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X-Ray powder diffraction and crystallographic data of Nebivolol Hydrochloride

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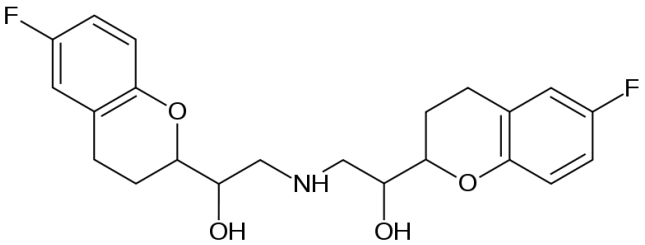
Abstract:

The powder diffraction pattern and crystallographic data of Nebivolol HCl is studied. From the resulting unit cell values a=8.112(3)^{0}A, b=9.080(3)^{0}A, c=15.605(5)^{0}A, α=81.336(6)^0, β=85.270(6)^0, γ=68.677(6)^0, V=1058.1(6) ^{0}A^3, Z=2, 1.387mg/m^3 a Triclinic P1 structure proposed.

Keywords: Powder diffraction, Nebivolol HCl, Crystallographic data

I. INTRODUCTION

Beta blockers for many years have been established as first line therapy in management of hypertension [1, 2]. Nebivolol is a third generation, highly selective β adrenoceptor antagonist indicated for treatment of essential hypertension [3]. Essential hypertension is a condition associated with endothelial dysfunction which is caused by production of oxygen free radicals that destroy nitric oxide and impair its beneficial and protective effects on vessel wall [4]. In addition to its beta blocking effects, nebivolol has an endothelium dependent vasodilator property which is mediatede via L-arginine/ NO pathway[5].Apart from vasodilatation, NO also serves important functions like inhibition of platelet and leucocyte adhesion to vascular endothelium, inhibits smooth muscle hyperplasia following vascular injury and scavenging superoxide anion. Endothelial dysfunction is a marker of cardiovascular disorders [6].



HCl

Polymorphism is of great importance for the pharmaceutical industry and other applications where crystals of small organic molecules are used. Different polymorphs may have different solubility, and different rates of uptake in the body leading to lower or higher biological activity than the desired. In this paper X-Ray powder diffraction data and crystal data of Nebivolol HCl is reported.

II.RESULTS

The high resolution X-ray powder diffraction pattern of Nebivolol HCl is shown in Figure-1.The powder diffraction data presented in Table-I.

The crystal structure of Nebivolol HCl is shown in Figure-2.The crystal data and structure refinement for Nebivolol HCl is presented in Table-II

