the high melting point (531–532 K) and very low solubility of the crystalline material in organic solvents. The crystal structure of the related compound, racemic 3,6-dimethyl-1,4-dioxane-2,5-dione, has been reported (van Hummel, Harkema, Kohn & Feijen, 1982) and it also has close C—H$\cdots$O contacts [H$\cdots$O 2·33 (2) Å]. The occurrence and the geometry of C—H$\cdots X$-type H bonds, where X is O, N or Cl, has been discussed by Taylor & Kennard (1982).

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Structure of a Dimer Ketone Formed *via* Iron Carbonyl-Promoted Coupling of 7-Phenylnorbornadiene with Carbon Monoxide

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Abstract. *trans-exo*-10,11-Diphenyl-1,4,4a,4b,5,8,8a,9a-octahydro-1,4:5,8-dimethanofluoren-9-one, $C_{27}H_{24}O$, M_r = 364·49, triclinic, $P\bar{1}$, a = 9·673 (2), b = 9·748 (2), c = 12·522 (3) Å, α = 91·80 (2), β = 107·12 (2), γ = 118·23 (1)°, V = 973·6 (3) Å^3, Z = 2, D_x = 1·243 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·68 cm^{-1}, F(000) = 388, T = 295 K, R = 0·0642 for 1666 reflections. The two norbornene rings are *trans*-fused to a cyclopentanone ring. The norbornene bonds adjacent to the carbonyl average 1·577 (2) Å while those attached opposite the carbonyl average 1·558 (2) Å. The sp^2 C atoms of the norbornene moieties are pyramidalized with the two double bonds exhibiting folding angles of about 10 (1)°.

Experimental. A small, colorless crystal of dimensions 0·07 × 0·15 × 0·60 mm was mounted on a Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer; data collected in the ω mode ($3 \leq 2\theta \leq 45°$) using a variable scan rate (4 to 29·3° min^{-1}) and graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($25{\cdot}46 \leq 2\theta \leq 36{\cdot}40°$); no systematic absences and intensity statistics consistent with space group $P\bar{1}$; monitored reflections (021 and $2\bar{1}\bar{2}$) showed a random maximum 2% variation during data collection; $-10 \leq h \leq 9$; $-10 \leq k \leq 10$; $0 \leq l \leq 13$; 3540 reflections measured, 2576 independent ($R_{\rm merge}$ = 0·008), 1666 with $I \geq 3\sigma(I)$; Lorentz–polarization corrections, ψ-scan-based empirical absorption correction (transmission factors 0·895 to 0·956); structure solved by direct methods, H atoms located in difference maps and refined with isotropic thermal parameters; final R = 0·0642, wR = 0·0551 for 349 parameters (R = 0·0885, wR = 0·0673 for all 2576 observed reflections), S = 1·298, $(\Delta/\sigma)_{\rm max}$ = 0·021, largest peaks in the final difference map of −0·27

