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 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, $\alpha = 78.145(12)^\circ$, $\beta = 79.181(15)^\circ$, $\gamma = 76.108(14)^\circ$, $V = 581.1(1)$ Å$^3$, $Z = 1$, $D_x = 1.199$ Mg m$^{-3}$. Cu Kα radiation (graphite crystal monochromator, $\lambda = 1.54184$ Å), $\mu$(Cu Kα) = 5.17 cm$^{-1}$, $T = 290$K. Final conventional $R$-factor = 0.039, $R_w = 0.042$ for 2795 observed reflections and 377 variables. The structure was solved using MULTAN and DIF. With a view to determining the conformational preference of the piperidine ring when it is highly substituted 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(rl)ol(equatorial) and trans, trans-2,6-diphenylpiperidin-4(rl)ol(axial). How-
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 × 0.22 × 0.07 mm was used during the measurements. Throughout the experiment Cu Kα radiation was used with a graphite crystal monochromator on a Nonius CAD4 single-crystal diffractometer (λ = 1.54184 Å). The unit cell dimensions, 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 = 581.1(1) Å³, 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 θ = 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), μ(Cu Kα) = 5.17 cm⁻¹ (correction factors were in the range 0.97–1.00). Some double measured reflections were averaged, R_{int} = \Sigma (I - \langle I \rangle) / \Sigma I = 0.027, resulting in 4328 unique reflections of which 2795 were observed with I > 3σ(I). Lorentz and polarization corrections were applied and the data were reduced to |F_o| values.