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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1293). Services for accessing these data are described at the back of the journal.

References

Dudek, B. O. & Holm, R. H. (1961). *J. Am. Chem. Soc.* **83**, 2099–2104.
Nathan, L. C., Balzer, B. L., Larsen, S. K. & Casalnuovo, A. L. (1993). *Microchem. J.* **47**, 48–54.
Sheldrick, G. M. (1990). *SHELXTL*. Version 4.20. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1994). *SHELXTL*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Ueno, K. & Martell, A. E. (1955). *J. Phys. Chem.* **59**, 998–1004.

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6,6-Diphenylfulvene at 140 K

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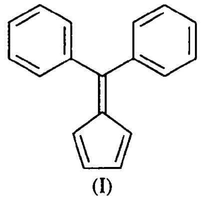
Abstract

The title compound, $C_{18}H_{14}$, crystallizes with two molecules in the asymmetric unit, which differ only in the angle between the phenyl rings and the fulvene moiety. The two double bonds of the cyclopentadiene moiety are shortened [1.352 (2) and 1.353 (2) Å] in the title compound compared with the range found in its metal complexes (1.381–1.520 Å). The bond joining these two double bonds is generally shorter in the metal complexes than in the title compound [1.467 (2) and 1.468 (2) Å], but there are exceptions. A conformational analysis of the title compound with the molecular modelling program *MOMO* [Bolte, Beck & Egert (1991). *Molecular Modelling Program MOMO*. University of Frankfurt, Germany] shows only one minimum on the energy hypersurface, where both phenyl rings form an angle of 56.6° with the fulvene moiety.

Comment

Crystallographic information about 6,6-diphenylfulvene, (I), as retrieved from the Cambridge Structural Database (Allen & Kennard, 1993), is so far only available for structures where the molecule acts as a ligand for various metal atoms. In all cases, at least one of the fulvene double bonds, but never a phenyl ring, complexes the metal atom.



(I)

We have determined the crystal structure of (I), which crystallizes with two independent but very similar molecules in the asymmetric unit, in order to compare it with those where the fulvene moiety acts as a ligand to Ni ($C_{26}H_{26}Ni$: Edelmann, Lubke & Behrens, 1982), Fe ($C_{21}H_{14}O_3Fe$: Edelmann, Lubke & Behrens, 1982; $C_{26}H_{14}O_8Fe_2$: Behrens, 1976), Pt ($C_{61}H_{52}P_2Pt$: Christofides, Howard, Spencer & Stone, 1982), Co ($C_{23}H_{19}Co$: Wadepohl & Pritzkow, 1991) and

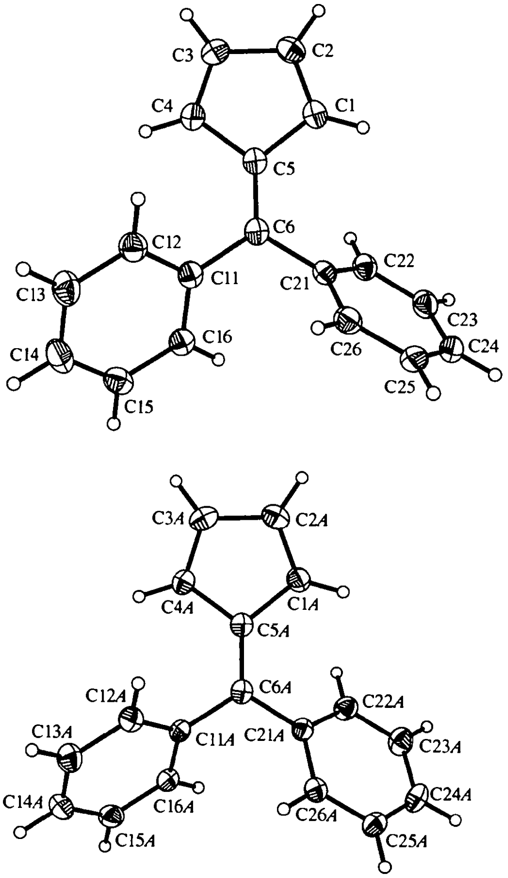


Fig. 1. A perspective view (not the relative orientation in the crystal) of the independent molecules of the title compound with the atom-numbering scheme; displacement ellipsoids are at the 50% probability level.

Ru ($C_{34}H_{14}O_{15}Ru_6$: Blake, Dyson, Johnson, Parsons, Reed & Shephard, 1995).

