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The Crystal and Molecular Structures of 2,2,7,7-Tetramethyl-1,4-diaza-5-cycloheptanone (2,2,7,7-Tetramethyl-5-homopiperazinone) and the Corresponding Seven-Membered-Ring Nitroxide

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Abstract

The title compounds are, respectively, monoclinic, space group $P2_1/c$ [$a = 11.722$ (2), $b = 7.777$ (1), $c = 11.534$ (2) Å, $\beta = 109.88$ (1)°] and orthorhombic, space group $P2_12_12_1$ [$a = 8.123$ (1), $b = 10.858$ (2), $c = 11.561$ (1) Å]. The two molecules [(1) and (2) respectively] have flattened chair conformations. By hydrogen bonding, they are arranged in pairs in (1) and chains in (2). The geometrical parameters of the nitroxide group in (2) are compared with those reported earlier for five- and six-membered-ring nitroxides.

Much structural information about pyrrolidinoxyl (five-membered-ring) and piperidinoxyl (six-membered-ring) free radicals is available. However, no structural study has been reported on seven-membered-ring nitroxides. Since the reactivity and physical properties

of cyclic nitroxides, as in the corresponding cyclic amines, depend upon ring size, it is unfortunate that nothing is known about the structure and conformation of these free radicals.

The most common method used in the determination of molecular conformation is NMR spectroscopy which, however, gives rather qualitative data for molecules in solution and does not always lead to unequivocal answers. Furthermore, for paramagnetic species, it is often difficult to assign a resonance signal to the right proton group without selective deuterium labeling which requires much synthetic work. A quantitative determination of the shape of molecules in the solid state is possible by diffraction methods.

The simplest representative of seven-membered-ring nitroxides described up to now is 2,2,7,7-tetramethyl-5-oxohomopiperazin-1-oxyl (2), the synthesis of which was reported several years ago (Rozantsev & Papko, 1962, 1963). This compound is interesting, not only because of the oxyl group, but also because of its parent amine (1), both of which give general information on the 1,4-diazacycloheptane system.

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Reported here are the molecular structures of the amine (1) and the nitroxide (2). Three special features will be discussed. First, these two compounds, considered as models for the crowded 1,4-diazacycloheptane system, will be compared to the analogous hydrocarbon. Second, the changes of conformation between the nitroxide and the parent amine will be analyzed. Third, comparison with the five- and six-membered-ring nitroxides will show how the geometrical parameters of the C₂N—O group vary with the ring size.

Experimental

(1), m.p. 421 K, is readily obtained by Beckmann rearrangement of triacetonamine oxime.* Oxidation of (1) by hydrogen peroxide and phosphotungstic acid affords (2), m.p. 436 K, in quantitative yield. Single crystals of (1) (0.24 × 0.20 × 0.21 mm) and (2) (0.18 × 0.23 × 0.25 mm) giving Weissenberg photographs of a satisfactory quality were both recrystallized from hot benzene. The crystals were assigned to the monoclinic system, space group *P2₁/c* (*h0l* absent for *l* odd, and *0k0* for *k* odd), for (1), and to the orthorhombic system, space group *P2₁2₁2₁* (absence of *h00* for *h* odd, *0k0* for *k* odd and *00l* for *l* odd), for (2), on the basis of the Weissenberg photographs. The cell dimensions and their e.s.d.'s were determined by least squares from 25 reflexions measured with graphite-monochromatized Mo *K*α radiation on an Enraf-Nonius CAD-4 diffractometer. They are reported in Table 1. The intensities of 2020 independent reflexions ($3 < \theta < 30^\circ$) for (1), and 1727 for (2) were recorded. Standard reflexions were checked periodically but no discrepancies appeared. The intensities were corrected for the Lorentz-polarization factor but not for absorption, extinction or anomalous dispersion.

* It was not possible to reproduce the Rozantsev synthesis of (1). Our detailed synthetic procedure will be published elsewhere.

