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4,17-Diacetyl-3α,8,12α,18β-tetramethyl-4,5,7,14,16,17-hexaazapentacyclo[10.6.2.$0^{2,11}$.$0^{3,8}$.$0^{13,18}$]icosa-5,7,9,13,15,19-hexaene (I*a*) 0·5-Methanol Solvate and Trimethyl 2α,8β-Dimethyl-3,4,6-triazatricyclo[6.2.2.$0^{2,7}$]dodeca-4,6,9,11-tetraene-3,9,10-tricarboxylate (II*b*)

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Abstract. (I*a*): $C_{22}H_{26}N_6O_2.\frac{1}{2}CH_4O$, $M_r = 422{\cdot}5$, triclinic, $P\bar{1}$, $a = 10{\cdot}307\,(2)$, $b = 10{\cdot}378\,(2)$, $c = 11{\cdot}959\,(2)$ Å, $\alpha = 69{\cdot}28\,(2)$, $\beta = 69{\cdot}07\,(1)$, $\gamma = 69{\cdot}57\,(1)°$, $V = 1080{\cdot}6\,(4)$ Å^3, $Z = 2$, $D_m = 1{\cdot}28$, $D_x = 1{\cdot}298$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 450$, room temperature, $R = 0{\cdot}047$, $wR = 0{\cdot}046$ for 3235 observed reflections with $I > \sigma(I)$. (II*b*): $C_{17}H_{19}N_3O_6$, $M_r = 361{\cdot}4$, orthorhombic, *Pbca*, $a = 8{\cdot}313\,(2)$, $b = 16{\cdot}156\,(7)$, $c = 26{\cdot}094\,(9)$ Å, $V = 3505\,(2)$ Å^3, $Z = 8$, density not measured, $D_x = 1{\cdot}370$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 1520$, room temperature, $R = 0{\cdot}083$, $wR = 0{\cdot}062$ for 1321 observed reflections with $I > \sigma(I)$. The two structures demonstrate the existence of 1,8a-dihydro-1,2-benzodiazines, which cannot be isolated in the oxidation reaction of N^2-alkylamino-N^1-(2,6-dimethylphenyl)formamidines with MnO_2 in CH_2Cl_2. In fact, the reaction either continues to form the dimer (I) or, in the presence of an excess of dimethyl acetylendicarbonate, gives (II). The dienophilic attack occurs in the *endo* position. The 'common' part of both molecules shows no substantial differences in geometrical terms.

Introduction. Within a framework of research on triazadienes of the type (IV), as precursors of 1,2,4-benzotriazines or 1,2,4-benzotriazepines, Garanti (1986) oxidized the azoketimine (III) with MnO_2 in CH_2Cl_2. The reaction product was a dimer of (IV), to which the structure (I) was tentatively attributed on the basis of NMR and mass spectroscopy. The hypothetical intermediate (V) was not isolated and its

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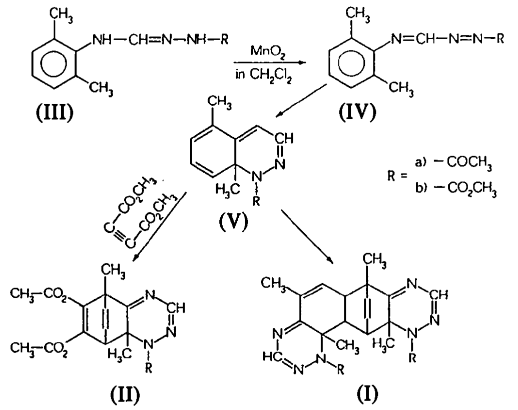
Table 1. *Details of data collection and structure refinement*

