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X-ray structure analysis of tridimenol

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ABSTRACT

The unit cell parameters a = 8. 130(2) Å b = 16. 790(2)Å c=21. 990Å. The space group is determined to be $P2_1/n$. The measured density is 1. 3215g/cm^3 and calculated density is 1. 3102g/cm^3. The average bond distances of C-H and N-H types are 0. 96(2)Å and 0. 90(1)Å respectively. The asymmetric unit comprises two crystallography independent molecule of tridimenol.

INTRODUCTION

Colorless well formed crystals were grown by slow evaporation technique from a solution of cyclohexanone at 278ºK. Chemical structure of Tridimenol is given in figure1 and crystal data is shown in TABLE 1. The three-dimensional intensity data are collected using a computerized automatic 4-circle CAD-4 Enraf-Nonius Diffractometer using graphite filtered MoK(α)

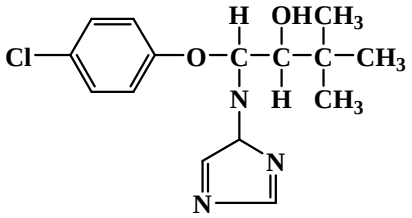

Figure 1: β-{4-Chlorophenxy}-α-(1,1-dimethylethy)1H-1,2,4-triazole-1ehtanol

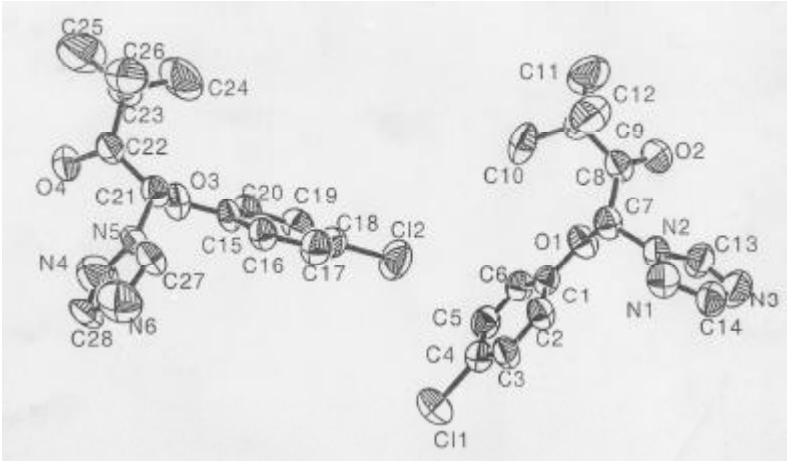

Figure 2 : OPTEP diagram

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TABLE 1

Preliminary	Crystal data
Chemical name	β-4(-chlorophenoxy)-α-(1,1-dimethylethyl)1H-1,2,4-trizole-1-ethanol
Chemical formula	C_{14} H_{18} Cl N_3 O_2
Molecular weight	295. 76
System	Monoclinic
Space group	$P2_1/n$
A	8.130(2)Å
B	16. 790(2)Å
C	21.990(1)Å
α	90 (1) ^{o}
β	92. 52 (1) ^{o}
γ	90(1) ^{o}
V	2998. 8 $Å^3$
Dm	1.3215 g/cm^3
Dc	1.3102 g/cm^3
Mw	295. 76
λ(Mokα)	0.71075Å
Z	4
Mode of data	CAD-4Enraf Nonious 4-circle automatic diffractrometer
Structure solution	SHELXS-97
Structure refinemen t	SHELXL-97
Intensity reflection	4 0 0 0 6-6 0 6 0
Mode of data collection	ω-2θ
Theta range	1-73°
No. of unique reflections	6345
Temp. of crystal during Data collection	293°K
μ	0. 260mm^{-1}
Lp correction	Applied
absorption coefficient	Not applied
Symmetry element	X,Y,Z 1/2-X, 1/2+Y,1/2-Z, 1/2+X ,1/2-Y,1/2+Z

(71075Å) radiations, at 293°K. Three standard reflections (4,0,0) (0,6,-6), (0,6,0) are measured,where h varies from 0 to 10, k from 0 to 15 and l from 0 to 18 The total number of reflections measured was 7542. The crystal structure is solved using SHELXS-97. The ORTEP diagram is shown in figure 2. Bond Lengths (Angstrom) involving Non-Hydrogen atoms is shown in TABLE 2. Bond Angles (Degrees)-(Angles are ordered on the middle label, left to right and top to bottom) involving Non-Hydrogen atoms is shown in TABLE 3.

