The Analytical method (HPLC) used for identification, assay of triclabendazole, related substances and preservatives used in finished product Tricladem 5, in SC Delos Impex 96 SRL*

Metoda analitică (HPLC), utilizată pentru identificarea, dozarea triclabendazolului, a impurităților înrudite chimic și a conservanților utilizați în produsul finit Tricladem 5, în cadrul SC Delos Impex '96 SRL

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SC Delos Impex '96 SRL

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Cuvinte cheie: *triclabendazol, HPLC, identificare, substanțe înrudite chimic, methylparaben, propylparaben*

Key words: *triclabendazole, HPLC, identification, related substances, methyparaben, propylparaben*

Abstract

Because, triclabendazole is an active pharmaceutical ingredient without compendial monography (European Pharmacopoeia, United Stated Pharmacopoeia) in SC Delos Impex '96 SRL, the API identification, assay, related substances, and, preservatives, assay and identification is efectuated used the method presented below. It permit, all these determination in a relative short time (the chromatogram time recorded is 25 min.).

Rezumat

Deoarece, triclabendazolul este o substanță farmaceutică activă fără monografie compendiala (European Pharmacopoeia, United Stated Pharmacopoeia), în cadrul **SC Delos Impex '96 SRL,** identificarea si dozarea triclabendazole, impuritatile inrudite chimic, dar si identificarea si dozarea conservantilor folositi in formularea farmaceutica a produsului finit **Tricladem 5** se efectueaza folosind metoda descrisă mai jos. Metoda de analiza prezentata (HPLC), permite toate aceste determinari intr-un timp relativ scurt (timpul de inregistrare al unei cromatograme este de 25 min.)

Introduction

Triclabendazole is an active pharmaceutical ingredient without compendial monography (*European Pharmacopoeia, US Pharmacopoeia*).

Triclabendazole is an antihelmintic of benzimidazolic class, used both, in human medicine and in veterinary medicine.

Materials and methods

Method presentation

Operational parameters of the chromatographic method are summarized in Table 1.

Equipment used

Chromatographic equipment used for the study was an Agilent 1200 system with the following parameters:

Agilent 1200 with modulus:	- Solvents cabinet; - Quaternary pump high presure G 1354A with degassing G 1379B, serie 1200; - Thermostat for column G 1316A, serie 1200; - Spectrometric Detector (DAD) G 1314B, serie 1200, or another similarly; - Autosampler G 1329A; - Thermostat for autosampler G1330B, serie 1200;

Table 1.

Operational parameters of the method

Chromatographic column:	**octylsilyl silica gel for chromatography R, 250 mm L x 4,6 mm i.d. x 5 µm d.p.**
Column temperature:	25°C
Injection volume:	100 µL
Elution:	Isocratic
Compozition for mobile phase:	Solvent A: Ammonium acetate R 30mM, Solvent B: Acetonitrile R
Ratio of solvents in mobile phase:	**Composition:** % Solvent A: 40; % Solvent B: 60
Flow:	1.0 mL/min
Detection at λ:	305 nm for triclabendazole and related substances 254 nm for methyparaben and propylparaben
Chromatogram recorded	25 min.