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Table 1. *Atomic coordinates* (× 10^4) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	*U*
C(1)	3313 (5)	4046 (4)	3080 (3)	39 (2)*
C(2)	4094 (5)	4260 (5)	2292 (4)	50 (2)
C(3)	5127 (5)	3645 (6)	2298 (4)	60 (3)
C(4)	5396 (6)	2803 (5)	3130 (4)	61 (3)
C(5)	4657 (6)	2591 (5)	3929 (4)	61 (3)
C(6)	3627 (5)	3224 (5)	3925 (4)	53 (3)
C(1*A*)	−2695 (5)	7920 (4)	1913 (4)	42 (2)
C(2*A*)	−2969 (5)	8459 (5)	2821 (4)	51 (3)
C(3*A*)	−4170 (6)	8907 (5)	2664 (4)	59 (3)
C(4*A*)	−5090 (6)	8832 (5)	1582 (5)	63 (3)
C(5*A*)	−4877 (6)	8289 (6)	656 (4)	64 (3)
C(6*A*)	−3650 (5)	7841 (5)	823 (4)	56 (3)
C(7)	−422 (5)	2376 (5)	2299 (4)	47 (2)
C(8)	−22 (5)	2631 (5)	3395 (4)	49 (3)
C(9)	1241 (5)	4376 (4)	3854 (3)	43 (2)
C(10)	219 (5)	5255 (4)	3461 (3)	41 (2)
C(11)	1295 (5)	7047 (4)	3851 (3)	40 (2)
O(11)	1969 (4)	7733 (3)	4838 (2)	60 (2)
C(12)	1406 (4)	7853 (4)	2856 (3)	39 (2)
C(13)	460 (5)	8818 (4)	2701 (3)	41 (2)
C(14)	743 (5)	9585 (4)	1698 (4)	44 (2)
C(15)	−132 (5)	8466 (5)	772 (3)	45 (3)
C(16)	−1026 (5)	6898 (4)	1142 (3)	41 (2)
C(17)	369 (5)	6530 (4)	1771 (3)	39 (2)
C(18)	−293 (4)	4912 (4)	2154 (3)	38 (2)
C(19)	538 (5)	3934 (4)	1968 (3)	41 (2)
C(20)	2172 (4)	4731 (4)	3017 (3)	37 (2)
C(21)	−1359 (5)	7453 (4)	2149 (3)	39 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·375 (7)	C(1)—C(6)	1·396 (6)
C(1)—C(20)	1·523 (7)	C(2)—C(3)	1·386 (9)
C(3)—C(4)	1·386 (8)	C(4)—C(5)	1·360 (9)
C(5)—C(6)	1·397 (9)	C(1*A*)—C(2*A*)	1·383 (7)
C(1*A*)—C(6*A*)	1·381 (6)	C(1*A*)—C(21)	1·518 (8)
C(2*A*)—C(3*A*)	1·388 (9)	C(3*A*)—C(4*A*)	1·362 (7)
C(4*A*)—C(5*A*)	1·368 (9)	C(5*A*)—C(6*A*)	1·412 (9)
C(7)—C(8)	1·293 (7)	C(7)—C(19)	1·507 (6)
C(8)—C(9)	1·508 (5)	C(9)—C(10)	1·579 (7)
C(9)—C(20)	1·522 (7)	C(10)—C(11)	1·517 (5)
C(10)—C(18)	1·539 (5)	C(11)—O(11)	1·209 (4)
C(11)—C(12)	1·498 (6)	C(12)—C(13)	1·575 (8)
C(12)—C(17)	1·543 (5)	C(13)—C(14)	1·510 (6)
C(13)—C(21)	1·535 (4)	C(14)—C(15)	1·312 (5)
C(15)—C(16)	1·526 (6)	C(16)—C(17)	1·556 (7)
C(16)—C(21)	1·531 (7)	C(17)—C(18)	1·549 (6)
C(18)—C(19)	1·560 (8)	C(19)—C(20)	1·542 (5)

