Complexation with Hydroxy Host Compounds, Part 5. Structures and Thermal Analyses of 1,2,3,4,5-Pentaphenylcyclopenta-2,4-dien-1-ol and Its Inclusion Compounds with Water and Dimethylsulphoxide

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Abstract. The crystal and molecular structures of the title host compound (**I**), $C_{35}H_{26}O$, and its inclusion complexes with water (**II**), $C_{35}H_{26}O \cdot H_2O$ and dimethylsulphoxide (**III**), $C_{35}H_{26}O \cdot C_2H_6OS$ are reported. (**I**) is monoclinic $C2/c$, $a = 9.725(1)$, $b = 20.031(3)$, $c = 26.545(3)$ Å, $\beta = 90.60(1)^0$, $V = 5170$ Å^3, $Z = 8$; (**II**) is triclinic, $P\bar{1}$, $a = 10.206(1)$, $b = 10.324(2)$, $c = 14.425(2)$ Å, $\alpha = 101.68(1)$, $\beta = 89.92(2)$, $\gamma = 116.34(1)^0$, $V = 1327$ Å^3, $Z = 2$; (**III**) is monoclinic $P2_1/n$, $a = 17.832(5)$, $b = 10.109(3)$, $c = 17.901(5)$ Å, $\beta = 111.93(2)^0$, $V = 2993$ Å^3, $Z = 4$. The structures were solved by direct methods and refined to final residuals of 0.045, 0.048, and 0.071, respectively. Thermal analysis of the complexes corroborates the crystallographic results.

Key words. Inclusion compound, hydroxy compound, crystal structure, thermal analysis.

1. Introduction

Hydroxy compounds have proved to be versatile hosts in the formation of inclusion compounds. Toda has reviewed these compounds and has emphasised their uses in the optical resolution of racemates and the separation of close isomers [1], as well as their role in entrapping various guest compounds in topologically-favourable positions and thus controlling the reactions of the latter [2]. We have studied the complexation of the host compound *trans*-9,10-dihydroxy-9,10-diphenyl-9,10-dihydroanthracene with a variety of ketones and substituted pyridines [3–5], and have elucidated the structures of inclusion compounds of a suberol-derived host with dioxane and acetone [6]. Recently we have studied the dynamics of enclathration of the host 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol with acetone, as well as the kinetics of the solid–solid reaction of this host with benzophenone [7].

We now present our investigation of the host compound 1,2,3,4,5-pentaphenylcyclopenta-2,4-dien-1-ol in its non-porous alpha phase, as well as the structures and thermal analyses of its inclusion compounds with water and dimethyl sulphoxide.

2. Experimental

2.1. PREPARATION OF COMPLEXES (I), (II) AND (III)

The host compound (I) was prepared by a modification of an established procedure
[8]. 2,3,4,5-Tetraphenylcyclopenta-2,4-dien-1-one (2.00 g, 5.2 mmol), dissolved
in dry benzene (10 mL) was added dropwise and with vigorous stirring to a solution
of phenylmagnesium bromide which was cooled in an ice bath. The latter solution
was prepared from bromobenzene (7.2 g, 46 mmol) and magnesium (1.10 g,
45 mmol) in dry ether (25 mL). The rate of addition was determined by the
disappearance of the red colour of the added solution of the ketone. After
the addition, the mixture was heated under reflux for 30 min, cooled to room
temperature, and a 5% aqueous solution of ammonium chloride was added
dropwise until the pH of the aqueous phase was approximately 8 (*ca.* 15 mL).
The organic layer was separated, dried over magnesium sulphate and *ca.* 90%
of the solvent was removed under reduced pressure. Petroleum ether (b.p. 100–
120°C, 30 mL) was added to the residue and the mixture was allowed to crystallize
in a refrigerator. The crystalline product was washed with cold petroleum ether,
yielding pale yellow crystals (1.9 g, 81%). The product could be recrystallized
from petroleum ether, m.p. 177.8–178.2°C, or glacial acetic acid, m.p. 177.5–
178°C.

