Determination of Octopirox in Shampoos by HPLC

Summary

Octopirox is determined by reversed phase (RP) HPLC after reduction with Ti(III) chloride. The evaluation is carried out via an external standard.

Contents

Erstellt	Geprüft	Freigegeben	gültig ab
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1. Purpose

The method is used for the determination of the Octopirox content in shampoos in the range from 0.1 to 5 g/100 g.

2. Scope of application

Division Functional Chemicals, RQA

3. Definitions

Eluent = mobile phase

4. Responsibilities

The determination is carried out by a trained employee. The head of the group shall ensure a procedure in accordance with these operating instructions. Any deviations necessary shall be approved by him and recorded by the employee.

5. Description

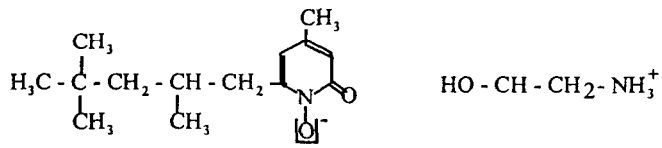
5.1 Task and field of application

The method is used for determination of Octopirox in shampoos in the range from 0.1 to 5 g/100 g.

5.2 Principle

Octopirox is determined by reversed phase (RP) HPLC after reduction with Ti(III) chloride. The quantitative evaluation is done out via an external standard.

Octopirox:



5.3 Apparatus

- HPLC apparatus with autosampler and UV detector
- HPLC column: Purospher RP-18, 5 µm, 125 x 4.0 mm, e.g. Merck art. no. 50142
- usual laboratory equipment

5.4 Reagents

5.4.1 Methanol CHROMASOLV, e.g. Riedel de Haen no. 34860

5.4.2 HPLC mobile phase: methanol/water = 95/5, degassed

5.4.3 Octopirox

5.4.4 Titanium(III) chloride solution 15%, e.g. Riedel de Haen no. 14010

5.4.5 Octopirox standard solution

Weigh approx. 10 mg Octopirox (5.4.3) accurately to 0.1 mg into a 10 ml volumetric flask and make up to volume with the mobile phase. Transfer 1 ml of this solution into a 100 ml volumetric flask, add 100 µl Ti(III) chloride solution (5.4.4) and, after a reaction time of 5 min, make up to volume with the mobile phase.

5.5 Procedure

5.5.1 Sample preparation

- weigh approx. 50 mg, accurately to 0.1 mg, of the shampoo to be analyzed into a 25 ml measuring flask
- add 1 ml mobile phase and 100 µl Ti(III) solution
- leave the sample to react for 5 min, make up to volume with the mobile phase
- filter the sample over a membrane filter
- inject 25 µl of the filtered sample
- inject 25 µl of the external standard solution

5.5.2 HPLC conditions

Column : Purospher RP-18, 5 µm, 125 x 4.0 mm
Eluent : MeOH/H_2O = 95/5 (v/v), degassed
Flow rate : 0.5 ml/min (pressure: approx. 180 bar)
Detector : UV detector
Wavelength : 300 nm
Injection : 25 µl

5.5.3 Result of the determination

$$\text{Octopirox content (g/100 g)} = \frac{A_s \cdot E_{St} \cdot 100}{A_{St} \cdot E_s}$$

In this equation:

A_s = peak area of the Octopirox in the sample
A_{St} = peak area of the standard
E_s = mass of the sample in mg/ml
E_{St} = mass of the standard in mg/ml

5.6 Validation of the Method

Precision

The precision is checked by injecting an Octopirox standard solution (0.01 mg/ml) six times.

