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Crystal and molecular structure of 1-methyl-2,3,5,6-tetraphenylpiperidin-4-ol, $C_{30}H_{29}NO$

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Abstract

The structure of the the title compound, $C_{30}H_{29}NO$, was determined by X-rays. M_r = 419.57, triclinic, space group $P1$, a = 5.8922(12), b = 8.5855(11), c = 12.2216(20) Å, α = 78.145(12)°, β = 79.181(15)°, γ = 76.108(14)°, V_c = 581.1(1) Å^3, Z = 1, D_x = 1.199 Mg m^{-3}. Cu $K\alpha$ radiation (graphite crystal monochromator, λ = 1.54184 Å), μ(Cu $K\alpha$) = 5.17 cm^{-1}, T = 290K. Final conventional R-factor = 0.039, R_w = 0.042 for 2795 observed reflections and 377 variables. The structure was solved using MULTAN and DIRDIF. With a view to determining the conformational preference of the piperidine ring when it is highly sustituted with a bulky group like the phenyl group, the present X-ray investigation was undertaken.

Introduction

Conformationally homogeneous 4-piperidones containing an anchoring group (bulky group) yield on reduction axial and equatorial alcohols which are separable. The relative yields of these alcohols depend upon the nature of the reducing agent employed (Balasubramanian and Padma, 1963). Reduction of 2,6-diphenylpiperidone gives a mixture of *cis*, *cis*-2,6-diphenyl-4-piperidin-4(r)ol(equatorial) and *trans*, *trans*-2,6-diphenylpiperidin-4(r)ol (axial). How-

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ever, the reduction of 1-methyl-2,3,5,6-tetraphenyl-piperidin-4-one gave only one alcohol. The present work indicates the nature of preferred conformation of the piperidinol by the single-crystal X-ray structure determination. Previous studies indicate that most of the piperidine derivatives exist in chair conformation (Ramalingam *et al.*, 1979).

Experimental

Condensation of dibenzyl ketone with benzaldehyde and ammonium acetate afforded 2,3,5,6-tetraphenylpiperidin-4-one (m.p. 205–206°C. Analysis: $C_{29}H_{25}NO$ requires C 86.32, H 6.44; found C 86.35, H 6.27). *N*-Methylation was carried out by methyl iodide in acetone in the presence of anhydrous potassium carbonate to yield 1-methyl-2,3,5,6-tetraphenylpiperidin-4-one (m.p. 220–221°C. Analysis: $C_{30}H_{27}NO$ requires C 86.28, H 6.52; found C 86.33; H 6.47). Reduction of the above piperidone with sodium and *n*-butanol gave only one isomer of the alcohol, 1-methyl-2,3,5,6-tetraphenylpiperidin-4-ol (m.p. 265–267°C. Analysis: $C_{30}H_{29}NO$ requires C 85.88, H 6.97; found C 85.52, H 7.33). The OH frequency of the alcohol appears as a sharp band at 3550 cm^{-1} which corresponds to the O–H stretching frequency of equatorially oriented hydroxyl groups. The NMR spectrum could not be recorded since the compound is insoluble in most of the NMR solvents. A crystal of approximately $0.19 \times 0.22 \times 0.07$ mm was used during the measurements. Throughout the experiment Cu $K\alpha$ radiation was used with a graphite crystal monochromator on a Nonius CAD4 single-crystal diffractometer ($\lambda = 1.54184$ Å). The unit cell dimensions, $a = 5.8922(12)$, $b = 8.5855(11)$, $c = 12.2216(20)$ Å, $\alpha = 78.145(12)°$, $\beta = 79.181(15)°$, $\gamma = 76.108(14)°$, $V_c = 581.1(1)$ Å^3, were determined from the angular settings of 25 reflections. The space group was determined to be $P1$ from the structure determination. The intensity data of 4395 reflections (the full sphere up to $\theta = 65°$), *hkl* range $(-6,-10,-14)$–$(6,10,14)$, were measured, using the ω–2θ scan technique, with a scan angle of 1.50° and a variable scan rate with a maximum scan time of 60 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 0.99 and 1.04. On all reflections profile analysis was performed (Lehman and Larsen, 1974; Grant and Gabe, 1978); empirical absorption correction was applied, using psi-scans (North *et al.*, 1968), $\mu(\text{Cu } K\alpha) = 5.17$ cm^{-1} (correction factors were in the range 0.97–1.00). Some double measured reflections were averaged, $R_{\text{int}} = \Sigma(I - \langle I \rangle)/\Sigma I = 0.027$, resulting in 4328 unique reflections of which 2795 were observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied and the data were reduced to $|F_o|$ values.

