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COMMUNICATION

A New Method for Quality Control of Zinc Pyritione Pharmaceutical and Cosmetic Forms

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ABSTRACT

A simple high-performance liquid chromatographic (HPLC) method was developed to determine zinc pyritione in pharmaceutical and cosmetic products. Reversed-phase chromatography was conducted using a C_{18} column with an isocratic mobile phase consisting of a suitable mixture of methanol, acetonitrile, and water (30:2.5:20). The effluent was monitored on a ultraviolet (UV) detector at 243 nm. The method was validated following International Conference on Harmonisation (ICH) suggestions and proved accurate, precise, and specific.

INTRODUCTION

Zinc pyritione is used for the treatment of dandruff (1), seborrhea (2), and a great number of skin disorders, such as psoriasis (3), acne (4), and itch (3). Such properties may be due to its ionic zinc form promoting the precipitation of bacterial protein, but its mechanism is still unclear.

This drug is found in pharmaceutical and cosmetic products such as shampoos and other hair care creams and lotions and also in sprays in concentrations ranging from 0.1% to 2%.

Quantitative high-performance liquid chromatographic (HPLC) methods for zinc pyritione have been described, but all are very cumbersome and need special equipment, which led us to develop the method reported here.

EXPERIMENTAL

Reagents and Materials

Zinc pyritione spray formulations for external use, shampoo, and other hair care products were obtained in

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the Argentine and Spanish markets. All solvents were HPLC grade and were used as received.

Chromatography

The HPLC system consisted of a dual-piston reciprocating pump (model KNK-500 G), an ultraviolet-visible (UV-VIS) detector (model KNK-029-757), an integrator (model SP 4600), and a Rheodyne injector (model 7125), all from Konik. LiChroCART® 250 × 4 mm HPLC cartridge and LiChrospher® 100 RP-18 (5 μm), Merck (Darmstadt, Germany) were used. The mobile phase was a suitable mixture of methanol, acetonitrile, and water (30:2.5:20), filtered through a 0.45-μm membrane and degassed with helium. The flow rate was 1.2 ml/min, and the run time was 6 min at room temperature. The wavelength was set at 243 nm, and the volume of each injection was 20 μl.

Standard Solutions

Five solutions of zinc pyritione were prepared in methanol (concentrations from 25 to 72 μg/ml) to study system linearity response.

Accuracy was evaluated by preparing six different samples in the same way as before to obtain the same final concentration (100% of assay solution), and three consecutive injections of each sample were performed with the same equipment on the same day and by the same operator.

Precision was considered at two levels: repeatability and intermediate precision. Repeatability was evaluated by analyzing 10 injections of a homogenous sample with the same equipment and by the same operator, and intermediate precision was evaluated by carrying out two accuracy assays using standard working solution 1 day apart and 1 week apart using two different operators and different equipment in the same laboratory.

Method specificity and selectivity were studied under the following conditions: (*a*) by performing an UV derivative spectrum of each component of the pharmaceutical and/or cosmetic form; (*b*) by performing HPLC of the degradation products and all components of pharmaceutical or cosmetic forms in the conditions described above; and (*c*) by performing the UV derivative spectrum and HPLC described above on stressed samples of zinc pyritione treated with light, acid, alkali, 24-hr UV lamp exposure, and heat.

Sample stability was evaluated by leaving the diluted sample for 24 hr at room temperature and protecting it from light.

Procedure

Solutions were prepared using volumetric flasks as suitable containers to minimize solvent evaporation and to optimize the amount. Prior to running the solutions, the column was equilibrated for at least 15 min with mobile phase flowing through the system. Acceptable results for the number of theoretical plates, tailing factor, precision calculated using USP 23 equation, and detector linearity criteria were required before samples were analyzed.

Quantitation was accomplished using an external standard method, and each solution was injected in triplicate.

RESULT AND DISCUSSION

Figure 1 shows a typical chromatogram obtained following the analysis of zinc pyritione raw material. Using the above chromatographic conditions, zinc pyritione was eluted at about 3.6 min.

Linearity

The regression curve of peak areas versus concentrations was linear with a coefficient of correlation $r = 0.99351$ and with confidence intervals at $p = .05$ (Table 1).

$$Y = -3470.371 + 928.419\,X$$

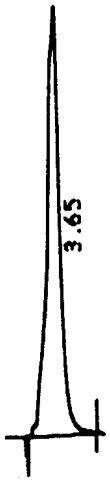

Figure 1. Typical chromatogram obtained following the analysis of zinc pyritione raw material.

Table 1

Linearity

Zinc Pyritione (μg/ml)		Mean Peak Area Response
24.0		19,741
30.0		24,832
48.0		40,628
60.0		49,449
72.0		65,698
Zinc Pyritione Slope (a)	Linearity sb rel. % (c)	Intercept (b)
923.73 ± 466.2	4.1	3161.13 ± 327.7

Accuracy

Mean data recovery was 101.0% with a SD of 1.186 and a coefficient of variation (CV) of 1.17%. Test *t* of mean versus true value with 95% confidence shows that the experimental mean was not significantly different from the true value (t_{n-1}, $\alpha/2$ from tables = 1.4723 for 4 degrees of freedom) (Table 2).

Precision

Repeatability

The mean area obtained was 57338 with a SD of 699.30 and a CV of 1.22%.

Table 2

*Accuracy (*n = *5) Result of the Recovery Analysis of Zinc Pyritione*

Zinc Pyritione (mg/ml)	Amount Recovered (mg/ml)	Recovery (%)
60.0	59.92	99.87
60.0	61.58	102.63
60.0	59.89	99.82
60.0	60.74	101.23
60.0	60.90	101.50

Mean recovery, 101.01%; SD, 1.186; RSD, 1.174.

Intermediate Precision

For each accuracy assay, results were as follows: mean accuracy 1 was 101.01%, SD 1.186, CV 1.17%; mean accuracy 2 was 99.63%, SD 1.425, CV 1.43%. Test *t*, comparing two sample means with 95% confidence for 10 degrees of freedom, disclosed that both samples were not significantly different from each other (t_f; $\alpha/2$ = 1.6345).

Specificity

Zinc pyritione rendered degradation products following alkaline and acid hydrolysis, reduction, oxidation, and photolysis. Selectivity was demonstrated, showing that the zinc pyritione peak was free of interference of degradation products and indicating that the proposed method can also be used in a stability assay.

Sample Stability

The assay preparation failed to undergo significant degradation over a span of 6 hr, during which the difference between two determinations remained below 2%.

CONCLUSION

Our proposed method for zinc pyritione quantification described here was validated following ICH suggestions and provides a simple, rapid, and reproducible system for quality control of this drug as a raw material and in pharmaceutical and cosmetic forms.

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