

# Complexation with Diol Host Compounds. Part 1. Structures of the 1:2 Molecular Complexes of *trans*-9,10-Dihydroxy-9,10-Diphenyl-9,10- Dihydroanthracene with Acetophenone and with 3-Methylcyclopentanone

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**Abstract.** The structures of the 1:2 molecular complexes of *trans*-9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene with acetophenone (**1**), (C<sub>26</sub>H<sub>20</sub>O<sub>2</sub>·2 C<sub>8</sub>H<sub>8</sub>O) and with 3-methylcyclopentanone (**2**), (C<sub>26</sub>H<sub>20</sub>O<sub>2</sub>·2C<sub>6</sub>H<sub>10</sub>O) have been determined by X-ray crystallography. The crystal data are as follows: Compound (**1**):  $P\bar{1}$ ,  $a = 8.979(5) \text{ \AA}$ ,  $b = 9.316(3) \text{ \AA}$ ,  $c = 11.12(1) \text{ \AA}$ ,  $\alpha = 94.40(6)^\circ$ ,  $\beta = 106.53(6)^\circ$ ,  $\gamma = 109.92(5)^\circ$ ,  $V = 822.94 \text{ \AA}^3$ ,  $Z = 1$ ,  $R = 0.097$  for 2549 unique reflections. Compound (**2**):  $P\bar{1}$ ,  $a = 8.958(7) \text{ \AA}$ ,  $b = 9.815(4) \text{ \AA}$ ,  $c = 9.807(4) \text{ \AA}$ ,  $\alpha = 96.88(3)^\circ$ ,  $\beta = 109.21(8)^\circ$ ,  $\gamma = 103.33(7)^\circ$ ,  $V = 774.10 \text{ \AA}^3$ ,  $Z = 1$ ,  $R = 0.059$  for 2494 unique reflections. The intermolecular arrangements in both structures are characterised by host-to-guest hydrogen bonding interactions. The thermal properties of compound (**2**) have been characterised by DTA and TGA thermograms.

**Key words.** Inclusion compounds, crystal structures, *trans*-9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene, acetophenone, 3-methylcyclopentanone, guest molecules.

**Supplementary Data** relating to this article are deposited with the British Library as supplementary publication No. SUP 82079 (31 pages).

## 1. Introduction

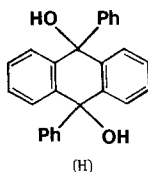
The separation and purification of materials are important processes both in the laboratory and in industry. Recently researchers have recognised that if the method of inclusion compound formation can be applied, then these processes must be simplified and rendered economically attractive. To date the lattice inclusion phenomenon has been successfully applied to problems of molecular separation [1–4]. Studies on molecular inclusion by hydroxy host systems indicate that these compounds form crystalline complexes with a variety of alcohols and glycols [5–7]. X-ray crystallographic investigations have provided important information which is crucial to the design of new and efficient host compounds. The compound *trans*-

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9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene (H) was designed to meet all requirements necessary for molecular inclusion formation [8]. It is a rigid molecule and has an anti-diol function to allow for possible H-bond formation and has bulky hydrophobic phenyl groups for surrounding and thus sandwiching potential guest molecules.

We are undertaking a systematic study of this host compound with a variety of guest compounds in order to better understand the type of host-guest interactions and their strengths.

We present the structures of the 1:2 molecular complexes of the host (H) with acetophenone ( $C_8H_8O$ ) (1) and with 3-methylcyclopentanone ( $C_6H_{10}O$ ) (2).



## 2. Experimental

The host compound (H) was obtained by reacting anthraquinone with a Grignard reagent prepared from bromobenzene. On acidification the product *trans*-9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene (H) precipitated from solution. Recrystallisation from benzene produced pure white crystals which melted at 260–262°C. (Yield = 36%), anal. Found: C 85.30%; H 5.6%. Calcd. for  $C_{26}H_{20}O_2$ : C 85.70%; H 5.5%.

The 1:2 inclusion complexes were obtained as transparent single crystals by slow evaporation from dilute solutions of the host in the guest.

Evidence for the presence of the acetophenone guest was obtained by visually following the thermal decomposition of the complex using a Fisher Johns hot-stage melting point apparatus. The transparent crystals released acetophenone at ca 110°C and became white and opaque and finally melted at the expected temperature of 262°C.

The thermal properties of compound (2) were characterised by differential thermal analysis (DTA) and thermal gravimetric analysis (TGA). The thermograms were obtained on a Stanton-Redcroft Thermal Analyser (Model STA 780) operating at a uniform heating rate of 10°C/min. Nitrogen was passed through the cell at a rate of 60 mL/min during the run. Only the furnace temperature could be measured directly as the platinum sample container is located directly above the thermocouple. The temperature lag was estimated by accurate measurement of the melting points of a series of standards.

The results of the thermal analysis are shown in Figure 1. The inclusion compound lost the 3-methylcyclopentanone guest molecules (in a single step) between 64°C and 115°C as shown by peak A on the DTA curve. This corresponds to a 35.4% weight loss in agreement with the required stoichiometry (35.0%). Thereafter melting of the host molecule occurred between 256°C and 277°C, giving rise to peak B on the DTA curve.