The structure was solved using MULTAN (Main *et al.*, 1980). Isotropic least-squares refinement, using SHELX (Sheldrick, 1976), converged to R = 0.090. At this stage, an empirical absorption correction was applied (Walker and Stuart, 1983) which resulted in a further decrease of R to 0.080.

All hydrogen atoms were located by difference Fourier synthesis. During the final stages of the refinement the positional parameters of all atoms and the anisotropic thermal parameters of the nonhydrogen atoms were refined. The hydrogen atoms had fixed isotropic temperature factors of 0.05, 0.07, and 0.06 $Å^2$ for the central ring, the methyl group and oxygen, and the rest of the hydrogens respectively. The final conventional agreement factors were $R = 0.039$ and $R = 0.042$ for the 2795 observed reflections and 377 variables. The function minimized was $\Sigma w^*(F_o - F_c)^2$ with $w = 1/(\sigma^2|F_o| + 0.00040\ |F_o|^2)$ with $\sigma|F_o|$ from counting statistics. The maximum shift-over-error ratio in the last full-matrix least-squares cycle was less than 0.21. The final difference Fourier map showed no peaks higher than 0.25 e $Å^{-3}$. Atomic scattering factors were taken from the *International Tables for X-ray Crystallography* (1974).

Description of the Structure

Final positional and thermal parameters are given in Table 1. Molecular geometry data are collected in Tables 2 and 3. A stereoview of the molecule, showing the molecular configuration, is given in Fig. 1. The crystallographic numbering scheme is given in Fig. 2. The figures are drawn with the PLUTO program (Motherwell, 1976). The atomic arrangement in the molecule is almost centrosymmetric; nevertheless the geometries around N(1) and C(4) are significantly different and enable the assignment of N and O atomic species. The central six-membered ring exists in a chair conformation. Values for the main torsion angles and the angles between phenyl rings are given in Tables 4 and 5 respectively. No unusual geometric features are present in the molecule, which showed agreement with similar compounds found in the literature (Cigler, 1981).

An analysis of the torsion angles indicates that all of the substituents, the phenyl, methyl, and hydroxyl groups, are equatorially oriented. The torsion angle between the two C–H bonds is found to be close to 175°, indicating that all the piperidine ring H atoms are axially oriented. Similarly, the torsion angles involving the N atom indicate equatorial orientation of the *N*-methyl group. The torsion angles of O(41)–C(4)–C(3)–C(31) [61.7(3)°] and O(41)–C(4)–C(3)–H(3) [−64.(2)°] suggest that the –OH group is in equatorial disposition. Physicochemical and single crystal analysis confirm the chair conformation for the piperidine with all the substituents in the equatorial orientation.