C(2)—C(1)—C(6)	118·1 (5)	C(2)—C(1)—C(20)	119·4 (4)
C(6)—C(1)—C(20)	122·5 (4)	C(1)—C(2)—C(3)	121·9 (5)
C(2)—C(3)—C(4)	119·1 (5)	C(3)—C(4)—C(5)	120·3 (6)
C(4)—C(5)—C(6)	120·5 (5)	C(1)—C(6)—C(5)	120·1 (5)
C(2*A*)—C(1*A*)—C(6*A*)	118·1 (5)	C(2*A*)—C(1*A*)—C(21)	119·2 (4)
C(6*A*)—C(1*A*)—C(21)	122·7 (5)	C(1*A*)—C(2*A*)—C(3*A*)	122·0 (4)
C(2*A*)—C(3*A*)—C(4*A*)	199·0 (6)	C(3*A*)—C(4*A*)—C(5*A*)	121·1 (6)
C(4*A*)—C(5*A*)—C(6*A*)	119·6 (5)	C(1*A*)—C(6*A*)—C(5*A*)	120·2 (5)
C(8)—C(7)—C(19)	108·2 (3)	C(7)—C(8)—C(9)	107·7 (4)
C(8)—C(9)—C(10)	105·2 (3)	C(8)—C(9)—C(20)	101·0 (4)
C(10)—C(9)—C(20)	100·1 (3)	C(9)—C(10)—C(11)	112·8 (3)
C(9)—C(10)—C(18)	102·9 (4)	C(11)—C(10)—C(18)	106·4 (3)
C(10)—C(11)—O(11)	124·3 (4)	C(10)—C(11)—C(12)	111·3 (3)
O(11)—C(11)—C(12)	124·5 (3)	C(11)—C(12)—C(13)	112·1 (4)
C(11)—C(12)—C(17)	106·8 (3)	C(13)—C(12)—C(17)	102·7 (3)
C(12)—C(13)—C(14)	105·1 (4)	C(12)—C(13)—C(21)	100·1 (3)
C(14)—C(13)—C(21)	101·1 (3)	C(13)—C(14)—C(15)	108·1 (3)
C(14)—C(15)—C(16)	106·9 (4)	C(15)—C(16)—C(17)	105·5 (3)
C(15)—C(16)—C(21)	101·0 (4)	C(17)—C(16)—C(21)	101·2 (3)
C(12)—C(17)—C(16)	102·5 (4)	C(12)—C(17)—C(18)	107·6 (3)
C(16)—C(17)—C(18)	114·0 (3)	C(10)—C(18)—C(17)	107·8 (3)
C(10)—C(18)—C(19)	102·2 (3)	C(17)—C(18)—C(19)	115·3 (4)
C(7)—C(19)—C(18)	104·9 (4)	C(7)—C(19)—C(20)	100·1 (3)
C(18)—C(19)—C(20)	101·6 (3)	C(1)—C(20)—C(9)	117·8 (4)
C(1)—C(20)—C(19)	114·8 (3)	C(9)—C(20)—C(19)	93·0 (3)
C(1*A*)—C(21)—C(13)	115·9 (4)	C(1*A*)—C(21)—C(16)	119·0 (3)
C(13)—C(21)—C(16)	93·0 (3)		

and 0·20 e $Å^{-3}$; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00047F_o^2]^{-1}$. All computer programs supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration (Nicolet Instrument Corporation, 1986); atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters while Table 2 lists selected bond distances and valence angles.* Fig. 1 shows a thermal ellipsoid plot.

Related literature. The $Fe(CO)_5$ promoted reactions of norbornadiene derivatives with carbon monoxide have been described (Marchand, Earlywine, & Heeg, 1986; Marchand & Hayes, 1977; Marchand, Goodin, Hossain & van der Helm, 1984; Flippen-Anderson, Gilardi, George, Marchand & Dave, 1989). The structures of a hydrogenated *o*-methoxyphenyl derivative (Marchand, Goodin, Hossain & van der Helm, 1984) and of a $Fe(CO)_3$ complex of a related exocyclic diene (Watson, Nagl, Marchand & Chenera, 1988) may be used for comparison.

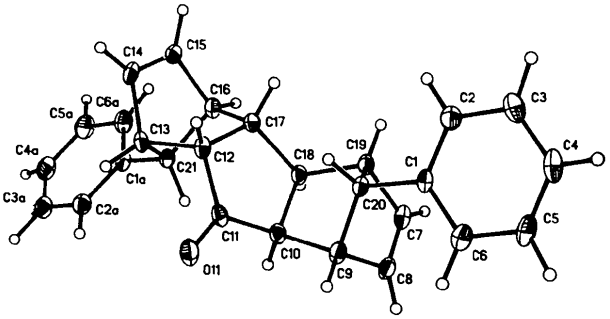

Fig. 1. Dimer ketone with thermal ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary size.

