

GUSTAFSSON, T., LUNDGREN, J.-O. & OLOVSSON, I. (1977). *Acta Cryst.* **B33**, 2373–2376.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
 JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
 LALANCETTE, R. A., CEFOLA, M., HAMILTON, W. C. & LAPLACA, S. J. (1967). *Inorg. Chem.* **6**, 2127–2134.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
 TAESLER, I. & OLOVSSON, I. (1969). *J. Chem. Phys.* **51**, 4213–4219.

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The Structure of 4-Hydroxy-2,3,4-triphenyl-2-cyclopenten-1-one Benzene Solvate, $C_{23}H_{18}O_2 \cdot \frac{1}{2}C_6H_6$

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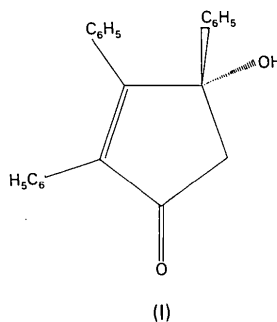
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Abstract. $M_r = 365.14$, triclinic, $P\bar{1}$, $a = 6.068$ (1), $b = 13.110$ (1), $c = 13.527$ (1) Å, $\alpha = 110.63$ (1), $\beta = 100.41$ (2), $\gamma = 82.62$ (2)°, $V = 988.09$ Å³, $Z = 2$, $D_m = 1.21$, $D_x = 1.23$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 5.53$ cm⁻¹, $F(000) = 386$. The structure was solved by direct methods and refined to a final R value of 0.075 for 2558 reflections collected with Cu $K\alpha$ radiation at 295 K. The five-membered ring is puckered in a half-chair conformation. The benzene molecule lies on the centre of symmetry.

Introduction. The title compound (I) belongs to the class of hydroxycyclopentenones which are important in the preparation of cyclopentadienes and related compounds (Freeman, Gagan & Lloyd, 1973). Moreover, the dehydration of compound (I) gives various interesting cyclopentadienone dimers (Conner, Dunston, Le Goff & Yates, 1971), which contain this molecule as the basic skeleton. The structural analysis was undertaken to study the effect of the substituents on the conformation of the five-membered ring and to compare it with the conformation in the dimers.

Experimental. Compound prepared by condensation of benzil with phenylacetone in the presence of alcoholic KOH (Koelsch & Geissman, 1939) and characterized by IR and NMR. Single crystals obtained by slow evaporation of the solution of the compound in benzene–petrol. Density measured by flotation in aqueous KI solution. Needle-shaped crystal, $0.15 \times 0.125 \times 0.45$ mm elongated along **a**, CAD-4 diffractometer; cell parameters based on 15 centred reflections within the θ range 10–40°. Intensity data collected for



4252 reflections, $\omega/2\theta$ scan, $0 \leq h \leq 7$, $-15 \leq k \leq 15$, $-16 \leq l \leq 16$, $(\sin \theta/\lambda)_{\max} = 0.61$ Å⁻¹. Variation in two standard reflections (131 and 111) < 15%; reflections scaled by the use of standards. 3189 unique reflections ($R_{\text{int}} = 0.024$), 631 of which were considered unobserved [$I < 3\sigma(I)$]; Lp correction but not absorption. Direct methods, refinement by full-matrix least squares on F . H positions from a difference map but not refined (their temperature factors fixed at $U = 0.063$ Å²). 253 parameters refined. Final $R = 0.093$ for all data, $wR = 0.097$, $S = 3.27$; R for 2558 observed reflections is 0.075. $w = 2.974/[\sigma^2(F) + 0.00018F^2]$ where $\sigma(F)$ was derived from counting statistics. In the final cycle of refinement $(\Delta/\sigma)_{\max} = 0.13$, maximum and minimum $\Delta\rho$ excursions 0.10 and -0.15 e Å⁻³ respectively. Scattering factors from *International Tables for X-ray Crystallography* (1974). *MULTAN* (Germain, Main & Woolfson, 1971) used for structure determination, *SHELX 76* (Sheldrick, 1976) for refinement and *ORTEP* (Johnson, 1976) for diagrams.

Discussion. The fractional coordinates of the non-hydrogen atoms are given in Table 1.*

* List of structure factors, anisotropic thermal parameters, phenyl-ring dimensions, H-atom coordinates, distances and angles involving H atoms and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38844 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{\AA}^2 \times 10^2$) *for the non-H atoms, with e.s.d.'s in parentheses*