	Compound (I*a*)	Compound (II*b*)
(*a*) Data collection		
Crystal size (mm)	0·35 × 0·35 × 0·20 (pale yellow prism)	0·20 × 0·20 × 0·10 (colourless irregular prism)
Cell-parameter determination		
Number; range of reflections (°)	24; 12–14	24; 10–12
Range *h*	0,13	0,8
k	−13,13	0,17
l	−15,15	0,28
θ (°)	0·0–30	0·0–22·5
Scan technique	ω/θ	ω/θ
Scan range (°)	1·1 + 0·25 tanθ	1·4 + 0·35 tanθ
Scan speed (° min^{-1})	1·6–10	2·2–10
Standard reflections		
Number; variation of intensity (%)	3; 6	1; 1
Number of intensity measurements	4963	2286
(*b*) Refinement		
Refined reflections	3235 if $I > \sigma(I)$	1321 if $I > \sigma(I)$
R	0·047	0·083
wR	0·046	0·062
S	1·96	2·05
Weighting scheme	$w = 4F_o\|\sigma^2(F_o^2) + 0{\cdot}0004(F_o^4)\|^{-1}$	
$(\Delta/\sigma)_{max}$ in the last cycle	0·2	0·05
$\Delta\rho$ in final difference Fourier map (e Å^{-3})	0·5	0·3
Extinction coefficient *g* (× 10^6) (Larson, 1967, equation 3)	4 (1)	Not refined

presence was indirectly confirmed by product (II) obtained in small quantities by adding to the solution an excess of dimethyl butynedioate.



Experimental. Density of (I*a*) measured using fresh crystals by flotation in dilute K_2HgI_4 solution: after one month, crystals become opaque by loss of methanol solvate. Density of (II*b*) not measured because of lack and smallness of crystals. Details of data collection and refinement in Table 1. Intensity data collected with an Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo *K*α radiation. Correction for Lorentz and polarization, not for absorption. For (I*a*) correction for decay was applied. Structures solved using direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971). For both compounds, full-matrix least-squares refinement was performed, minimizing the quantity $\sum w(|F_o| - |F_c|)^2$ and including scale factor, anisotropic C, N, O atoms, isotropic H atoms. Refinement of (I*a*) also included extinction parameter, but not the H's of solvate methanol; multiplicity of methanol molecule was kept to $\frac{1}{2}$. In the refinement of (II*b*), the methyl H's were put in calculated positions; those of C(22) and C(26) were split into six positions each with multiplicity 0·5. Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used include Enraf–Nonius (1979) *SDP*, *ORTEP* (Johnson, 1965), *PARST* (Nardelli, 1983) and various in-house programs for refinement and geometrical analysis running on a Gould 32/97 computer.

