

Crystal and molecular structure of 2,6-bis(4-chlorophenyl)-3-phenylpiperidin-4-one

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Received 3 December 2002, revised 10 January 2003, accepted 29 January 2003

Published online 15 September 2003

Key words piperidone, bactericidal, fungicidal and herbicidal.

PACS 61.10.Nz

The crystal structure of the title compound is described. The chemical formula of the compound is C₂₃H₁₉Cl₂NO. The compound is found to crystallize in monoclinic system with space group *P*2₁/*c*, *Z* = 4. The unit cell dimensions are *a* = 15.137(3) Å, *b* = 8.9171(18) Å, *c* = 14.779(3) Å, β = 91.461(4)° and *V* = 1994.2(7) Å³, *D*_{calc} = 1.320 gcm⁻³. The final R factor is 4.4%. The central piperidone ring of the molecule adopts a slightly distorted chair conformation, the mean torsion angle being 52.3°; the phenyl rings are planar.

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Introduction

In this paper we report the X-ray crystal structure determination of 2,6-bis(4-chlorophenyl)-3-phenylpiperidin-4-one. 4-Piperidones are found to possess medicinal and fungicidal properties, the actual property possessed being determined by the nature and position of substituents attached to the ring [1-3]. They are also useful intermediates for the synthesis of biologically important compounds such as vicinal diamines [4].

Experimental

Synthesis

Ammonium acetate (0.475 g; 0.0075 mol) was dissolved in ethanol (3 ml) by heating. 4-Chlorobenzaldehyde (2.109 g; 0.015 mol) and phenylacetone (1 g, 0.0075 mol) were added to this solution and the mixture heated until the color of the solution changed to yellow. The solution was kept at room temperature for 2-3 days. The solid precipitated was filtered off, washed with ethanol and recrystallized from ethanol and ethyl acetate. The pure compound was obtained in 60% yield. Melting point of the compound was found to be 174°C. The chemical diagram of the compound is shown in Figure 1.

Data Collection, Structure Solution and Refinement

At room temperature, single crystal X-ray diffraction data is collected on a Bruker AXS single crystal X-ray diffractometer using MoK_α radiation and a SMART APEX CCD detector. Data is collected with ω scan width of 0.3°. A total of 606 frames are collected in each of three different settings of φ (0, 90 and 180) keeping the

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sample to detector distance at 6.03 cm and the Θ value is also fixed at -25° . The data is reduced by SAINTPLUS [5] and an empirical absorption correction is applied using the package SADABS [5]. The structure was solved and refined using SHELXTL [5]. Molecular and packing diagrams are generated by ORTEP32 [6] and CAMERON [7] present in the WINGX [8] program suite. The geometric calculations are done by PARST95 [9]. The refinement converged to a final R-factor of 0.044. The crystal data has been deposited at Cambridge Crystallographic Data Centre [CCDC 197609]. Details of data collection and refinement are given in Table 1.

Table 1 Summary of crystal data, data collection and refinement

Crystal data	
Empirical formula	$C_{23}H_{19}Cl_2NO$
Formula weight	396.29
Temperature/K	293(2)
Wavelength/Å	0.71073
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
a/Å	15.137(3)
b/Å	8.9171(18)
c/Å	14.779(3)
$\beta/^\circ$	91.461(4)
Volume/ Å ³	1994.2(7)
Z	4
Calculated density/gcm ⁻³	1.320
Absorption coefficient (μ)/ mm ⁻¹	0.338
F(000)	824
Crystal size/mm	0.35 x 0.3 x 0.25
Data collection	
Diffractometer	Bruker AXS CCD
Goniometer	Bruker AXS
Detector	SMART APEX
θ for data collection/ °	2.65 to 28.00
$h_{\min,\max}; k_{\min,\max}; l_{\min,\max}$	-19,19; -11,11; -19,19.
Reflections collected	17008
Unique reflections	4661
R_{int}	0.0824
Completeness to $\theta = 28.00$	96.9 %
Refinement	
Refinement method	Full-matrix least-squares on F^2
Parameters refined	320
Goodness-of-fit on F^2	0.789
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0443, wR_2 = 0.0675$
R indices (all data)	$R_1 = 0.1339, wR_2 = 0.0828$
Largest diff. peak and hole/eÅ ⁻³	0.264 and -0.218

Results and discussion

The title compound crystallizes in the space group $P2_1/c$ with $Z = 4$. The Figure 2 shows the ORTEP plot drawn at 50% probability displacement ellipsoids and the atom-numbering scheme. The two *p*-chlorophenyl rings on C2 and C6 are twisted with two different torsion angles with respect to C2-C7 ($N1-C2-C7-C8 = -37.2(3)^\circ$) and C6-C19 ($N1-C6-C19-C20 = 21.1(3)^\circ$) bonds, whereas the torsion angle of the phenyl ring on C3

about C3-C13 is $68.8(3)^\circ$ ($C2-C3-C13-C14 = 68.8(3)^\circ$). The planarity of the piperidone ring generally depends on the specific positional attachment of the bulky groups. It can adopt a chair conformation when the bulky groups are attached to the positions 2 and 6 and it will take up a sofa conformation whenever they are attached to positions 3 and 5{10}. The central piperidone ring of the title compound is in a slightly distorted chair conformation (torsion angles in Table 2). The mean torsion angle of the piperidone is calculated in this case as 52.3° .