Table 1. *Crystal data for 2,2,7,7-tetramethyl-5-homopiperazinone (1) and 2,2,7,7-tetramethyl-5-oxo-homopiperazin-1-oxyl (2)*

(1) C ₉ H ₁₈ N ₂ O, space group <i>P2₁/c</i>	
<i>a</i> = 11.722 (2) Å	<i>V</i> = 988.8 Å ³
<i>b</i> = 7.777 (1)	<i>Z</i> = 4
<i>c</i> = 11.534 (2)	<i>T</i> = 293 K
β = 109.88 (1)°	<i>D_c</i> = 1.27 Mg m ⁻³
(2) C ₉ H ₁₇ N ₂ O ₂ , space group <i>P2₁2₁2₁</i>	
<i>a</i> = 8.123 (1) Å	<i>V</i> = 1019.7 Å ³
<i>b</i> = 10.858 (2)	<i>Z</i> = 4
<i>c</i> = 11.561 (1)	<i>T</i> = 293 K
	<i>D_c</i> = 1.21 Mg m ⁻³

Structure determination and refinement

Both structures were determined with *MULTAN* (Germain, Main & Woolfson, 1971). The solution was based on 200 reflexions with *E* > 1.64 for (1) and on 117 with *E* > 1.5 for (2). In both cases, all non-hydrogen atoms were located from the *E* map with the best figure of merit. The structures were refined with *ORFLS* (Busing & Levy, 1959) with the C, N, O atoms anisotropic until $R_w = \sqrt{\sum w(F_o - F_c)^2 / \sum wF_o^2}$ and $R = \sum |F_o - F_c| / \sum |F_o|$ reached 0.112 and 0.123 for (1), 0.080 and 0.085 for (2). At this stage all H atoms were located on a difference map. The final *R* values were 0.080 and 0.086 for (1), 0.064 and 0.067 for (2).*

The atomic coordinates are listed in Tables 2 and 3, bond lengths and angles in Tables 4 and 5.

* Lists of structure factors and anisotropic thermal parameters for both compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 34452 (20 pp.) Copies may be obtained through The Executive Secretary International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Fractional atomic coordinates (× 10⁴) for the non-hydrogen atoms, with their e.s.d.'s, in (1)*

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	2982 (4)	9575 (5)	9218 (3)
C(2)	1997 (4)	10858 (7)	8845 (4)
C(3)	790 (5)	10077 (7)	7968 (5)
N(4)	731 (4)	9785 (6)	6707 (4)
C(5)	1392 (4)	8635 (7)	6363 (5)
C(6)	2279 (5)	7560 (7)	7386 (5)
C(7)	3339 (4)	8530 (7)	8325 (4)
C(8)	4049 (5)	9586 (9)	7661 (5)
C(9)	4192 (5)	7154 (8)	9142 (5)
C(10)	1786 (6)	11402 (8)	10056 (5)
C(11)	2324 (5)	12478 (8)	8294 (5)
O(12)	1294 (3)	8430 (5)	5261 (3)

Table 3. *Fractional atomic coordinates (× 10⁴) for the non-hydrogen atoms, with their e.s.d.'s, in (2)*

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	3868 (7)	4121 (5)	3668 (5)
C(2)	2062 (8)	4361 (6)	3687 (6)
C(3)	1644 (8)	5666 (6)	3238 (6)
N(4)	2104 (6)	6651 (4)	4022 (5)
C(5)	3675 (8)	6977 (6)	4227 (6)
C(6)	4927 (8)	6335 (6)	3483 (6)
C(7)	5291 (8)	4989 (6)	3831 (6)
C(8)	5827 (9)	4922 (7)	5094 (7)
C(9)	6602 (10)	4520 (9)	3010 (9)
C(10)	1399 (10)	4174 (7)	4928 (7)
C(11)	1342 (9)	3424 (6)	2839 (7)
O(12)	4249 (7)	2987 (4)	3846 (6)
O(13)	4029 (6)	7759 (4)	4934 (5)

Table 4. Bond lengths (Å) with their *e.s.d.*'s

2,2,7,7-Tetramethyl-5-homopiperazinone (1)			
N(1)—C(2)	1.474 (6)	N(4)—C(5)	1.329 (8)
N(1)—C(7)	1.481 (7)	C(5)—O(12)	1.247 (7)
C(2)—C(3)	1.556 (7)	C(5)—C(6)	1.529 (7)
C(2)—C(10)	1.558 (8)	C(6)—C(7)	1.540 (7)
C(2)—C(11)	1.518 (8)	C(7)—C(8)	1.545 (9)
C(3)—N(4)	1.450 (8)	C(7)—C(9)	1.547 (7)
2,2,7,7-Tetramethyl-5-oxohomopiperazin-1-oxyl (2)			
N(1)—C(2)	1.489 (8)	C(3)—N(4)	1.450 (8)
N(1)—C(7)	1.504 (8)	N(4)—C(5)	1.345 (9)
N(1)—O(12)	1.287 (7)	C(5)—C(6)	1.503 (9)
C(2)—C(3)	1.548 (9)	C(5)—O(13)	1.213 (8)
C(2)—C(10)	1.546 (10)	C(6)—C(7)	1.545 (9)
C(2)—C(11)	1.528 (9)	C(7)—C(8)	1.525 (10)
		C(7)—C(9)	1.515 (10)