Table 2. *Final coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = (1/6\pi^2)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}(Å^2)
Compound (I*a*)				
O(24)	0·2046 (2)	0·1394 (2)	0·4690 (1)	0·0556 (8)
O(29)	0·4189 (2)	0·1506 (2)	0·2030 (1)	0·0543 (8)
O(31)	0·451 (2)	0·625 (1)	−0·041 (1)	0·21 (1)
N(5)	0·0953 (2)	0·3574 (2)	0·3677 (2)	0·0388 (8)
N(6)	0·0959 (2)	0·4995 (2)	0·3301 (2)	0·0510 (9)
N(8)	−0·1255 (2)	0·5652 (2)	0·2741 (2)	0·0481 (9)
N(15)	0·1283 (2)	−0·1223 (2)	0·1799 (2)	0·0412 (8)
N(17)	0·3498 (2)	−0·1571 (2)	0·2228 (2)	0·0424 (8)
N(18)	0·3098 (2)	−0·0127 (2)	0·2179 (1)	0·0350 (7)
C(1)	0·2194 (2)	0·0858 (2)	0·1337 (2)	0·0316 (8)
C(2)	0·1331 (2)	0·2297 (2)	0·1682 (2)	0·0330 (8)
C(3)	0·0287 (2)	0·1924 (2)	0·2998 (2)	0·0301 (8)
C(4)	−0·0262 (2)	0·3089 (2)	0·3701 (2)	0·0326 (8)
C(7)	−0·0083 (3)	0·5895 (3)	0·2888 (2)	0·056 (1)
C(9)	−0·1299 (2)	0·4338 (2)	0·3084 (2)	0·0371 (8)
C(10)	−0·2488 (2)	0·3996 (2)	0·2955 (2)	0·0428 (9)
C(11)	−0·2335 (2)	0·2670 (2)	0·2935 (2)	0·0416 (9)
C(12)	−0·0964 (2)	0·1514 (2)	0·2901 (2)	0·0333 (8)
C(13)	−0·0473 (2)	0·1092 (2)	0·1629 (2)	0·0376 (9)
C(14)	0·1035 (2)	0·0132 (2)	0·1576 (2)	0·0331 (8)
C(16)	0·2653 (2)	−0·1990 (2)	0·1953 (2)	0·045 (1)
C(19)	0·0535 (2)	0·3090 (2)	0·0703 (2)	0·0431 (9)
C(20)	−0·0327 (2)	0·2463 (2)	0·0638 (2)	0·046 (1)
C(21)	0·3140 (2)	0·1049 (3)	−0·0006 (2)	0·042 (1)
C(22)	−0·1076 (3)	0·2548 (3)	0·5061 (2)	0·045 (1)
C(23)	0·2029 (2)	0·2651 (2)	0·4221 (2)	0·046 (1)
C(25)	0·3168 (4)	0·3251 (4)	0·4193 (4)	0·068 (2)
C(26)	−0·3837 (3)	0·5174 (3)	0·2842 (4)	0·073 (2)
C(27)	−0·1488 (3)	0·0359 (3)	0·1585 (3)	0·055 (1)
C(28)	0·4067 (2)	0·0288 (2)	0·2451 (2)	0·043 (1)
C(30)	0·4917 (3)	−0·0820 (3)	0·3308 (3)	0·061 (1)
C(32)	0·559 (1)	0·510 (2)	−0·028 (2)	0·24 (1)
Compound (II*b*)				
O(15)	0·2011 (6)	−0·0328 (3)	0·4465 (2)	0·077 (3)
O(16)	0·4475 (6)	0·0138 (3)	0·4308 (2)	0·060 (2)
O(20)	0·0922 (9)	0·1512 (5)	0·2572 (3)	0·116 (3)
O(21)	−0·0883 (8)	0·0506 (4)	0·2529 (3)	0·109 (3)
O(24)	−0·0904 (7)	−0·0978 (4)	0·3703 (2)	0·095 (3)
O(25)	0·1048 (7)	−0·0517 (3)	0·3177 (2)	0·075 (2)
N(3)	0·2430 (7)	0·1024 (4)	0·4280 (2)	0·041 (2)
N(4)	0·3643 (7)	0·1633 (4)	0·4296 (2)	0·057 (3)
N(6)	0·1755 (8)	0·2514 (4)	0·3860 (2)	0·051 (2)
C(1)	−0·062 (1)	0·0695 (5)	0·4202 (3)	0·052 (3)
C(2)	0·0756 (8)	0·1340 (4)	0·4352 (3)	0·040 (3)
C(5)	0·317 (1)	0·2347 (5)	0·4125 (3)	0·062 (4)
C(7)	0·0629 (9)	0·1992 (4)	0·3935 (3)	0·040 (3)
C(8)	−0·0803 (9)	0·1934 (4)	0·3576 (3)	0·047 (3)
C(9)	−0·0430 (8)	0·1097 (5)	0·3327 (3)	0·051 (3)
C(10)	−0·0377 (8)	0·0449 (5)	0·3660 (3)	0·046 (3)
C(11)	−0·215 (1)	0·1193 (6)	0·4239 (4)	0·061 (4)
C(12)	−0·223 (1)	0·1822 (6)	0·3920 (4)	0·064 (4)
C(13)	0·0552 (9)	0·1676 (5)	0·4889 (3)	0·069 (3)
C(14)	0·291 (1)	0·0221 (5)	0·4362 (3)	0·044 (3)
C(17)	0·5076 (9)	−0·0704 (5)	0·4370 (3)	0·075 (4)
C(18)	−0·097 (1)	0·2667 (5)	0·3206 (3)	0·084 (4)
C(19)	−0·008 (1)	0·1070 (6)	0·2757 (4)	0·070 (4)
C(22)	−0·040 (1)	0·0523 (8)	0·1960 (3)	0·146 (6)
C(23)	−0·011 (1)	−0·0430 (5)	0·3516 (3)	0·057 (4)
C(26)	0·120 (1)	−0·1315 (6)	0·2953 (4)	0·121 (5)