Crystal densities were obtained by flotation using a mixture of a saturated KI solution and  $H_2O$ .

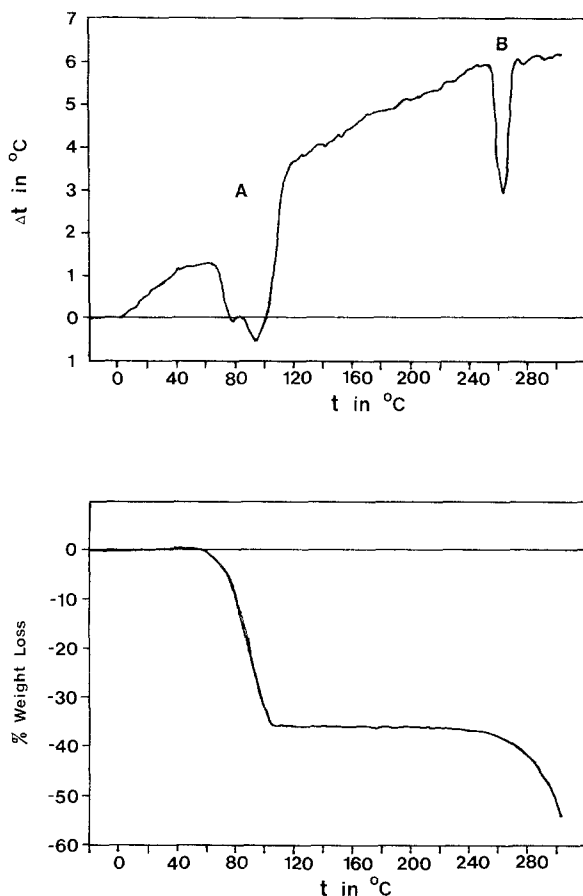


Fig. 1. DTA and TGA thermograms for compound (2).

Suitable crystals of each complex were sealed in a 0.5 mm Lindemann glass capillary surrounded by mother liquor and mounted on a CAD4 diffractometer. Intensity data were collected at  $21^{\circ}\text{C}$  using graphite monochromated  $\text{MoK}_{\alpha}$  radiation ( $\lambda = 0.71069 \text{ \AA}$ ). An  $\omega-2\theta$  scan mode was employed at a maximum recording time of 40 s. The intensities of three standard reflections were checked every hour and recentering was carried out every 200 measured reflections. Intensities were corrected for Lorentz and polarisation factors and absorption corrections were applied. Both structures were solved using the Direct Methods program SHELXS-86 [9] and refined using SHELX-76 [10].

All non-hydrogen atoms were varied anisotropically in the structure refinements. The host aromatic hydrogens were placed in calculated positions with a common temperature factor. The host hydroxy hydrogens were located from the difference Fourier map and the O—H length was constrained to a value of  $0.97(1) \text{ \AA}$  in both structures. This value was chosen as representing the typical hydrogen bonding geometry as extrapolated from a graph depicting O—H versus O $\cdots$ O distances for a large number of crystal structures [11].

In the final refinement of structure (1) the guest phenyl hydrogens were placed in calculated positions and tied with a common temperature factor. The methyl hydrogens were treated as a rigid group with a single temperature factor. In the refinement of the 3-methylcyclopentanone guest, compound (2), the methylene protons were generated geometrically and assigned a single temperature factor. The methyl protons were constrained as for compound (1). The geometry of C(22) is planar and the position of the methine proton H(22) could not be uniquely defined. It was therefore omitted from the final model.

Complex neutral-atom scattering factors were employed: for non-hydrogen atoms from Cromer and Mann [12]; for hydrogen atoms from Stewart, Davidson and Simpson [13]. Molecular parameters were obtained from Parst [14] drawings with PLUTO [15]. All computations were carried out on a Sperry 1100 computer. Further details are supplied in Table I.