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C(1)	6704 (6)	4257 (3)	9250 (2)	4.21
C(2)	5711 (5)	4505 (2)	8270 (2)	4.05
C(3)	4567 (5)	3668 (2)	7602 (2)	4.05
C(4)	4449 (6)	2793 (2)	8115 (2)	4.55
C(5)	6327 (7)	3113 (3)	9091 (2)	5.39
O(1)	7686 (4)	4894 (2)	10067 (2)	5.15
O(2)	2288 (5)	2910 (2)	8433 (2)	5.95
C(11)	5914 (6)	5587 (2)	8186 (2)	4.15
C(12)	3988 (6)	6153 (3)	7825 (3)	4.81
C(13)	4150 (7)	7180 (3)	7792 (3)	5.83
C(14)	6156 (8)	7661 (3)	8102 (3)	6.27
C(15)	8037 (7)	7120 (3)	8478 (3)	5.99
C(16)	7905 (6)	6076 (3)	8509 (3)	5.13
C(21)	3359 (6)	3552 (2)	6516 (2)	4.31
C(22)	1285 (7)	3145 (3)	6159 (3)	6.22
C(23)	192 (8)	3066 (4)	5132 (3)	7.88
C(24)	1186 (8)	3412 (4)	4471 (4)	7.32
C(25)	3306 (9)	3770 (3)	4814 (3)	7.09
C(26)	4403 (7)	3869 (3)	5833 (3)	5.25
C(31)	4863 (7)	1625 (3)	7378 (3)	5.04
C(32)	6723 (8)	1343 (3)	6846 (3)	6.42
C(33)	7200 (8)	299 (3)	6220 (3)	7.78
C(34)	5834 (11)	-505 (3)	6107 (3)	8.58
C(35)	3956 (10)	-248 (3)	6624 (4)	8.42
C(36)	3481 (8)	829 (3)	7261 (3)	6.33
†C(S1)	9304 (11)	289 (4)	9118 (3)	8.47
†C(S2)	7869 (8)	469 (3)	9836 (4)	7.93
†C(S3)	8542 (10)	174 (4)	10714 (4)	8.35

† Atoms of the benzene molecule.

Table 2. *Selected bond lengths* (\AA), *bond angles* ($^\circ$) *and torsion angles* ($^\circ$) *with e.s.d.'s in parentheses*

C(1)–C(2)	1.480 (4)	C(3)–C(4)	1.550 (4)
C(1)–C(5)	1.480 (6)	C(3)–C(21)	1.483 (4)
C(1)–O(1)	1.230 (4)	C(4)–C(5)	1.551 (5)
C(2)–C(3)	1.332 (4)	C(4)–O(2)	1.426 (5)
C(2)–C(11)	1.485 (4)	C(4)–C(31)	1.522 (5)
C(2)–C(1)–C(5)	109.0 (3)	C(4)–C(3)–C(21)	120.5 (3)
C(2)–C(1)–O(1)	125.8 (3)	C(3)–C(4)–C(5)	101.7 (2)
C(5)–C(1)–O(1)	125.2 (3)	C(3)–C(4)–O(2)	108.9 (2)
C(1)–C(2)–C(3)	109.0 (3)	C(3)–C(4)–C(31)	113.8 (3)
C(1)–C(2)–C(11)	120.8 (3)	C(5)–C(4)–O(2)	111.8 (3)
C(3)–C(2)–C(11)	130.0 (3)	C(5)–C(4)–C(31)	111.8 (3)
C(2)–C(3)–C(4)	112.0 (3)	O(2)–C(4)–C(31)	108.8 (3)
C(2)–C(3)–C(21)	127.4 (3)	C(1)–C(5)–C(4)	104.6 (3)
C(5)–C(1)–C(2)–C(3)	+7.2 (4)	C(3)–C(4)–C(5)–C(1)	+18.9 (3)
C(1)–C(2)–C(3)–C(4)	+6.1 (3)	C(4)–C(5)–C(1)–C(2)	-16.8 (4)
C(2)–C(3)–C(4)–C(5)	-16.0 (3)		

The five-membered ring assumes a half-chair conformation, which is confirmed by the deviations of C(4) and C(5) from the plane containing C(1), C(2) and C(3) by -0.153 (3) and 0.175 (4) \AA respectively. The conformation can also be described in terms of pseudo-rotation parameters (Altona, Geise & Romers, 1968), $\Delta = 3.74^\circ$, $\varphi_m = 18.9^\circ$ and the asymmetry parameters $\Delta C_{2(4-5)} = 0.96^\circ$ (Duax, Weeks & Rohrer, 1976). The torsion angles are given in Table 2.