Table 5. Bond angles (°) with their *e.s.d.*'s

2,2,7,7-Tetramethyl-5-homopiperazinone (1)			
C(2)—N(1)—C(7)	123.2 (3)	N(4)—C(5)—C(6)	116.7 (5)
N(1)—C(2)—C(3)	112.0 (4)	N(4)—C(5)—O(12)	122.0 (4)
N(1)—C(2)—C(10)	105.6 (4)	C(6)—C(5)—O(12)	121.3 (5)
N(1)—C(2)—C(11)	113.5 (4)	C(5)—C(6)—C(7)	116.7 (4)
C(3)—C(2)—C(10)	107.6 (5)	N(1)—C(7)—C(6)	114.0 (4)
C(3)—C(2)—C(11)	110.8 (4)	N(1)—C(7)—C(8)	112.3 (5)
C(10)—C(2)—C(11)	106.8 (5)	N(1)—C(7)—C(9)	103.5 (4)
C(2)—C(3)—N(4)	115.8 (5)	C(6)—C(7)—C(8)	110.7 (4)
C(3)—N(4)—C(5)	124.8 (4)	C(6)—C(7)—C(9)	106.8 (4)
		C(8)—C(7)—C(9)	108.9 (4)
2,2,7,7-Tetramethyl-5-oxohomopiperazin-1-oxyl (2)			
C(2)—N(1)—C(7)	130.2 (5)	C(3)—N(4)—C(5)	123.1 (5)
C(2)—N(1)—O(12)	113.7 (5)	N(4)—C(5)—C(6)	114.8 (6)
C(7)—N(1)—O(12)	113.3 (5)	N(4)—C(5)—O(13)	121.8 (6)
N(1)—C(2)—C(3)	111.8 (5)	C(6)—C(5)—O(13)	123.4 (6)
N(1)—C(2)—C(10)	109.6 (6)	C(5)—C(6)—C(7)	114.8 (5)
N(1)—C(2)—C(11)	104.6 (5)	N(1)—C(7)—C(6)	114.4 (5)
C(3)—C(2)—C(10)	110.7 (5)	N(1)—C(7)—C(8)	108.0 (6)
C(3)—C(2)—C(11)	108.2 (5)	N(1)—C(7)—C(9)	104.5 (6)
C(10)—C(2)—C(11)	111.9 (6)	C(6)—C(7)—C(8)	110.4 (5)
C(2)—C(3)—N(4)	114.3 (5)	C(6)—C(7)—C(9)	106.9 (6)
		C(8)—C(7)—C(9)	112.5 (6)

Description of the structures and discussion

The amine (1) and nitroxide (2) are shown in Fig. 1 projected on their mean planes, with the numbering of the atoms. In Fig. 2 they are projected along the C(2)—C(7) direction.

In (1) and (2), the ring adopts, in the solid state, a chair conformation. The crystal structures, however, are very different. For (1), molecules are arranged in pairs around centers of symmetry; the amide group of one molecule is linked to the amide group of another by two hydrogen bonds (O—H = 1.994 Å; O—H—N = 167°). In (2) the molecules are arranged in chains; the amide group of one molecule now forms two hydrogen bonds with the amide group of two other molecules (O—H = 2.00 Å; O—H—N = 153°). These chains are

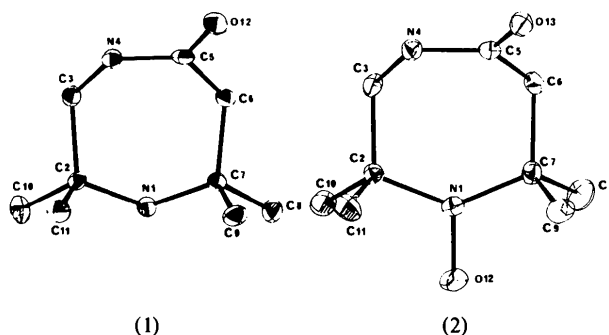


Fig. 1. Projections of (1) and (2) on their mean planes.