Table I. Crystal data, experimental and refinement parameters

Compound	1	2
Molecular formula	C <sub>26</sub> H <sub>20</sub> O·2 C <sub>8</sub> H <sub>8</sub> O	C <sub>26</sub> H <sub>20</sub> O <sub>2</sub> ·2 C <sub>6</sub> H <sub>10</sub> O
Molecular weight (g mol <sup>-1</sup> )	604.75	560.74
<i>D</i> <sub>c</sub> (g cm <sup>-3</sup> )	1.22	1.20
<i>D</i> <sub>m</sub> (g cm <sup>-3</sup> )	1.20	1.20
$\mu$ (MoK $\alpha$ )(cm <sup>-1</sup> )	0.42	0.42
<i>F</i> (000)	320	300
Data collection (21°C)		
Crystal dimensions	0.50 × 0.53 × 0.53	0.25 × 0.35 × 0.38
Range scanned $\theta$ (°)	1–25	1–25
Range of indices <i>h</i> , <i>k</i> , <i>l</i>	±10, ±11, +13	±10, ±11, +11
Reflections for lattice parameters no., $\theta$ range (°)	24, 16–17	24, 16–17
Stability of standard reflections (%)	1.6	4.5
Scan mode	$\omega$ -2 $\theta$	$\omega$ -2 $\theta$
Scan width in $\omega$ (°)	(1.05 + 0.35 tan $\theta$ )	(0.95 + 0.35 tan $\theta$ )
Vertical aperture length (mm)	6	6
Aperture width (mm)	(1.40 + 1.05 tan $\theta$ )	(1.15 + 1.05 tan $\theta$ )
Number of reflections collected (unique)	2549	2494
Number of reflections observed with $I_{\text{rel}} > 2\sigma I_{\text{rel}}$	2085	2143
Final refinement		
Number of parameters	217	200
<i>R</i>	0.097	0.059
$\omega R$	0.123	0.082
$\omega$	( $\sigma^2 F + 0.02 F^2$ ) <sup>-1</sup>	( $\sigma^2 F + 0.002 F^2$ ) <sup>-1</sup>
<i>S</i>	1.30	3.62
Max. shift/e.s.d.	0.1	0.1
Max. height in difference Fourier map (e Å <sup>-3</sup> )	0.39	0.48
Min. height in difference Fourier map (e Å <sup>-3</sup> )	-0.40	-0.35
Absorption correction		
Max., Min. % values	99.93, 98.72	99.92, 96.73

### 3. Results and Discussion

Final fractional atomic coordinates for both compounds are listed in Tables II and III. Lists of anisotropic thermal parameters, torsion angles and structure factors have been deposited. Bond lengths and angles are listed in Tables IV and V for structure (1) and in Tables VI and VII for structure (2). All bond lengths and angles are in agreement with literature values [5–7]. Any discrepancies are ascribed to vibrational effects.

Table II. Fractional atomic coordinates ( $\times 10^4$ ) and thermal parameters ( $\text{\AA}^2 \times 10^3$ ) with e.s.ds in parentheses for compound (1)

Atom	$x/a$	$y/b$	$z/c$	$U_{\text{iso}}/U_{\text{equ}}, *$
C(1)	413(4)	–1458(4)	10006(3)	40(1)*
O(1)	–150(3)	–2776(3)	9007(3)	52(1)*
H(1)	–641(–)	–2527(–)	8194(–)	103(22)
C(2)	1427(4)	2(4)	9645(3)	40(1)*
C(3)	2795(5)	–8(5)	9267(4)	54(2)*
H(3)	3048(5)	–974(5)	9237(4)	75(5)
C(4)	3777(5)	1258(5)	8946(5)	59(2)*
H(4)	4741(5)	1216(5)	8685(5)	75(5)
C(5)	3456(5)	2609(5)	8975(5)	64(2)*
H(5)	4169(5)	3542(5)	8725(5)	75(5)
C(6)	2129(5)	2646(5)	9359(5)	57(2)*
H(6)	1918(5)	3632(5)	9418(5)	75(5)
C(7)	1083(4)	1345(4)	9663(4)	40(1)*
C(11)	1567(4)	–1762(4)	11175(4)	40(2)*
C(12)	2386(6)	–611(5)	12266(5)	63(2)*
H(12)	2196(6)	386(5)	12281(5)	75(5)
C(13)	3464(7)	–834(7)	13331(5)	79(2)*
H(13)	4087(7)	21(7)	14100(5)	75(5)
C(14)	3687(6)	–2219(6)	13347(6)	75(3)*
H(14)	4444(6)	–2385(6)	14131(6)	75(5)
C(15)	2872(6)	–3365(5)	12287(6)	67(2)*
H(15)	3038(6)	–4373(5)	12291(6)	75(5)
C(16)	1820(5)	–3146(4)	11210(5)	54(2)*
H(16)	1228(5)	–4002(4)	10439(5)	75(5)
C(20)	–744(8)	–2604(7)	5526(6)	81(3)*
O(20)	–1331(7)	–2419(6)	6354(4)	112(3)*
C(30)	948(10)	–1465(10)	5644(8)	124(5)*
H(301)	1425(10)	–2071(10)	5191(8)	165(14)
H(302)	1620(10)	–1160(10)	6576(8)	165(14)
H(303)	990(10)	–509(10)	5279(8)	165(14)
C(21)	–1696(6)	–3916(6)	4424(5)	64(2)*
C(22)	–994(7)	–4305(7)	3548(5)	73(3)*
H(22)	194(7)	–3671(7)	3636(5)	165(14)
C(23)	–1933(10)	–5561(8)	2553(6)	96(4)*
H(23)	–1412(10)	–5859(8)	1941(6)	165(14)
C(24)	–3614(10)	–6423(8)	2401(7)	97(3)*
H(24)	–4309(10)	–7321(8)	1670(7)	165(14)
C(25)	–4292(9)	–6018(8)	3264(7)	95(3)*
H(25)	–5490(9)	–6630(8)	3170(7)	165(14)
C(26)	–3348(8)	–4789(7)	4246(7)	87(3)*
H(26)	–3872(8)	–4508(7)	4862(7)	165(14)

Table III. Fractional atomic coordinates ( $\times 10^4$ ) and thermal parameters ( $\text{\AA}^2 \times 10^3$ ) with e.s.ds in parentheses for compound (2)