Table 1. Fractional coordinates

Atom	x	y	z
N(1)	1.1370(4)	0.09002(26)	0.82657(17)
C(2)	0.9966(5)	0.2163(3)	0.89727(20)
C(3)	1.1299(4)	0.35610(27)	0.87478(19)
C(4)	1.1694(4)	0.4276(3)	0.75156(18)
C(5)	1.3017(4)	0.30043(28)	0.68679(19)
C(6)	1.1825(5)	0.1524(3)	0.70264(20)
C(11)	1.0105(6)	−0.0424(3)	0.84252(22)
C(21)	0.9649(4)	0.14444(27)	1.02033(19)
C(22)	0.7399(5)	0.1707(3)	1.08045(24)
C(23)	0.6986(6)	0.1141(4)	1.1929(3)
C(24)	0.8769(7)	0.0280(4)	1.24971(26)
C(25)	1.1116(6)	−0.0048(3)	1.19009(21)
C(26)	1.1511(5)	0.0639(3)	1.07311(20)
C(31)	0.9862(4)	0.4878(3)	0.94491(20)
C(32)	1.0744(5)	0.5174(3)	1.03188(23)
C(33)	0.9404(7)	0.6413(4)	1.0939(3)
C(34)	0.7149(7)	0.7210(4)	1.0689(3)
C(35)	0.6347(6)	0.6944(4)	0.9885(3)
C(36)	0.7643(5)	0.5799(3)	0.92039(24)
O(41)	1.3089(5)	0.55423(28)	0.74364(20)
C(51)	1.3488(4)	0.3671(3)	0.56027(20)
C(52)	1.5726(5)	0.3411(4)	0.49655(24)
C(53)	1.5998(6)	0.4032(4)	0.37984(24)
C(54)	1.4016(7)	0.4902(4)	0.32896(25)
C(55)	1.1916(7)	0.5096(5)	0.39038(27)
C(56)	1.1603(5)	0.4607(3)	0.50234(23)
C(61)	1.3180(5)	0.02628(28)	0.63714(19)
C(62)	1.5369(6)	−0.0568(4)	0.6595(3)
C(63)	1.6681(6)	−0.1743(4)	0.5999(3)
C(64)	1.5691(7)	−0.2085(4)	0.50578(26)
C(65)	1.3569(7)	−0.1222(4)	0.48465(24)
C(66)	1.2317(6)	−0.0107(3)	0.54853(22)

Anisotropic thermal parameters (× 100) for nonhydrogen atom

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	6.71(16)	5.12(12)	4.92(12)	−0.56(10)	−0.73(11)	−1.59(11)
C(2)	4.34(15)	4.76(13)	4.72(13)	−1.06(10)	−0.65(11)	−1.61(11)
C(3)	3.65(14)	3.95(12)	4.48(13)	−1.27(10)	−0.21(11)	0.29(10)
C(4)	3.77(13)	4.44(12)	4.02(12)	−0.49(10)	−0.65(10)	−0.90(11)
C(5)	3.73(14)	4.55(13)	4.25(13)	−0.23(10)	−1.12(11)	0.17(11)
C(6)	4.84(15)	5.23(14)	4.41(13)	0.78(11)	−2.18(11)	−2.40(12)
C(11)	6.60(19)	4.15(13)	4.47(14)	−0.93(11)	0.07(13)	−2.72(13)
C(21)	4.03(14)	4.12(12)	4.28(12)	−0.75(10)	0.91(11)	−1.32(11)
C(22)	5.32(18)	5.61(15)	5.80(16)	−1.24(13)	−0.72(13)	−0.21(13)
C(23)	6.45(23)	8.67(23)	7.48(22)	−2.39(18)	2.09(19)	−1.81(19)
C(24)	9.45(28)	9.38(24)	4.63(16)	−0.47(16)	1.26(18)	−4.29(21)
C(25)	7.85(21)	6.37(17)	3.97(13)	0.11(12)	−1.71(14)	−1.16(15)
C(26)	5.86(18)	5.75(16)	3.91(13)	0.19(11)	−0.32(12)	−1.62(14)
C(31)	3.84(14)	4.47(13)	5.11(14)	−0.84(11)	−0.11(11)	−1.08(11)
C(32)	6.20(19)	5.56(16)	5.45(15)	−1.25(12)	−1.28(14)	0.23(14)
C(33)	10.0(3)	7.63(21)	5.94(18)	−3.36(17)	0.86(18)	−1.36(20)

Table 1. (*Continued*)

