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4-Methyl-10-phenyl-1,4,8,10-tetraazabicyclo[5.3.0]dec-7-en-9-one

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Abstract. $C_{13}H_{16}N_4O$, $M_r = 244.30$, triclinic, $P\bar{1}$, $a = 7.986$ (1), $b = 8.941$ (1), $c = 10.409$ (2) Å, $\alpha = 114.59$ (1), $\beta = 103.78$ (1), $\gamma = 100.10$ (1)°, $V = 623.9$ (2) Å^3, $Z = 2$, $D_x = 1.300$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.51$ cm^{-1}, $F(000) = 260$, $T = 293$ K, $R = 0.034$ for 1591 observed reflections. The structure consists of antiparallel molecules which are stacked in columns parallel to [100]. Eight of the eleven intermolecular distances ≤3.6 Å are intracolumnar. The phenyl group is nearly planar, the maximum torsion angle being 2.2° (mean 1.4°) and the greatest deviation of an atom from the least-squares plane is 0.013 Å (mean 0.009 Å). The five-membered ring is less planar, maximum torsion angle 7° (mean 4.7°), greatest deviation from least-squares plane 0.037 Å (mean 0.025 Å). The seven-membered ring adopts a chair-like conformation.

Experimental. The title compound belongs to the class of 1,5-heteroannulated 1,2-dihydro-2-phenyl-3*H*-1,2,4-triazol-3-ones and the synthesis has been achieved by rearrangement of 4′,5′-dihydro-1-methyl-5′-oxo-1′-phenyl-spiro[piperidinium-4,3′-3′*H*-[1′,2′,4′]triazolium] bis(tetrafluoroborate) (Seil, 1989; Gstach & Seil, 1990). Crystals were grown from methanol. Approximate crystal dimensions 0.2 × 0.3 × 0.5 mm, Philips PW 1100 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ-scan mode, scan width 1°, scan speed 1.2° min^{-1}, $4 < 2\theta < 48°$, $-9 \le h \le 9$, $-10 \le k \le 10$, $-11 \le l \le 11$, 3918 reflections collected, 1959 independent reflections ($R_{int} = 0.018$), 1591 with $I > 3\sigma(I)$ observed. Three standard reflections (113, 123, 222) every 2 h, 2% variation in intensity. Data corrected for Lorentz and polarization effects, no correction for absorption. Accurate cell dimensions by least-squares fitting of 54 reflections with $10 \le 2\theta \le 40°$.

The structure was solved by direct methods and difference Fourier syntheses, and refined by least-squares methods based upon F with weights $w = [\sigma^2(F_o) + 0.00024F_o^2]^{-1}$ (Sheldrick, 1976), H atoms included using a riding model (C—H = 1.08 Å, H—C—H = 109.5°, phenyl H atoms on the external bisector of the appropriate C—C—C angle). C, N and O atoms anisotropic, H atoms isotropic. The scattering functions used were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.034$, $wR = 0.044$, maximum and minimum electron density in final difference Fourier map 0.12 and −0.14 e Å^{-3}, respectively. $(\Delta/\sigma)_{max} = 0.1$. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1;* bond distances, angles and intermolecular distances

Table 1. *Atomic coordinates and isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}(Å^2 × 10^3)
O	0.2087 (2)	−0.2981 (2)	0.3719 (1)	66 (1)
N(1)	0.2110 (2)	−0.0449 (2)	0.3592 (1)	50 (1)
N(2)	0.3346 (2)	−0.0251 (2)	0.5859 (1)	53 (1)
N(3)	0.2594 (2)	0.1269 (2)	0.4711 (1)	46 (1)
N(4)	0.3463 (2)	0.5220 (2)	0.6834 (2)	50 (1)
C(1)	0.0722 (2)	−0.1085 (2)	0.2185 (2)	45 (1)
C(2)	0.0742 (3)	−0.2492 (2)	0.0939 (2)	57 (1)
C(3)	−0.0662 (3)	−0.3197 (2)	−0.0424 (2)	62 (1)
C(4)	−0.2055 (2)	−0.2481 (2)	−0.0558 (2)	61 (1)
C(5)	−0.2027 (3)	−0.1047 (2)	0.0678 (2)	62 (1)
C(6)	−0.0661 (2)	−0.0351 (2)	0.2056 (2)	52 (1)
C(7)	0.2490 (2)	−0.1399 (2)	0.4345 (2)	50 (1)
C(8)	0.3407 (2)	0.1293 (2)	0.6006 (2)	47 (1)
C(9)	0.4309 (2)	0.2956 (2)	0.7432 (2)	52 (1)
C(10)	0.3291 (2)	0.4287 (2)	0.7689 (2)	55 (1)
C(11)	0.2383 (2)	0.4172 (2)	0.5233 (2)	54 (1)
C(12)	0.3051 (2)	0.2709 (2)	0.4385 (2)	52 (1)
C(13)	0.2919 (3)	0.6784 (2)	0.7469 (2)	71 (1)

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54507 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond lengths* (Å), *bond angles* (°) *and intermolecular distances* ≤3.6 Å *with e.s.d.'s in parentheses*

