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Structure of 2′,2′-Dimethylspiro[bicyclo[2.2.1]hept-5-ene-7,1′-cyclopropane]-2,3-dicarbonitrile, $C_{13}H_{14}N_2$

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Abstract. $M_r = 198{\cdot}13$, monoclinic, $P2_1/c$, $a = 10{\cdot}973\ (6)$, $b = 7{\cdot}611\ (3)$, $c = 13{\cdot}842\ (3)$ Å, $\beta = 91{\cdot}27\ (3)°$, $U = 1155{\cdot}7\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}14$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 6{\cdot}4$ mm^{-1}, $F(000) = 424$, $T = 298$ K. Final $R = 0{\cdot}092$ for 1285 observed unique reflections. The structure of the title compound has been established and correlates well with the observed NMR data. The bond lengths and angles are normal.

Introduction. During the course of our photochemical studies, we obtained a crystalline 1:1 adduct from irradiation (254 nm) of 3,3-dimethyl-6-methylenecyclohexa-1,4-diene (0·06 mol dm^{-3}) in acetonitrile in the presence of fumaronitrile (0·14 mol dm^{-3}). The ^{1}H NMR spectrum and other data did not distinguish among possible isomers. This X-ray structural study was undertaken to establish the structure.

Experimental. Platy crystals, dimensions 0·10 × 0·83 × 0·77 mm. Syntex $P2_1$ four-circle diffractometer. $2\theta_{max} = 50°$, scan range ± 1·0° (2θ) around $K\alpha_1$–$K\alpha_2$ angles, scan speed 2–29° min^{-1}, depending upon intensity of a 2 s pre-scan; backgrounds at each end of scan for 0·25 scan time. Three standard reflections monitored every 100 reflections showed irregularities during data collection (probably due to counter instability of electronic origin); data rescaled using a sliding point-to-point scale to correct for this. Unit-cell dimensions and standard deviations by least-squares fit to 15 high-angle reflections. 2299 reflections measured, 2023 unique, 738 unobserved [$I/\sigma(I) < 3{\cdot}0$]. Range of hkl: h −12→13, k 0→8, l 0→16. No absorption correction. Systematic absences indicated space group $P2_1/c$. Structure solution readily achieved using direct-methods link of *SHELXTL* (Sheldrick, 1981). Refinement by cascaded least squares on F (unit weights) using anisotropic temperature factors for all non-H atoms. H atoms (fixed isotropic temperature factors of 0·07–0·08 Å^2) inserted at calculated positions, with methyl groups treated as rigid bodies. Final $R = 0{\cdot}092$, $S = 1{\cdot}6$. The relatively high R value is attributed to the counter instability already noted. As the structure solution proceeded satisfactorily and bond e.s.d.'s were not excessive, it was not felt that recollection would be worthwhile. $(\Delta/\sigma)_{max} = 0{\cdot}18$. Max. and min. height in final ΔF map 0·3 and −0·4 e Å^{-3}. Computing with *SHELXTL* on a Data General Nova 3. Scattering factors from *International Tables for X-ray Crystallography* (1974).

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Discussion. The X-ray structure solution revealed that the compound had the title formula with structure

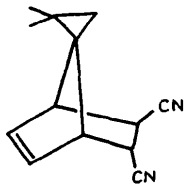

Fig. 1 shows an *ORTEP* view (Johnson, 1976) of the molecule. Final atomic coordinates are in Table 1 with bond lengths and angles in Table 2.* The dimensions of the molecule are standard. This structure correlates well with the observed NMR data† and the compound has since been synthesized unambiguously by a Diels–Alder reaction between fumaronitrile and 1,1-dimethylspiro[4.2]hepta-4,6-diene (Schröer & Friedrichsen, 1978). The latter compound is a known product of the irradiation of the methylenecyclohexadiene (Zimmerman, Juers, McCall & Schröder, 1971).

We thank the SERC for support.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39292 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† ^{1}H NMR ($CDCl_3$): δ 0·475 (1H, *d*, J = 5·7 Hz, 3′–H), 0·732 (1H, *d*, J = 5·7 Hz, 3′–H), 1·063 (3H, *s*, Me), 1·071 (3H, *s*, Me), 2·585 (1H, *d*, J = 4·3 Hz, 2–H), 2·900 (1H, *m*, 1 or 4–H), 3·021 (1H, *m*, 1 or 4–H), 3·198 (1H, *dd*, J = 4·3, 4·2 Hz, 3–H), 6·469 p.p.m. (2H, *m*, 5,6–H).