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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 52567 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of (4*R*)-*cis*-4,5-Dihydro-2-methyl-4,5-diphenyl-6*H*-cyclopenta[*b*]furan-6-one and *N*,*N*-Dimethyl-3,4-diphenyl-2,2′-bifuran-5-amine

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Abstract. (I) $C_{20}H_{16}O_2$, $M_r = 288·35$, orthorhombic, $P2_12_12_1$, $a = 8·665\,(1)$, $b = 8·808\,(1)$, $c = 19·766\,(1)$ Å, $V = 1508·6\,(1)$ Å^3, $Z = 4$, $D_x = 1·27$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1·5418$ Å, $\mu = 5·6$ cm^{-1}, $F(000) = 608$, $T = 123$ K, $R = 0·056$ for 1181 unique observed reflections. (II) $C_{22}H_{19}NO_2$, $M_r = 329·40$, monoclinic, $P2_1/c$, $a = 13·076\,(4)$, $b = 8·203\,(3)$, $c = 19·605\,(2)$ Å, $\beta = 124·95\,(3)°$, $V = 1723·6\,(1)$ Å^3, $Z = 4$, $D_x = 1·27$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1·5418$ Å, $\mu = 5·7$ cm^{-1}, $F(000) = 696$, $T = 123$ K, $R = 0·055$ for 2038 unique observed reflections. The C(4)—C(5) bond distance in structure (I) is unusually long at 1·59 Å. The two furan rings in structure (II) are coplanar; the dihedral angle between the planes is 1·9°.

Experimental. A clear, thin plate of dimensions 0·03 × 0·13 × 0·26 mm (I), and a clear, chunky plate of dimensions 0·10 × 0·08 × 0·05 mm (II) were used for intensity measurements on Syntex $P2_1$ (I) and $P1$ (II) diffractometers controlled by a Harris computer. Intensity measurements were made using graphite-monochromatized Cu $K\alpha$ radiation. The $\theta/2\theta$ step-scan technique was used with a scan speed of 2° min^{-1} and a scan width over 3·4° to $2\theta_{\max} = 138°$. Ten reflections monitored periodically showed no loss of intensity during the data collection. Of the 1626 (I), 2892 (II) unique reflections measured, 1181 (I), 2038 (II) had intensities greater than $3\sigma(I)$. Standard deviations in the intensities were approximated by the equation: $\sigma^2(I) = \sigma^2(I)_{\text{counting statistics}} + (DI)^2$ where the coefficient [$D = 0·0163$ (I), 0·0128 (II)] of I was calculated from the variations in intensities of the monitored reflections. Range of *hkl*: $h\ 0\rightarrow10$, $k\ 0\rightarrow10$, $l\ 0\rightarrow22$ (I), $h\ -15\rightarrow12$, $k\ 0\rightarrow9$, $l\ 0\rightarrow18$ (II). Unit-cell parameters were determined accurately by least-squares fit of Cu $K\alpha_1$ 2θ values [$\lambda(\mathrm{Cu}\,K\alpha_1) = 1·5406$ Å] for 25 high 2θ reflections ($120 < 2\theta < 138°$) (Duchamp, 1977). Lorentz and polarization corrections appropriate for a monochromator with 50% perfect character were applied but no correction for absorption was made. The structures were solved by direct methods, using *RANTAN*81 (Yao Jia-Xing, 1981) (I) and *DIREC* (Duchamp, 1984*a*) (II). H atoms found in difference maps were very close to positions generated using planar or tetrahedral geometry, so generated positions were used. The structure was refined by full-matrix least squares with all the coordinates and anisotropic thermal parameters for non-H atoms included in the refinement. Hydrogen thermal parameters were included in the calculations but not refined. The H atoms, with assigned temperature factors 0·5 Å^2 higher than the equivalent average isotropic values of the atoms of attachment, were included but only their positions were varied in the refinement. The function minimized in the refinement was $\sum w(F_o^2 - F_c^2)^2$, where weights w were $1/\sigma^2(F_o^2)$. Atomic form factors were from Doyle & Turner (1968), except for hydrogen which was from Stewart, Davidson & Simpson (1965). In the final cycle, Δ/σ was $<0·5$. The final R, wR were 0·056, 0·081 (I) and

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