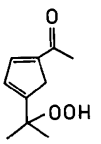

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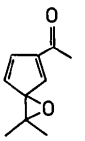

(IV)

Experimental. Experimental data and structure refinement are summarized in Table 1.† Both crystals were furnished by Firmenich S. A. Research Laboratories. Philips PW 1100 diffractometer, graphite-monochromated Mo $K\alpha$; $\omega/2\theta$ scans; Lorentz–polarization correction; no absorption correction; structures solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); no secondary-extinction correction; all calculations performed with a local version of *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *ORTEP*II (Johnson, 1976). Table 2 gives the positional parameters and Table 3 bond distances and torsion angles. Fig. 1 shows stereoviews of the two isomers.

* 3a′,5′-Diacetyl-3a′,4′,7′,7a′-tetrahydro-3,3,3″,3″-tetramethyldispiro[oxirane-2,1′-[4,7]-methano-[1*H*]-indene-8′,2″-oxirane] (I) and 3a′,6′-diacetyl-3a′,4′,7′,7a′-tetrahydro-3,3,3″,3″-tetramethyldispiro[oxirane-2,1′-[4,7]-methano-[1*H*]-indene-8′,2″-oxirane] (II).

† Lists of structure factors, atomic positional and thermal parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1985) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42706 (67 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Summary of crystal data, intensity measurement and structure refinement*

	(I)	(II)
Crystal size (mm)	0·18 × 0·33 × 0·38	0·30 × 0·33 × 0·33
Unit-cell determination	Least-squares fit from	
	22 reflections ($24° \le 2\theta \le 38°$)	21 reflections ($27° \le 2\theta \le 39°$)
$(\sin\theta/\lambda)_{max}$(Å^{-1})	0·53	0·53
h,k,l range	−13,13; 0,9; 0,15	0,7; 0,15; 0,18
Number of standard reflections (variation)	2 (0·9%)	2 (1·2%)
Number of measured reflections	2411	1306
Number of unique reflections	2190	1282
Criterion for observed reflections	$\|F\| \ge 3\sigma(F)$ and $\|F\| \ge 7$	$\|F\| \ge 4\sigma(F)$
Number of observed reflections	1193	1134
Refinement (on F)	Full matrix	
Number of parameters	217	217
Weighting scheme	$w(F) = \exp[18(\sin\theta/\lambda)^2]$	
Hydrogen atoms	Calculated	
Max., av. Δ/σ	0·15, 0·02	0·18, 0·03
Max., min. $\Delta\rho$(e Å^{-3})	0·22, −0·38	0·31, −0·32
S	3·9	8·5
R, wR (%)	4·4, 4·5	5·5, 6·4

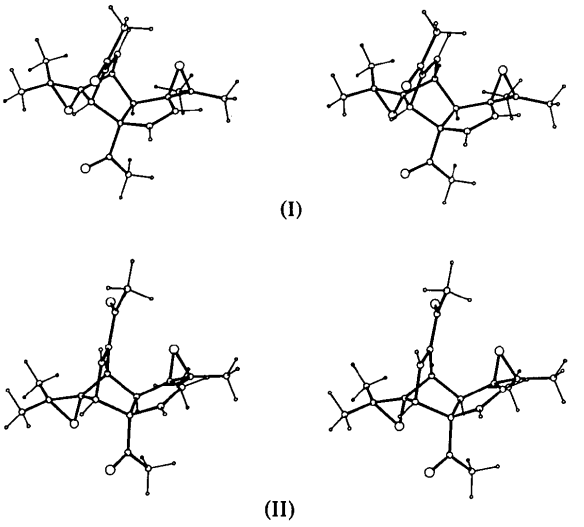
Table 2. *Fractional coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

U_{eq} is the average of the eigenvalues of U.