TABLE 2: Bond lengths (Angstrom) involving Non-Hydrogen atoms with estimated standard deviations in parentheses

Cl(1)	C(4)	1.7484(1)
O(1)	C(1)	1.3889(2)
O(1)	C(7)	1.4028(1)
O(2)	C(8)	1.2070(1)
N(1)	N(2)	1.3456(1)
N(1)	C(14)	1.2955(2)
N(2)	C(7)	1.4360(1)
N(2)	C(13)	1.3410(1)
N(3)	C(13)	1.3084(2)
N(3)	C(14)	1.3239(1)
C(1)	C(2)	1.3757(2)
C(1)	C(6)	1.3951(1)
C(2)	C(3)	1.3732(2)
C(3)	C(4)	1.3881(2)
C(4)	C(5)	1.3696(2)
C(5)	C(6)	1.3636(1)
C(7)	C(8)	1.5399(2)
C(8)	C(9)	1.5157(1)
C(9)	C(10)	1.5361(2)
C(9)	C(11)	1.5022(1)
C(9)	C(12)	1.5219(1)
Cl(2)	C(18)	1.7473(2)
O(3)	C(15)	1.3751(1)
O(3)	C(21)	1.4114(2)
O(4)	C(22)	1.1995(2)
N(4)	N(5)	1.3349(1)
N(4)	C(28)	1.3032(1)
N(5)	C(21)	1.4402(2)
N(5)	C(27)	1.3342(1)
N(6)	C(27)	1.3096(2)
N(6)	C(28)	1.2971(1)
C(15)	C(16)	1.3851(1)
C(15)	C(20)	1.3947(2)
C(16)	C(17)	1.3720(2)
C(17)	C(18)	1.3606(1)
C(18)	C(19)	1.3670(2)
C(19)	C(20)	1.3835(1)
C(21)	C(22)	1.5470(2)
C(22)	C(23)	1.5043(2)
C(23)	C(24)	1.5154(2)
C(23)	C(25)	1.4884(1)
C(23)	C(26)	1.5587(1)

RESULT AND DISCUSSION

The average bond distances of C-H and N-H types are 0.96(2)Å and. 090(1)Å respectively. The bond lengths and angles in the benzene rings show regular features in both the molecules. The C(4)-Cl(1) and Cl(2)-C(18) distances are 1.748(1)Å and 1. 747(2)Å comparable to other structures although similar structures are not available for comparison. These distances are short and shortening may be due to delocalization

TABLE 3 : Bond angles (Degrees) -(Angles are ordered on the middle label, left to right and top to bottom) involving non-hydrogen atoms with estimated standard deviations in parentheses

