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Comparison of two reversed phase LC methods for stability study of ciprofloxacin hydrochloride

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Table 1. Chromatographic parameters of a mixture of ciprofloxacin hydrochloride and its four potential impurities (method I)

	Parameters					
Molecules[a]	Retention time R_t (min)	Capacity factor k'	Separation factor α	Resolution factor R	Limit of detection	Limit of quantification
1	1.47 (1.45–1.49)	0.26			3 μg/mL	7 μg/mL
2	21.36 (21.25–21.47)	17.26			0.3 μg/mL	1 μg/mL
1 and 2			66.38	24.86		
3	25.13 (25.25–25.47)	20.48			20 ng/mL	40 ng/mL
2 and 3			1.19	2.90		
4	36.24 (36.13–36.35)	29.97			20 ng/mL	50 ng/mL
3 and 4			1.46	6.94		
5	49.01 (48.84–49.18)	40.89			0.2 μg/mL	0.4 ng/mL
4 and 5			1.36	5.80		

[a] 1, fluoroquinolinic acid; 2,1-cyclopropyl-1,4-dihydro-4-oxo-7-piperazine-1-ylquinoline-3-carboxylic acid; 3,desethyleneciprofloxacin; 4, ciprofloxacin hydrochloride; 5,7-chloro-1-cyclopropyl-1,4-dihydro-4-oxo-6-piperazine-1-ylquinoline-3-carboxylic acid.

PURPOSE

In this study, two LC procedures are presented and validated for rapid quantitation of ciprofloxacin hydrochloride and its impurities and degradation products.

MATERIAL AND METHODS

These two methods were performed on a Licrospher RP-18 column (250 × 4.0 mm i.d., 5 μm particle size), with UV detection at 278 nm, but mobile phases and chromatographic conditions were different.

The first method (I) used methanol/phosphoric acid 0.245% in water proviously adjusted to pH 3.0 with triethylamine (12:88, v/v) as mobile phase LC analysis was carried out isocratically at 40°C with a flow rate of 1.5 mL/min.

The second method (II) was performed with acetonitrile/water adjusted to pH 2.5 with phosphoric acid and containing 2.5 mM sodium heptane sulphonate as mobile phase. Elution was carried out at 30°C by a gradient from 17% acetonitrile to 19% and from 19% to 13% in water over 28 min at a flow rate of 1.5 mL/min.

RESULTS AND DISCUSSION

Both methods were used to study the stability properties of pure ciprofloxacin hydrochloride and of Euciprin capsules (250 mg) producted by Europharm Brasov, Romania. In Tables 1 and 2 are summarized the cromatographic parameters obtained in a mixture of ciprofloxacin hydrochloride and its four potential impurities.

CONCLUSIONS

These methods allow separation of the potential impurities of ciprofloxacin hydrochloride and identification of two of them in Romanian-manufactured ciprofloxacin.

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Table 2. Chromatographic parameters of a mixture of ciprofloxacin hydrochloride and its four potential impurities (method II)

	Parameters					
Molecules[a]	Retention time R_t (min)	Capacity factor k'	Separation factor α	Resolution factor R	Limit of detection	Limit of quantification
1	1.47 (1.45–1.49)	0.16			3 μg/mL	7 μg/mL
2	16.11 (15.86–16.36)	11.69			0.3 μg/mL	1 μg/mL
1 and 2			73.06	24.86		
3	18.42 (18.17–18.67)	13.50			15 ng/mL	30 ng/mL
2 and 3			1.15	2.90		
4	23.67 (23.37–23.97)	17.64			15 ng/mL	40 ng/mL
3 and 4			1.31	6.94		
5	36.73 (36.43–37.03)	27.92			0.1 μg/mL	0.2 ng/mL
4 and 5			1.58	5.80		

[a] 1, fluoroquinolinic acid; 2,1-cyclopropyl-1,4-dihydro-4-oxo-7-piperazine-1-ylquinoline-3-carboxylic acid; 3, desethyleneciprofloxacin; 4, ciprofloxacin hydrochloride; 5,7-chloro-1-cyclopropyl-1,4-dihydro-4-oxo-6-piperazine-1-ylquinoline-3-carboxylic acid.

The second method is characterized by shorter retention times and lower detection and quantification limits for compounds 3, 4 and 5. According to the results obtained, these two procedures appear to be reliable for degradation kinetic studies of ciprofloxacin in various storage conditions.