Atom	$x/a$	$y/b$	$z/c$	$U_{\text{iso}}/U_{\text{equiv}}^*$
O(1)	3909(2)	6865(2)	8498(2)	54(1)*
H(1)	3892(-)	6230(-)	7665(-)	98(11)
C(1)	4376(3)	6322(2)	9820(2)	42(1)*
C(2)	3249(3)	4822(2)	9563(2)	42(1)*
C(3)	1552(3)	4662(3)	9161(3)	52(1)*
H(3)	1144(3)	5526(3)	9069(3)	69(3)
C(4)	444(3)	3348(3)	8888(3)	60(1)*
H(4)	-760(3)	3255(3)	8589(3)	69(3)
C(5)	998(3)	2150(3)	9029(3)	60(1)*
H(5)	194(3)	1188(3)	8831(3)	69(3)
C(6)	2659(3)	2288(3)	9442(3)	54(1)*
H(6)	3055(3)	1418(3)	9550(3)	69(3)
C(7)	3803(3)	3623(2)	9711(2)	43(1)*
C(11)	4153(2)	7324(2)	11001(2)	42(1)*
C(12)	3764(3)	8589(3)	10752(3)	51(1)*
H(12)	3627(3)	8862(3)	9778(3)	69(3)
C(13)	3566(3)	9470(3)	11863(3)	59(1)*
H(13)	3291(3)	10379(3)	11676(3)	69(3)
C(14)	3742(3)	9121(3)	13197(3)	60(1)*
H(14)	3583(3)	9763(3)	13977(3)	69(3)
C(15)	4140(3)	7873(3)	13464(3)	58(1)*
H(15)	4272(3)	7608(3)	14442(3)	69(3)
C(16)	4354(3)	6988(3)	12370(3)	51(1)*
H(16)	4657(3)	6094(3)	12574(3)	69(3)
O(20)	3489(3)	5280(3)	5706(3)	88(1)*
C(20)	2397(4)	4206(4)	4899(3)	70(1)*
C(21)	1107(5)	3297(5)	5289(4)	101(2)*
H(211)	507(5)	3894(5)	5674(4)	225(14)
H(212)	1607(5)	2776(5)	6054(4)	225(14)
C(22)	-56(8)	2318(8)	3896(6)	195(4)*
C(23)	657(6)	2314(5)	2848(4)	111(2)*
H(231)	1001(6)	1425(5)	2703(4)	225(14)
H(232)	-182(6)	2364(5)	1899(4)	225(14)
C(24)	2156(5)	3612(5)	3328(4)	98(2)*
H(241)	1933(5)	4327(5)	2699(4)	225(14)
H(242)	3145(5)	3333(5)	3278(4)	225(14)
C(221)	-1438(6)	1180(5)	3905(5)	111(2)*
H(221)	-1879(6)	1695(5)	4558(5)	224(18)
H(222)	-1350(6)	249(5)	4191(5)	224(18)
H(223)	-2203(6)	993(5)	2853(5)	224(18)

A perspective view of the host molecule and its two associated guest molecules is shown in Figures 2 and 3 for compounds (1) and (2) respectively. In both structures the 9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene molecules occupy centrosymmetric sites. The symmetry is met by the *trans* orientation of the two phenyl and two hydroxy substituents. The conformation of the tricyclic carbon skeleton is planar. The central 1,4-cyclohexadiene ring is positioned on the centre of symmetry

Table IV. Bond lengths (Å) with e.s.ds in parentheses for compound (I)

C(1)—O(1)	1.431(5)
C(1)—C(2)	1.513(5)
C(1)—C(11)	1.528(5)
O(1)—H(1)	.968(51)
C(2)—C(3)	1.410(7)
C(2)—C(7)	1.388(6)
C(3)—H(3)	1.000(7)
C(3)—C(4)	1.353(6)
C(4)—H(4)	1.000(8)
C(4)—C(5)	1.384(7)
C(5)—H(5)	1.000(7)
C(5)—C(6)	1.385(8)
C(6)—H(6)	1.000(7)
C(6)—C(7)	1.391(6)
C(11)—C(12)	1.384(6)
C(11)—C(16)	1.385(6)
C(12)—H(12)	.999(8)
C(12)—C(13)	1.381(8)
C(13)—H(13)	1.000(7)
C(13)—C(14)	1.373(9)
C(14)—H(14)	1.000(8)
C(14)—C(15)	1.358(7)
C(15)—H(15)	1.000(7)
C(15)—C(16)	1.375(8)
C(16)—H(16)	1.000(6)
C(20)—O(20)	1.213(10)
C(20)—C(30)	1.490(10)
C(20)—C(21)	1.480(7)
C(30)—H(301)	1.000(15)
C(30)—H(302)	1.000(11)
C(30)—H(303)	1.000(14)
C(21)—C(22)	1.388(9)
C(21)—C(26)	1.374(8)
C(22)—H(22)	1.000(8)
C(22)—C(23)	1.379(8)
C(23)—H(23)	1.000(13)
C(23)—C(24)	1.399(11)
C(24)—H(24)	1.000(9)
C(24)—C(25)	1.366(13)
C(25)—H(25)	1.000(10)
C(25)—C(26)	1.357(9)
C(26)—H(26)	1.000(12)
C(1)—C(7)'	1.524(6)

has internal torsion angles of less than  $3^\circ$  in both structures and thus its asymmetry parameters [ $\Delta C_s$  and  $\Delta C_2$ ] are less than 0.3. In describing the conformations of cyclic systems we employ the method of asymmetry parameters developed by Duax [16]. These parameters define the conformations of any ring relative to ideal forms and related torsion angles are compared so that the parameter value is zero if the symmetry in question is present.