The molecular structure (view down **a**) with atomic numbering is shown in Fig. 1. The bond lengths and bond angles of the five-membered ring (Table 2) are normal (Harlow & Simonsen, 1977). The peripheral phenyl rings are planar with average C–C bond lengths 1.381 (e.s.d.'s 0.005 to 0.009) \AA and C–C–C bond angles 120.1 (e.s.d.'s 0.3 to 0.5) $^\circ$. The molecules are arranged in rows with molecular planes approximately perpendicular to **a** (Fig. 2). The orientation of the phenyl rings is apparently determined by van der Waals contacts, which are normal. A short contact of 2.885 (4) \AA observed between the ketonic oxygen and the hydroxyl oxygen of the symmetry-related molecule is a possible hydrogen bond with the following parameters $O \cdots O = 2.885$ (4) \AA , $O-H = 1.167$ \AA , $O \cdots H = 1.897$ \AA and $\angle O-H \cdots O = 139^\circ$. The

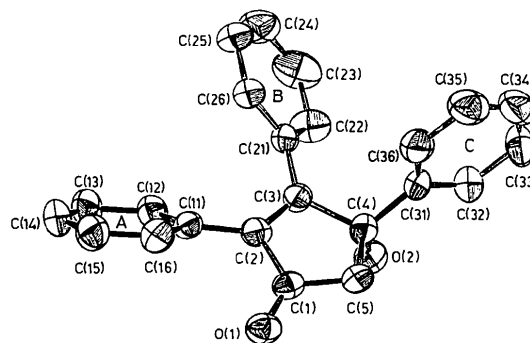


Fig. 1. Molecular structure (view down **a**).

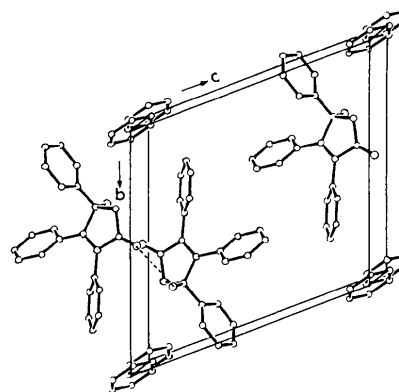


Fig. 2. Molecular packing (view down a^* axis). The hydrogen bond is shown by a broken line.

dihedral angles between the plane through C(1), C(2), C(3) of the five-membered ring and the phenyl rings *A*, *B* and *C* are 44.7 (4), 43.8 (5), 98.4 (5)° respectively. The benzene molecules lie on centres of symmetry and stack in channels parallel to *a*.

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References

ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–29.
 CONNER, M. K., DUNSTON, J. M., LE GOFF, E. & YATES, P. (1971). *Tetrahedron*, **27**, 1821–1831.

DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. ALLINGER & E. L. ELIEL, pp. 279–286. New York: John Wiley.
 FREEMAN, B. H., GAGAN, J. M. F. & LLOYD, D. (1973). *Tetrahedron*, **29**, 6307–6312.
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
 HARLOW, R. L. & SIMONSEN, S. H. (1977). *Cryst. Struct. Commun.* **6**, 695–698.
International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.
 JOHNSON, C. K. (1976). *ORTEP-II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
 KOELSCH, C. F. & GEISSMAN, T. A. (1939). *J. Org. Chem.* **3**, 484–485.
 SHELDRICK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.

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A 1:2 Host–Guest Complex Between 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6) and *N,N'*-Diformohydrazide, C₁₂H₂₄O₆·2C₂H₄N₂O₂

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Abstract. $M_r = 440.5$, monoclinic, $P2_1/n$, $a = 14.176$ (2), $b = 9.968$ (2), $c = 8.121$ (1) Å, $\beta = 103.34$ (1)°, $V = 1116.6$ (3) Å³, $Z = 2$, $D_x = 1.310$, $D_m = 1.29$ (1) Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.94$ mm⁻¹, $F(000) = 472$, $T = 293$ K. Final $R = 0.044$ for 1218 unique observed reflections. The 18-crown-6 molecule lies on an inversion center and its conformation deviates only slightly from D_{3d} symmetry. The conformation of the guest *N,N'*-diformohydrazide approximates C_2 symmetry with a torsion angle of 105.4 (4)° around the N–N bond. Two inversion-related crown ether O atoms are each hydrogen-bonded to an N–H group of the guest molecule. Guest molecules are linked by two identical N–H...O=C hydrogen bonds which are related by another inversion center.

Introduction. Although complexes between uncharged organic molecules play a fundamental role in biochemical processes, it is an area that remains relatively

unexplored. Many data have been gathered on interactions involving hydrogen bonding; however, it is still difficult to suggest specific hosts (receptors) for the complexation of neutral (uncharged) guest molecules. The importance of 'acidic' methyl and methylene groups in stabilizing complexes between neutral molecules has not been appreciated (Goldberg, 1975; Bandy, Truter & Vögtle, 1981; Kaufmann, Knochel, Kopf, Oehler & Rudolph, 1977; Watson, Galloy, Grossie, Vögtle & Müller, 1983). While such interactions are weak, they lead to well-ordered crystalline solids and may be of importance in orienting and directing substrate molecules at active sites. When the guest molecule contains only one hydrogen capable of interacting with an 18-crown-6 host, usually a molecule of water is incorporated to form a ternary complex (Grossie, Watson, Vögtle & Müller, 1982; Watson *et al.*, 1983) or a complex of unusual stoichiometry is formed. In order to provide additional data for the development of theories for predicting structures of hosts for specific guests, we have examined the title compound.

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