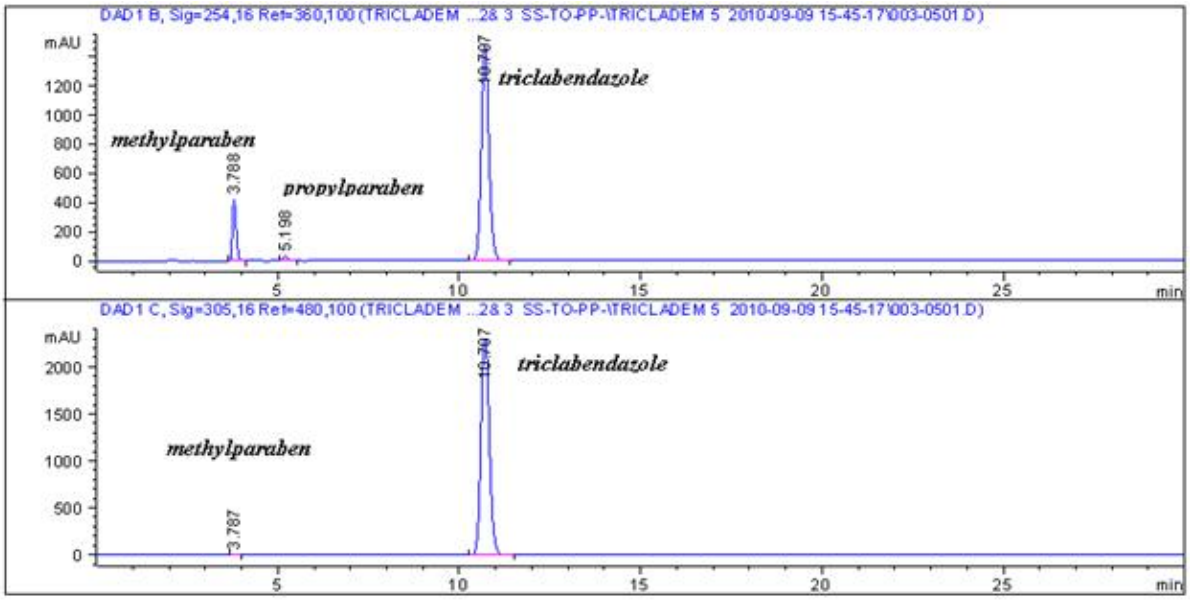

Figure 1. Chromatograms for test solution, recorded at 254 nm and 305 nm

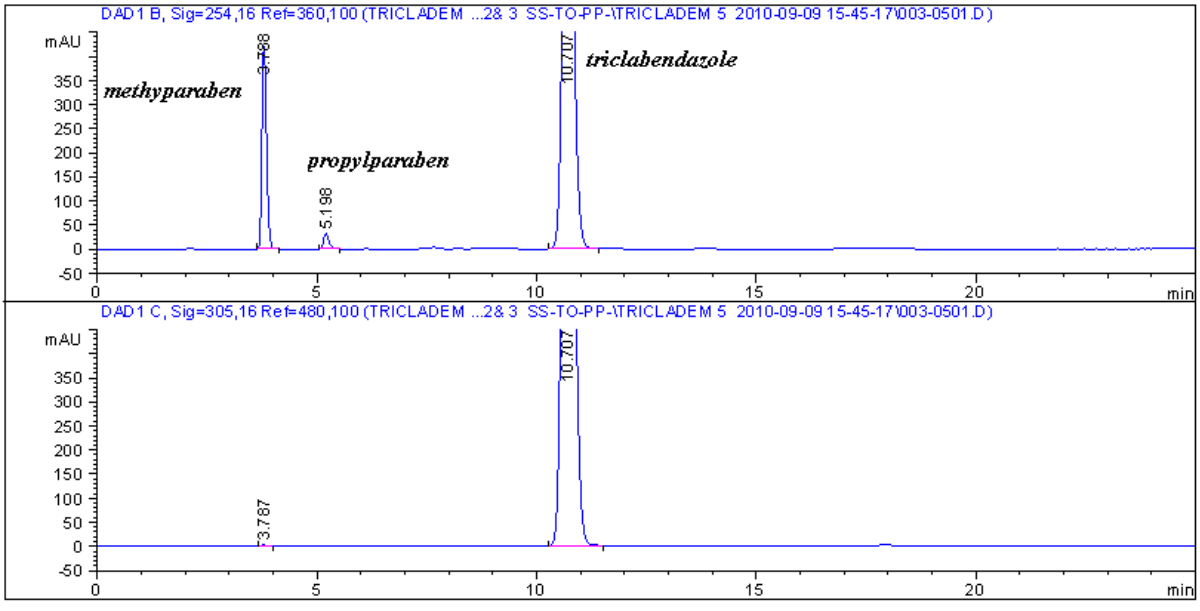

Figure 2. the chromatogram obtained for test solution (167 ppm).