Table V. Bond angles (deg) with e.s.ds in parentheses for compound (1)

C(2)—C(1)—C(11)	108.1(4)
O(1)—C(1)—C(11)	106.6(4)
O(1)—C(1)—C(2)	110.6(4)
C(1)—O(1)—H(1)	109.6(43)
C(1)—C(2)—C(7)	123.7(5)
C(1)—C(2)—C(3)	118.0(5)
C(3)—C(2)—C(7)	118.3(5)
C(2)—C(3)—C(4)	121.6(6)
C(2)—C(3)—H(3)	119.2(7)
H(3)—C(3)—C(4)	119.3(7)
C(3)—C(4)—C(5)	120.6(6)
C(3)—C(4)—H(4)	119.7(7)
H(4)—C(4)—C(5)	119.7(7)
C(4)—C(5)—C(6)	118.6(6)
C(4)—C(5)—H(5)	120.7(7)
H(5)—C(5)—C(6)	120.7(7)
C(5)—C(6)—C(7)	121.6(6)
C(5)—C(6)—H(6)	119.2(7)
H(6)—C(6)—C(7)	119.3(7)
C(2)—C(7)—C(6)	119.3(5)
C(1)—C(11)—C(16)	123.3(5)
C(1)—C(11)—C(12)	118.7(5)
C(12)—C(11)—C(16)	118.0(5)
C(11)—C(12)—C(13)	120.2(6)
C(11)—C(12)—H(12)	119.9(7)
H(12)—C(12)—C(13)	119.9(7)
C(12)—C(13)—C(14)	120.7(6)
C(12)—C(13)—H(13)	119.6(8)
H(13)—C(13)—C(14)	119.7(8)
C(13)—C(14)—C(15)	119.4(6)
C(13)—C(14)—H(14)	120.3(8)
H(14)—C(14)—C(15)	120.3(8)
C(14)—C(15)—C(16)	120.5(6)
C(14)—C(15)—H(15)	119.8(8)
H(15)—C(15)—C(16)	119.8(7)
C(11)—C(16)—C(15)	121.1(6)
C(15)—C(16)—H(16)	119.4(7)
C(11)—C(16)—H(16)	119.5(7)
C(30)—C(20)—C(21)	121.1(7)
O(20)—C(20)—C(21)	120.7(8)
O(20)—C(20)—C(30)	118.2(7)
C(20)—C(30)—H(303)	116.9(11)
C(20)—C(30)—H(302)	106.8(10)
C(20)—C(30)—H(301)	104.5(10)
H(302)—C(30)—H(303)	109.5(12)
H(301)—C(30)—H(303)	109.5(12)
H(301)—C(30)—H(302)	109.5(13)
C(20)—C(21)—C(26)	119.1(7)
C(20)—C(21)—C(22)	122.8(7)
C(22)—C(21)—C(26)	118.1(6)
C(21)—C(22)—C(23)	120.2(7)
C(21)—C(22)—H(22)	119.9(7)
H(22)—C(22)—C(23)	119.9(8)
C(22)—C(23)—C(24)	120.1(7)
C(22)—C(23)—H(23)	119.9(10)
H(23)—C(23)—C(24)	120.0(9)
C(23)—C(24)—C(25)	119.0(8)
C(23)—C(24)—H(24)	120.5(11)
H(24)—C(24)—C(25)	120.5(10)
C(24)—C(25)—C(26)	120.2(9)
C(24)—C(25)—H(25)	119.9(9)
H(25)—C(25)—C(26)	119.9(10)
C(21)—C(26)—C(25)	122.4(8)
C(25)—C(26)—H(26)	118.9(9)
C(21)—C(26)—H(26)	118.8(9)
C(2)—C(1)—C(7)	113.6(5)
C(2)—C(7)—C(1)	122.7(5)



Table VI. Bond lengths (Å) with e.s.ds in parentheses for compound (2)

