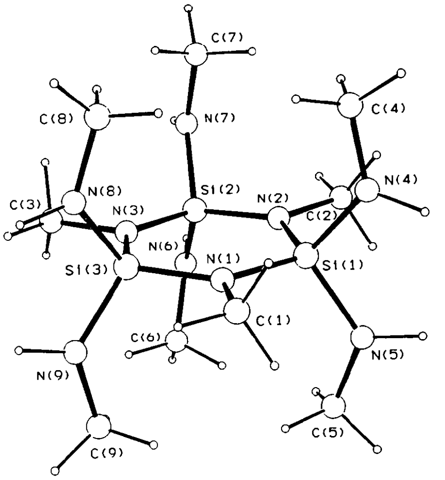

Fig. 1. The molecular structure of the title compound.

perspective view of the molecule, Fig. 2 crystal packing.

Related literature. With respect to the Si_3N_3 core, related silazanes are 2,2,4,4,6,6-hexaisopropylcyclotrisilazane (Klingebiel & Vater, 1983), 2,4,6-tri-*tert*-butyl-2,4,6-trifluorocyclotrisilazane (Clegg, Sheldrick & Stalke, 1984*b*) and 2,2,4,4,6,6-hexa-*tert*-butylcyclotrisilazane (Clegg, Sheldrick & Stalke, 1984*a*), all showing a planar six-membered ring in agreement with our results.

The reactions of $SiCl_4$ with CH_3NH_2 in the gas phase (Drake & Westwood, 1971), without solvent at room temperature (Hagen & Callaway, 1972) and in petroleum ether (Adrianov, Il'in, Talanov, Isakova & Sidorenko, 1976) have been reported in the literature. Based on an elementary analysis, Adrianov *et al.* (1976) suggested for one of their products the same composition as that of the title compound; however, no information on the molecular structure was given.

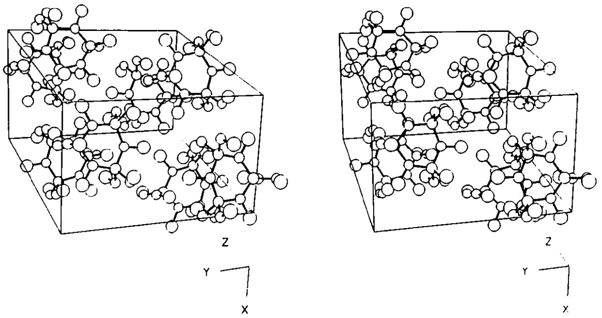

Fig. 2. Stereoplot of the unit cell. H atoms are omitted.

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Acta Cryst. (1990). C**46**, 1181–1183

9-(2,4-Cyclopentadienylidene)bicyclo[3.3.1]nonane, a Ring-Strained Pentafulvene

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(*Received* 5 *September* 1989; *accepted* 16 *January* 1990)

Abstract. 9-(2,4-Cyclopentadien-1-ylidene)bicyclo-[3.3.1]nonane, $C_{14}H_{18}$, M_r = 186·3, monoclinic, $P2_1/c$, a = 10·6088 (11), b = 12·251 (3), c = 9·2343 (14) Å, β = 111·23 (1)°, V = 1118·7 (6) Å^3, Z = 4, D_x = 1·106 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 0·425 mm^{-1}, F(000) = 408, T = 295 K, R = 0·042 for 1636 observations with $I > 3\sigma(I)$ (of 2300 unique data). The bicyclo[3.3.1]nonane adopts a twin-chair conformation. The bond angle of the C═C exocyclic to the cyclopentadienylidene ring is 110·95 (9)°. This bond angle is very close to the corresponding bond angle in 2-(2,4-cyclopentadien-1-ylidene)adamantane or adamantylidenefulvene. The cyclopentadienylidene ring is planar, with maximum deviation of 0·005 (2) Å.

Experimental. The title compound was prepared by condensing bicyclo[3.3.1]nonan-9-one and 1,3-

