Crystals were grown for X-ray analysis in diethyl ether. Details of the data collection and structural refinement are given in Table 1. Final positional and equivalent isotropic thermal parameters are given in Table 4, and both lengths, bond angles, and torsion angles, are listed in Table 5.* Fig. 2 shows the molecule and the atomic numbering scheme.

Compound (III) was prepared by passing oxygen through a vigorously stirred solution of 2,6-di-*tert*-butylphenol, CuCl and *N,N,N',N'*-tetramethylethylenediamine in 95% ethanol (Hay, 1969). Crystals were grown for X-ray analysis in ethyl acetate. Details of the data collection and structural refinement are given in Table 1. Final positional and equivalent isotropic thermal parameters are given in Table 6.* Fig. 3 shows the molecule and the atomic numbering scheme.

Related literature. Original crystal structure of (I): Akhmed, Farag & Amin (1971). The structure of hydroquinone: Maartmann–Moe (1966); structure of 2,6-di-*tert*-butyl-4-phenylphenol: Bekkouch, Perrin & Thozet (1988) related to our compounds (I) and (II); structure of *p*-benzoquinone: Trotter (1960). Original crystal structure of (III): Khan, Osman & Tuck (1986).

* See deposition footnote

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A Bichromophoric Difulvene from 1,5-Cyclooctadione

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Abstract. 1,5-Bis(2,4-cyclopentadien-1-ylidene)cyclooctane, $C_{18}H_{20}$, $M_r = 236{\cdot}4$, monoclinic, $P2_1/n$, $a = 7{\cdot}498\ (2)$, $b = 11{\cdot}415\ (3)$, $c = 16{\cdot}555\ (3)$ Å, $\beta = 97{\cdot}80\ (2)°$, $V = 1403{\cdot}7\ (10)$ Å^3, $Z = 4$, $D_x = 1{\cdot}118$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}58$ cm^{-1}, $F(000) = 512$, $T = 296$ K, $R = 0{\cdot}050$ for 1550 observations with $I > 1\sigma(I)$ (of 2471 unique data). The eight-membered ring of the title molecule adopts a boat–chair conformation with considerable bond angle distortions observed for all the methylenes forming the eight-membered ring. These six bond angles range from 113·3 (2)–116·1 (2)°. The bond angles exocyclic to the cyclopentadienylidene ring are 116·7 (2) and 116·6 (2)°. The intramolecular distance, 2·957 (2) Å, between the carbons that are exocyclic to the cyclopentadienylidene ring is well within the sum of van der Waals radii. The title compound exhibits a molar absorptivity which is less than half that of a single fulvene chromophore. The cyclopentadienylidene rings are nearly planar, with maximum deviations of 0·008 (2) and 0·013 (2) Å, and they form a dihedral angle of 32·2 (1)°.

Experimental. The title compound was prepared by condensing 1,5-cyclooctadione and 1,3-cyclopentadiene with pyrrolidine as a catalyst in methanol (Stone & Little, 1985). Crystals grown by slow cooling of refluxing hexane, m.p. 396–397 K, were suitable; a yellow crystal 0·10 × 0·48 × 0·75 mm was mounted in a capillary on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)*
C1	0·5957 (2)	0·1275 (2)	0·5698 (1)	4·85 (5)
C2	0·6964 (3)	0·0612 (2)	0·6402 (1)	6·10 (6)
C3	0·8243 (3)	0·1369 (2)	0·6991 (1)	5·94 (5)
C4	0·7372 (3)	0·2362 (2)	0·7416 (1)	6·03 (5)
C5	0·6498 (2)	0·3277 (2)	0·6839 (1)	4·95 (5)
C6	0·4479 (3)	0·3271 (2)	0·6684 (1)	6·47 (6)
C7	0·3611 (3)	0·2076 (2)	0·6526 (1)	7·15 (6)
C8	0·4012 (2)	0·1516 (2)	0·5736 (1)	6·33 (6)
C9	0·6767 (2)	0·1571 (2)	0·5047 (1)	4·59 (5)
C10	0·8614 (2)	0·1326 (2)	0·4925 (1)	5·53 (5)
C11	0·8885 (3)	0·1759 (2)	0·4205 (1)	6·66 (6)
C12	0·7252 (3)	0·2286 (2)	0·3826 (1)	6·76 (6)
C13	0·6008 (3)	0·2200 (2)	0·4316 (1)	5·65 (5)
C14	0·7512 (2)	0·4069 (2)	0·6505 (1)	4·67 (5)
C15	0·9443 (2)	0·4209 (2)	0·6656 (1)	5·40 (5)
C16	0·9933 (3)	0·5061 (2)	0·6178 (1)	6·61 (6)
C17	0·8343 (3)	0·5525 (2)	0·5708 (1)	7·00 (6)
C18	0·6906 (3)	0·4977 (2)	0·5899 (1)	5·85 (5)

