Determination of active pharmaceutical ingredient – chloropyramine in dragées

Vesna Kostik, Biljana Gjorgeska, Sofija Petkovska

University Goce Delcev, Faculty of Medical Sciences

Introduction

Chloropyramine is a first generation antihistamine drug approved in some Eastern European countries for the treatment of allergic conjunctivitis, allergic rhinitis, bronchial asthma, and other allergic conditions .

Chloropyramine is known as a competitive reversible H1-receptor antagonist. By blocking the effects of histamine, the drug inhibits the vasodilatation, increased vascular permeability, and tissue edema associated with histamine release in the tissue .

Materials and methods

Commercially available samples, Suprastin® for oral use containing 25 mg chloropyramine in the form of chloropyramine hydrochloride were used in this study. In addition to the active ingredient, each tablet contains stearic acid, gelatine, sodium carboxymethyl starch (type A) talc, potato starch, and lactose monohydrate. Chloropyramine hydrochloride purity ≥ 99.8% was obtained from Fluka, Germany. Chloropyramine base purity ≥99.7% was obtained from Alkaloid, Macedonia.

Chloroform (Merck, Germany) gas chromatography grade was used for the extraction and preparation of standard solutions. Ammonium hydroxide (25%, V/V) was obtained from Fluka, Germany. Hydrochloric acid (37%, V/V) was obtained from Sigma Aldrich, Germany. Anhydrous sodium sulfate was obtained from Merck, Germany. Water was obtained by double distillation.

Goals

The aim of this study was to develop a simple, rapid, specific, precise and accurate method based on GC-MS technique for the determination of chloropyramine hydrochloride in tablets. The method was validated in respect of system suitability, specificity, linearity range, accuracy, precision, limit of detection (LOD), limit of quantification (LOQ), selectivity, robustness and stability .

Results

System suitability

Parameter	Results	Parameter limit
RSD of retention time	0.12%	<1%
RSD of peak area	0.25%	<1%
Capacity factor k·	5.52	>2%
Theoretical plate	12,345	>2000

Linearity and range

Parameter	Chloropyramine[a] API	Chloropyramine + excipients
Linearity range (µg/L)	0.4 – 4.0	0.8 – 8.0
Slope	11178	11454
Intercept	32	65
Determination coefficient (r^2)	0.9999	0.9999
SE^b of the intercept	29.432	24.332
SE of the slope	65.433	71.343
P – value of the slope	0.395	0.386

Precision

	Parameter	% of declared content (X)	RSD (%)	Bias (%)
Intra – assay precision[a]	25 mg tablets	99.75	0.75	0.25
Intermediate precision[a]	Day 1	96.15	0.95	3.85
	Day 2	96.45	0.88	3.55
	Day 3	96.58	0.93	3.42
	X	96.39	0.10	3.60

Accuracy

Amount added (µg/L)	Amount found (µg/L)	Recovery (%)	RSD (%)	Bias (%)
0.402	0.398	99.00	0.75	0.99 3.43 0.81
0.553	0.572	103.44	1.05	
0.617	0.612	99.19	0.98	
0.720	0.715	99.30	0.95	0.69
0.795	0.790	99.37	0.66	0.63 0.93
0.855	0.847	99.06	0.78	
	X	99.89	0.15	0.105

Conclusions

The proposed GC-MS method allows a simple, accurate, precise and rapid determination of chloropyramine API in tablets. Furthermore, operating in SIM mode provides additional selectivity of the method in terms of interferences. The proposed method could be applicable for routine analysis in pharmaceutical analytical laboratories.