Discussion. The final positional parameters and their e.s.d.'s are given in Table 2.* Tables 3 and 4 give bond distances and angles. Fig. 1 shows the molecules with

Table 3. *Bond distances* (Å)

Compound (I*a*)

O(24)–C(23)	1·220 (3)	O(29)–C(28)	1·218 (3)
O(31)–C(32)	1·32 (2)	N(5)–N(6)	1·383 (2)
N(5)–C(4)	1·492 (3)	N(5)–C(23)	1·380 (3)
N(6)–C(7)	1·275 (3)	N(8)–C(7)	1·395 (4)
N(8)–C(9)	1·290 (3)	N(15)–C(14)	1·282 (2)
N(15)–C(16)	1·399 (3)	N(17)–N(18)	1·394 (2)
N(17)–C(16)	1·276 (4)	N(18)–C(1)	1·484 (2)
N(18)–C(28)	1·386 (4)	C(1)–C(2)	1·561 (2)
C(1)–C(14)	1·519 (3)	C(1)–C(21)	1·536 (2)
C(2)–C(3)	1·560 (2)	C(2)–C(19)	1·501 (3)
C(3)–C(4)	1·545 (3)	C(3)–C(12)	1·544 (3)
C(4)–C(9)	1·512 (2)	C(4)–C(22)	1·540 (3)
C(9)–C(10)	1·459 (4)	C(10)–C(11)	1·337 (3)
C(10)–C(26)	1·509 (3)	C(11)–C(12)	1·499 (2)
C(12)–C(13)	1·589 (3)	C(13)–C(14)	1·519 (2)
C(13)–C(20)	1·510 (3)	C(13)–C(27)	1·517 (5)
C(19)–C(20)	1·308 (4)	C(23)–C(25)	1·495 (6)
C(28)–C(30)	1·499 (4)		

Compound (II*b*)

O(15)–C(14)	1·19 (1)	O(16)–C(14)	1·32 (1)
O(16)–C(17)	1·46 (1)	O(20)–C(19)	1·20 (1)
O(21)–C(19)	1·27 (1)	O(21)–C(22)	1·54 (1)
O(24)–C(23)	1·21 (1)	O(25)–C(23)	1·32 (1)
O(25)–C(26)	1·42 (1)	N(3)–N(4)	1·41 (1)
N(3)–C(2)	1·49 (1)	N(3)–C(14)	1·37 (1)
N(4)–C(5)	1·30 (1)	N(6)–C(5)	1·39 (1)
N(6)–C(7)	1·27 (1)	C(1)–C(2)	1·60 (1)
C(1)–C(10)	1·48 (1)	C(1)–C(11)	1·51 (1)
C(2)–C(7)	1·52 (1)	C(2)–C(13)	1·51 (1)
C(7)–C(8)	1·52 (1)	C(8)–C(9)	1·53 (1)
C(8)–C(12)	1·50 (1)	C(8)–C(18)	1·53 (1)
C(9)–C(10)	1·36 (1)	C(9)–C(19)	1·52 (1)
C(10)–C(23)	1·49 (1)	C(11)–C(12)	1·32 (1)

Table 4. *Bond angles* (°)

