for H atoms, $(\Delta/\sigma)_{\max} = 0{\cdot}4$, $(\Delta\rho)_{\max} = 0{\cdot}47$, $(\Delta\rho)_{\min} = -0{\cdot}49$ e Å^{-3}. For (2): 1561 reflections for 181 variables; $R = 0{\cdot}031$, $wR = 0{\cdot}026$, $U_{\rm iso} = 0{\cdot}086\,(6)$ Å^2 for H atoms, $(\Delta/\sigma)_{\max} = 0{\cdot}1$, $(\Delta\rho)_{\max} = 0{\cdot}20$, $(\Delta\rho)_{\min} = -0{\cdot}38$ e Å^{-3}.

Fractional coordinates for the non-H atoms of both compounds are given in Table 1, bond lengths and angles in Table 2.* The structures are shown in Fig. 1.

Related literature. The crystal structures of three related ylides are described by Dillen, Meth-Cohn & van Rooyen (1987), and reactions of possible interest in Dillen, Meth-Cohn, Moore & van Rooyen (1988).

References

DILLEN, J. L. M., METH-COHN, O., MOORE, C. & VAN ROOYEN, P. H. (1988). *Tetrahedron*, **44**, 3127–3138.

DILLEN, J. L. M., METH-COHN, O. & VAN ROOYEN, P. H. (1987). *S. Afr. J. Chem.* **40**, 51–56.

DILLEN, J. L. M., METH-COHN, O. & VAN VUUREN, G. (1987). *J. Chem. Soc. Perkin Trans.* 1, pp. 2659–2661.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52619 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Bichromophoric Difulvene from 3,7-Bicyclo[3.3.0]octadione

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Abstract. 3,7-Bis(2,4-cyclopentadien-1-ylidene)bicyclo[3.3.0]octane, $C_{18}H_{18}$, $M_r = 234{\cdot}3$, monoclinic, $I2/a$, $a = 15{\cdot}2830\,(11)$, $b = 5{\cdot}1918\,(3)$, $c = 16{\cdot}8990\,(13)$ Å, $\beta = 96{\cdot}397\,(6)^\circ$, $V = 1332{\cdot}6\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}168$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 4{\cdot}58$ cm^{-1}, $F(000) = 504$, $T = 295$ K, $R = 0{\cdot}036$ for 1042 observations with $I > 3\sigma(I)$ (of 1377 unique data). The bicyclo[3.3.0]octane ring system adopts a flattened half-chair conformation with a crystallographic twofold axis bisecting the ring fusion bond. The bond angle of the C=C exocyclic to the cyclopentadienylidene ring is $108{\cdot}72\,(8)^\circ$. The intramolecular distance between the electrophilic fulvene carbons is $3{\cdot}822\,(2)$ Å. The cyclopentadienylidene rings are planar, with maximum deviation of $0{\cdot}001\,(1)$ Å.

Experimental. The title compound was prepared by condensing 3,7-bicyclo[3.3.0]octadione and 1,3-cyclopentadiene with pyrrolidine as a catalyst in methanol (Stone & Little, 1984). Suitable crystals (m.p. 395–397 K) were grown by slow cooling of refluxing hexane; a yellow crystal with dimensions $0{\cdot}06 \times 0{\cdot}35 \times 0{\cdot}43$ mm was mounted in a capillary on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 20^\circ$. The ω–2θ scans were designed for $I = 50\sigma(I)$, subject to maximum scan time = 90 s, scan rates varied $0{\cdot}61$–$3{\cdot}30^\circ$ min^{-1}. Two quadrants of data having $2 \le \theta \le 75^\circ$, $-26 \le h \le 26$, $0 \le k \le 6$, $-19 \le l \le 19$, were measured and corrected for background, Lorentz, polarization and absorption. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 0·884. Three standard reflections (200, 020, 002) showed only a random fluctuation in intensity so no correction for decay was applied. Data from two equivalent quadrants were averaged, $R_{\rm int} = 0{\cdot}018$. Systematic absences hkl with $h + k + l$ odd and $h0l$ with h odd indicated space groups $I2/a$ or Ia. The former was confirmed by successful refinement of a centrosymmetric model. The structure was solved by direct methods, refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer

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& Waber (1974), anomalous coefficients of Cromer (1974). C-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0{\cdot}036$ for 1042 observed data (0·052 for all 1377 data), $wR = 0{\cdot}042$, $S = 1{\cdot}994$ for 119 variables. Maximum shift $< 0{\cdot}01\sigma$ in the final cycle, max. residual density 0·10, min. $-0{\cdot}10$ e Å^{-3}, extinction coefficient $g = 7{\cdot}2\,(5) \times 10^{-6}$ 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 structural diagram and Fig. 2 is a perspective drawing showing the atom numbering. Bond distances, angles and torsion angles are presented in Table 2.*

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 like 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)°.