O(1)—H(1)	0.961(32)
O(1)—C(1)	1.431(3)
C(1)—C(2)	1.520(3)
C(1)—C(11)	1.523(3)
C(2)—C(3)	1.405(4)
C(2)—C(7)	1.384(3)
C(3)—H(3)	1.000(5)
C(3)—C(4)	1.368(4)
C(4)—H(4)	1.000(4)
C(4)—C(5)	1.382(5)
C(5)—H(5)	1.000(4)
C(5)—C(6)	1.376(4)
C(6)—H(6)	1.001(5)
C(6)—C(7)	1.398(4)
C(11)—C(12)	1.393(4)
C(11)—C(16)	1.384(4)
C(12)—H(12)	0.999(4)
C(12)—C(13)	1.392(4)
C(13)—H(13)	1.000(4)
C(13)—C(14)	1.359(4)
C(14)—H(14)	1.000(4)
C(14)—C(15)	1.383(4)
C(15)—H(15)	1.001(4)
C(15)—C(16)	1.390(4)
C(16)—H(16)	1.000(4)
O(20)—C(20)	1.229(4)
C(20)—C(21)	1.475(6)
C(20)—C(24)	1.505(5)
C(21)—H(211)	1.000(7)
C(21)—H(212)	1.000(7)
C(21)—C(22)	1.471(6)
C(22)—C(23)	1.379(9)
C(22)—C(221)	1.467(9)
C(23)—H(231)	1.000(8)
C(23)—H(232)	0.999(6)
C(23)—C(24)	1.516(6)
C(24)—H(241)	1.001(7)
C(24)—H(242)	1.000(7)
C(221)—H(221)	1.000(8)
C(221)—H(222)	1.000(7)
C(221)—H(223)	1.000(6)
C(1)—C(7)'	1.528(4)

Surveys of available structural data for 9,10-substituted-9,10-dihydroanthracene compounds [17, 18] have revealed that the conformational preference of the central 1,4-cyclohexadiene ring (planar or folded) depends on the degree and stereochemistry as well as on the steric bulk of the substituents. To date, including the present study, all except one of the *trans*-9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene molecules exhibit a preferential planar conformation of the

Table VII. Bond angles (degrees) with e.s.ds in parentheses for compound (2)

H(1)—O(1)—C(1)	111.9(17)
O(1)—C(1)—C(11)	106.6(3)
O(1)—C(1)—C(2)	109.7(2)
C(2)—C(1)—C(11)	109.5(3)
C(1)—C(2)—C(7)	123.8(4)
C(1)—C(2)—C(3)	117.4(3)
C(3)—C(2)—C(7)	118.8(4)
C(2)—C(3)—C(4)	121.3(4)
C(2)—C(3)—H(3)	119.3(5)
H(3)—C(3)—C(4)	119.3(6)
C(3)—C(4)—C(5)	119.8(5)
C(3)—C(4)—H(4)	120.1(5)
H(4)—C(4)—C(5)	120.1(5)
C(4)—C(5)—C(6)	119.8(4)
C(4)—C(5)—H(5)	120.0(6)
H(5)—C(5)—C(6)	120.2(5)
C(5)—C(6)—C(7)	121.0(4)
C(5)—C(6)—H(6)	119.5(5)
H(6)—C(6)—C(7)	119.5(5)
C(2)—C(7)—C(6)	119.3(4)
C(1)—C(11)—C(16)	119.8(3)
C(1)—C(11)—C(12)	121.8(3)
C(12)—C(11)—C(16)	118.4(3)
C(11)—C(12)—C(13)	119.8(3)
C(11)—C(12)—H(12)	120.0(4)
H(12)—C(12)—C(13)	120.2(4)
C(12)—C(13)—C(14)	121.3(4)
C(12)—C(13)—H(13)	119.3(4)
H(13)—C(13)—C(14)	119.4(4)
C(13)—C(14)—C(15)	119.6(4)
C(13)—C(14)—H(14)	120.2(4)
H(14)—C(14)—C(15)	120.3(4)
C(14)—C(15)—C(16)	119.8(3)
C(14)—C(15)—H(15)	120.1(5)
H(15)—C(15)—C(16)	120.2(4)
C(11)—C(16)—C(15)	121.1(4)
C(15)—C(16)—H(16)	119.4(4)
C(11)—C(16)—H(16)	119.5(4)
O(20)—C(20)—C(24)	125.0(5)
O(20)—C(20)—C(21)	126.2(4)
C(21)—C(20)—C(24)	108.8(4)
C(20)—C(21)—C(22)	105.5(4)
C(20)—C(21)—H(212)	110.5(7)
C(20)—C(21)—H(211)	110.4(6)
H(212)—C(21)—C(22)	112.5(6)
H(211)—C(21)—C(22)	108.5(7)
H(211)—C(21)—H(212)	109.4(6)
C(21)—C(22)—C(221)	119.9(5)
C(21)—C(22)—C(23)	110.1(6)
C(23)—C(22)—C(221)	126.1(5)
C(22)—C(23)—C(24)	109.4(5)
C(22)—C(23)—H(232)	107.5(7)
C(22)—C(23)—H(231)	111.4(6)
H(232)—C(23)—C(24)	109.9(5)
H(231)—C(23)—C(24)	109.2(7)
H(231)—C(23)—H(232)	109.5(6)
C(20)—C(24)—C(23)	103.5(4)
C(23)—C(24)—H(242)	111.3(6)
C(23)—C(24)—H(241)	110.7(5)
C(20)—C(24)—H(242)	111.1(5)
C(20)—C(24)—H(241)	110.7(5)
H(241)—C(24)—H(242)	109.4(7)
C(22)—C(221)—H(223)	100.1(6)
C(22)—C(221)—H(222)	124.8(8)
C(22)—C(221)—H(221)	102.6(6)
H(222)—C(221)—H(223)	109.4(7)
H(221)—C(221)—H(223)	109.5(8)
H(221)—C(221)—H(222)	109.4(7)
C(2)—C(1)—C(7)'	112.9(3)
C(2)—C(7)—C(1)'	123.3(3)

