parallel, with the angle between the two plane normals being 0(3)°.

The O atoms of the $CH_3O_3S^-$ anion form O··· HN-type hydrogen bonds. The O(1) atom in this structure is unmistakably hydrogen-bonded to H(3) with an H(3)···O(1) distance of 1·91 (5) Å (Table 2). In addition, O(1) has close contacts with two mirror-related H(5) atoms with a distance of 2·61 (4) Å. This distance is much longer than the usual hydrogen-bond value of 2·0 Å given by Hamilton & Ibers (1968) for the NH···O-type bond. A value of 1·92 (2) Å was reported in the structure of hycanthone methanesulfonate (Wei & Einstein, 1978) and another value of 2·04 (4) Å was found in the structure of IA-4 methanesulfonate (Wei, 1981). According to these criteria, N—H(5)···O(1) is probably not a hydrogen bond. In summary, each O atom of the anion is hydrogen-bonded to a neighboring H atom while three of the four H atoms of the NH_4^+ ion participate in the bonding scheme, resulting in an infinite chain around the crystallographic twofold screw axis ($x = \frac{1}{4}$ or $\frac{3}{4}$, $z = 0$) in the **b** direction, as shown in Fig. 2.

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Structure of a Novel 3+2 Enone Cyclodimer

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Abstract. 3a-Hydroxytrispiro[octahydropentalene-1,1′:2,1″:3,1‴-triscyclopentane]-2‴-one, $C_{20}H_{30}O_2$, $M_r = 302{\cdot}46$, monoclinic, $P2_1/c$, $a = 11{\cdot}626$ (3), $b = 10{\cdot}761$ (2), $c = 12{\cdot}989$ (3) Å, $\beta = 92{\cdot}10$ (2)°, $V = 1623{\cdot}93$ Å^3, $Z = 4$, $D_x = 1{\cdot}237$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7093 Å, μ(Mo $K\alpha$) = 0·72 cm^{-1}, $F(000) = 664$, $T = 298$ K, $R = 0{\cdot}048$ for 1030 observed reflections. The structure reveals the bicyclo[3.3.0]octan-7-ol skeleton

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with three spiro five-membered rings attached to the 1, 2, and 3 positions and indicates the 2+3 enone photodimerization of two 2-cyclopentylidenecyclopentanone molecules.

Introduction. The photochemical dimerization of α, β-unsaturated ketones is a well known process (Eaton, 1968). The 2+2 cyclodimerization of 2-cyclohexenone was also observed when the photochemical reaction of 2-cyclohexenone was carried out in the presence of tertiary amines (Pienta & Mckimmey, 1982). We have studied the photochemical reaction of 2-cyclopentylidenecyclopentanone with several tertiary amines such as triethylamine, *N*-methyldiisopropylamine and *N*-ethyldiisopropylamine. A novel 3+2 cyclodimerization product, $C_{20}H_{30}O_2$, was isolated and its structure has been determined by single-crystal X-ray diffraction studies.

Experimental. The photochemical reaction was carried out in a quartz reaction vessel containing 2-cyclopentylidenecyclopentanone (0·2 *M*) and tertiary amine (2 *M*) in acetonitrile solution. The reaction vessel was irradiated with a Hanovia 450 W medium pressure mercury lamp. The products were isolated with column chromatography (silica gel, Merck Kieselgel 60, 70–230 mesh). Single crystals were grown from *n*-hexane as solvent at room temperature.

A colorless crystal of dimensions 0·05 × 0·15 × 0·50 mm was used for data collection. CAD-4 diffractometer, graphite-monochromated Mo *K*α radiation. Unit-cell dimensions determined from a least-squares refinement of 25 reflections ($18{\cdot}44 < 2\theta < 22{\cdot}8^\circ$). Intensity data within $2\theta < 50^\circ$ were collected at various scan speeds of 20/16–20/3° min^{-1}, by using the $\omega/2\theta$ scan technique, with a scan range $(0{\cdot}7 + 0{\cdot}35\tan\theta)^\circ$. Three reflections monitored every 2 hours and fluctuation within 2%. No corrections for absorption or extinction. 2847 unique reflections were collected, 1030 observed reflections with $I \geq 3\sigma(I)$.

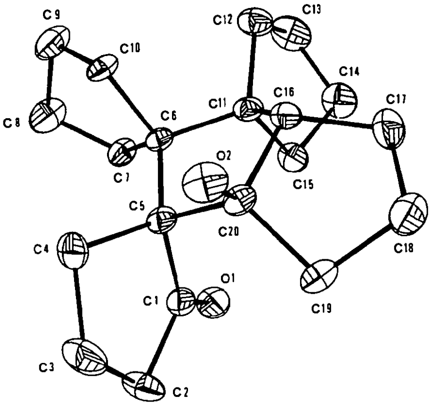

Fig. 1. The molecular structure and labeling scheme of $C_{20}H_{30}O_2$.