$Area_1$	=	458.85 counts	Area (n = 6)	=	458.25 counts
$Area_2$	=	458.47 counts	std	=	0.43 counts
$Area_3$	=	458.68 counts	VC	=	0.09%
$Area_4$	=	457.82 counts			
$Area_5$	=	458.28 counts			
$Area_6$	=	459.00 counts			

Linearity

The linearity is checked by duplicate injection of 5 Octopirox standard solutions in the concentration range from 0.001 to 0.06 mg/ml.

x mg/ml	$Area_1$ counts	$Area_2$ counts
0.0584	2550.5	2549.9
0.0292	1284.1	1284.7
0.0103	458.8	458.5
0.0021	92.0	92.8
0.0011	46.2	46.5

Standard deviation of the method: 0.00014 mg/ml

Relative standard deviation of the method: 0.68%

Sensitivity: 43674 $\frac{\text{counts}}{\text{mg/ml}}$

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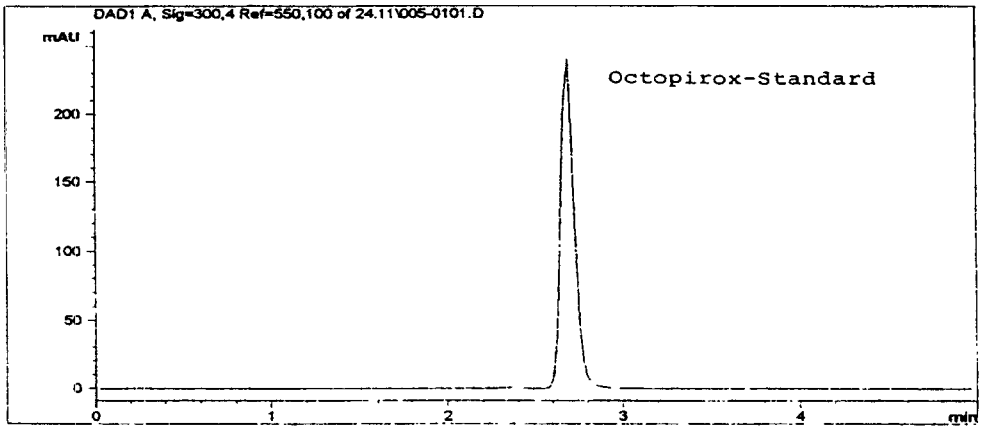
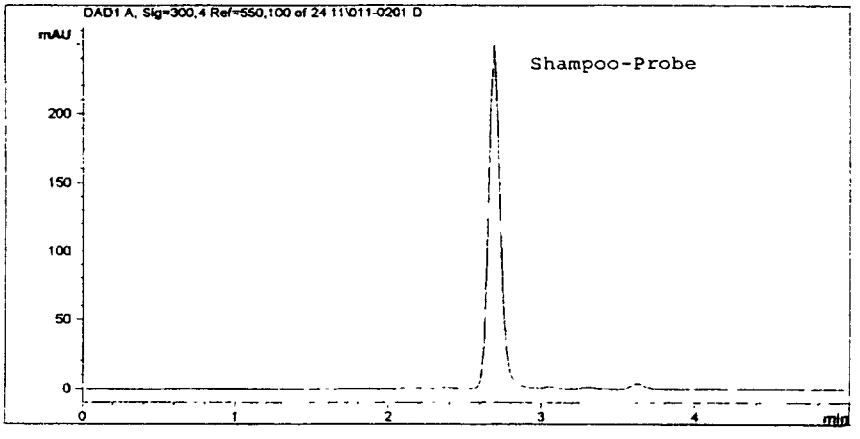
5.7 Remarks

The procedure is described for Octopirox contents of approx. 0.5 %. For higher Octopirox contents the sample masses are to be adjusted accordingly. If conventional RP-18 columns are used, a slight increase in the retention time of Octopirox may occur in the chromatography of shampoos. In such cases, it should be ensured that the correct signal is evaluated by standard addition of Octopirox to the appropriate samples.

5.8 Appendix

typical chromatogram





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6. Notes

6.1 Further applicable documents

Monitoring of balances (EQH-1110-AA-0018)

6.2 Literature references

Research report from Hoechst AG no. 06-Z30-0120-75 of 03.02.1975

7. Documentation

The head of the group is responsible for the electronic documentation of the Standard Operation Procedure. The documentation of the paper version is delegated to the archive employee.

8. Updating service

The head of the group is responsible for the update of this Standard Operation Procedure.

9. Distribution

This Standard Operation Procedure is to via the document management system accessible for all involved employees of RQA.