The notable difference is that in (I), the two double bonds of the cyclopentadiene moiety are shortened [1.352 (2) Å for C3—C4 and C3*A*—C4*A*, and 1.353 (2) Å for C1—C2 and C1*A*—C2*A*] compared with their counterparts in the other structures, which range from 1.381 to 1.520 Å. The bond joining the two double bonds of the cyclopentadiene moiety is generally shorter in (I) [1.467 (2) Å for C2—C3 and 1.468 (2) Å for C2*A*—C3*A*] than in the complexes (1.399–1.443 Å), but there are exceptions in $C_{61}H_{52}P_2Pt$ and $C_{26}H_{14}O_8Fe_2$ (1.489 and 1.473 Å, respectively).

The angles between the fulvene ring and the two phenyl rings [46.30 (6) and 57.97 (6)° for molecule *A*, and 44.74 (6) and 53.22 (5)° for molecule *B*] are in the same range (39.8–64.6°) for all structures. These two angles adopt the same value in $C_{26}H_{14}O_8Fe_2$ (53.9°) because the molecule is exactly C_2 symmetric, but they differ from each other in the other structures. A conformational analysis of (I) with the molecular modelling program *MOMO* (Bolte, Beck & Egert, 1991) showed only one minimum on the energy hypersurface, representing a conformation where both phenyl rings form an angle of 56.6° with the fulvene ring.

It can be concluded that the complexation to a metal atom slightly alters three bond lengths of the molecule and the angle between the fulvene moiety and phenyl rings can vary over an appreciable range.

Experimental

The title compound was obtained by dissolving 0.1 mol benzophenone in a warm solution of 0.1 mol metallic sodium in 40 ml absolute ethanol and slowly adding 0.1 mol cyclopentadiene at 313 K. After cooling to 273 K, the red crystalline solid was isolated by filtration and suitable crystals were obtained by slow evaporation from an ether/petrol ether solution.

Crystal data

$C_{18}H_{14}$
$M_r = 230.29$
Triclinic
$P\bar{1}$
$a = 10.0033\,(1)$ Å
$b = 10.0382\,(1)$ Å
$c = 14.3045\,(1)$ Å
$\alpha = 90.619\,(1)^\circ$
$\beta = 99.805\,(1)^\circ$
$\gamma = 113.053\,(1)^\circ$
$V = 1297.61\,(2)$ Å^3
$Z = 4$
$D_x = 1.179$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\theta = 1$–25°
$\mu = 0.067$ mm^{-1}
$T = 140$ K
Block
0.40 × 0.30 × 0.20 mm
Red

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996*a*)
$T_{\min} = 0.784$, $T_{\max} = 1.000$
20 486 measured reflections

5196 independent reflections
3978 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 26.43^\circ$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.102$
$S = 1.030$
5196 reflections
325 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.3825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.159$ e Å^{-3}
$\Delta\rho_{\min} = -0.228$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

C1—C2	1.353 (2)	C1*A*—C2*A*	1.353 (2)
C1—C5	1.473 (2)	C1*A*—C5*A*	1.479 (2)
C2—C3	1.467 (2)	C2*A*—C3*A*	1.468 (2)
C3—C4	1.352 (2)	C3*A*—C4*A*	1.352 (2)
C4—C5	1.480 (2)	C4*A*—C5*A*	1.476 (2)
C5—C6	1.368 (2)	C5*A*—C6*A*	1.364 (2)

The data collection nominally covered over a sphere of reciprocal space, by a combination of eight sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.95 cm. Coverage of the unique set is over 90% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analyzing the duplicate reflections. The structure was solved by building a model of the title compound using the program *MOMO* (Bolte, Beck & Egert, 1991), performing a conformational analysis and using the resulting structure as a search model for a fragment search with the program *PATSEE* (Egert & Sheldrick, 1985). After locating one molecule in the asymmetric unit with *PATSEE*, a tangent expansion with *SHELXS*96 (Sheldrick, 1996*b*) yielded the complete structure. All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [$U(\rm H) = 1.2U_{\rm eq}(\rm C)$] using a riding model with C—H = 0.95 Å.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *PATSEE* (Egert & Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*c*). Molecular graphics: *XP* (Siemens, 1994).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1206). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Behrens, U. (1976). *J. Organomet. Chem.* **107**, 103–111.
Blake, A. J., Dyson, P. J., Johnson, B. F. G., Parsons, S., Reed, D. & Shephard, D. S. (1995). *Organometallics*, **14**, 4199–4208.
Bolte, M., Beck, H. & Egert, E. (1991). *Molecular Modelling Program MOMO*. University of Frankfurt, Germany.