	Anisotropic thermal parameters (× 100) for nonhydrogen atom					
Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(34)	6.92(24)	6.01(19)	8.05(23)	−0.34(17)	0.23(19)	0.40(17)
C(35)	4.98(19)	5.30(17)	10.52(27)	−1.79(17)	1.21(19)	−0.98(15)
C(36)	4.41(16)	4.78(14)	6.63(17)	−0.90(12)	−2.13(13)	−0.29(12)
O(41)	12.63(22)	7.47(15)	8.50(16)	−0.72(13)	0.41(15)	−4.80(15)
C(51)	4.66(16)	4.31(13)	5.03(14)	−0.85(10)	−2.22(12)	−0.59(11)
C(52)	4.58(16)	6.91(18)	6.43(17)	−1.98(14)	0.97(13)	−2.57(14)
C(53)	7.80(24)	8.39(20)	5.04(16)	−2.45(15)	1.50(16)	−4.20(19)
C(54)	9.06(27)	7.32(20)	4.38(15)	−0.52(14)	−1.94(17)	−2.77(19)
C(55)	8.41(26)	8.83(24)	6.26(20)	0.17(17)	−1.53(19)	−2.38(20)
C(56)	5.06(17)	5.23(15)	5.43(16)	−0.09(12)	−1.68(13)	−0.06(13)
C(61)	5.08(16)	4.07(13)	4.38(13)	0.25(10)	−1.35(12)	−1.11(11)
C(62)	6.56(21)	5.33(17)	9.83(25)	−1.53(16)	−1.94(18)	−0.94(15)
C(63)	6.15(21)	5.55(18)	9.62(24)	−0.28(17)	−2.47(18)	0.23(15)
C(64)	8.55(24)	5.05(16)	6.67(18)	−2.39(14)	2.27(17)	−2.83(16)
C(65)	9.71(26)	6.16(18)	5.16(16)	−0.28(14)	−2.95(17)	−0.67(18)
C(66)	6.62(20)	6.15(17)	4.64(14)	−0.83(12)	−1.61(14)	−1.49(15)

	Fractional coordinates for hydrogen atoms			
Atom	x	y	z	U_{iso} (× 100)
H(2)	0.820(4)	0.2614(27)	0.8695(18)	5.00
H(3)	1.289(4)	0.305(3)	0.8938(19)	5.00
H(4)	0.987(4)	0.4952(27)	0.7222(18)	5.00
H(5)	1.450(5)	0.255(3)	0.7118(20)	5.00
H(6)	1.019(4)	0.1959(27)	0.6639(19)	5.00
H(22)	0.613(5)	0.263(3)	1.0318(21)	6.00
H(23)	0.518(5)	0.160(3)	1.2368(21)	6.00
H(24)	0.808(5)	−0.021(3)	1.3325(21)	6.00
H(25)	1.267(5)	−0.038(3)	1.2372(20)	6.00
H(26)	1.316(5)	0.052(3)	1.0293(21)	6.00
H(32)	1.227(5)	0.448(3)	1.0670(21)	6.00
H(33)	0.979(5)	0.653(3)	1.1598(23)	6.00
H(34)	0.667(4)	0.813(3)	1.1285(21)	6.00
H(35)	0.473(5)	0.728(3)	0.9796(21)	6.00
H(36)	0.683(5)	0.534(3)	0.8814(21)	6.00
H(41)	1.241(5)	0.618(3)	0.6612(23)	7.00
H(52)	1.725(5)	0.299(3)	0.5342(21)	6.00
H(53)	1.748(5)	0.404(3)	0.3392(21)	6.00
H(54)	1.386(5)	0.528(3)	0.2553(24)	6.00
H(55)	1.062(5)	0.608(3)	0.3596(21)	6.00
H(56)	0.996(5)	0.492(3)	0.5493(22)	6.00
H(62)	1.592(4)	−0.049(3)	0.7394(22)	6.00
H(63)	1.827(5)	−0.271(3)	0.6249(20)	6.00
H(64)	1.713(5)	−0.286(3)	0.4684(22)	6.00
H(65)	1.237(5)	−0.158(3)	0.4311(22)	6.00
H(66)	1.063(5)	0.047(3)	0.5436(21)	6.00
H(111)	1.123(5)	−0.125(3)	0.8062(22)	7.00
H(112)	0.918(5)	−0.078(3)	0.9206(24)	7.00
H(113)	0.841(5)	0.018(3)	0.8290(23)	7.00