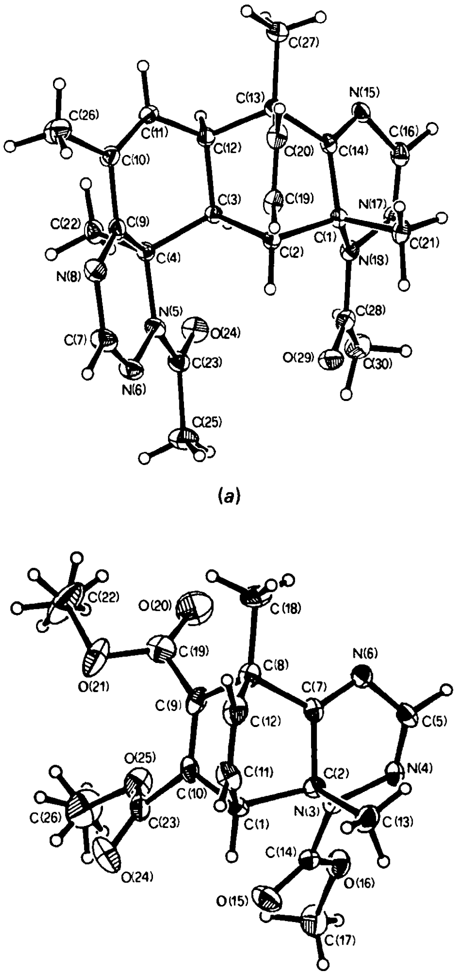
Compound (I*a*)

C(4)–N(5)–C(23)	121·3 (2)	N(6)–N(5)–C(23)	115·2 (2)
N(6)–N(5)–C(4)	122·7 (2)	N(5)–N(6)–C(7)	117·2 (2)
C(7)–N(8)–C(9)	115·7 (2)	C(14)–N(15)–C(16)	114·7 (2)
N(18)–N(17)–C(16)	115·5 (2)	N(17)–N(18)–C(28)	113·5 (2)
N(17)–N(18)–C(1)	117·4 (2)	C(1)–N(18)–C(28)	122·5 (2)
N(18)–C(1)–C(21)	108·6 (2)	N(18)–C(1)–C(14)	104·9 (1)
N(18)–C(1)–C(2)	113·8 (1)	C(14)–C(1)–C(21)	112·1 (2)
C(2)–C(1)–C(21)	113·4 (2)	C(2)–C(1)–C(14)	103·6 (2)
C(1)–C(2)–C(19)	103·5 (2)	C(1)–C(2)–C(3)	106·8 (1)
C(3)–C(2)–C(19)	111·8 (2)	C(2)–C(3)–C(12)	109·4 (1)
C(2)–C(3)–C(4)	114·6 (1)	C(4)–C(3)–C(12)	111·7 (2)
N(5)–C(4)–C(3)	110·9 (2)	C(3)–C(4)–C(22)	111·4 (2)
C(3)–C(4)–C(9)	108·6 (2)	N(5)–C(4)–C(22)	108·8 (2)
N(5)–C(4)–C(9)	109·4 (2)	C(9)–C(4)–C(22)	107·7 (2)
N(6)–C(7)–N(8)	128·9 (2)	N(8)–C(9)–C(4)	125·5 (2)
C(4)–C(9)–C(10)	115·4 (2)	N(8)–C(9)–C(10)	119·0 (2)
C(9)–C(10)–C(26)	117·8 (2)	C(9)–C(10)–C(11)	119·1 (2)
C(11)–C(10)–C(26)	123·1 (2)	C(10)–C(11)–C(12)	124·8 (2)
C(3)–C(12)–C(11)	114·5 (2)	C(11)–C(12)–C(13)	108·4 (2)
C(3)–C(12)–C(13)	108·0 (2)	C(12)–C(13)–C(27)	111·6 (2)
C(12)–C(13)–C(20)	104·7 (2)	C(12)–C(13)–C(14)	104·3 (2)
C(20)–C(13)–C(27)	116·0 (2)	C(14)–C(13)–C(27)	112·5 (2)
C(14)–C(13)–C(20)	106·9 (2)	C(1)–C(14)–C(13)	116·1 (2)
N(15)–C(14)–C(13)	121·2 (2)	N(15)–C(14)–C(1)	122·6 (2)
N(15)–C(16)–N(17)	127·0 (2)	C(2)–C(19)–C(20)	115·9 (2)
C(13)–C(20)–C(19)	115·0 (2)	O(24)–C(23)–N(5)	120·6 (2)
N(5)–C(23)–C(25)	117·3 (2)	O(24)–C(23)–C(25)	122·0 (3)
O(29)–C(28)–N(18)	121·1 (2)	N(18)–C(28)–C(30)	116·8 (2)
O(29)–C(28)–C(30)	122·1 (3)		