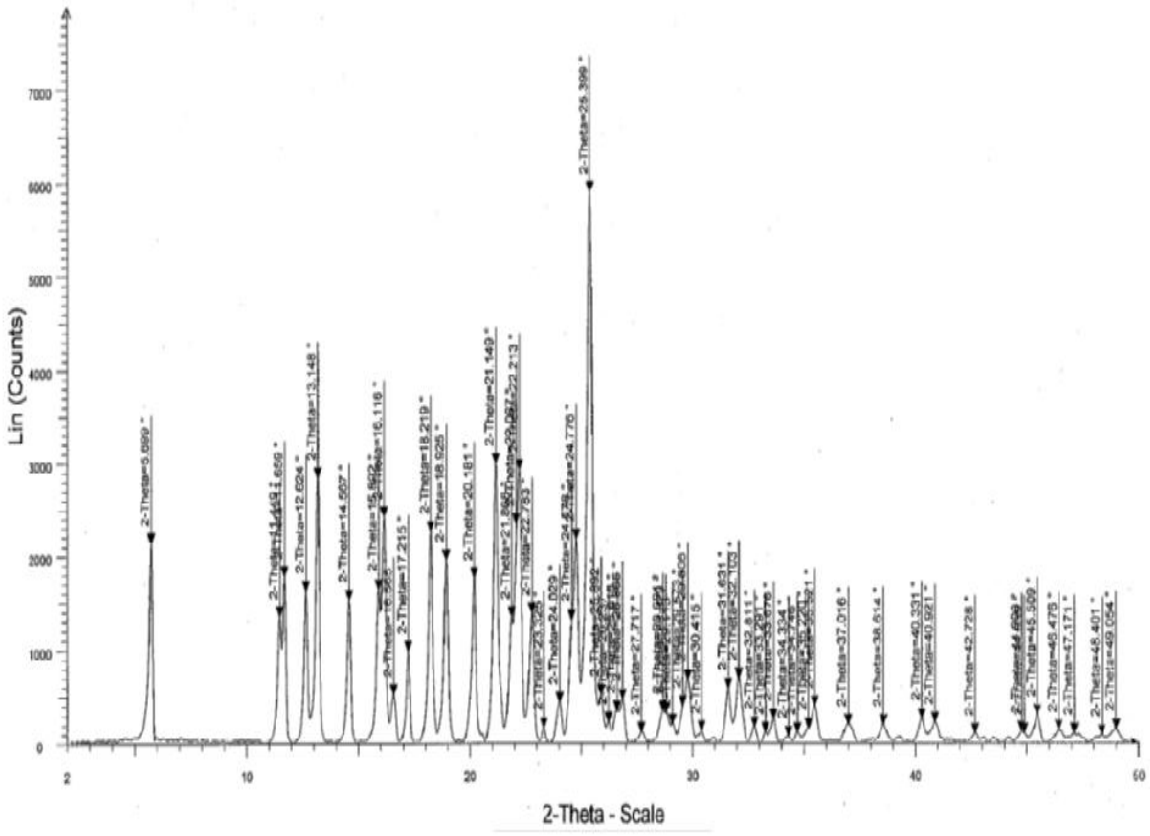


Figure-1: X-ray powder diffraction pattern of Nebivolol HCl

Table-1: The powder diffraction data

S.No	2-θ Angle^0	d-Value Angstrom	% Intensity		S.No	2-θ Angle	d-Value Angstrom	% Intensity
1	5.699	15.494	36.30		21	24.578	3.619	22.10
2	11.449	7.722	22.90		22	24.776	3.590	36.80
3	11.659	7.584	30.10		23	25.399	3.503	100.00
4	12.624	7.006	27.50		24	25.892	3.438	8.60
5	13.148	6.728	48.20		25	26.237	3.393	3.60
6	14.567	6.076	25.80		26	26.616	3.346	5.70
7	15.892	5.572	27.70		27	26.865	3.316	7.80
8	16.116	5.495	41.00		28	28.691	3.108	5.60
9	16.565	5.347	8.70		29	28.829	3.094	5.40
10	17.215	5.146	16.40		30	29.573	3.018	6.60
11	18.219	4.865	38.30		31	29.806	2.995	11.30
12	18.925	4.685	33.10		32	31.631	2.826	9.70
13	20.181	4.396	29.70		33	32.103	2.785	11.60
14	21.149	4.197	50.60		34	32.811	2.727	2.70
15	21.868	4.061	22.70		35	33.676	2.659	4.20
16	22.067	4.024	39.70		36	35.521	2.525	6.50
17	22.213	3.998	49.50		37	37.016	2.426	3.20
18	22.783	3.900	23.30		38	40.331	2.234	4.20
19	23.325	3.810	2.60		39	40.921	2.203	3.60
20	24.029	3.700	7.30		40	45.509	1.991	4.90