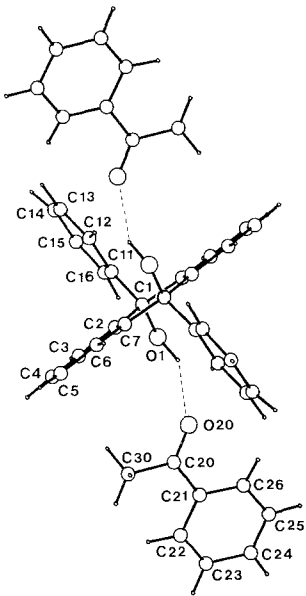


Fig. 2. A perspective view of the host-guest inclusion complex (1).

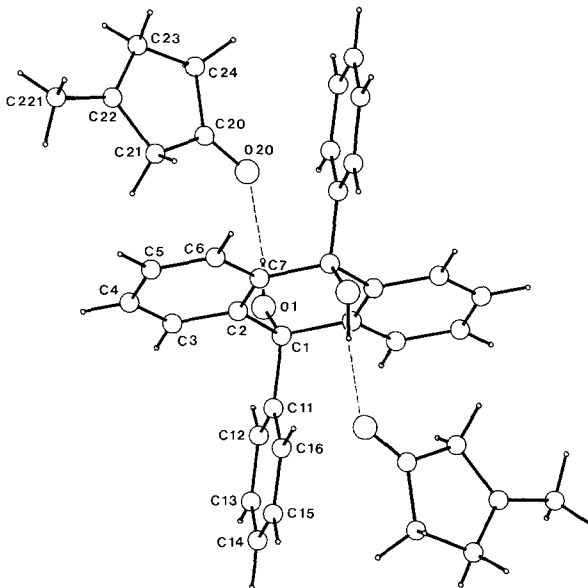


Fig. 3. A perspective view of the host molecule and its two associated guest molecules (2).

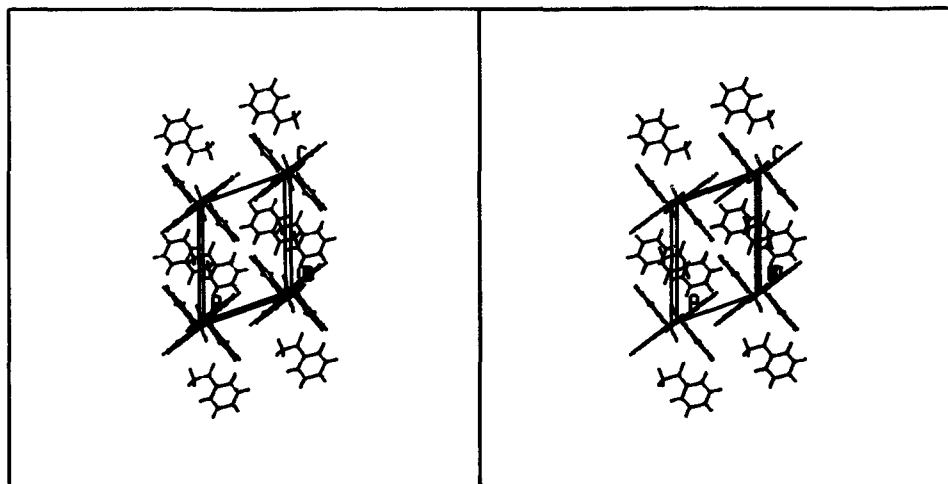


Fig. 4. A stereoview of the crystal structure of compound (1) viewed along [010] (*c* is vertical).

tricyclic molecular skeleton [18]. The phenyl substituents of the host molecules are planar to within 0.01 Å and are perpendicularly orientated with respect to the tricyclic backbone (the dihedral angles are 87.8° in structure (1) and 85.1° in structure (2)).

The conformation of the acetophenone guest molecule (compound (1)) shows distortion from the essentially planar geometry elucidated for the acetophenone molecule itself [19]. In compound (1) the methyl carbon atom of the acetophenone guest deviates from the plane of the phenyl ring by 0.20 Å whereas the oxygen atom is displaced by -0.19 Å. The torsion angles C(22)C(21)C(20)C(30) and C(26)C(21)C(20)O(20) are 9.9(11)° and 9.5(11)° respectively. The phenyl moiety of the acetophenone molecule is planar to within 0.01 Å and makes a dihedral angle of 78.4° with respect to the phenyl substituent of the adjacent host molecule. This arrangement precludes any stacking interactions between phenyl moieties of host and guest molecules.