* The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $B_{eq} = (8\pi^2/3) \times \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

from setting angles of 25 reflections having $12 > \theta > 10°$. The ω–2θ scans were designed for $I = 50\sigma(I)$, subject to max. scan time 180 s, scan rates varied 0·35–4·0° min^{-1}. Data having $1 \le \theta \le 25°$, $0 \le h \le 8$, $0 \le k \le 13$, $-19 \le l \le 19$ were measured and corrected for background, Lorentz, polarization and decay. Absorption was negligible. Three standard reflections (200,020,004) showed a 3·0% decrease of intensity, so a linear correction was applied. Redundant $0kl$ and $0k\bar{l}$ data were averaged, $R_{int} = 0·017$. The space group was determined from systematic absences $h0l$ with $h + l$ odd and $0k0$ with k odd. The structure was solved by direct methods, refined by full-matrix least squares based upon F, using data for which $I > 1\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0·02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), and scattering factors of Cromer & Waber (1974). C atoms were refined anisotropically; H atoms were located by difference synthesis and refined isotropically. Final $R = 0·050$ (0·120 for all data), $wR = 0·044$, $S = 1·879$ for 244 variables. Maximum shift $0·01\sigma$ in the final cycle, max. residual density 0·16, min. $-0·12$ e Å^{-3}, extinction coefficient $g = 3·4\,(11) \times 10^{-7}$ where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a line drawing and Fig. 2 is a perspective drawing showing the atom numbering. Bond distances, angles and torsion angles are presented in Table 2.*

* Lists of anisotropic thermal parameters, H-atom parameters, bond distances and angles involving H, structure-factor amplitudes and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52490 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *angles* (°) *and torsion angles* (°)

C1—C2	1·504 (2)	C9—C10	1·453 (2)
C1—C8	1·494 (2)	C9—C13	1·456 (2)
C1—C9	1·349 (2)	C10—C11	1·331 (2)
C2—C3	1·538 (3)	C11—C12	1·431 (3)
C3—C4	1·527 (3)	C12—C13	1·320 (2)
C4—C5	1·504 (2)	C14—C15	1·444 (2)
C5—C6	1·501 (2)	C14—C18	1·470 (2)
C5—C14	1·348 (2)	C15—C16	1·337 (2)
C6—C7	1·518 (3)	C16—C17	1·433 (3)
C7—C8	1·523 (3)	C17—C18	1·321 (3)

C2—C1—C8	116·7 (2)	C1—C9—C13	128·3 (2)
C2—C1—C9	120·8 (2)	C10—C9—C13	104·6 (2)
C8—C1—C9	122·4 (2)	C9—C10—C11	108·5 (2)
C1—C2—C3	114·4 (2)	C10—C11—C12	108·9 (2)
C2—C3—C4	116·1 (2)	C11—C12—C13	109·4 (2)
C3—C4—C5	113·6 (2)	C9—C13—C12	108·6 (2)
C4—C5—C6	116·6 (2)	C5—C14—C15	127·7 (2)
C4—C5—C14	120·3 (2)	C5—C14—C18	127·9 (2)
C6—C5—C14	123·0 (2)	C15—C14—C18	104·5 (2)
C5—C6—C7	115·6 (2)	C14—C15—C16	109·1 (2)
C6—C7—C8	113·3 (2)	C15—C16—C17	108·4 (2)
C1—C8—C7	114·9 (2)	C16—C17—C18	109·9 (2)
C1—C9—C10	127·1 (2)	C14—C18—C17	108·1 (2)

C8—C1—C2—C3	−105·2 (2)	C9—C1—C2—C3	78·6 (2)
C2—C1—C8—C7	51·2 (3)	C9—C1—C8—C7	−132·6 (2)
C2—C1—C9—C10	−0·4 (3)	C2—C1—C9—C13	179·6 (2)
C8—C1—C9—C10	−176·4 (2)	C8—C1—C9—C13	3·5 (3)
C1—C2—C3—C4	60·7 (2)	C2—C3—C4—C5	−63·3 (2)
C3—C4—C5—C6	105·8 (2)	C3—C4—C5—C14	−75·9 (2)
C4—C5—C6—C7	−45·2 (2)	C14—C5—C6—C7	136·5 (2)
C4—C5—C14—C15	−3·0 (3)	C4—C5—C14—C18	176·3 (2)
C6—C5—C14—C15	175·2 (2)	C6—C5—C14—C18	−5·5 (3)
C5—C6—C7—C8	−66·2 (2)	C6—C7—C8—C1	61·9 (2)
C1—C9—C10—C11	179·7 (2)	C13—C9—C10—C11	−0·2 (2)
C1—C9—C13—C12	−178·9 (2)	C10—C9—C13—C12	1·1 (2)
C9—C10—C11—C12	−0·6 (3)	C10—C11—C12—C13	1·4 (3)
C11—C12—C13—C9	−1·5 (3)	C5—C14—C15—C16	177·3 (2)
C18—C14—C15—C16	−2·2 (2)	C5—C14—C18—C17	−177·0 (2)
C15—C14—C18—C17	2·5 (2)	C14—C15—C16—C17	1·1 (2)
C15—C16—C17—C18	0·6 (3)	C16—C17—C18—C14	−1·9 (2)