C(1)	O(1)	C(7)	119.63(1)
N(2)	N(1)	C(14)	103.80(2)
N(1)	N(2)	C(7)	121.85(2)
N(1)	N(2)	C(13)	107.77(1)
C(7)	N(2)	C(13)	130.36(2)
C(13)	N(3)	C(14)	103.31(1)
O(1)	C(1)	C(2)	125.09(1)
O(1)	C(1)	C(6)	114.80(2)
C(2)	C(1)	C(6)	119.92(1)
C(1)	C(2)	C(3)	120.82(2)
C(2)	C(3)	C(4)	117.98(1)
Cl(1)	C(4)	C(3)	118.81(2)
Cl(1)	C(4)	C(5)	119.23(1)
C(3)	C(4)	C(5)	121.96(1)
C(4)	C(5)	C(6)	119.49(2)
C(1)	C(6)	C(5)	119.74(1)
O(1)	C(7)	N(2)	111.71(2)
O(1)	C(7)	C(8)	100.96(2)
N(2)	C(7)	C(8)	113.64(1)
O(2)	C(8)	C(7)	119.12(1)
O(2)	C(8)	C(9)	123.55(1)
C(7)	C(8)	C(9)	117.34(2)
C(8)	C(9)	C(10)	109.77(1)
C(8)	C(9)	C(11)	109.49(1)
C(8)	C(9)	C(12)	107.05(2)
C(10)	C(9)	C(11)	109.95(1)
C(10)	C(9)	C(12)	109.85(2)
C(11)	C(9)	C(12)	110.69(1)
N(2)	C(13)	N(3)	110.29(1)
N(1)	C(14)	N(3)	114.77(1)
C(15)	O(3)	C(21)	118.87(2)
N(5)	N(4)	C(28)	112.37(1)
N(4)	N(5)	C(21)	130.51(2)
N(4)	N(5)	C(27)	107.91(1)
C(21)	N(5)	C(27)	121.56(1)
C(27)	N(6)	C(28)	118.71(2)
O(3)	C(15)	C(16)	125.05(1)
O(3)	C(15)	C(20)	114.45(2)
C(16)	C(15)	C(20)	120.50(1)
C(15)	C(16)	C(17)	119.72(1)
C(16)	C(17)	C(18)	119.53(2)
Cl(2)	C(18)	C(17)	119.71(2)
Cl(2)	C(18)	C(19)	118.39(1)
C(17)	C(18)	C(19)	121.89(2)
C(18)	C(19)	C(20)	119.78(1)
C(15)	C(20)	C(19)	118.56(2)
O(3)	C(21)	N(5)	111.56(1)
O(3)	C(21)	C(22)	102.56(2)
N(5)	C(21)	C(22)	112.55(1)
O(4)	C(22)	C(21)	119.37(2)
O(4)	C(22)	C(23)	123.16(1)
C(21)	C(22)	C(23)	117.31(2)
C(22)	C(23)	C(24)	112.53(1)
C(22)	C(23)	C(25)	112.27(2)
C(22)	C(23)	C(26)	105.10(1)
C(24)	C(23)	C(25)	112.81(2)
C(24)	C(23)	C(26)	107.22(1)
C(25)	C(23)	C(26)	106.27(2)
N(5)	C(27)	N(6)	100.90(1)
N(4)	C(28)	N(6)	100.06(1)

TABLE 4: Hydrogen bond parameters and intermolecular distances less than 3.50(Å)

Hydrogen bonds					
Donor	**H…..A**	**D -H**	**H...A**	**D...A**	**∠ D-H...A**
N(1)	H(1A)..O(4a)	0.900(2)	2.592(1)	3.191(1)	124.6(1)
N(4)	H(4A)..O(2b)	0.900(1)	2.541(2)	3.169(2)	127.3(2)
Some non bonded contacts					
	C(9)		O(4a)		3.3709
	C(24)		O(2b)		3.2614
Symmetry codes					
[a] = 3/2-x,1/2+y,1/2-z					
[b] = 1/2-x,-1/2+y,1/2-z					

of electrons from the benzene rings. The whole molecules appeared to be twisted and folded and reason may be due to stacking constraints. The bond distances around C(7) and C(21) are as usual shorter than single bond values. They may also appears to bear a partial double bond character. The C(7)-O(1) and C(21)-O(3) distances are 1.4028(1)Å and 1.4114(2)Å respectively.

Hydrogen bonding and molecular packing

The hydrogen bond parameter and some other non-bonded contact distances upto 3.50Å are given in TABLE 4. The crystal structure consists of parallel sheets stacked along *a-axis*. The molecules overlap while running along the a-axis. The parallel sheets are hydrogen bonded through the center of inversion where N(4) and N(1) works as donor while the symmetry related O(2) works as an acceptor.

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REFERENCES

[1] G.M.Sheldrich; SHELXS-97.Program for the solution of Crystal Structure, **(1997)**.
[2] G.M.Sheldrich; SHELXSL-97.Program for Crystal Structure determination, **(1997)**.
[3] C.K.Johnson; ORTEP.Report ORNL-3794.OAK Ridge National Lab, Tennessee, U.S.A, **(1965)**.
[4] M.Alleaume, J.Decap; Acta.Cryst., **B24**, 214, **(1968)**.
[5] M.Alleaume, A.Gulko, F.H.Herbstein, M.Kapon, R.E.Marsh; Acta.Cryst., **B32**, 669 **(1976)**.