In compound (2) the 3-methylcyclopentanone guest molecule has an envelope conformation. A single mirror plane of symmetry passing through the out-of-plane atom C(22), predominates ( $\Delta C_S^{22} = 0.61$ ,  $\Delta C_2^{20} = 7.92$  and  $\Delta C_S^{23} = 12.38$ ) [16]. The internal torsion angles average to 8.5°. C(22) deviates significantly, by 0.23 Å, from the least-squares plane through the remaining ring atoms, as expected for an envelope conformation.

Stereodiagrams of the molecular packing for both structures are shown in Figures 4 and 5. In both the crystal structures of (1) and (2) the host molecules are arranged so as to provide infinite channels along [010] in which guest molecules are included. Each guest ketonic function is hydrogen bonded to the hydroxy group of the neighbouring host molecule such that the O(1)⋯O(20) distances are 2.923(6) Å and 2.844(4) Å and the O(1)—H(1)⋯O(20) angles are 163° and 164° in structures (1) and (2), respectively. Other non-bonded distances between host and guest molecules are greater than 3.5 Å in both structures and thus unremarkable.

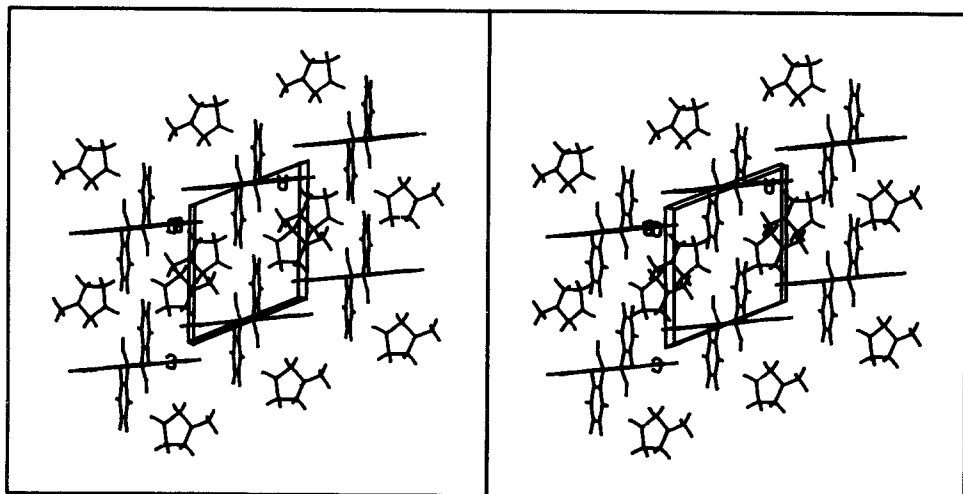


Fig. 5. A stereoview of the crystal structure of compound (2) viewed along [010] (*c* is vertical).

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### References

1. F. Toda, K. Tanaka, G. U. Dumas and M. C. Sanchez: *Chem. Lett.* 1521 (1983).
2. F. Toda, K. Tanaka, Y. Tagami and T. C. W. Mak: *Chem Lett.* 195 (1985).
3. F. Toda, K. Tanaka and T. C. W. Mak: *Bull. Chem. Soc. Jpn.* **58**, 2221 (1985).
4. K. Tanaka and F. Toda: *J. Chem. Soc. Jpn.* 932 (1986).
5. K. Tanaka, F. Toda and T. C. W. Mak: *J. Incl. Phenom.* **2**, 99 (1984).
6. J. M. Shin, F. Toda and M. S. Jhon: *J. Incl. Phenom.* **5**, 567 (1987).
7. F. Toda, K. Tanaka and T. C. W. Mak: *J. Incl. Phenom.* **13**, 225 (1985).
8. F. Toda: *Topics in Current Chemistry*, **140**, 43 (1987).
9. G. M. Sheldrick: SHELXS-86. *Crystallographic Computing 3* (Eds. G. M. Sheldrick, C. Kruger and R. Goddard.) Oxford University Press, 1985, p. 175.
10. G. M. Sheldrick: *The SHELX Program System*, Univ. Chem. Lab., Cambridge (1976).
11. P. Schuster, G. Zundel and C. Sandorfy (Eds.): *The Hydrogen Bond*, North-Holland Publ. Co., Vol. II, 1976, p. 411.
12. D. T. Cromer and J. B. Mann: *Acta Crystallogr.*, **A24**, 321 (1968).
13. R. F. Stewart, E. R. Davidson and W. T. Simpson: *J. Chem. Phys.* **42**, 3175 (1965).
14. M. Nardelli: *Comput. Chem.* **7**, 95 (1983).
15. W. D. S. Motherwell: *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge., England. Unpublished.
16. W. L. Duax and D. A. Norton (Eds.): *Atlas of Steroid Structure*, Vol. 1, Plenum Press, London, 1975.
17. F. Toda, K. Tanaka, S. Nagamatsu and T. C. W. Mak: *Isr. J. Chem.* **25**, 346 (1985).
18. F. H. Herbstein, M. Kapon and G. M. Reisner: *Acta Crystallogr.*, **B42**, 181 (1986).
19. Y. Tanimoto, H. Kobayashi, S. Nagakura and Y. Saito: *Acta Crystallogr.*, **B29**, 1822 (1973).