Table 4 (*cont.*)

Compound (II*b*)

C(14)–O(16)–C(17)	114·9 (6)	C(19)–O(21)–C(22)	107·6 (8)
C(23)–O(25)–C(26)	116·0 (7)	C(2)–N(3)–C(14)	125·0 (6)
N(4)–N(3)–C(14)	116·6 (6)	N(4)–N(3)–C(2)	115·1 (5)
N(3)–N(4)–C(5)	113·2 (6)	C(5)–N(6)–C(7)	114·6 (7)
C(10)–C(1)–C(11)	108·8 (7)	C(2)–C(1)–C(11)	103·9 (6)
C(2)–C(1)–C(10)	108·0 (6)	N(3)–C(2)–C(1)	114·5 (6)
C(1)–C(2)–C(13)	112·4 (6)	C(1)–C(2)–C(7)	103·1 (6)
N(3)–C(2)–C(13)	110·1 (6)	N(3)–C(2)–C(7)	102·3 (5)
C(7)–C(2)–C(13)	114·0 (6)	N(4)–C(5)–N(6)	126·8 (8)
N(6)–C(7)–C(2)	121·2 (7)	C(2)–C(7)–C(8)	117·0 (6)
N(6)–C(7)–C(8)	121·5 (6)	C(7)–C(8)–C(18)	114·3 (6)
C(7)–C(8)–C(12)	105·1 (6)	C(7)–C(8)–C(9)	99·1 (6)
C(12)–C(8)–C(18)	113·6 (7)	C(9)–C(8)–C(18)	115·5 (6)
C(9)–C(8)–C(12)	107·9 (6)	C(8)–C(9)–C(19)	118·7 (7)
C(8)–C(9)–C(10)	114·4 (7)	C(10)–C(9)–C(19)	126·8 (8)
C(1)–C(10)–C(9)	113·5 (7)	C(9)–C(10)–C(23)	125·4 (7)
C(1)–C(10)–C(23)	121·2 (7)	C(1)–C(11)–C(12)	114·5 (8)
C(8)–C(12)–C(11)	115·5 (8)	O(16)–C(14)–N(3)	111·4 (7)
O(15)–C(14)–N(3)	123·8 (8)	O(15)–C(14)–O(16)	124·7 (7)
O(21)–C(19)–C(9)	112·3 (8)	O(20)–C(19)–C(9)	120·6 (8)
O(20)–C(19)–O(21)	126·9 (9)	O(25)–C(23)–C(10)	112·3 (7)
O(24)–C(23)–C(10)	121·3 (8)	O(24)–C(23)–O(25)	126·4 (8)



(*a*)

(*b*)

Fig. 1. (*a*) Compound (I*a*); (*b*) compound (II*b*). Thermal ellipsoids for heavy atoms are drawn at 50% of probability level; H atoms not to scale.