The inclusion complex with water was obtained in a variety of ways: dissolving
the host in wet methylethylketone or benzene or acetonitrile followed by slow
evaporation; while the DMSO complex crystallized from a concentrated solution of
the host in DMSO and subsequent slow evaporation.

2.2. X-RAY ANALYSIS

In each case, a suitable single crystal was selected, mounted in a Lindemann tube
with mother liquor and irradiated with Mo K_{α} ($\lambda = 0.7107 \text{ Å}$) radiation on an
Enraf–Nonius CAD4 diffractometer. Accurate cell parameters were obtained by
least-squares refinement on the setting angles of 24 reflections ($16 < \theta < 17°$). Data
were collected with variable scan width and scan speed with a maximum recording
time of 40 s. Three reference reflections were periodically monitored for intensity
and orientation control. The data were corrected for Lorentz-polarization effects
but not for absorption. The crystal data and parameters for data collection are
reported in Table I.

2.3. STRUCTURE SOLUTION AND REFINEMENT

All three structures were solved by direct methods using SHELXS-86 [9] and
refined using SHELX-76 [10]. For (I) and (III) the structure solutions were
obtained in the space groups in which they were ultimately refined. For (II) the
solution was only obtained in $P1$; inspection of the result showed that the two
host–guest complexes in the cell were related by a centre of symmetry and hence
the subsequent refinement was carried out in $P\bar{1}$. Compound (I) has a single host
molecule in the asymmetric unit; (II) and (III) each have a single host–guest
complex in the asymmetric unit with a host–guest ratio of 1 : 1.

Table I. Crystal data, details of the data collection and final refinement of **I**, **II** and **III**.

	I	**II**	**III**
Molecular formula	$C_{35}H_{26}O$	$C_{35}H_{26}O \cdot H_2O$	$C_{35}H_{26}O \cdot C_2H_6OS$
M_r	462.59	480.61	540.73
Space group	$C2/c$	$P\bar{1}$	$P2_1/n$
a (Å)	9.725(1)	10.206(1)	17.832(5)
b (Å)	20.031(3)	10.324(2)	10.109(3)
c (Å)	26.545(3)	14.425(2)	17.901(5)
α (°)	90	101.68(1)	90
β (°)	90.60(1)	89.92(2)	111.93(2)
γ (°)	90	116.34(1)	90
V (Å^3)	5170	1327	2993
Z	8	2	4
D_c (g mL^{-3})	1.19	1.20	1.20
$F(000)$	1952	508	1144
μ (Mo K_α)	0.36	0.39	1.02
Dim. of crystal (mm)	$0.25 \times 0.41 \times 0.50$	$0.31 \times 0.34 \times 0.41$	$0.25 \times 0.34 \times 0.41$
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$	$\omega - 2\theta$
Scan width	$.8 + .35 \tan \theta$	$.8 + .35 \tan \theta$	$.8 + .35 \tan \theta$
Aperture width	$1.12 + 1.05 \tan \theta$	$1.12 + 1.05 \tan \theta$	$1.12 + 1.05 \tan \theta$
θ range scanned	1–25	1–25	1–25
$h\,k\,l$ range	± 11; 0, 23:0, 31	± 12; ± 12; 0, 17	± 19; 0, 11; 0, 19
Tot. reflns. collected	3785	4011	2908
Reflns obs $(I_{rel} > 2\sigma I_{rel})$	2870	3215	1446
No. of parameters	329	344	367
$R = F_0 - F_c/F_0$	0.045	0.048	0.071
R_W	0.043	0.047	0.071
w	$(\sigma^2 F)^{-1}$	$(\sigma^2 F)^{-1}$	$(\sigma^2 F)^{-1}$
S	2.44	2.03	2.42