	x	y	z	U_{eq}(Å^2 ×10^3)
Compound (I)				
O(1)	0·36878 (22)	0·5554 (3)	0·10614 (18)	44·3 (8)
O(2)	0·67011 (21)	0·9508 (3)	0·09178 (17)	41·2 (8)
O(3)	0·5273 (4)	0·7591 (5)	0·40854 (22)	83·8 (16)
O(4)	0·5235 (3)	0·3370 (3)	0·21842 (23)	58·7 (10)
C(1)	0·5741 (3)	0·5949 (4)	0·18966 (23)	35·2 (11)
C(2)	0·4779 (3)	0·6496 (5)	0·23637 (23)	37·4 (11)
C(3)	0·4060 (3)	0·6803 (4)	0·15798 (23)	35·2 (11)
C(4)	0·4739 (3)	0·8032 (4)	0·11945 (23)	35·5 (11)
C(5)	0·5704 (3)	0·6995 (4)	0·10841 (23)	32·4 (10)
C(6)	0·6726 (3)	0·7861 (4)	0·1120 (3)	36·4 (11)
C(7)	0·7277 (3)	0·7380 (5)	0·1914 (3)	40·1 (11)
C(8)	0·6760 (3)	0·6348 (4)	0·23399 (23)	39·1 (11)
C(9)	0·4970 (3)	0·8147 (5)	0·2649 (3)	38·1 (12)
C(10)	0·4939 (3)	0·9040 (4)	0·1954 (3)	37·6 (11)
C(11)	0·2958 (3)	0·6580 (5)	0·1466 (3)	47·4 (13)
C(12)	0·2400 (4)	0·5780 (7)	0·2145 (4)	66·2 (17)
C(13)	0·2306 (3)	0·7535 (6)	0·0857 (3)	55·5 (14)
C(14)	0·7209 (3)	0·8463 (4)	0·0362 (3)	42·7 (12)
C(15)	0·8340 (4)	0·8683 (6)	0·0364 (3)	59·1 (15)
C(16)	0·6652 (4)	0·8248 (5)	−0·0496 (3)	57·5 (15)
C(17)	0·5232 (3)	0·8575 (5)	0·3529 (3)	45·3 (13)
C(18)	0·5470 (4)	1·0220 (6)	0·3745 (3)	61·3 (16)
C(19)	0·5701 (3)	0·4198 (5)	0·17336 (25)	40·6 (11)
C(20)	0·6316 (4)	0·3569 (5)	0·1057 (3)	57·7 (15)
Compound (II)				
O(1)	0·5496 (7)	−0·1426 (3)	0·17898 (24)	46·1 (12)
O(2)	0·7619 (7)	0·1653 (3)	0·0574 (3)	46·2 (12)
O(3)	0·9546 (7)	0·0035 (3)	−0·0523 (3)	54·6 (14)
O(4)	0·1135 (7)	−0·0560 (4)	0·1884 (3)	61·4 (16)
C(1)	0·3622 (8)	0·0156 (4)	0·1153 (3)	37·7 (16)
C(2)	0·3777 (9)	−0·0709 (4)	0·0628 (3)	38·6 (15)
C(3)	0·5579 (9)	−0·1103 (4)	0·1002 (3)	36·2 (15)
C(4)	0·6909 (7)	−0·0309 (3)	0·0824 (3)	33·0 (14)
C(5)	0·5753 (8)	0·0427 (3)	0·1290 (3)	33·5 (15)
C(6)	0·5953 (8)	0·1398 (3)	0·1010 (3)	33·1 (15)
C(7)	0·4025 (9)	0·1625 (4)	0·0663 (3)	44·7 (17)
C(8)	0·2775 (9)	0·0970 (4)	0·0749 (3)	44·6 (17)
C(9)	0·4601 (9)	−0·0427 (4)	−0·0139 (3)	42·2 (17)
C(10)	0·6421 (8)	−0·0190 (3)	−0·0046 (3)	34·4 (15)
C(11)	0·6104 (11)	−0·2047 (4)	0·1156 (3)	47·3 (19)
C(12)	0·4693 (15)	−0·2781 (5)	0·0986 (5)	68 (3)
C(13)	0·8150 (13)	−0·2317 (4)	0·1198 (5)	63·5 (23)
C(14)	0·7249 (9)	0·2054 (4)	0·1337 (4)	41·1 (16)
C(15)	0·6770 (11)	0·3053 (4)	0·1317 (5)	60·5 (22)
C(16)	0·8669 (10)	0·1785 (4)	0·1961 (4)	55·1 (19)
C(17)	0·7824 (9)	0·0110 (4)	−0·0640 (3)	41·1 (17)
C(18)	0·7074 (11)	0·0502 (8)	−0·1385 (4)	79 (3)
C(19)	0·2468 (8)	−0·0022 (4)	0·1893 (3)	41·4 (16)
C(20)	0·2894 (9)	0·0492 (5)	0·2619 (4)	54·8 (19)