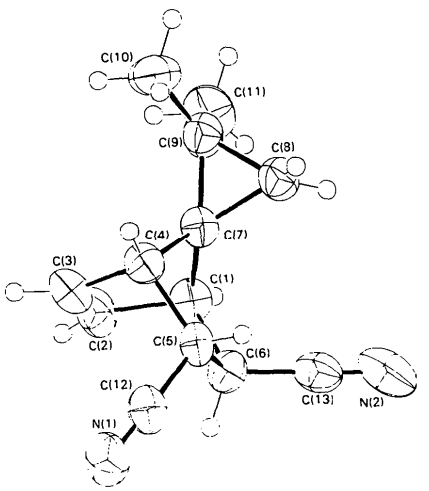

Fig. 1. View of the molecule, showing the atomic numbering.

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2 $\times 10^3$)

	x	y	z	U_{eq}*
N(1)	6158 (6)	5518 (9)	1743 (5)	67 (3)
N(2)	3608 (6)	10075 (10)	−652 (5)	77 (3)
C(1)	2398 (6)	6361 (9)	391 (5)	47 (2)
C(2)	2583 (6)	4505 (10)	819 (6)	58 (3)
C(3)	2962 (6)	4660 (10)	1713 (5)	52 (3)
C(4)	3073 (6)	6598 (9)	1950 (5)	45 (2)
C(5)	4184 (6)	7323 (9)	1360 (4)	43 (2)
C(6)	3729 (6)	7116 (9)	278 (4)	45 (2)
C(7)	2057 (6)	7358 (9)	1313 (4)	43 (2)
C(8)	1609 (6)	9245 (10)	1339 (5)	52 (3)
C(9)	769 (6)	7740 (10)	1655 (5)	55 (3)
C(10)	528 (7)	7513 (13)	2719 (5)	67 (3)
C(11)	−287 (7)	7307 (13)	969 (7)	73 (3)
C(12)	5321 (7)	6334 (10)	1579 (5)	50 (2)
C(13)	3686 (6)	8810 (11)	−230 (5)	54 (3)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

N(1)–C(12)	1·128 (10)	N(2)–C(13)	1·128 (11)
C(1)–C(2)	1·544 (10)	C(1)–C(6)	1·581 (10)
C(1)–C(7)	1·539 (9)	C(2)–C(3)	1·303 (11)
C(3)–C(4)	1·515 (10)	C(4)–C(5)	1·583 (9)
C(4)–C(7)	1·521 (9)	C(5)–C(6)	1·577 (9)
C(5)–C(12)	1·483 (10)	C(6)–C(13)	1·469 (11)
C(7)–C(8)	1·518 (10)	C(7)–C(9)	1·527 (10)
C(8)–C(9)	1·540 (10)	C(9)–C(10)	1·513 (11)
C(9)–C(11)	1·518 (11)		
C(2)–C(1)–C(6)	104·9 (5)	C(2)–C(1)–C(7)	99·5 (5)
C(6)–C(1)–C(7)	98·4 (5)	C(1)–C(2)–C(3)	108·5 (6)
C(2)–C(3)–C(4)	108·5 (6)	C(3)–C(4)–C(5)	106·7 (5)
C(3)–C(4)–C(7)	101·0 (5)	C(5)–C(4)–C(7)	97·5 (5)
C(4)–C(5)–C(6)	102·9 (5)	C(4)–C(5)–C(12)	111·8 (5)
C(6)–C(5)–C(12)	112·9 (5)	C(1)–C(6)–C(5)	102·5 (5)
C(1)–C(6)–C(13)	110·3 (6)	C(5)–C(6)–C(13)	111·9 (6)
C(1)–C(7)–C(4)	96·0 (5)	C(1)–C(7)–C(8)	124·8 (6)
C(4)–C(7)–C(8)	125·4 (6)	C(1)–C(7)–C(9)	126·5 (6)
C(4)–C(7)–C(9)	124·3 (6)	C(8)–C(7)–C(9)	60·7 (5)
C(7)–C(8)–C(9)	59·9 (5)	C(7)–C(9)–C(8)	59·3 (5)
C(7)–C(9)–C(10)	117·6 (6)	C(8)–C(9)–C(10)	118·7 (7)
C(7)–C(9)–C(11)	117·6 (6)	C(8)–C(9)–C(11)	115·9 (7)
C(10)–C(9)–C(11)	115·9 (6)	N(1)–C(12)–C(5)	177·1 (8)
N(2)–C(13)–C(6)	176·4 (7)		

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