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq} (Å^2)
C1	0·2862 (1)	0·0538 (1)	0·0973 (2)	4·15 (3)
C2	0·3022 (1)	0·1720 (1)	0·1126 (2)	4·48 (3)
C3	0·3663 (1)	0·1962 (1)	0·2630 (2)	5·11 (4)
C4	0·3930 (1)	0·0969 (1)	0·3525 (2)	5·52 (4)
C5	0·3466 (2)	0·0120 (1)	0·2565 (2)	5·15 (4)
C6	0·2306 (1)	−0·0044 (1)	−0·0345 (2)	4·27 (3)
C7	0·2233 (2)	−0·1271 (1)	−0·0413 (2)	5·57 (4)
C8	0·0739 (2)	−0·1623 (1)	−0·1109 (2)	6·19 (4)
C9	−0·0064 (2)	−0·1095 (1)	−0·2658 (2)	5·61 (4)
C10	0·0221 (1)	0·0123 (1)	−0·2675 (2)	5·20 (4)
C11	0·1723 (1)	0·0440 (1)	−0·1942 (2)	4·45 (3)
C12	0·3109 (2)	−0·1671 (1)	−0·1319 (2)	7·42 (5)
C13	0·2772 (2)	−0·1155 (2)	−0·2909 (2)	6·84 (5)
C14	0·2588 (2)	0·0067 (2)	−0·2876 (2)	5·86 (4)

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

C1—C2	1·459 (1)	C4—C5	1·339 (2)
C7—C12	1·539 (2)	C12—C13	1·518 (2)
C2—C3	1·340 (1)	C13—C14	1·512 (2)
C10—C11	1·538 (1)	C1—C5	1·467 (1)
C6—C7	1·506 (1)	C8—C9	1·519 (2)
C7—C8	1·541 (2)	C3—C4	1·440 (2)
C1—C6	1·348 (1)	C11—C14	1·539 (1)
C9—C10	1·524 (2)	C6—C11	1·499 (1)
C2—C1—C5	104·84 (9)	C6—C7—C12	108·0 (1)
C2—C1—C6	127·59 (9)	C8—C7—C12	114·6 (1)
C5—C1—C6	127·56 (9)	C7—C8—C9	114·5 (1)
C1—C2—C3	108·5 (1)	C8—C9—C10	112·4 (1)
C2—C3—C4	109·1 (1)	C9—C10—C11	114·88 (9)
C3—C4—C5	109·1 (1)	C6—C11—C10	109·05 (9)
C1—C5—C4	108·3 (1)	C6—C11—C14	108·42 (9)
C1—C6—C7	124·37 (9)	C10—C11—C14	114·24 (9)
C1—C6—C11	124·65 (9)	C7—C12—C13	114·6 (1)
C7—C6—C11	110·95 (9)	C12—C13—C14	112·3 (1)
C6—C7—C8	109·0 (1)	C11—C14—C13	114·4 (1)
C1—C6—C7—C8	119·8 (2)	C1—C6—C7—C12	−115·1 (2)
C11—C6—C7—C8	−62·2 (2)	C6—C7—C8—C9	53·0 (2)
C12—C7—C8—C9	−68·2 (2)	C6—C7—C12—C13	−53·5 (2)
C7—C8—C9—C10	−44·0 (2)	C8—C9—C10—C11	43·8 (2)

cyclopentadiene with pyrrolidine as a catalyst in methanol (Stone & Little, 1984). Crystals from the reaction solvent, m.p. 321–322 K, were suitable; a yellow crystal with dimensions 0·20 × 0·48 × 0·55 mm was mounted in a capillary on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 25°$. The ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0·46–3·30° min^{-1}. Data having $2 \leq \theta \leq 75°$, $0 \leq h \leq 13$, $0 \leq k \leq 15$, $-11 \leq l \leq 11$ were measured, and background, Lorentz, polarization and absorption corrections were applied. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 0·958. Three standard reflections (400, 060, 002) showed only random fluctuations of intensity so no correction for decay was applied. Equivalent $0kl$ and $0k\bar{l}$ data were averaged, $R_{int} = 0{\cdot}022$. The space group is uniquely determined by systematic absences $h0l$ with 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 $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors from Cromer & Waber (1974), anomalous coefficients from Cromer (1974), 664 unobserved data. C-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0{\cdot}042$ (0·066 for all data), $wR = 0{\cdot}052$, $S = 2{\cdot}604$ for 200 variables. Maximum shift $0{\cdot}06\sigma$ in the final cycle, max., min. residual density 0·14, $-0{\cdot}11$ e Å^{-3}, extinction coefficient $g = 1{\cdot}08\,(4) \times 10^{-5}$ where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the C atoms are given in Table 1. Fig. 1 is a structural diagram and Fig. 2 is a perspective drawing showing the atomic numbering scheme. Bond distances, angles and selected torsion angles are presented in Table 2.*

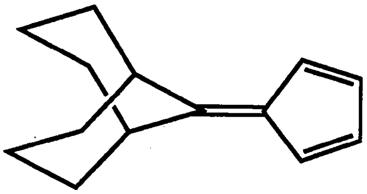

Fig. 1. 9-(2,4-Cyclopentadien-1-ylidene)bicyclo[3.3.1]nonane.