In the final refinements of all three structures the non-hydrogen atoms of the host molecule were treated anisotropically; phenyl hydrogens were placed in calculated positions and modelled isotropically with a single temperature factor for all. The hydroxyl hydrogen was located in a difference map and constrained to ride at 1.00(2) Å from the oxygen atom. For (**II**), modelling of the guest water was straightforward; the oxygen was treated anisotropically and the hydrogens located in a difference map were constrained to ride at 1.00(2) Å from the guest oxygen with a single temperature factor linked to that of the host hydrogens. For (**III**), the DMSO molecule was found to be disordered and was modelled with variable site occupancy factors on the sulphur atom, as shown in Figure 1. A similar type of disorder has been observed previously [11]. Complex neutral atom scattering factors were taken from Cromer and Mann [12] for non-hydrogen atoms and from Stewart, Davidson and Simpson [13] for H, with dispersion corrections from Cromer and Liberman [14]. Molecular parameters were calculated using PARST [15] and drawings obtained using PLUTO [16]. All computations were performed at the Computer Centre of the University of Cape Town on a VAX computer. Further details of the data collections, structure solutions and refinements are given in Table I.
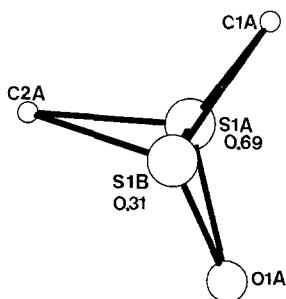
Fig. 1. A depiction of the disordered model of DMSO employed in structure (**III**), showing site occupancies.

2.4. THERMAL ANALYSIS

Differential scanning calorimetry (DSC) and thermogravimetry (TG) were performed on a Perkin Elmer PC7 Series system. Crystals were removed from their mother liquor, blotted dry on filter paper and crushed before analysis. Sample weights varied between 2 and 4 mg. The temperature ranges were from 30–200°C at heating rates of 10°C min^{-1} for (**II**) and 5°C min^{-1} for (**III**). The purge gas was dry nitrogen at a flow rate of 40 mL min^{-1}.

3. Results and Discussion

Final fractional atomic coordinates are reported in Table II. A perspective view of (**I**) with atomic labelling is shown in Figure 2. An examination of molecular parameters shows that standard deviations for (**I**) and (**II**) are much lower than those of (**III**); this is clearly a consequence of crystal quality and the disorder of the DMSO in (**III**). Therefore structures (**I**) and (**II**) are discussed separately from (**III**).

The bond lengths of the central ring for (**I**) and (**II**) are similar. The double bonds C(2)—C(3) and C(4)—C(5) are in the range 1.344(3) to 1.347(4) Å. C(3)—C(4) is 1.486(4) and 1.481(4) Å in (**I**) and (**II**), respectively, and C(1)—C(5) and C(4)—C(5) are between 1.518 (3) and 1.543(5) Å. The C(sp^2)—C(ar) bonds from the central ring to the phenyl groups are in the range 1.466(3) to 1.486(3) Å. All these values compare well with those tabulated by Allen *et al.* [18]. The bond lengths in (**III**) are not as well defined, but the double bonds in the central ring have lengths of 1.34(2) and 1.34(2) Å and can be differentiated from the single bonds, which have values of 1.47(1) Å for C(3)—C(4), 1.54(1) Å for C(1)—C(2) and 1.53(1) Å for C(1)—C(5).

The conformation of the host molecules in these three structures can be described in terms of the torsion angles formed between the phenyl rings and the central cyclopentadiene ring. These are reported in Table III. The conformation of the four phenyl rings attached to the C(sp^2) atoms have generally similar torsion angles, but we note a distinct difference in the torsion angle of the phenyl ring bonded to C(1). This has a value of +62.5° in (**I**) but −54.8° and −51.0° in structures (**II**) and (**III**), respectively.

Table II. Fractional atomic coordinates ($\times 10^4$) and thermal parameters ($\text{Å}^2 \times 10^3$) with e.s.d.s in parentheses for (**I**).