Table 3. *Interatomic distances* (Å) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

	(I)	(II)
O(1)–C(3)	1·423 (5)	1·427 (7)
O(1)–C(11)	1·478 (5)	1·484 (7)
O(2)–C(6)	1·460 (5)	1·431 (7)
O(2)–C(14)	1·451 (5)	1·453 (7)
O(3)–C(17)	1·222 (6)	1·223 (8)
O(4)–C(19)	1·203 (5)	1·228 (8)
C(1)–C(2)	1·575 (6)	1·572 (8)
C(1)–C(5)	1·569 (5)	1·558 (8)
C(1)–C(8)	1·507 (5)	1·515 (8)
C(1)–C(19)	1·537 (6)	1·520 (8)
C(2)–C(3)	1·530 (5)	1·527 (9)
C(2)–C(9)	1·514 (6)	1·489 (8)
C(3)–C(4)	1·537 (5)	1·535 (8)
C(3)–C(11)	1·457 (6)	1·476 (8)
C(4)–C(5)	1·569 (5)	1·575 (7)
C(4)–C(10)	1·492 (5)	1·532 (8)
C(5)–C(6)	1·532 (5)	1·529 (7)
C(6)–C(7)	1·467 (6)	1·508 (9)
C(6)–C(14)	1·485 (6)	1·443 (8)
C(7)–C(8)	1·330 (6)	1·317 (9)
C(9)–C(10)	1·342 (6)	1·327 (9)
C(9)–C(17)	1·459 (6)	—
C(10)–C(17)	—	1·477 (8)
C(11)–C(12)	1·505 (7)	1·499 (11)
C(11)–C(13)	1·493 (6)	1·485 (12)
C(14)–C(15)	1·495 (7)	1·524 (9)
C(14)–C(16)	1·509 (6)	1·509 (9)
C(17)–C(18)	1·492 (7)	1·493 (10)
C(19)–C(20)	1·484 (7)	1·484 (9)
C(5)–C(1)–C(2)–C(3)	36·0 (3)	36·0 (5)
C(2)–C(1)–C(5)–C(4)	1·0 (3)	0·4 (5)
C(8)–C(1)–C(5)–C(6)	1·9 (4)	3·9 (5)
C(5)–C(1)–C(8)–C(7)	−1·1 (4)	−1·5 (6)
C(5)–C(1)–C(19)–O(4)	−142·7 (4)	−150·3 (6)
C(1)–C(2)–C(3)–C(4)	−60·0 (3)	−60·0 (5)
C(2)–C(3)–C(4)–C(5)	60·8 (3)	59·9 (4)
C(3)–C(4)–C(5)–C(1)	−37·5 (3)	−36·4 (5)
C(1)–C(5)–C(6)–C(7)	−2·1 (4)	−4·7 (5)
C(5)–C(6)–C(7)–C(8)	1·5 (5)	4·0 (6)
C(6)–C(7)–C(8)–C(1)	−0·3 (5)	−1·6 (7)
C(2)–C(9)–C(10)–C(4)	−0·5 (4)	0·0 (5)
C(10)–C(9)–C(17)–O(3)	−176·2 (5)	—
C(9)–C(10)–C(17)–O(3)	—	−156·8 (6)



(I)

(II)

Fig. 1. Stereopairs showing the conformations of the two isomers of $C_{20}H_{24}O_4$.

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