Table 2. Bond lengths (Å) with esd's in parentheses

C(4)-C(3)	1.502(3)	C(6)-C(5)	1.556(4)	C(21)-C(22)	1.381(4)	C(63)-C(64)	1.490(6)
C(4)-C(5)	1.463(3)	C(6)-C(61)	1.467(3)	C(21)-C(26)	1.347(4)	C(64)-C(65)	1.333(5)
C(4)-O(41)	1.491(4)	C(5)-C(51)	1.532(3)	C(22)-C(23)	1.357(4)	C(65)-C(66)	1.350(4)
C(3)-C(2)	1.539(4)	C(31)-C(32)	1.359(4)	C(23)-C(24)	1.350(5)	C(51)-C(52)	1.393(4)
C(3)-C(31)	1.548(3)	C(31)-C(36)	1.406(3)	C(24)-C(25)	1.429(5)	C(51)-C(56)	1.419(4)
C(2)-N(1)	1.514(3)	C(32)-C(33)	1.426(4)	C(25)-C(26)	1.429(3)	C(52)-C(53)	1.410(4)
C(2)-C(21)	1.499(3)	C(33)-C(34)	1.399(5)	C(61)-C(62)	1.363(4)	C(53)-C(54)	1.404(5)
N(1)-C(6)	1.495(3)	C(34)-C(35)	1.249(6)	C(61)-C(66)	1.399(4)	C(54)-C(55)	1.313(5)
N(1)-C(11)	1.465(4)	C(35)-C(36)	1.407(4)	C(62)-C(63)	1.365(5)	C(55)-C(56)	1.337(4)
C(4)-H(4)	1.17(2)	C(34)-H(34)	1.13(3)	C(26)-H(26)	1.01(3)	C(65)-H(65)	1.17(3)
C(3)-H(3)	0.99(3)	C(35)-H(35)	0.95(3)	C(11)-H(111)	0.96(3)	C(66)-H(66)	1.00(3)
C(2)-H(2)	1.11(3)	C(36)-H(36)	0.93(3)	C(11)-H(112)	1.03(3)	C(52)-H(52)	1.04(3)
C(6)-H(6)	1.10(3)	C(22)-H(22)	1.11(3)	C(11)-H(113)	1.04(3)	C(53)-H(53)	0.92(3)
C(5)-H(5)	0.94(3)	C(23)-H(23)	1.12(3)	C(62)-H(62)	1.11(3)	C(54)-H(54)	0.91(3)
O(41)-H(41)	1.14(3)	C(24)-H(24)	1.06(3)	C(63)-H(63)	1.13(3)	C(55)-H(55)	1.05(3)
C(32)-H(32)	1.06(3)	C(25)-H(25)	1.12(3)	C(64)-H(64)	1.04(3)	C(56)-H(56)	1.03(3)
C(33)-H(33)	0.91(3)						

Table 3. Selected bond angles (deg)

C(3)-C(4)-C(5)	109.7(2)	C(2)-N(1)-C(11)	109.1(2)
C(3)-C(4)-O(41)	105.6(2)	C(6)-N(1)-C(11)	108.0(2)
C(5)-C(4)-O(41)	110.8(2)	N(1)-C(6)-C(5)	107.7(2)
C(4)-C(3)-C(2)	112.3(2)	N(1)-C(6)-C(61)	113.1(2)
C(4)-C(6)-C(61)	110.7(2)	C(5)-C(6)-C(61)	113.2(2)
C(2)-C(3)-C(31)	108.2(2)	C(4)-C(5)-C(6)	114.1(2)
C(3)-C(2)-N(1)	107.5(2)	C(4)-C(5)-C(51)	111.9(2)
C(3)-C(2)-C(21)	111.2(2)	C(3)-C(4)-C(5)	109.7(2)
N(1)-C(2)-C(21)	111.2(2)	C(4)-O(41)-H(41)	89(2)
C(2)-N(1)-C(6)	114.8(2)	O(41)-C(4)-H(4)	107(2)