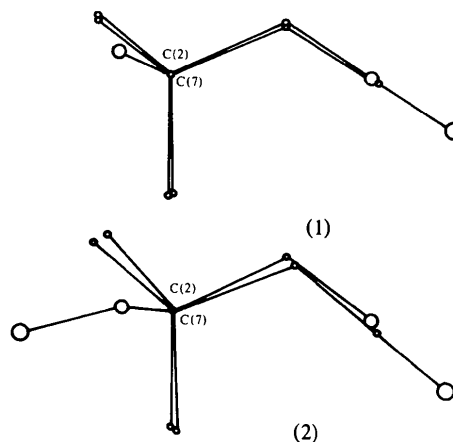


Fig. 2. Projections of (1) and (2) along the C(2)—C(7) direction.

approximately parallel to **a**, the mean plane of the molecules being roughly parallel to the *ab* plane. All other intermolecular distances were calculated up to 5 Å. With the van der Waals radii of Pauling (1960) no abnormal contacts were detected. Neither the N(1)—H in (1) nor the N(1)—O groups in (2) are engaged in hydrogen bonding. The crystal organization is shown in Fig. 3(a,b).

The molecular fragment C(2), C(3), N(4), C(5), C(6) is very similar in both compounds. Atoms 2, 3, 6 and 7 are almost in the same plane, and the distances of these atoms to their least-squares plane are < 0.01 Å in (1) and < 0.03 Å in (2). The angle between this plane and the C(2)—N(1)—C(7) plane is 132.2° in (1) and 152.9° in (2). Torsion angles are given in Table 6 and compared with those recently predicted for cycloheptane in the chair and twist-chair conformations (Bocian & Strauss, 1977).

The very low *t*₄ value is most likely due to the planarity of the amide group. The discrepancies observed for the other *t*'s are accounted for by the flattening of the chair, induced by the lowering of interaction between the methyl groups.

Table 6 shows that the nitroxide is more flattened at N(1) than is the corresponding amine; the

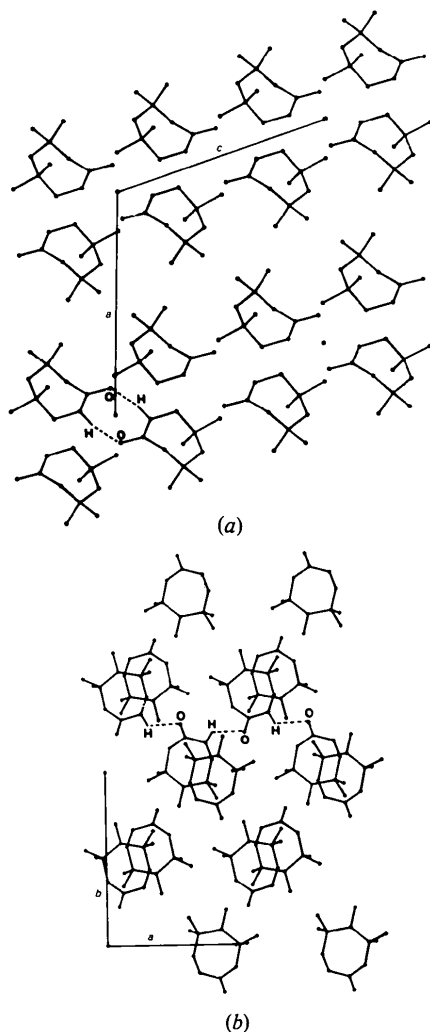


Fig. 3. Projection of the crystal structures on (a) the *ac* plane for (1) and (b) the *ab* plane for (2).

$C(2)-N(1)-C(7)$ angle is 123.1° for (1) and 130.2° for (2). The O atom adopts an equatorial position with an out-of-plane angle α of 20.6 (10) $^\circ$. In Fig. 4(a,b) some intramolecular distances depicting the environment of the N—H and the N—O groups are shown. The steric hindrance due to the flanking 2,2,7,7 methyl groups is very similar in both molecules.

The same intramolecular distances are reported in Fig. 4(c,d) for, respectively, a six- and a five-membered-ring nitroxide. The steric hindrance is similar in all these compounds. Although $\alpha,\alpha,\alpha',\alpha'$ -tetramethylamines have been modified without great difficulty in many chemical reactions, the corresponding nitroxides are unusually stable in many of these conditions. It is likely that the remarkable stability of the nitroxide group can be accounted for, in major part, by the intrinsic stability of the N—O three-electron bond (Linnett, 1961). This stability in solution, however,