O—C(7)	1.225 (2)	N(4)—C(13)	1.470 (3)
N(1)—N(3)	1.398 (2)	C(1)—C(2)	1.386 (2)
N(1)—C(1)	1.417 (2)	C(1)—C(6)	1.387 (3)
N(1)—C(7)	1.401 (3)	C(2)—C(3)	1.384 (2)
N(2)—C(7)	1.378 (2)	C(3)—C(4)	1.385 (3)
N(2)—C(8)	1.315 (2)	C(4)—C(5)	1.379 (3)
N(3)—C(8)	1.339 (2)	C(5)—C(6)	1.382 (2)
N(3)—C(12)	1.471 (3)	C(8)—C(9)	1.486 (2)
N(4)—C(10)	1.464 (3)	C(9)—C(10)	1.526 (3)
N(4)—C(11)	1.460 (2)	C(11)—C(12)	1.511 (3)

C(1)—N(1)—N(3)	120.9 (1)	C(2)—C(3)—C(4)	120.5 (2)
C(1)—N(1)—C(7)	125.6 (1)	C(3)—C(4)—C(5)	119.4 (2)
N(3)—N(1)—C(7)	106.1 (1)	C(4)—C(5)—C(6)	120.9 (2)
C(7)—N(2)—C(8)	106.1 (2)	C(5)—C(6)—C(1)	119.3 (2)
N(1)—N(3)—C(8)	105.6 (1)	O—C(7)—N(1)	123.9 (1)
N(1)—N(3)—C(12)	121.0 (1)	O—C(7)—N(2)	128.0 (2)
C(8)—N(3)—C(12)	124.8 (1)	N(1)—C(7)—N(2)	108.1 (1)
C(10)—N(4)—C(11)	113.0 (1)	N(2)—C(8)—N(3)	113.6 (1)
C(10)—N(4)—C(13)	109.6 (2)	N(2)—C(8)—C(9)	125.6 (2)
C(11)—N(4)—C(13)	109.2 (1)	N(3)—C(8)—C(9)	120.8 (2)
N(1)—C(1)—C(2)	118.7 (2)	C(8)—C(9)—C(10)	115.7 (1)
N(1)—C(1)—C(6)	120.9 (1)	N(4)—C(10)—C(9)	113.8 (2)
C(2)—C(1)—C(6)	120.3 (1)	N(4)—C(11)—C(12)	114.3 (2)
C(1)—C(2)—C(3)	119.5 (2)	N(3)—C(12)—C(11)	111.6 (2)

Atom in molecule 1 at x, y, z	Atom in molecule 2	Distance (Å)	Molecule 2 at
N(2)	C(6)	3.354 (2)	$-x, -y, 1-z$
O	C(9)	3.371 (2)	$1-x, -y, 1-z$
C(7)	C(8)	3.372 (2)	$1-x, -y, 1-z$
O	C(3)	3.429 (2)*	$-x, -1-y, -z$
N(1)	N(2)	3.436 (1)	$1-x, -y, 1-z$
N(2)	C(7)	3.487 (2)	$1-x, -y, 1-z$
O	C(11)	3.520 (2)*	$x, -1+y, z$
C(6)	C(8)	3.530 (2)	$-x, -y, 1-z$
O	C(8)	3.543 (2)	$1-x, -y, 1-z$
N(4)	C(4)	3.543 (2)*	$1+x, 1+y, 1+z$
N(2)	N(2)	3.589 (1)	$1-x, -y, 1-z$

* Intercolumnar distance.

≤3.6 Å are given in Table 2. Fig. 1 shows the atom-numbering scheme of the molecule. The crystal packing is illustrated in Fig. 2.

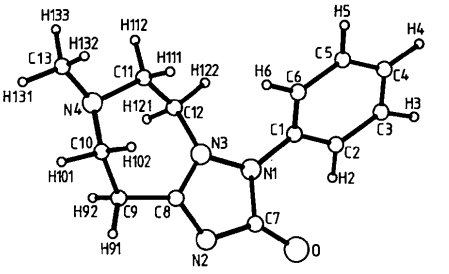

Fig. 1. Perspective view of the molecule showing the atom-numbering scheme. Radii are arbitrary.

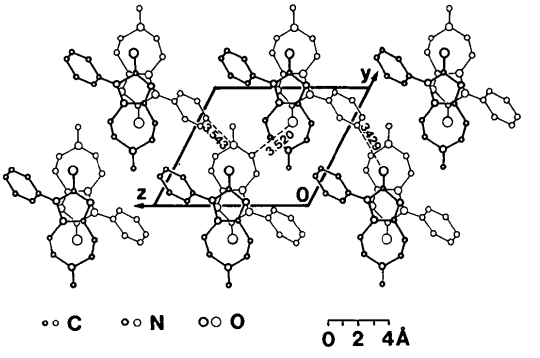

Fig. 2. Packing plot projected down x. H atoms omitted for clarity.

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8,12-Diethyl-2,3,7,13,17,18-hexamethyl-20-phenylporphyrin

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Abstract. $C_{36}H_{38}N_4$, $M_r = 526.7$, orthorhombic, *Iba*2, $a = 12.870$ (6), $b = 55.71$ (3), $c = 7.837$ (2) Å, $V = 5619$ (4) Å^3, $Z = 8$, $D_x = 1.245$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.7107$ Å, $\mu = 0.069$ mm^{-1}, $F(000) = 2256$, $T = 130$ K, $R = 0.076$ for 1428 independent reflections [$I > 2\sigma(I)$]. The title compound consists of the slightly

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