STRUCTURAL INVESTIGATION OF L-VALINE TRICHLORO ACETIC ACID

[1]MAHALAKSHMI G, [2]JEYALAXMI M, [3]SUGANYA V,[4]Dr P SHARMILA

[1]*Assistant Professor, Department of Physics, Prince Shri Venkateshwara Padmavathy Engineering College,Tamilnadu,India.*
[2]*Assistant Professor, Department of Physics, Prince Shri Venkateshwara Padmavathy Engineering College,Tamilnadu,India.*
[3]*Assistant Professor, Department of Physics, Prince Shri Venkateshwara Padmavathy Engineering College,Tamilnadu,India.*
[4]*Assistant Professor, Department of Physics, Prince Shri Venkateshwara Padmavathy Engineering College,Tamilnadu,India.*

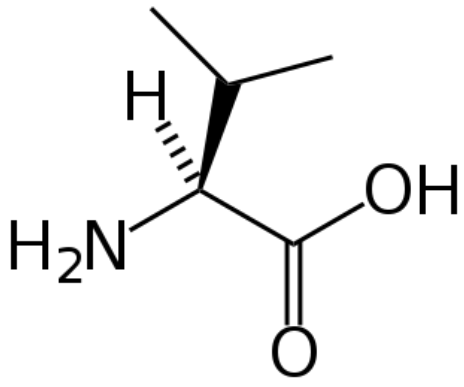
ABSTRACT

L-valinum trichloro acetic acid is a monoclinic space group p21 with a=1.3452Åb=20.3055Åc=11.9131Å α=90.0°β=95.667°γ=90.0°,V=2490(13) Å^3and Z=8.The structure has been solved by direct and difference Fourier methods and refined by full matrix least squares to an R value 0.1993for 1456 reflections with intensities I>2σ(I) measured using CCD diffractometer. The amino nitrogen of the four valine residues belongs to class I, II and III types. The amino nitrogen of the valine residue of trichloroacetic acid through a strong hydrogen bonding. The amino nitrogen of valine residue II form a chelated three-centered hydrogen bonding with residue C of TCAA. The amino nitrogen of valine residue III forms two three –centered hydrogen bonding. The chlorine atoms C14, C16, C18and C19 of trichloroacetic acid exhibit positional order with site occupancies 0.55and 0.45 respectively.

Keywords: *Trichloro acetyl, Valine,crystal and Molecular structure*

1.INTRODUCTION

Valine (abbreviated as Val or V) is an -amino acid with the chemical formula HO2CCH(NH2)CH(CH3)2. L-Valine is one of 20 proteinogenic amino acids is a branched-chain amino acid. Valine is an aliphatic and extremely hydrophobic essential amino acid in humans related to leucine, Valine is found in many proteins, mostly in the interior of globular proteins helping to determine three-dimensional structure. Valine is obtained from soy, cheese, fish, meats and vegetables. Valine supplements are used for muscle growth, tissue repair, and energy. The lack of L-valine may influence the growth of body, cause neuropathic obstacle, anaemia. It has wide applications in the field of pharmaceutical and food industry.



2. EXPERIMENTAL WORK

Saturated solution of L-valine was prepared using water as solvent .similarly prepare a saturated solution of trichloro acetic acid .stir both these mixture in a beaker using a magnetic stirrer .filter the solution and keep it undisturbed in a petri dish .after a forth night, colourless ,needle shape single crystals of L-valine trichloro acetic acid appear .the density was determined using a mixture of xylene and bromoform. Colourless , transparent ,needle shaped crystals of size0.3x0.2x0.2mm was mounted on the goniometer. precise cell dimensions were determined by least squares refinement of the observed bragg angles(θ)of 5006 reflections in therange2.64 °=θ=30.07°.the intensities were collected using CAD-4(Enraf-Nonius diffractometer) with MOK**α** radiation . Lorentz and polarization corrections were applied and the corrected intensities were used for the crystal structure determination and the subsequent refine ment of the structure. multi scan absorption correction were applied with T min=0.93T max=0.96 and the absorption coefficient μ=0.730mm¹(SADABS: Sheldrick, 2004).Nearly 5006 reflections were collected ,of which 4648 reflections were unique .out of 4648 unique reflections only 1456 reflections satisfy the condition I>2σ(I).