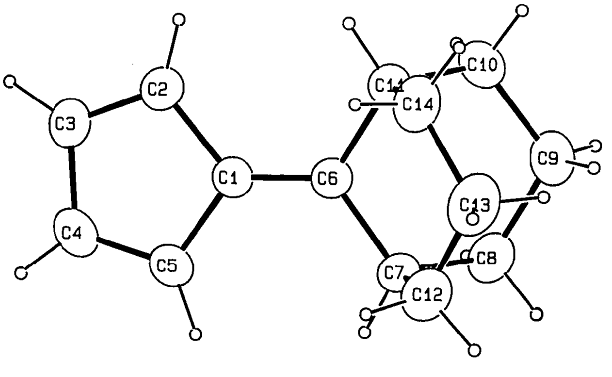

Fig. 2. *ORTEP* (Johnson, 1965) drawing of the molecule. C atoms are represented by 40% probability ellipsoids and H atoms by circles of arbitrary radius.

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* Lists of H-atom coordinates and thermal parameters, anisotropic thermal parameters, bond distances and angles involving H atoms, structure-factor amplitudes, torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52618 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structures of dimethylfulvene at 248 K (Norman & Post, 1961) and 2-(2,4-cyclopentadien-1-ylidene)adamantane or adamantylidenefulvene (Garcia, McLaughlin & Fronczek, 1989) exhibit the expected single–double bond alternation within the fulvene ring system as does the title compound. The bond angle C7—C6—C11 = 110·95 (9)° of the title compound is similar to the analogous bond angle in adamantylidenefulvene, 111·5 (2)°, and smaller than that of dimethylfulvene, 114·0 (6)°, indicative of ring strain.

The bicyclo[3.3.1]nonane ring system of the title molecule exhibits a close intramolecular contact, 1·95 (2) Å, between the axial C9 and C13 H atoms in the twin chair conformation similar to those observed by Watson, Grossie & Taylor (1982), Sim (1983), and Narasimhan & Chacko (1984). The true internuclear H···H distance is even shorter, since the X-ray experiment underestimates C—H distances, and the C—H bonds are convergent.

We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, the LSU-Center for Energy Studies, and the Louisiana Educational Quality Support Fund Grant # LEQSF(1987-90)-RD-A-5 for support of this research.

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Acta Cryst. (1990). C46, 1183–1184

Hydroquinone–1,4-Dioxane (1/1)

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(*Received* 27 *November* 1989; *accepted* 6 *February* 1990)

Abstract. $C_6H_6O_2.C_4H_8O_2$, $M_r = 198{\cdot}22$, monoclinic, $P2_1/a$, $a = 7{\cdot}388\ (2)$, $b = 7{\cdot}684\ (1)$, $c = 9{\cdot}264\ (2)$ Å, $\beta = 78{\cdot}83\ (2)°$, $V = 515{\cdot}95\ (19)$ Å^3, $Z = 2$, $D_x = 1{\cdot}37$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}09$ mm^{-1}, $F(000) = 212$, $T = 293$ K, $R = 0{\cdot}048$ for 601 reflections. The complex occurs as chains parallel to **c** with O···H—O bonding [2·722 (4) Å]. O···H 1·830 (5) Å, with O···H—O 168·8 (6)°. The torsion angles H···O—C—C average 25·7 (15)°. The short C—C distance observed in the dioxan molecule [1·489 (2) Å] may indicate some disorder.

Experimental. This previously unreported complex was prepared by allowing a solution of hydroquinone in 1,4-dioxane to evaporate at room temperature. Transparent colourless crystals up to 2 mm along their edges are stable indefinitely when stored under mother liquor but become opaque, losing dioxane within minutes when exposed to air at room temperature. Even specimens mounted dry in Lindemann-glass capillaries decomposed completely overnight. The crystal (dimensions 0·20 × 0·35 × 0·43 mm) used for data collection was mounted in a capillary partially filled with mother liquor not in contact with the crystal. This crystal showed no significant change in the intensities of three standard reflections checked hourly during data collection. Unit-cell dimensions were refined from 20 accurately centred reflections with $\theta \simeq 12°$ on an Enraf–Nonius CAD-4 diffractometer. Data were collected for a hemisphere of reciprocal space to $\theta = 25°$. 1367 measured reflections gave 764 unique data of which 601 with $F_o \geq 2\sigma(F)$ were used in the final refinement. $R_{\text{int}} = 0{\cdot}015$. Ranges of indices $-8 \leq h \leq$

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