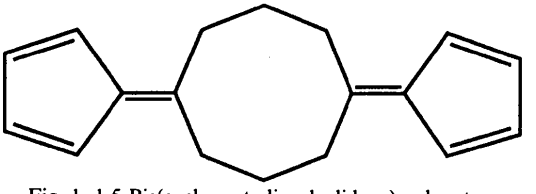

Fig. 1. 1,5-Bis(cyclopentadien-1-ylidene)cyclooctane.

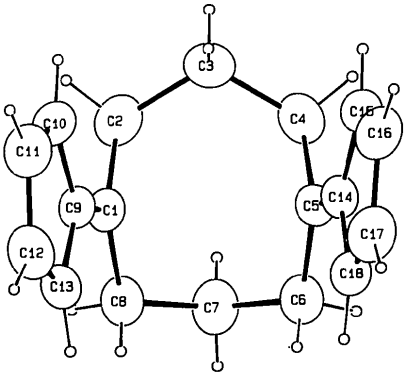

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the molecule, representing C atoms as 30% probability ellipsoids and H atoms as circles of arbitrary radius.

Related literature. The structures of dimethylfulvene at 248 K (Norman & Post, 1961), and 5-(2,4-cyclopentadien-1-ylidene)cyclooctanone (Cronan, Fronczek & McLaughlin, 1989) exhibit the expected single–double bond alternation within the fulvene ring system, as does the title compound. The bond angles C2—C1—C8 116·7 (2) and C4—C5—C6 116·6 (2)° of the title compound are similar to the analogous bond angles of dimethylfulvene, 114·0 (6)°, and 5-(2,4-cyclopentadien-1-ylidene)cyclooctanone, 116·6 (2)°.

The eight-membered ring of the title molecule adopts a chair–boat conformation with bond distances and angles similar to those found for 1,5-cyclooctadione (Miller & McPhail, 1979) and 5-(2,4-cyclopentadien-1-ylidene)cyclooctanone (Cronan, Fronczek & McLaughlin, 1989). The close intermolecular contact between the 1 and 5 positions of the eight-membered ring of 1,5-cyclooctadione, 2·963 (8) Å, 5-(2,4-cyclopentadien-1-ylidene)cyclooctanone, 3·018 (2) Å, and the title compound, 2·957 (2) Å, are similar.

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7,8-Dihydro-1,6,11-trimethoxy-5,12-naphthacenedione

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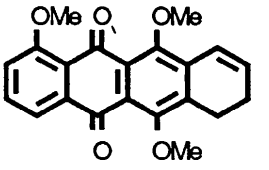
Abstract. $C_{21}H_{18}O_5$, $M_r = 350{\cdot}4$, monoclinic, $P2_1/n$, $a = 12{\cdot}853$ (3), $b = 8{\cdot}183$ (2), $c = 17{\cdot}506$ (4) Å, $\beta = 109{\cdot}45$ (2)°, $V = 1736{\cdot}1$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}34$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}89$ cm^{-1}, $F(000) = 736$, $T = 295$ K. Final $R = 0{\cdot}052$ for 1863 observed reflections. The average bond distances are C=O = 1·216 (4), O—C_{benz} = 1·367 (4), O—CH_3 = 1·426 (5), C—C_{benz} = 1·397 (5), C—C = 1·489 (5) Å and C=C = 1·346 (5) Å. The X-ray structure confirms the structure of one of the regioisomers formed in the reaction of aryne generated from 2-bromo-5,6-dihydro-1,4-dimethoxynaphthalene and 7-methoxycyanophthalide.

Experimental. The title compound (1) was prepared along with the other regioisomer, 9,10-dihydro-1,6,11-trimethoxy-5,12-naphthacenedione, by a reaction of 7-methoxy-1-oxo-1,3-dihydro-3-isobenzofurancarbonitrile and 2-bromo-1,5-dimethoxy-5,6-dihydronaphthalene in the presence of lithium diisopropylamide (LDA) and THF (Khanapure & Biehl, 1987). Two regioisomers were readily separated by flash chromatography. Crystals of (1) are pale-yellow plates, unit-cell parameters by least-squares fit of 25 reflections in the range $10 < 2\theta < 25°$, crystal dimensions 0·30 × 0·40 × 0·10 mm, space group $P2_1/n$ from systematic absences ($0k0$, k odd; $h0l$, $h + l$ odd); automatic Nicolet $R3m/V$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan mode (scan rate 3·0–15·0° min^{-1}, depending on intensity), 4436 measured reflections, 4015 independent reflections in the range $3 < 2\theta < 55°$,



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