3. CRYSTAL STRUCTURE DETERMINATION

The skeleton of the title compound was obtained by direct methods using SHELXS-97(Sheldrick,1990).but the temperature factors of some of the chlorine atoms seems to be extraordinarily high hence these atoms were treated for positional disorder. The chlorine atoms C14,C16,C17,C18and C19 of 0.55and 0.45.subsequently the isotropic thermal parameters of all the non hydrogen atoms were converted into anisotropictherma factors and refined.all the hydrogen atoms were fixed using geometric HFIX constraints.Least squares refinement of F^2 against all reflections was carried out using SHELXS-97(Sheldrick,1997).in the final stages of least square refinement, an overall scale factor ,positional parameters for hydrogen atoms were refined .H-H=0.95—0.99Åand with U_{iso}(H)=1.2(1.5 for methyl groups) times U_{eq}(c).the extinction coefficientis 0.0032(16).this has resulted in a final R=0.0752,WR=0.1993 and GOOF=0.949 for 1456 reflections satisfying the condition I>2σ(I).the r value and WR value are 0.3191 and 0.3031 for 4648 unique reflections.

3.1 CRYSTALLOGRAPHIC DATA FOR L-VALINIUM TRICHLORO ACETATE

Chemical formula : c7H12Cl3NO4

Molecular weight : 280.53

Cell dimensions a: 10.3425(4)Å

b: 20.3055(6) Å

c: 11.9131(3) Å

α: 90.0°

β: 95.667°

γ: 90.0°

Volume of the unit cell V: 2490(13) Å^3

No.of molecules in the unit cell : Z=8

Linear absorption coefficient μ:0.730mm-1

Density(calculated) ρ:1.497mg.m-1

Space group :$P2_1$

F(000) :1152

Systematic absences :0 k 0:k odd absent

3.2 DETAILS OF INTENSITY DATA COLLECTION FOR l-VALINIUM TRICLOACETATE

Diffractometer : CAD -4(Enraf- Nonius)

Wavelength of radiation : 0.71073

Monochromator : Graphite

Scan mode : ω-2θ

Crystal size :0.21x 0.19x 0.18mm

Absorption corrections : None

Number of monitor reflections: 3

Reflections measured : h:0≤h≤12

k: 0≤k≤28

l:-16≤l≤16

θ range for data collection :2.64°to30.07°

Total number of reflections for which intensity data collected: 5006

R_{int} From merging equivalent reflections: 0.1731

3.3 DETAILS OF STRUCTURE REFINEMENT OF FOR L-VALINIUM TRICLOACETATE

Number of unique reflections collected: 4648

Number of reflections observed I>2 σ(I):1456

Number of parameters refined: 589

Final R factor for unique reflections: 0.3191

Final WR factor for unique reflections: 0.3031

Final Goof value for unique reflections: 0.949

Final R factor for reflections I>2σ(I):0.0752

Final WR factor for unique reflections I>2σ(I):0.1993

Final GooF value for unique reflections I>2σ(I):0.949

Mean shift/standard deviation :< 0.01

Extinction coefficient: 0.0032(16)

Flack parameter: 0.0(2)

Program used: SHELXS(Sheldrick,1990)

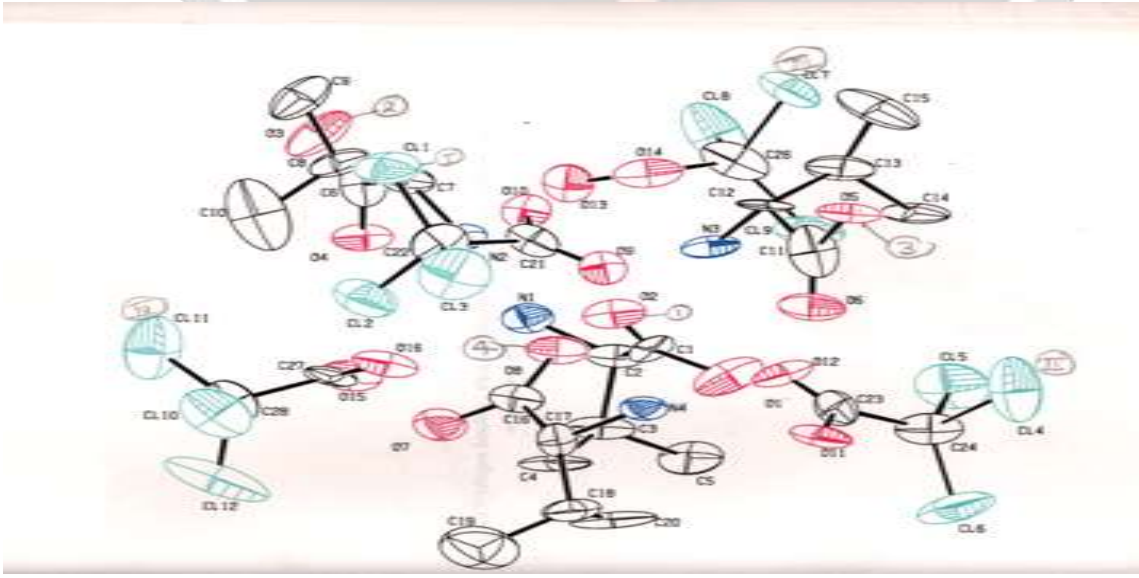
SHELXL-97(Sheldrick,1997)

