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1,6,7,7-Tetrachloro-5-methyl-2-phenyl-3-azabicyclo[4.1.0]heptan-4-one

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Abstract. C₁₃H₁₁Cl₄NO, $M_r = 339.05$, triclinic, $P\bar{1}$, $a = 8.180$ (1), $b = 8.318$ (1), $c = 11.542$ (2) Å, $\alpha = 87.85$ (1), $\beta = 96.32$ (2), $\gamma = 110.27$ (2)°, $V = 732.2$ (2) Å³, $Z = 2$, $D_x = 1.54$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 7.99$ cm⁻¹, $F(000) = 344$, $T = 291$ K, $R = 0.031$ for 1862 observed reflections. The configuration of the bicyclic system is *exo*. The six-membered piperidinone adopts a slightly distorted boat conformation with endocyclic torsion angles of -50 (1), 57 (1), -7 (1), -47 (1), 48 (1) and 0 (1)°. The three cyclopropane bonds have very similar lengths: 1.520 (3), 1.511 (3) and 1.521 (3) Å. Dimers are formed through hydrogen bonds between the piperidyl hydrogen and the carbonyl oxygen: $N\cdots O = 2.908$ (3), $H\cdots O = 2.08$ (2) Å and $N-H\cdots O = 168.5$ (2)°.

Experimental. The title compound results from a [4 + 2] cycloaddition of 1-phenyl-3-trimethylsilyloxy-2-aza-1,3-pentadiene to tetrachlorocyclopropene. Since stereochemical assignments based on ¹H NMR coupling constants were uncertain, an X-ray analysis was undertaken in order to assess the configuration of the bicyclic system.

Crystals were obtained by evaporation from acetonitrile. D_m not measured. Parallelepiped crystal of dimensions $0.22 \times 0.20 \times 0.40$ mm. Lattice parameters refined using 17 reflections in the range $5 \leq 2\theta \leq 25^\circ$. Huber four-circle diffractometer, monochromatized $Mo K\alpha$ radiation. ω scans. $2168 h \pm k \pm l$ independent reflections with $\sin\theta/\lambda \leq 0.56$ Å⁻¹; $0 \leq h \leq 9$, $-9 \leq k \leq 9$, $-12 \leq l \leq 12$, 1862 with $I \geq 2.5\sigma(I)$. Standard reflection 114 checked every 50

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (Å²)

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

	x	y	z	B_{eq}
C1	2748 (3)	7472 (3)	2365 (2)	2.95 (4)
C2	3108 (3)	8555 (3)	1255 (2)	2.81 (4)
N3	1549 (2)	9030 (3)	928 (2)	3.09 (3)
C4	1015 (3)	9868 (3)	1687 (2)	3.04 (4)
C5	1997 (3)	10067 (3)	2905 (2)	2.96 (4)
C6	2124 (3)	8325 (3)	3282 (2)	3.05 (4)
C7	3878 (3)	8052 (3)	3503 (2)	3.55 (4)
C18	1743 (1)	5275 (1)	2117 (1)	4.23 (1)
C19	5839 (1)	9814 (1)	3523 (1)	4.73 (1)
C110	4120 (1)	6519 (1)	4513 (1)	5.60 (2)
C111	438 (1)	7066 (1)	4083 (1)	4.67 (1)
C12	1234 (4)	10905 (4)	3752 (2)	3.87 (5)
O13	-149 (2)	10478 (3)	1432 (1)	4.33 (4)
C14	3625 (3)	7731 (3)	277 (2)	3.06 (4)
C15	5334 (3)	7728 (3)	354 (2)	3.63 (5)
C16	5871 (4)	6996 (3)	-517 (3)	4.36 (5)
C17	4735 (4)	6286 (4)	-1479 (3)	4.71 (6)
C18	3057 (4)	6283 (4)	-1566 (3)	5.46 (7)
C19	2488 (4)	6994 (4)	-689 (2)	4.51 (5)

reflections: no significant deviation. Structure solved by *SHELXS86* (Sheldrick, 1985). All H atoms from difference Fourier synthesis; anisotropic least-squares refinement (*SHELX76*; Sheldrick, 1976) using F ; H atoms isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0.00096F^2)$. $R = 0.031$, $wR = 0.038$ for 1862 observed reflections. Final max. shift/e.s.d. = 0.32 (U_{22} of C12). $S = 1.30$. Max. and min. heights in final difference Fourier synthesis = 0.23 and -0.18 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 2. Bond distances (Å) and angles (°)