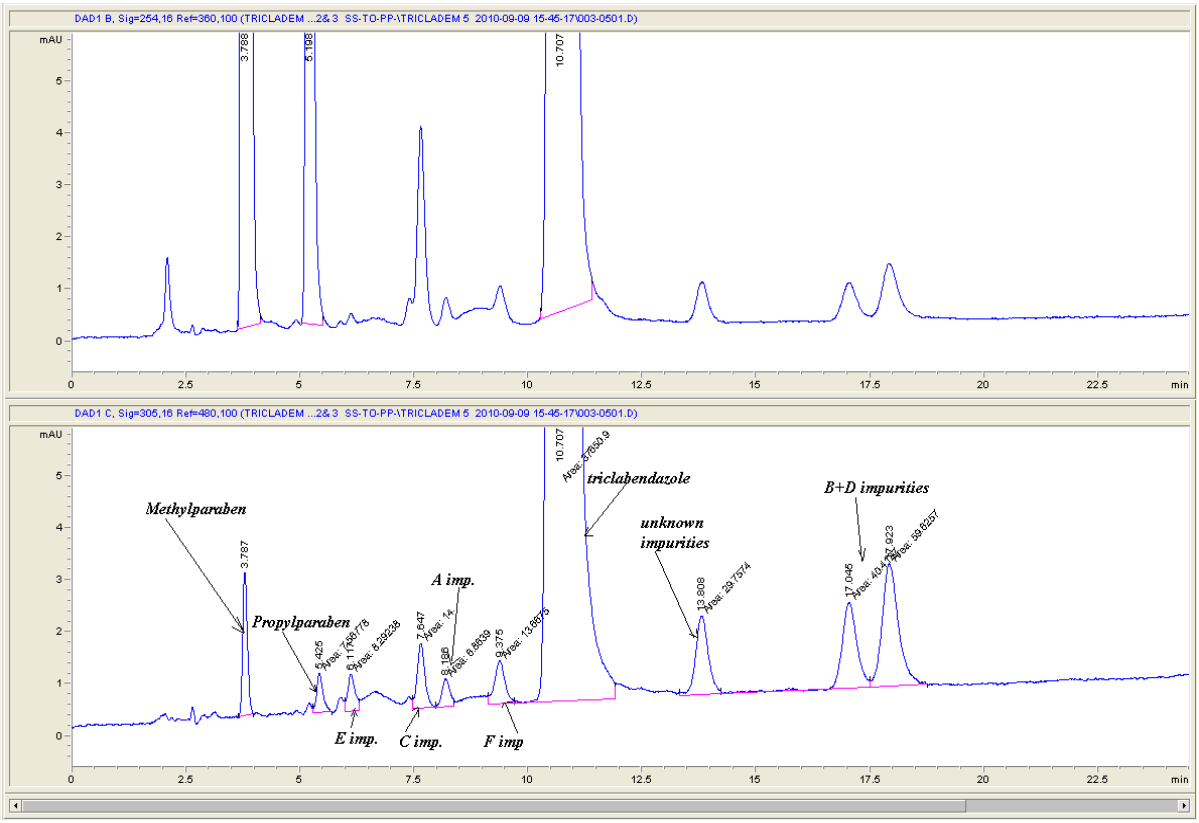

Figure 3. Identification and determination of triclabendazole and preservatives used in Tricladem 5

For triclabendazole assay and identification, assay and identification related substances, the chromatogram is recorded at λ= 305 nm. For assay and identification methylparaben and propylparaben, the chromatogram is recorded at λ= 254 nm.

Injected solutions that are injected and their concentrations, are show in table 2

Table 2

Injected solutions and their concentrations

Type of determination	Sample type	Sample concentration (ppm)
Active substance identification / assay	**Reference solution 1**	Triclabendazole SRS – 167
	Test solution	Triclabendazole - 167
Related substances / preservatives assay and identification	**Reference solution 2**	Triclabendazole SRS – 0.334 Methylparaben – 5.664 Propylparaben -0.6
	Test solution	Triclabendazole - 167

Limit of detection, respectively limit of quantification, for the three substances, are listed in table 3

Table 3.

Detection limits and quantification for the three substances injected

Structure	Limit of quantification (ppm)	Limit of detection (ppm)
Methylparaben	0.2138	0.0641
Propylparaben	0.0386	0.0116
Triclabendazole	0.2900	0.087

Conclusions

1. Method permits determining and identifying of the active substance and of the chemically related impurities,
2. Method allows simultaneous identification and determination of preservatives used in pharmaceutical formulation of the finished product Tricladem 5.

Bibliography

1. http://online.pheur.org/EN/entry.htm