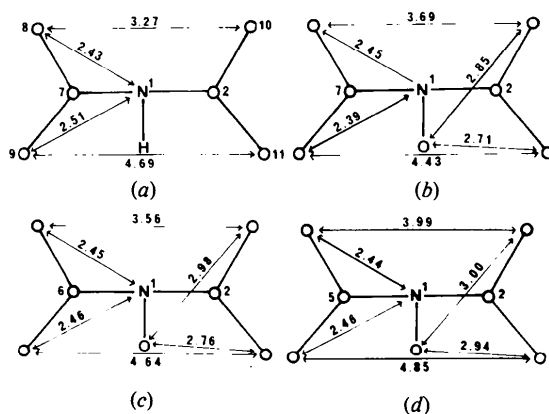


Fig. 4. Intramolecular distances depicting the C—N—C fragment with the flanking methyl groups. (a) (1); (b) (2) (this work); (c) 2,2,6,6-tetramethyl-4-hydroxypiperidin-1-oxyl (Berliner, 1970); (d) 2,2,5,5-tetramethyl-1-aza-3-cyclopentanone oxime (Chion & Thomas, 1975). E.s.d.'s are < 0.01 Å.

Table 6. Ring torsion angles ($^\circ$)

	Compound (1)	Compound (2)	Chair*	Twist-chair*
t_1^\dagger	53.4 (6)	31.7 (9)	63.8	-40.0
t_2	-75.4 (6)	-71.7 (6)	-85.6	89.4
t_3	67.3 (7)	70.8 (8)	68.5	-73.0
t_4	-0.3 (8)	6.5 (8)	-0.2	55.4
t_5	-65.7 (7)	-76.2 (7)	-68.3	-72.7
t_6	73.9 (6)	66.4 (7)	85.8	89.4
t_7	-52.8 (6)	-27.5 (9)	-63.9	-40.4

* Expected values for the chair and twist-chair conformations of cycloheptane (Bocian & Strauss, 1977).

$^\dagger t_i$ is the torsion angle around the bond between the *i*th and the (*i* + 1)th atoms (see Fig. 1).

Table 7. Geometrical parameters of nitroxides

	N—O	C—N—C	α	References
Open form	1.28 Å	136°	0°	(1)
Five-membered ring	1.27	112–117	0–5	(2) (3) (4) (5) (6)
Six-membered ring	1.27–1.31	123–126	15–20	(7) (8) (9) (10) (11) (12)
Seven-membered ring	1.287 (7)	130.2 (5)	20.6 (10)	(13)

References: (1) Andersen & Andersen (1966); (2) Turley & Boer (1972); (3) Ament, Wetherington, Moncrief, Flohr, Mochizuki & Kaiser (1973); (4) Boeyens & Kruger (1970); (5) Chion, Capiomont & Lajzerowicz (1972); (6) Bordeaux & Lajzerowicz (1977); (7) Capiomont, Bordeaux & Lajzerowicz (1972); (8) Berliner (1970); (9) Lajzerowicz Bonneteau (1968); (10) Bordeaux & Lajzerowicz (1974); (11) Shibaeva, Atovmjan, Neigauv, Novakovskaya & Ginzburg (1972); (12) Capiomont (1972); (13) this work.

decreases as the ring size increases (Rassat & Rey, to be published). *In solution*, large rings are more likely to have conformation changes, in which the steric hindrance of the N—O group is released; this leads to enhanced reactivity.

Other geometrical parameters of the N—O group in (2) are summarized in Table 7 with those previously published for some linear five- and six-membered-ring nitroxides.

The N—O length is almost the same for all compounds, whereas the C—N—C angle increases with the ring size. It is clear that increasing the ring size will make the value of this angle close to that found for the open form. Nevertheless, the values of the C—N—C angle, and of the out-of-plane angle α are directly related to the hybridization of the N atom and the spin density on that atom: ESR experiments show an increase of the N atom hyperfine splitting a_N with the ring size.

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Structure Cristalline et Moléculaire d'un Spirophosphorane à Liaison P—H Contenant les Ligands (+) ψ -Ephédrine et D-Phénylglycine: le Diméthyl-3,4 Diphényl-2,8 Dioxo-1,6 Diaza-4,9 Phospha(V)-5 Spiro[4.4]nonanone-7. Configuration Absolue de l'Atome de Phosphore

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Abstract

$C_{18}H_{21}N_2O_3P$, $M_r = 344.35$, is orthorhombic, space group $P2_12_12_1$ with $a = 11.315$ (2), $b = 17.455$ (3) and $c = 8.748$ (2) Å, $V = 1728$ Å³, $Z = 4$, $d_m = 1.30$ (2), $d_c = 1.311$ Mg m⁻³, $\mu(Cu K\alpha) = 1.55$ mm⁻¹. The

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crystal and molecular structure of the title compound separated in a pure enantiomeric form has been solved. The three-dimensional data were recorded at room temperature with a kappa-geometry diffractometer. The atomic parameters were refined by full-matrix least-squares calculations; the final R index is 0.056.

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