Atom	x/a	y/b	z/c	U_{equiv}
O(1)	−87(2)	775(1)	1919(1)	54(1)
C(1)	−1082(2)	1084(1)	1599(1)	41(1)
C(2)	−1770(2)	1674(1)	1861(1)	41(1)
C(3)	−1646(2)	2220(1)	1571(1)	42(1)
C(4)	−833(2)	2058(1)	1116(1)	43(1)
C(5)	−471(2)	1409(1)	1127(1)	32(1)
C(11)	−2116(2)	546(1)	1436(1)	41(1)
C(12)	−3277(3)	712(1)	1163(1)	50(1)
C(13)	−4211(3)	228(1)	1015(1)	61(1)
C(14)	−3993(3)	−427(2)	1131(1)	70(1)
C(15)	−2852(4)	−596(1)	1399(1)	87(2)
C(16)	−1897(3)	−114(1)	1555(1)	72(1)
C(21)	−2326(2)	1632(1)	2376(1)	42(1)
C(22)	−3050(3)	1079(1)	2544(1)	57(1)
C(23)	−3567(3)	1059(2)	3028(1)	69(1)
C(24)	−3365(3)	1584(2)	3352(1)	68(1)
C(25)	−2622(3)	2127(2)	3195(1)	69(1)
C(26)	−2094(3)	2149(1)	2713(1)	58(1)
C(31)	−2201(3)	2892(1)	1690(1)	48(1)
C(32)	−1319(3)	3425(1)	1762(1)	68(1)
C(33)	−1840(4)	4043(1)	1905(1)	90(1)
C(34)	−3235(4)	4124(2)	1969(1)	92(2)
C(35)	−4103(4)	3596(2)	1893(1)	89(2)
C(36)	−3594(3)	2980(1)	1754(1)	65(1)
C(41)	−487(3)	2561(1)	731(1)	45(1)
C(42)	866(3)	2647(1)	583(1)	52(1)
C(43)	1203(3)	3112(1)	220(1)	66(1)
C(44)	197(4)	3492(1)	0(1)	75(1)
C(45)	−1141(3)	3419(1)	142(1)	73(1)
C(46)	−1491(3)	2958(1)	506(1)	59(1)
C(51)	280(2)	1021(1)	745(1)	48(1)
C(52)	115(3)	1159(1)	234(1)	61(1)
C(53)	845(4)	805(2)	−122(1)	79(2)
C(54)	1735(4)	314(2)	24(2)	89(2)
C(55)	1900(3)	160(2)	523(2)	82(2)
C(56)	1162(3)	509(1)	886(1)	62(1)
O(1)	4097(2)	8570(2)	5762(1)	51(1)
C(1)	4191(3)	8528(3)	6747(2)	42(1)
C(2)	2930(3)	8708(3)	7253(2)	41(1)
C(3)	3531(3)	9931(3)	7979(2)	41(1)
C(4)	5153(3)	10635(3)	7999(2)	41(1)
C(5)	5551(3)	9861(3)	7289(2)	43(1)
C(11)	4125(3)	7043(3)	6802(2)	43(1)
C(12)	3940(3)	5971(3)	5996(2)	52(1)
C(13)	3873(3)	4626(3)	6068(2)	64(2)
C(14)	3993(4)	4335(3)	6937(3)	71(2)
C(15)	4182(4)	5402(4)	7747(2)	79(2)
C(16)	4240(4)	6742(3)	7684(2)	64(2)
C(21)	1382(3)	7623(3)	7001(2)	45(1)
C(22)	768(3)	7024(3)	6064(2)	59(2)
C(23)	−685(4)	6042(4)	5843(2)	73(2)
C(24)	−1597(4)	5612(4)	6537(3)	75(2)

Table II. (continued.)

