Determination of Palladium Content in Nebivolol HCl Bulk Drug by ICP-OES

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The aim of the present work is to develop a validated analytical method for the determination of palladium content in nebivolol HCl bulk drug by inductive coupled plasma-optical emission spectroscopy (ICP-OES). Samples were analyzed after a preparation of sample solution by dissolving in suitable diluents of nitric acid along with hydrogen peroxide. In the present method, RF power of 1500 watts, plasma flow of 15 L/min, nebulizer flow of 0.7 L/min and plasma view at axial mode were used. The wavelength was monitored at 340.46 nm. This allowed a rapid determination of amount of palladium metal present in nebivolol bulk drug. The developed method was validated according to ICH guidelines. Calibration curve for palladium determination were conducted in the range of 10-100 µg/mL. The correlation coefficient for palladium was found to be 0.999970 which indicates good linearity. Recovery rates for palladium were in the range of 86.66-102.66. Limit of detection (LOD) and limit of quantification (LOQ) present in the sample was found to be 3 µg/mL and 10 µg/mL, respectively. The method developed and validated was selective, sensitive, rapid and capable of detecting palladium in the presence of other elements in bulk drugs.

Keywords: Nebivolol HCl, Inductive coupled plasma-optical emission spectroscopy, Metal analysis.

INTRODUCTION

Nebivolol is a β_1 receptor blocker with nitric oxide potentiating vasodilator effect used in treatment of hypertension and in Europe, also for left ventricular failure [1]. It is highly cardio selective under certain circumstances. β -Blockers help patients with cardiovascular disease by blocking β receptors, while many of the side-effects of these medications are caused by their blockade of β_2 receptors [2]. Nebivolol is unique as a β -blocker. Unlike carvedilol, it has a nitric oxide potentiating vasodilator effect [3,4]. Along with labetalol, celiprolol and carvedilol, it is one of four β -blockers to cause dilation of blood vessels in addition to effects on heart [5]. However, recent studies question the clinical relevance of this property to Nebivolol's efficacy [6,7].

Various analytical techniques were employed for the determination of inorganic metal impurities include titration, ion-exchange chromatography, capillary electrophoresis and spectroscopic techniques like flame photometry, fluorimetry, atomic absorption spectroscopy and inductive coupled plasma. Among the above mentioned techniques, inductive coupled plasma is a versatile tool for detection and quantification of elements in accurate manner and it works on the basis of atomic spectrometry. Inductive coupled plasma also further include 2 types which are ICP-optical emission spectroscopy and ICP-

mass spectroscopy. Most specifically, ICP-OES is emission spectrometric technique that exploits the fact that excited atoms emit energy at a given wavelength as the electrons return to their ground state from excited state. A given element emits energy at specific wavelengths peculiar to its chemical nature. The intensity of the energy emitted at that wavelength is proportional to the amount of that element in the analyzed sample. When compared with the other techniques ICP-OES has an additional advantage in terms of detection limits (where it can detect to a concentration range of ppm or ppb) and a rapid analysis. In ICP-OES, sample experiences temperature estimated to be in the vicinity of 10,000 K. These results in atomization and excitation of even most refractory elements with high efficiency so that detection limits for these elements with ICP-OES can be well over and order of magnitude better than the corresponding values of other techniques [8].

The titration methods are not accurate whereas ion exchange chromatography and capillary electrophoresis stabilization is a time taking process and sensitivities are low when compared to ICP-OES. For the above reasons, ICP-OES has become a tool of choice for estimating metals. Literatures revealed that few methods like determination of metals in food samples like vinegar [9], environmental samples (soil and drinking water) [10-16], potato chips [17], honey [18] eliphos tablets [19] and escitalopram oxalate bulk drug [20] are reported.

TABLE-4
METHOD PRECISION VALUES

Sample	Repetitions						Results	
	1	2	3	4	5	6		
Palladium ppm	0.051 ppm	0.051 ppm	0.050 ppm	0.051 ppm	0.051 ppm	0.050 ppm	Mean	0.0505
							SD	0.0005
							RSD (%)	0.990099

TABLE-5
ACCURACY OF PALLADIUM

Spike level (%)	Amount added (ppm)	Amount recovered (ppm)	Recovery (%)	Mean recovery (%)
LOQ level Sample	0.01	0.090	90	86.66
	0.01	0.090	90	
	0.01	0.080	80	
100	0.05	0.051	102	102.66
	0.05	0.052	104	
	0.05	0.051	102	
200	0.10	0.097	97	97.00
	0.10	0.097	97	
	0.10	0.097	97	

Sample analysis: The previously prepared sample was aspirated in the instrument, the concentration of palladium was found to be 1 ppm.

Conclusion

A validated and accurate ICP-OES method has been developed to estimate palladium content in nebivolol drug. The method is selective and is capable to quantitate Palladium in the presence of other trace elements. The method has been validated in terms of specificity, precision, linearity, accuracy and limit of detection and quantification. The validated method can be used to estimate elements not only in nebivolol but also in other drug substances.

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