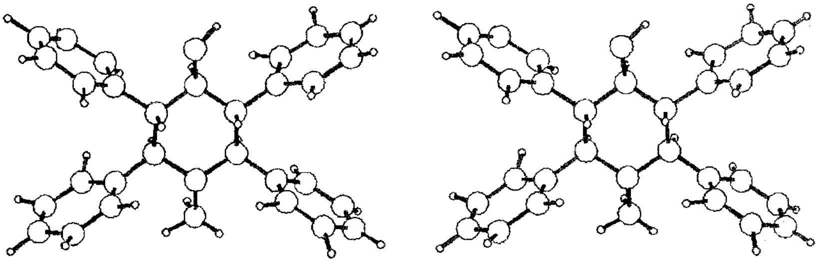

Fig. 1. Stereoview of the molecule.

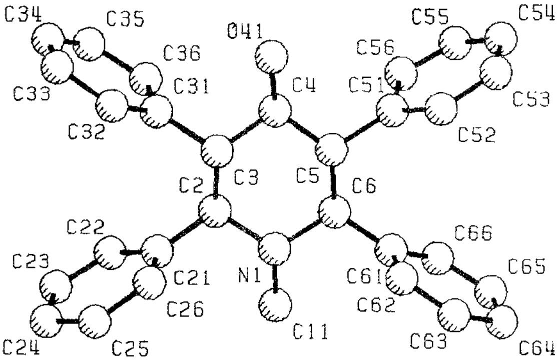

Fig. 2. Crystallographic atomic numbering scheme.

Table 4. Selected torsion angles (deg)

C(5)-C(4)-C(3)-C(2)	−58.0(3)	C(11)-N(1)-C(6)-C(5)	176.3(2)
C(5)-C(4)-C(3)-C(31)	−179.0(2)	N(1)-C(6)-C(5)-C(4)	−54.1(3)
O(41)-C(4)-C(3)-C(2)	−177.4(2)	N(1)-C(6)-C(5)-C(51)	−179.7(2)
C(3)-C(4)-C(5)-C(6)	56.6(3)	C(61)-C(6)-C(5)-C(4)	−180.0(2)
C(3)-C(4)-C(5)-C(51)	−179.5(2)	H(4)-C(4)-C(3)-H(3)	−178(2)
O(41)-C(4)-C(5)-C(6)	172.7(2)	H(4)-C(4)-C(5)-H(5)	177(2)
C(3)-C(4)-O(41)-H(41)	−156(1)	H(3)-C(3)-C(2)-H(2)	−174(2)
C(5)-C(4)-O(41)-H(41)	85(1)	H(6)-C(6)-C(5)-H(5)	−173(2)
H(4)-C(4)-O(41)-H(41)	−40(2)	H(2)-C(2)-N(1)-C(11)	−59(1)
C(4)-C(3)-C(2)-N(1)	56.6(3)	C(21)-C(2)-N(1)-C(11)	60.2(3)
C(4)-C(3)-C(2)-C(21)	178.6(2)	C(11)-N(1)-C(6)-C(6)	−57.8(3)
C(31)-C(3)-C(2)-N(1)	179.0(2)	C(11)-N(1)-C(6)-H(6)	58(1)
C(3)-C(2)-N(1)-C(6)	−56.5(3)	O(41)-C(4)-C(3)-C(31)	61.7(3)
C(3)-C(2)-N(1)-C(11)	−177.9(2)	O(41)-C(4)-C(3)-H(3)	−64(2)
C(21)-C(2)-N(1)-C(6)	−178.5(2)	O(41)-C(4)-C(5)-C(51)	−63.4(3)
C(2)-N(1)-C(6)-C(5)	54.3(3)	O(41)-C(4)-C(5)-H(5)	56(2)
C(2)-N(1)-C(6)-C(61)	−179.7(2)		

Table 5. Dihedral angles between phenyl rings

Plane P_i: atoms C_{ij} (j = 1, 6)

Plane	Plane	Angle (deg)
*P*2	*P*3	127.14(9)
*P*2	*P*5	1.8(1)
*P*2	*P*6	126.49(9)
*P*3	*P*5	128.9(1)
*P*3	*P*6	0.98(9)
*P*5	*P*6	128.27(9)

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