Atom	x/a	y/b	z/c	U_{equiv}
C(25)	−1007(4)	6167(4)	7464(3)	89(2)
C(26)	447(3)	7156(3)	7698(2)	71(2)
C(31)	2732(3)	10516(3)	8653(2)	44(1)
C(32)	3055(3)	10798(3)	9635(2)	52(1)
C(33)	2303(4)	11365(3)	10249(2)	64(1)
C(34)	1251(4)	11673(3)	9899(3)	73(2)
C(35)	932(3)	11421(3)	8933(3)	70(2)
C(36)	1679(3)	10843(3)	8318(2)	55(1)
C(41)	6128(3)	12003(3)	8704(2)	41(1)
C(42)	5827(3)	13211(3)	8897(2)	51(2)
C(43)	6713(3)	14464(3)	9586(2)	62(2)
C(44)	7859(3)	14513(3)	10087(2)	67(2)
C(45)	8182(3)	13333(3)	9892(2)	65(2)
C(46)	7321(3)	12081(3)	9203(2)	51(1)
C(51)	7022(3)	10292(3)	6948(2)	45(1)
C(52)	7855(3)	11726(3)	6840(2)	60(2)
C(53)	9214(4)	12153(4)	6492(2)	74(2)
C(54)	9744(4)	11144(4)	6230(2)	76(2)
C(55)	8945(3)	9715(4)	6327(2)	70(2)
C(56)	7583(3)	9274(3)	6685(2)	56(2)
O(11)	3725(3)	10953(3)	5517(2)	77(2)
O(1)	7955(5)	1198(8)	9032(8)	68(4)
C(1)	7410(6)	432(11)	9271(6)	49(5)
C(2)	7776(6)	153(11)	10184(6)	48(5)
C(3)	7295(6)	741(10)	10524(6)	55(5)
C(4)	6603(6)	1398(10)	9910(6)	51(5)
C(5)	6659(6)	1251(10)	9187(6)	47(5)
C(11)	7210(7)	−825(11)	8791(6)	54(5)
C(12)	7576(7)	−1131(12)	8258(7)	76(7)
C(13)	7358(9)	−2261(15)	7790(8)	96(9)
C(14)	6786(9)	−3071(16)	7838(8)	98(9)
C(15)	6429(8)	−2773(14)	8364(8)	96(8)
C(16)	6642(8)	−1676(13)	8853(7)	81(7)
C(21)	8475(7)	−688(11)	10582(6)	54(5)
C(22)	9049(7)	−809(12)	10233(7)	72(6)
C(23)	9718(8)	−1592(14)	10590(9)	90(8)
C(24)	9832(8)	−2287(13)	11279(9)	93(8)
C(25)	9259(9)	−2174(13)	11616(7)	86(7)
C(26)	8596(7)	−1393(12)	11272(7)	68(6)
C(31)	7431(7)	848(11)	11400(6)	57(5)
C(32)	6891(7)	394(11)	11720(7)	69(6)
C(33)	7067(10)	510(14)	12543(8)	99(8)
C(34)	7774(10)	1121(14)	13017(7)	93(8)
C(35)	8314(7)	1619(13)	12709(8)	84(7)
C(36)	8133(7)	1460(11)	11900(7)	72(6)
C(41)	5957(6)	2088(10)	10080(6)	50(5)
C(42)	5153(7)	1747(11)	9674(6)	62(6)
C(43)	4532(7)	2370(13)	9812(7)	73(7)
C(44)	4701(9)	3389(14)	10355(9)	85(8)
C(45)	5508(9)	3709(12)	10799(7)	79(7)
C(46)	6121(7)	3085(12)	10647(7)	67(6)
C(51)	6148(6)	1852(13)	8482(7)	59(6)
C(52)	5951(8)	3177(14)	8370(7)	86(7)

Table II. (continued.)