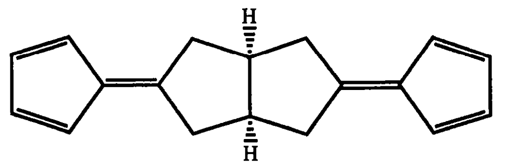

Fig. 1. 3,7-Bis(cyclopentadien-1-ylidene)bicyclo[3.3.0]octane.

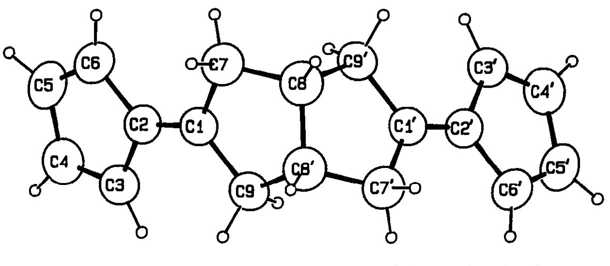

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the molecule. C atoms are represented by 40% probability ellipsoids and H atoms by circles of arbitrary radius.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)*
C1	0·82737 (7)	0·2877 (2)	0·41860 (6)	3·87 (2)
C2	0·87056 (7)	0·1195 (2)	0·37659 (6)	3·99 (2)
C3	0·83119 (8)	−0·0481 (3)	0·31306 (7)	4·85 (3)
C4	0·89532 (9)	−0·1893 (3)	0·28652 (7)	5·70 (3)
C5	0·97866 (8)	−0·1219 (3)	0·33128 (7)	5·49 (3)
C6	0·96449 (8)	0·0607 (3)	0·38460 (7)	4·79 (3)
C7	0·86765 (8)	0·4651 (3)	0·48332 (7)	4·50 (3)
C8	0·79130 (8)	0·5325 (2)	0·53061 (7)	4·45 (3)
C9	0·72864 (8)	0·3240 (3)	0·40937 (6)	4·48 (3)

* The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation:

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

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

C1—C2	1·344 (2)	C4—C5	1·450 (2)
C1—C7	1·508 (2)	C5—C6	1·342 (2)
C1—C9	1·511 (2)	C7—C8	1·527 (2)
C2—C3	1·459 (2)	C8—C8′	1·540 (2)
C2—C6	1·459 (2)	C8—C9′	1·537 (2)
C3—C4	1·341 (2)		
C2—C1—C7	126·52 (9)	C4—C5—C6	108·8 (8)
C2—C1—C9	124·74 (9)	C2—C6—C5	108·4 (1)
C7—C1—C9	108·72 (8)	C1—C7—C8	104·22 (8)
C1—C2—C3	126·13 (9)	C7—C8—C8′	105·40 (9)
C1—C2—C6	128·52 (9)	C7—C8—C9′	113·80 (9)
C3—C2—C6	105·34 (9)	C8′—C8—C9	103·61 (8)
C2—C3—C4	108·4 (1)	C1—C9—C8′	106·78 (8)
C3—C4—C5	109·0 (1)		
C1—C7—C8—C9′	80·6 (1)	C9—C1—C7—C8	19·4 (1)
C9′—C8—C8′—C7′	32·7 (1)	C1—C7—C8—C8′	−32·3 (1)
C7—C8—C8′—C7′	152·5 (1)	C9′—C8—C8′—C9	−87·2 (1)

The bicyclo[3.3.0]octane ring system adopts a flattened half-chair conformation with a molecular twofold axis bisecting the ring fusion between C8—C8′ like that observed in a related compound (Burnell, Freer, Grewal, Hayes, Sawyer & Yates, 1988).

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References

Burnell, D. J., Freer, V. J., Grewal, R. S. Hayes, P. C., Sawyer, J. F. & Yates, P. (1988). *Acta Cryst.* C**44**, 296–300.

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

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* Lists of H-atom coordinates and thermal parameters, bond distances and angles involving hydrogen, anisotropic thermal parameters, structure-factor amplitudes, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52638 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Cronan, J. M. Jr, Fronczek, F. R. & McLaughlin, M. L. (1989). *Acta Cryst.* **C45**, 1063–1065.
Frenz, B. A. & Okaya, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Norman, N. & Post, B. (1961). *Acta Cryst.* **14**, 503–507.
Stone, K. J. & Little, R. D. (1984). *J. Org. Chem.* **49**, 1849–1853.