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* 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 43306 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

numbering scheme used. The structure of the two molecules confirms the reaction path of the scheme. It is worth noting that the dienophilic attack occurs in the *endo* position. For molecule (I*a*), the dienophile is the unsubstituted double bond. Comparison of the 'common' part of (I*a*) and (II*b*) shows no substantial difference in geometrical terms. Molecule (II*b*) is loosely packed: no relevant intermolecular contact is found. In (I*a*) there is one short intermolecular contact, namely N(15)···H(7) 2·47 (3) Å with C(7)—H(7)···N(15) 155 (2)°.

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Structure of 2-Phenyladamantan-2-ol

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Abstract. 2-Phenyltricyclo[3.3.1.1^{3,7}]decan-2-ol, $C_{16}H_{20}O$, $M_r = 228{\cdot}33$, tetragonal, $I4_1/a$, $a = 22{\cdot}794\ (5)$, $c = 10{\cdot}004\ (2)$ Å, $V = 5198\ (2)$ Å^3, $Z = 16$, $D_x = 1{\cdot}167$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54183$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 5{\cdot}1$ cm^{-1}, $F(000) = 1984$, room temperature, $R = 0{\cdot}062$ for 2174 unique reflections. Four symmetry-related molecules are linked by intermolecular hydrogen bonds forming a tetramer. The tetramers are held together by van der Waals forces. The adamantane skeleton fixes the phenyl ring in such a position that the C—O bond rotates out of the phenyl plane by 82°. The position of the hydroxyl H atom suggests intramolecular OH···π interaction.

Introduction. An infrared study has demonstrated that intramolecular OH···π interaction in benzyl alcohols and anthracylmethanols depends on the position of the phenyl ring relative to the C—O axis (Visser & Van der Maas, 1986). It appears that OH···π interaction occurs if the C—O bond is twisted out of the phenyl plane by 90°. Ahmed & Huber (1981) have investigated the structure of 1-phenylcyclohexanol by means of X-rays. This compound also has a phenyl ring and a hydroxyl group substituted at the same C atom. However, the torsion angles involving the phenyl rings and the C—O bonds are small (between −11·7 and 27·3°). The X-ray analysis of the title compound was undertaken to determine the molecular stereochemistry, especially the position of the phenyl ring in connection with possible OH···π interaction.

Experimental. Data were measured on a crystal of dimensions 0·8 × 1·1 × 0·4 mm on an Enraf–Nonius CAD-4F diffractometer with Ni-filtered Cu $K\alpha$ radiation. Lattice constants from eight reflections in the range $62 < 2\theta < 64°$, ω–2θ mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}15\tan\theta)°$. The crystal is tetragonal with space group $I4_1/a$. 2460 unique reflections were measured up to $\theta = 70°$, $0 \le h \le 27$, $0 \le k \le 27$, $0 \le l \le 12$, 2178 of these were considered observed [$I > 1{\cdot}5\sigma(I)$] and used for structure determination and refinement. Three periodically measured standard reflections (600, 060, 004) showed intensity variations less then 6%; Lp correction, no correction for absorption or secondary extinction. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$ where $P(=0{\cdot}014)$ is the instability constant (McCandlish, Stout & Andrews, 1975). The structure was solved with *SHELXS*84 (Sheldrick, 1984); the best E map gave all non-H atoms. H atoms were placed on calculated positions except the hydrogen H atom which was located on a difference map. The temperature parameters of H atoms were set equal to the equivalent isotropic temperature factors of the carrier atoms. All refinements were full-matrix least-squares refinements using *SHELX*76 (Sheldrick, 1976). Hydrogen parameters were kept fixed except for the coordinates of the hydroxyl H atom. The reflections 040, 020, $\bar{2}40$ and 240 were omitted [$\Delta F/\sigma(F) > 10$] and the refinement on F converged at $R = 0{\cdot}0624$, $wR = 0{\cdot}0789$ where $w = 1/\sigma^2(F)$, and $S = 4{\cdot}3$. $|\Delta/\sigma| = 0{\cdot}03$ (av.) and 0·09 (max.) for non-H-atom parameters; final electron density showed a maximum and minimum of 0·13 and

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