4. SALIENT FEATURES

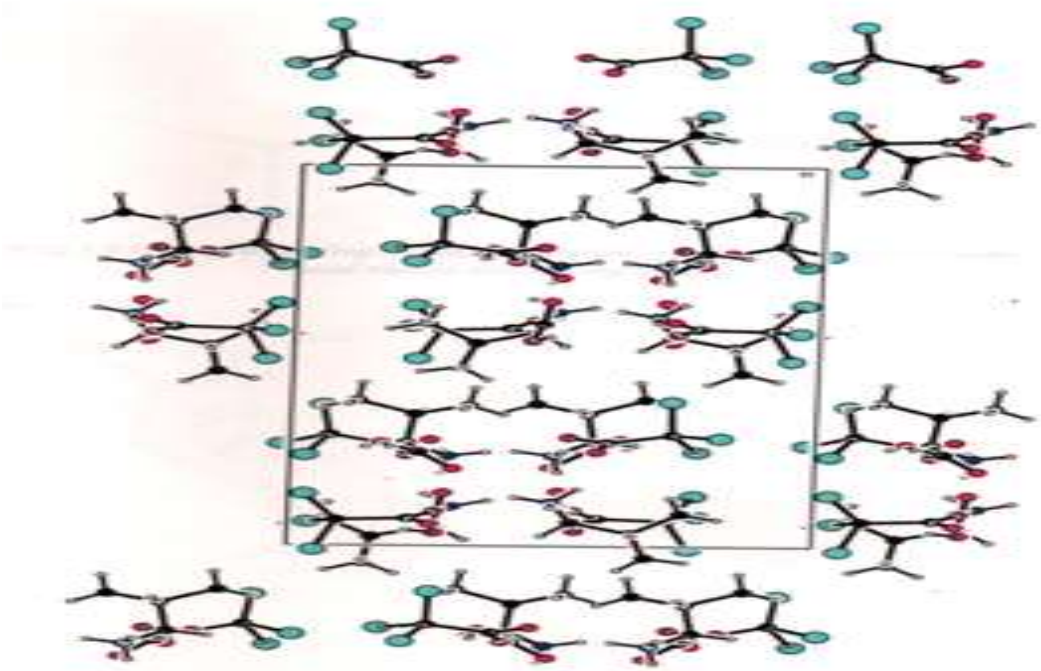
- The chlorine atoms C14, C16, C17, C18 and C19have positional disorders in the structure with occupational factors 0.54/0.47.
- The Trichloroacetate ion plays a vital role in forming hydrogen bonds with Valine molecule and stabilizing structure.
- The amino nitrogen of Valine residue II form chelated three centered hydrogen bondingwith TCAA.
- The single bonded carboxyl oxygen atomsO2, O4, O6, O8) for cis conformation with their respective amino nitrogens.
- The N- H…O and O-H…O strong hydrogen bonding forms bonding between Valine residue and trichloroacetic acid which stabilizes the crystal structure.

5. RESULTS

Fig shows 50%probability Thermal Ellipsoidal diagram of L-Valine Trichloro acetic acid



Hydrogen bonding : Packing diagram of L-valinum Trichloroacetic acid viewed along a-axis



REFERENCES

1. Alagar,M.,KrishnaKumar,R.V.Mostad,A&Natarajan,S.(2001).ActaCryst.E57,01101-01104.

2. Allen,F.H.&Kennard ,O.(1993).Chem.Des.Autom.News,8,1,31-37.

3. Braconnot,HM.:Ann Chim Phys Ser2 13,(1820),113-25.

4. Srinivasan,N&Rajaram,R.K.(1997).Z.Kristallographie212,313-314.

5.Tori,K.&Iitaka,Y.(1970).ActaCryst.B26,1317-1326.

6.Vermeer,C., The Biochemical Journal266(3),(march1990)625-36.

7.Wollaston,WH.,:Philosophical Transaction of the Royal society of London100,1810)223-30.

8.Dalhus,R.O.:Tetrahedron 50,(1994),1539-1650.

9.Gross,R.A.,Kalra,B.:Science297,(2002),(5582),803-807

10.Mitra,S.N&Subramanian,E.(1993).Curr.Sci.65,980-983.