Atom	x/a	y/b	z/c	U_{equiv}
C(53)	5486(8)	3749(16)	7659(10)	111(9)
C(54)	5183(9)	3060(21)	6976(9)	115(10)
C(55)	5369(10)	1782(22)	7002(8)	155(11)
C(56)	5843(8)	1182(16)	7713(7)	115(8)
S(1A)	8773(4)	4572(6)	10075(4)	117(4)
S(1B)	8092(8)	4398(12)	9496(9)	89(6)
O(1A)	8701(6)	3181(11)	10053(6)	130(4)
C(1A)	8655(15)	5173(25)	9200(13)	242(11)
C(2A)	7932(12)	5237(21)	10161(12)	191(9)
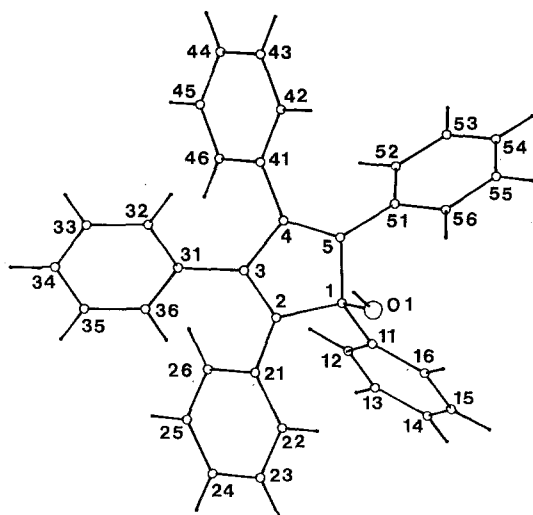
Fig. 2. A perspective view of (I) with atomic labelling.

As with most molecules capable of intermolecular hydrogen bonding and indeed with all inclusion complexes, the packing of the molecules is interesting and worthy of detailed analysis.

For (I), illustrated in the [010] projection in Figure 3, hydrogen bonding possibilities are apparent for the hydroxyl groups of adjacent host molecules. The

Table III. Torsion angles defining host conformation.

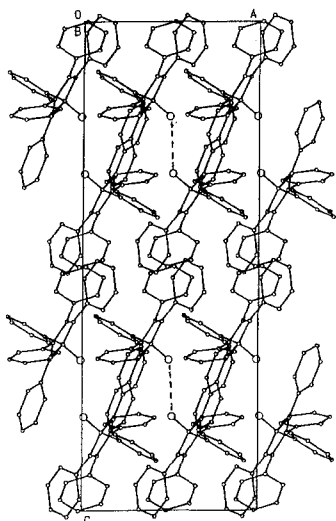|	I	II	III
O(1)—C(1)—C(11)—C(1X)	7.1	2.5	4.0
C(1)—C(2)—C(21)—C(2X)	41.1	45.1	28.3
C(2)—C(3)—C(31)—C(3X)	60.0	54.0	57.9
C(3)—C(4)—C(41)—C(4X)	52.6	47.2	54.3
C(4)—C(5)—C(51)—C(5X)	33.5	43.1	46.1
C(5)—C(1)—C(11)—C(1X)	62.5	−54.8	−51.0

Fig. 3. The [010] projection of the unit cell to illustrate the packing in (I).
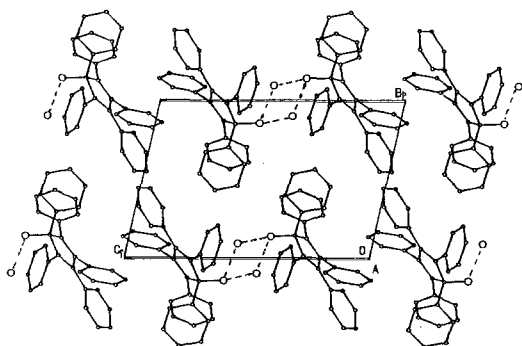

Fig. 4. A projection of the unit cell to illustrate the packing in (II).

intermolecular O···O distance, however, is 3.089 Å (symmetry operation
$-x, y, -z + 0.5$); this relatively long O···O distance indicates that the H-bonding
is weak.

To illustrate the packing in structure (II) a projection of the cell very close to
[100] is presented in Figure 4. The O···O close contacts between the host and water
molecules are at 2.824 Å (via $-x + 1, -y + 2, -z + 1$); the resulting cyclic pattern
of hydrogen bonding involves the host hydroxyl hydrogen and only one of the
water hydrogens; hence two host and two guest molecules are associated with each
other.