Christofides, A., Howard, J. A. K., Spencer, J. L. & Stone, F. G. A. (1982). *J. Organomet. Chem.* **232**, 279–292.
Edelmann, F., Lubke, B. & Behrens, U. (1982). *Chem. Ber.* **115**, 1325–1331.
Egert, E. & Sheldrick, G. M. (1985). *Acta Cryst.* A**41**, 262–268.
Sheldrick, G. M. (1996*a*). *SADABS. Program for Empirical Absorption Correction of Area Detector Data.* University of Göttingen, Germany.
Sheldrick, G. M. (1996*b*). *SHELXS96. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1996*c*). *SHELXL96. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XP. Interactive Molecular Graphics Program.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1995). *SMART* and *SAINT. Data Collection and Processing software for the SMART System.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wadepohl, H. & Pritzkow, H. (1991). *Acta Cryst.* C**47**, 2061–2063.

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6a,7-Dihydro-2,6-dimethyl-7-phenyl-6*H*-chromeno[4,3-*d*]-1,2,4-triazolo[1,5-*a*]-pyrimidin-6a-ol†

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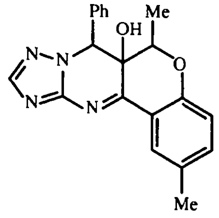
Abstract

In the title compound, $C_{20}H_{18}N_4O_2$, the dihydropyrimidine and dihydropyran rings adopt a distorted sofa conformation. The phenyl substituent at C7 and the hydroxy group at C6a adopt axial positions, whereas the methyl group at C6 is equatorial.

Comment

Derivatives of 1,2,4-triazolo[1,5-*a*]pyrimidine possess high biological activity, for example, as cardiovascular agents (Tsuda *et al.*, 1986). The chemical and physiological characteristics of these compounds are directly related to their conformations. However, relevant data on the structures of dihydro-1,2,4-triazolo[1,5-*a*]pyrimidines condensed with pyran rings, (I), or other heterocycles have not previously been available.



(I)

Torsion angles N7a—C10a—N11—C11a and C1—C1a—C11a—N11 are 15.4 (4) and 9.4 (4)°, respectively. This indicates some loss of planarity of the conjugated system of the molecule, although the C1a—C11a, N11—C11a and C10a—N11 bond lengths [1.453 (4), 1.294 (3) and 1.381 (3) Å, respectively] are evidence of the conjugation between the benzene ring, azomethine group and 1,2,4-triazole ring (Fig. 1).

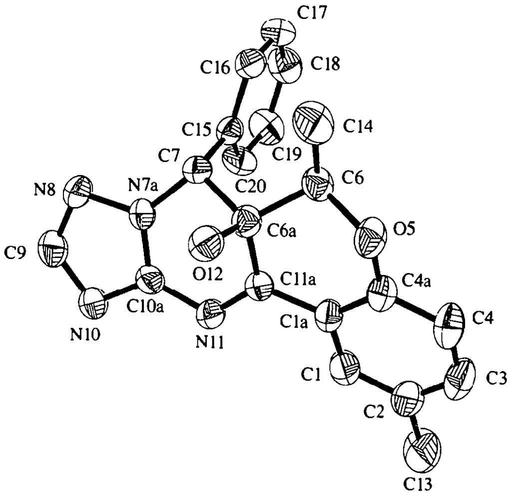


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 50% probability level. The H atoms are omitted for clarity.

The conformations of both the pyrimidine and pyran rings can be described as distorted sofas. The puckering coordinates (Zefirov, Palyulin & Dashevskaya, 1990) are $S = 0.63$, $\theta = 50.9$ and $\varphi = 18.3$ for the pyrimidine, and $S = 0.72$, $\theta = 44.7$ and $\varphi = 10.2$ for the pyran moiety, where S is the puckering degree, and θ and φ are polar angles which describe the ring conformation type (values of these angles for ideal conformations are: sofa $\theta = 45$, $\varphi = 0$; half chair $\theta = 45$, $\varphi = 30$). Comparison of the title compound with *cis*-3-bromo-2,3-dihydro-2-phenyl-4*H*-1-benzopyran-4-one and (2*R*,3*R*)-3,5,7-trihydroxy-2-(2*R**,3*R**)-3-(4-hydroxy-3-methoxyphenyl)-2-hydroxymethyl-(1,4-benzodioxan-6-yl)-4-chromanone

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† Alternative name: 6a,7-dihydro-2,6-dimethyl-7-phenyl-6*H*-[1,2,4]-triazolo[2′,3′:1,2]pyrimidino[5,4-*c*]chromen-6a-ol.