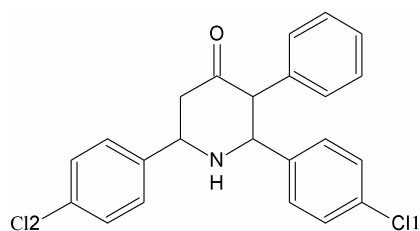


Fig. 1 Chemical diagram of the title compound.

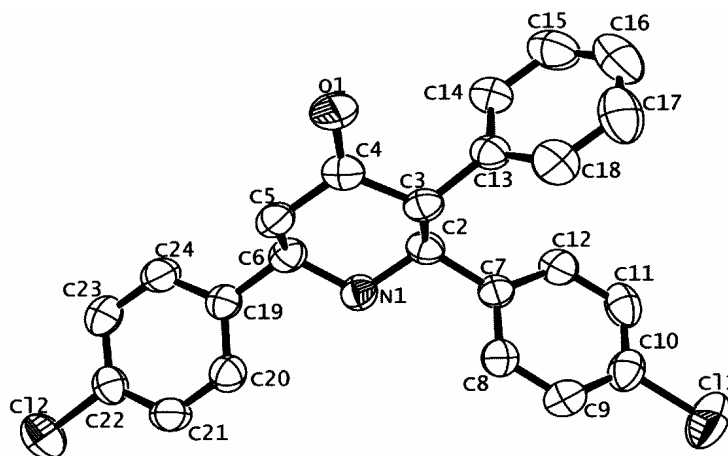


Fig. 2 ORTEP diagram with ellipsoids at the 50% probability level. H-atoms are omitted for clarity.

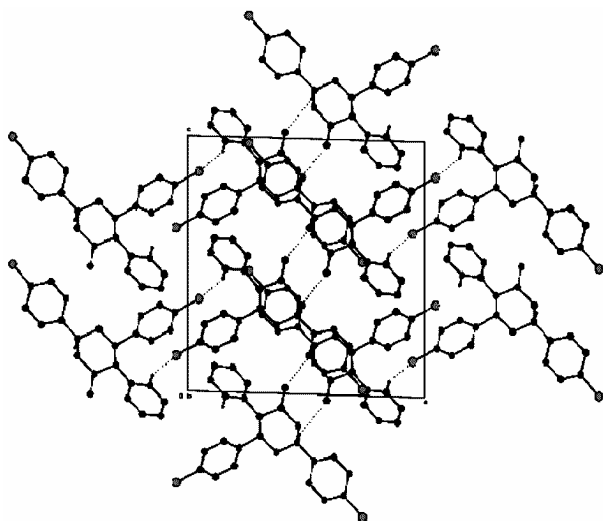


Fig. 3 Molecular packing diagram viewed down b-axis with weak interaction of C-H...Cl and C-H...O hydrogen bond marked.

Table 2 Torsion angle of piperidone ring.

Atoms	Angle($^\circ$)
N1-C2-C3-C4	-45.6(2)
C2-C3-C4-C5	40.6(3)
C3-C4-C5-C6	-45.8(3)
N1-C6-C5-C4	55.0(2)
C2-N1-C6-C5	-65.4(2)
C6-N1-C2-C3	61.2(2)

The out of plane deviations distances of the six atoms (N1, C2, C3, C4, C5 and C6) forming the central ring from the mean plane considered through them are 0.253(2), -0.245(2), 0.150(2), -0.114(2), 0.228(2) and -0.282(2) Å respectively. This shows that the carbons containing *p*-chlorophenyl rings are significantly away from the mean plane whereas C3 is much less deviated from mean plane. The crystal structure is held a) by a weak intermolecular C-H...O hydrogen bond and a C-H...Cl weak interaction (Table 3) forming layers parallel to (010), and b) by interaction between phenyl rings along [010]. The Figure 3 shows the hydrogen-bonding and weak interaction scheme in the packing.

Table 3 Hydrogen-bonding/ Weak Interaction geometry

D-H...A	D-H/Å	H...A/Å	D...A/Å	∠D-H...A°
C18-H18...C11	0.93(2)	2.90(2)	3.763(3)	155(2)
C6-H6...O1	0.94 (2)	2.66(2)	3.395(3)	135(1)

Acknowledgements The authors gratefully acknowledge the Department of Biotechnology (DBT), Government of India, supporting the use of the Bioinformatics Centre, the Interactive Graphics Based Molecular Modelling and Facilities at Madurai Kamaraj University, Madurai and Indian Institute of Science (IISc), Bangalore. The authors gratefully acknowledge the Department of Science and Technology (DST), Government of India for funding CCD diffractometer facility at IISc, Bangalore and for a major research project to S. Perumal.

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