C2—C1	1.534 (3)	C6—C1	1.520 (3)
C7—C1	1.511 (3)	C18—C1	1.745 (2)
N3—C2	1.468 (3)	C14—C2	1.513 (3)
C4—N3	1.336 (3)	C5—C4	1.525 (3)
O13—C4	1.230 (3)	C6—C5	1.532 (3)
C12—C5	1.522 (3)	C7—C6	1.521 (3)
C11—C6	1.750 (2)	C19—C7	1.757 (2)
C10—C7	1.743 (2)	C15—C14	1.392 (3)
C19—C14	1.383 (3)	C16—C15	1.379 (4)
C17—C16	1.374 (4)	C18—C17	1.365 (4)
C19—C18	1.389 (4)		
C6—C1—C2	112.3 (2)	C7—C1—C2	121.3 (2)
C7—C1—C6	60.3 (1)	C18—C1—C2	114.4 (2)
C18—C1—C6	119.4 (2)	C18—C1—C7	118.1 (2)
N3—C2—C1	106.0 (2)	C14—C2—C1	115.0 (2)
C14—C2—N3	113.7 (2)	C4—N3—C2	119.9 (2)
C5—C4—N3	115.0 (2)	O13—C4—N3	123.4 (2)
O13—C4—C5	121.6 (2)	C6—C5—C4	108.0 (2)
C12—C5—C4	112.4 (2)	C12—C5—C6	115.7 (2)
C5—C6—C1	113.7 (2)	C7—C6—C1	59.6 (1)
C7—C6—C5	121.8 (2)	C11—C6—C1	119.0 (2)
C11—C6—C5	114.5 (2)	C11—C6—C7	117.2 (2)
C19—C7—C1	119.8 (2)	C19—C7—C6	120.0 (2)
C19—C7—C10	110.2 (1)	C10—C7—C1	119.2 (2)
C6—C7—C1	60.2 (1)	C15—C14—C2	118.1 (2)
C10—C7—C6	119.6 (2)	C19—C14—C15	118.6 (2)
C19—C14—C2	123.2 (2)	C17—C16—C15	120.4 (2)
C16—C15—C14	120.3 (3)	C19—C18—C17	120.5 (3)
C18—C17—C16	119.8 (3)		
C18—C19—C14	120.3 (2)		

The atomic parameters are given in Table 1.* The bond lengths and angles are listed in Table 2. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978).

Related literature. Only one structure containing the 3-azabicyclo[4.1.0]heptan-4-one skeleton is classified

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54011 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Model for Spirochlorine (Antibiotic A30641)

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Abstract. 2*H*-5*a*,10*a*-Dihydro-10*a*,3-(iminomethano)-11-methoxy-2(4-methoxybenzyl)-1-benzofuran-

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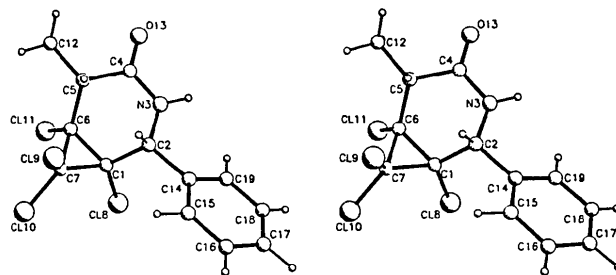


Fig. 1. Stereoscopic view of the molecule.

in the Cambridge Structural Database (Spek, 1978; Allen, Kennard & Taylor, 1983).

Substituent-induced bond-length asymmetry in cyclopropane derivatives has been extensively analysed (Allen, 1980; Schruppf & Jones, 1987). For chloro-substituted cyclopropanes, a lengthening of the distal bond is predicted. The geometry observed for the title derivative does not follow this expectation since we observe a symmetrical three-membered ring with C—C distances of 1.520 (3), 1.511 (3) and 1.521 (3) Å.

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