The structure was solved by direct methods using *MULTAN* with 392 largest *E*'s, 100 smallest *E*'s and 7512 $\sum_2$ phase relationships. All the non-hydrogen atoms located from the *E* map. The positions of H atoms were calculated from an idealized geometry and

Table 1. *Positional parameters with e.s.d.'s in parentheses and thermal parameters*

	x	*y*	*z*	B_{iso}
C(1)	0·8869 (5)	0·1846 (6)	0·5161 (4)	3·1 (3)
C(2)	1·0154 (6)	0·1957 (7)	0·5029 (5)	4·4 (4)
C(3)	1·0665 (6)	0·2028 (7)	0·6127 (5)	4·7 (5)
C(4)	0·9727 (5)	0·1491 (6)	0·6789 (4)	3·5 (4)
C(5)	0·8574 (5)	0·1971 (5)	0·6300 (4)	2·5 (3)
C(6)	0·7453 (5)	0·1301 (5)	0·6617 (4)	2·3 (3)
C(7)	0·7236 (6)	−0·0003 (6)	0·6146 (4)	3·2 (4)
C(8)	0·7507 (7)	−0·0945 (6)	0·6964 (5)	5·3 (5)
C(9)	0·7172 (7)	−0·0328 (6)	0·7943 (5)	5·0 (5)
C(10)	0·7550 (5)	0·1014 (6)	0·7805 (4)	3·0 (4)
C(11)	0·6471 (5)	0·2278 (6)	0·6394 (4)	2·7 (3)
C(12)	0·5335 (6)	0·2028 (7)	0·6962 (5)	4·0 (4)
C(13)	0·4397 (6)	0·1792 (8)	0·6161 (6)	5·6 (5)
C(14)	0·4761 (6)	0·2561 (7)	0·5261 (5)	4·7 (4)
C(15)	0·6044 (5)	0·2322 (6)	0·5242 (4)	3·5 (4)
C(16)	0·7051 (5)	0·3493 (5)	0·6771 (4)	2·9 (3)
C(17)	0·6666 (6)	0·4748 (6)	0·6329 (5)	4·5 (4)
C(18)	0·7766 (6)	0·5429 (6)	0·6072 (5)	4·1 (4)
C(19)	0·8565 (6)	0·4383 (6)	0·5747 (5)	3·4 (4)
C(20)	0·8348 (5)	0·3383 (5)	0·6565 (4)	2·6 (3)
O(1)	0·8212 (4)	0·1617 (4)	0·4440 (3)	4·2 (3)
O(2)	0·9053 (4)	0·3772 (4)	0·7432 (3)	3·7 (2)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·514 (8)	C(11)–C(16)	1·543 (8)
C(2)–C(3)	1·526 (9)	C(14)–C(15)	1·514 (9)
C(5)–C(6)	1·558 (7)	C(17)–C(18)	1·521 (9)
C(6)–C(10)	1·573 (7)	C(20)–O(2)	1·431 (6)
C(8)–C(9)	1·498 (9)	C(1)–O(1)	1·212 (7)
C(11)–C(15)	1·560 (7)	C(4)–C(5)	1·550 (8)
C(13)–C(14)	1·51 (1)	C(6)–C(7)	1·547 (8)
C(16)–C(20)	1·545 (8)	C(7)–C(8)	1·495 (9)
C(19)–C(20)	1·540 (8)	C(11)–C(12)	1·559 (8)
C(1)–C(5)	1·537 (7)	C(12)–C(13)	1·501 (9)
C(3)–C(4)	1·527 (9)	C(16)–C(17)	1·528 (8)
C(5)–C(20)	1·582 (8)	C(18)–C(19)	1·528 (9)
C(6)–C(11)	1·571 (8)		
C(9)–C(10)	1·522 (9)		