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Structure of 9-Oxo-1-thioxanthenecarbonitrile 10,10-Dioxide

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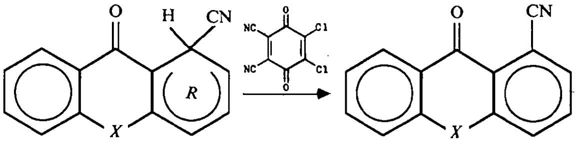
Abstract. $C_{14}H_7NO_3S$, $M_r = 269{\cdot}3$, triclinic, $P\bar{1}$, $a = 8{\cdot}069\,(3)$, $b = 8{\cdot}654\,(3)$, $c = 8{\cdot}973\,(3)$ Å, $\alpha = 102{\cdot}12\,(3)$, $\beta = 91{\cdot}16\,(3)$, $\gamma = 108{\cdot}19\,(3)°$, $V = 579{\cdot}5\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}543$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 2{\cdot}68$ cm^{-1}, $F(000) = 276$, $T = 298$ K, $R = 0{\cdot}0429$ and $wR = 0{\cdot}0494$ for 1854 reflections [$I \geq 3\sigma(I)$]. The bonding between C(9) and C(9a) and C(8a) is asymmetrical. C(9)—C(9a) [1·499 (3) Å] is longer than C(9)—C(8a) [1·475 (3) Å] due to the electron-withdrawing power of the cyano group. A considerable repulsion between the carbonyl and the cyano groups is accommodated by distortion of the cyano group and the geometry around C(1). Angle C(11)—C(1)—C(9a) [122·4 (2)°] is larger than C(11)—C(1)—C(2) [116·2 (2)°]. Additionally, angle C(1)—C(11)—N(11) is 174·0 (2)°, compared to an angle of 180° around an *sp*-hybridized carbon, and the N atom is pointing away from the carbonyl group. The dihedral angle between the two arene rings is 170·1 (2)°.

Experimental. The title compound (1) was synthesized in a demetallation–rearomatization reaction summarized in the scheme below (Sutherland, Chowdhury, Piórko & Lee, 1987). Crystals were grown by slow evaporation from an Me_2SO-d_6 solution and a pale-yellow prism of dimensions $0{\cdot}28 \times 0{\cdot}32 \times 0{\cdot}40$ mm was chosen for X-ray investigation. Data were collected on a Nicolet *R3m/V* diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation. 50 reflections with $15{\cdot}55 \leq 2\theta \leq 34{\cdot}99°$ were used to refine the cell parameters. 5108 reflections were collected using the ω-scan method (h, $-10\rightarrow10$; k, $-11\rightarrow10$; l, $-11\rightarrow11$), 2670 unique reflections, $R_{\rm int} = 0{\cdot}0249$; 2θ range $3\rightarrow55°$, 1·2° ω scan at 3–6° min^{-1}, depending upon intensity. Four reflections $(020, 112, 0\bar{2}0, \bar{1}\bar{1}\bar{2})$ were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 1·15%). Absorption corrections were applied based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1987); minimum and maximum transmission 0·9934 and 0·9959.



$X = SO_2$, R = FeCp (1)

The structure was solved by direct methods in *SHELXTL-Plus* from which the locations of all non-H atoms were obtained. The structure was refined [*SHELX76* (Sheldrick, 1976)] using full-matrix least squares and the positions of all H atoms were determined from a difference Fourier map. The non-H atoms were treated anisotropically, whereas the H atoms were refined with isotropic thermal parameters. 201 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$, I(intensity) = $(I_{\rm peak} - I_{\rm background})$(scan rate), and $\sigma(I) = (I_{\rm peak} + I_{\rm background})^{1/2}$(scan rate), k is the correction due to decay and Lp effects, 0·02 is a factor used to down weight intense reflections and to account for instrument instability. An extinction correction $\chi = 0{\cdot}0153\,(16)$ [where $F^* = F(1 + 0{\cdot}002\chi F^2/\sin2\theta)^{-1/4}$] was also applied (Sheldrick, 1987). Final $R = 0{\cdot}0429$, $wR = 0{\cdot}0494$ ($R_{\rm all} = 0{\cdot}0696$, $wR_{\rm all} = 0{\cdot}0567$) for 1854 reflections having $I \geq 3\sigma(I)$, and goodness-of-fit = 1·55. Maximum $\Delta/\sigma = 0{\cdot}001$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were $-0{\cdot}31$ and $0{\cdot}44$ e Å^{-3}, respectively.

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