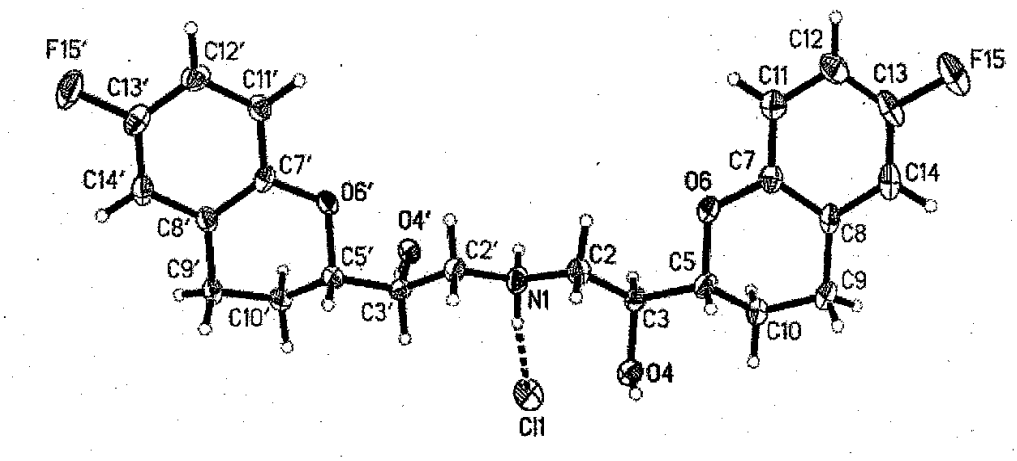


Figure-2: Crystal structure of Nebivolol HCl

Table-2: Crystal data and structure refinement for Nebivolol HCl

Empirical formula	$C_{22}H_{26}NO_4F_2^+$. Cl^-		Limiting indices	-9<=h<=9, -10<=k<=10, -18<=l<=18
Formula weight	414.89		Reflections collected /unique	9774 / 3637[R(int) = 0.0562]
Temperature	293K		Completeness to θ =25.00	97.3%
Wave length	0.71073^0A		Absorption correction	None
Crystal system	Triclinic		Refinement method	Full- matrix least – squares on F^2
Space group	$P\bar{1}$		Data/ restraints/ parameters	3637 / 0 / 273
Unit cell dimensions	a=$8.112(3)^0$A α=$81.336(6)^0$ b=$9.080(3)^0$A β=$85.270(6)^0$ c=$15.605(5)^0$A γ=$68.677(6)^0$		Goodness-of-fit on F^2	1.119
Volume	$1058.1(6)^0A^3$		Final R indices[I>2σ(I)]	R1 = 0.1026, wR2 = 0.2517
Z, Calculated density	2, 1.387 mg/m^3		R indices (all data)	R1 = 0.1323, wR2 = 0.2685
Absorption coefficient	0.227mm^-1		Largest diff.peak and hole	0.679 and -0.316 e.$^0A^3$
F(000)	464			
Crystal size	0.18 x 0.12 x 0.07 mm			
θ range for data collection	2.43 to 25.00^0			

III. EXPERIMENTAL PROCEDURE

The identity and quality of the material is confirmed which includes purity and assay by HPLC, physical characteristics such as differential scanning calorimetry and X-ray powder diffraction. The standard material mica NBS 675 was obtained from the national Institute of standards and technology.

PXRD pattern of the sample were recorded at Room temperature on Bruker's D8 Advance diffractometer (Karlsruche, West Germany) Cu Kα radiation (1.54 ^{0}A),at 40Kv,35mA passing through Nickel filter with divergence slit 0.3^0,anti-scattering slit $0.3^0$and receiving slit 1mm.The diffract meter was equipped with a 2θ compensating slit, and was calibrated for accuracy Korunprobe. Samples were subjected to X-ray powder diffraction analysis in continuous mode with a step size of $0.005^0$and step speed of 1 Sec/Step over an angular range of 5-50^0 2θ.Five hundred milligrams powder mixture was loaded in a 25mm holder made of poly methyl methacrylate and

pressed by a clean glass slide to ensure co planarity of the powder surface with the surface of the holder. The sample holder was rotated in a plane parallel to its surface at 30rpm during the measurements. Obtained diffractograms were analyzed with DIFFRAC^{plus}EVA(ver.9.0) diffraction software.

Single crystal X-ray diffraction data were collected at Room temperature with a Bruker smart Apex CCD diffractometer. Program used of crystal resolution and refinement is SHELXTL-PLUS [7].

IV: CONCLUSION

Polycrystalline crystals of Nebivolol HCl are indexed with a Triclinic P1 structure with unit cell parameters a=8.112(3)^{0}A, b=9.080(3)^{0}A, c=15.605(5)^{0}A, α=81.336(6)^0, β=85.270(6)^0, γ=68.677(6)^0, V=1058.1(6) ^{0}A^3, Z=2, 1.387mg/m^3.

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