The [001] projection of (III) (Figure 5) shows the DMSO molecules hydrogen-
bonded to the hydroxyl moieties of the host with an O···O distance of 2.70(1) Å.
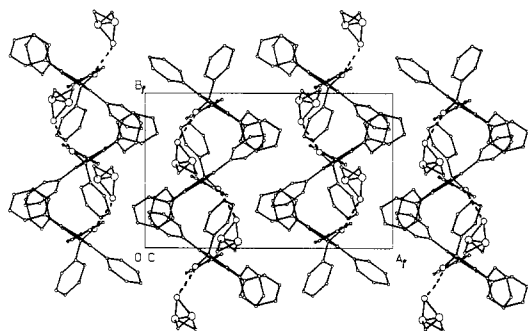The structure is hence built up of isolated host–guest pairs.

Fig. 5. The [001] projection of structure (**III**).

The TG experiments yielded a weight loss of 3.98% for the monohydrate, compound (**II**), which is close to the required value of 3.75%. For (**III**) the measured weight loss was 14.69%, while the calculated value is 14.45%. The excellent agreement between observed and calculated values confirms the chosen host : guest ratios of 1 : 1 in both compounds. The DSC of (**II**) was informative, yielding only one sharp endotherm with an onset at 178°C, corresponding to the melting point of the host compound. The DSC of (**III**), however, shows a single skewed endotherm, with an onset temperature of 115°C, and peaking at 139°C, and is shown in Figure 6. This corresponds to the release of the guest with a
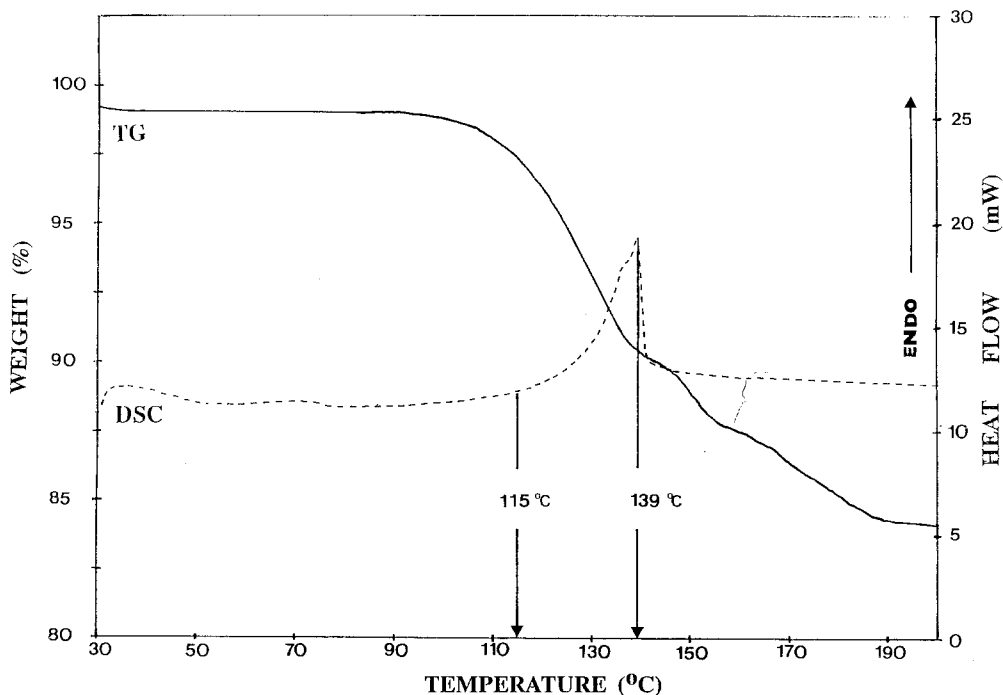

Fig. 6. The DSC and TG of compound (**III**).

concomitant dissolution of the host compound. We have observed this phenomenon in the decomposition of the inclusion compound of 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol with dioxane [17].

Further work involving this 2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ol host molecule with a variety of alcohols as potential guests is in progress.

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