C(2)–C(1)–C(5)	110·9 (5)	C(2)–C(1)–O(1)	121·7 (5)
C(5)–C(1)–O(1)	127·3 (5)	C(1)–C(2)–C(3)	104·5 (4)
C(2)–C(3)–C(4)	104·2 (5)	C(3)–C(4)–C(5)	105·5 (4)
C(1)–C(5)–C(4)	98·3 (4)	C(1)–C(5)–C(6)	115·6 (4)
C(1)–C(5)–C(20)	109·8 (4)	C(4)–C(5)–C(6)	117·1 (4)
C(4)–C(5)–C(20)	112·3 (4)	C(6)–C(5)–C(20)	103·9 (4)
C(5)–C(6)–C(7)	116·2 (4)	C(5)–C(6)–C(10)	108·6 (4)
C(5)–C(6)–C(11)	104·5 (4)	C(7)–C(6)–C(10)	102·4 (4)
C(7)–C(6)–C(11)	115·2 (4)	C(10)–C(6)–C(11)	109·7 (4)
C(6)–C(7)–C(8)	107·9 (4)	C(7)–C(8)–C(9)	104·3 (5)
C(8)–C(9)–C(10)	103·7 (5)	C(6)–C(10)–C(9)	106·9 (4)
C(6)–C(11)–C(12)	114·8 (4)	C(6)–C(11)–C(15)	113·7 (4)
C(6)–C(11)–C(16)	101·7 (4)	C(12)–C(11)–C(15)	102·3 (4)
C(12)–C(11)–C(16)	111·3 (4)	C(15)–C(11)–C(16)	113·5 (4)
C(11)–C(12)–C(13)	108·0 (4)	C(12)–C(13)–C(14)	103·1 (5)
C(13)–C(14)–C(15)	103·0 (5)	C(11)–C(15)–C(14)	105·6 (4)
C(11)–C(16)–C(17)	120·7 (5)	C(11)–C(16)–C(20)	107·3 (4)
C(17)–C(16)–C(20)	106·1 (4)	C(16)–C(17)–C(18)	105·7 (5)
C(17)–C(18)–C(19)	103·3 (5)	C(18)–C(19)–C(20)	101·9 (4)
C(5)–C(20)–C(16)	106·4 (4)	C(5)–C(20)–C(19)	119·2 (4)
C(5)–C(20)–O(2)	110·9 (4)	C(16)–C(20)–C(19)	104·6 (4)
C(16)–C(20)–O(2)	112·1 (4)	C(19)–C(20)–O(2)	103·6 (4)

checked with the difference map, except for the H atom attached to O(2), which was located from the difference map. All non-H atoms were refined anisotropically and all H atoms were fixed and not refined. The final $R(F) = 4{\cdot}81\%$ ($wR = 3{\cdot}33\%$). Weighting scheme from counting statistics, $w = 1/\sigma^2(F)$, $\Delta/\sigma < 0{\cdot}06$, peak in final difference map $< 0{\cdot}2$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All the calculations were performed using the NRCC *SDP* PDP-11 package (Gabe & Lee, 1981), *MULTAN* and *ORTEPB* from the Enraf–Nonius *Structure Determination Package* (Enraf–Nonius, 1979) on a PDP–11/23 computer. Fig. 1 shows the molecular structure and labeling scheme.

Discussion. The positional and isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. C(1)—O(1) is a double bond, while C(20)—O(2) is a single bond. All the C—C bonds are single, ranging from 1·495 to 1·582 Å. There is an intermolecular hydrogen bond between O(2) and O(1). The O(2)—H and O(1)···H distances are 1·00 (3) and 1·88 (3) Å respectively and the O(2)—H···O(1) angle is 165 (4)°.

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* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43194 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 5-Isopropyl-4-methyl-1,3-diphenyl-3a,4,5,9b-tetrahydropyrazolo[3,4-*c*]-isoquinoline

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Abstract. $C_{26}H_{27}N_3$, $M_r = 381{\cdot}5$, triclinic, $P\bar{1}$, $a = 9{\cdot}877\,(2)$, $b = 10{\cdot}493\,(2)$, $c = 11{\cdot}281\,(2)$ Å, $\alpha = 112{\cdot}18\,(3)$, $\beta = 98{\cdot}76\,(2)$, $\gamma = 93{\cdot}24\,(2)$°, $V = 1062$ Å^3, $Z = 2$, $D_x = 1{\cdot}19$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}47$ mm^{-1}, $F(000) = 408$, $T = 273$ K, $R = 0{\cdot}053$, $wR = 0{\cdot}064$ for 1759 independent reflections. The diastereoisomeric *trans* configuration is clearly evidenced. The pyrazolic ring is formed by regioselective 1,3-dipolar cycloaddition [C=N = 1·280 (4), N—N = 1·369 (3) Å]. The quasiplanar W-shaped H—C—N—C—H conformation explains the NMR coupling parameters.

Introduction. In a previous work (Kitane, Tshiamala, Laude, Vebrel & Cerutti, 1985), we discussed the stereochemistry of 1,3-dipolar cycloaddition of diphenylnitrilimine (1,3-dipole) to 1-isopropyl-2-methyl-1,2-dihydroisoquinoline (dipolarophile).

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