Hoechst

Hoechst Aktiengesellschaft

CONSUMER PRODUCTS DIVISION
Research

Stability Test of OCTOPIROX R Shampoo (0.5 % (w/w)) for 3 Years at Room Temperature

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1.1 Test Sample

Octopirox Shampoo Lot No.	Manufacturing Date
207-U-051	July 20, 1978
207-U-052	July 20, 1978
207-U-053	July 20, 1978

1.2 Composition

1)	Genapol LRO (28 %)	55.000 % (w/w)
2)	Comperlan KD	3.000
3)	Propylene Glycole	2.000
4)	Octopirox	0.500
5)	Disodium Edetate	0.100
6)	Citric Acid	0.160
7)	Sodium Chloride	0.400
8)	Hellbraun 1 % (C. I. No. 20170)	0.100
9)	Perfume Oil 21552	0.150
10)	Deionized Water	ad 100.000

1.3 Test Period

July 21, 1978 - July 30, 1981
(in 6 months intervals).

1.4 Container

White pigmented opaque plastic bottle (PE) with plastic screw cap.

1.5 Storage Conditions

Storage on a shelf in a non-airconditioned laboratory at room temperature (18 - 26 °C) and 50 to 60 % relative humidity (with rel. humidity extremes occasionally for short periods of 40 % and 80 % respectively).

2 Test Items

2.1 Appearance

2.2 Smell

2.3 pH Value of a 10 % aqueous solution

Remark: The measurement of the pH value was performed by using the Beckman digital pH meter Model 3500 (Beckman Instruments, Inc., Irvine, California, USA).

2.4 Thin Layer Chromatography

Stationary phase: Pre-coated TLC glass plates, type polyamide 11 F_{254} (Merck, Darmstadt, Fed. Rep. of Germany).

Plates are pre-activated for 1 hour at 110 °C before use.

Mobile phase No. 1:	Dioxane	10 % (v/v)
	0.2 M Disodium Edetate Solution	40
	Methanol	40
	Methyl Ethyl Ketone	5
	Ethanolamine	5
Mobile phase No. 2:	Methanol	75 % (v/v)
	Isopropanol	20
	28 % Aqueous Ammonia	5
Mobile phase No. 3:	Ether	30 % (v/v)
	Benzene	30
	Methanol	35
	28 % Aqueous Ammonia	5

Quantity applied:

18 µl of a solution of 1 g of Octopirox Shampoo (0.5 %) in 3 ml of Methanol.

Distance from start to solvent front: approximately 10 cm.

Detection: UV (254 nm).

Evaluation: No other spots should be detected when compared to the initial results.

0.2 M Disodium Edetate Solution:

74.448 g of disodium edetate dihydrate and 10 g of sodium

hydroxide are dissolved in water up to 1000 ml (e.g. = Idranal III, Riedel-de Haën, Seelze-Hannover, Fed. Rep. of Germany, or Titriplex III, Merck, Darmstadt, Fed. Rep. of Germany).

2.5 Assay

Dissolve 0.3 g of iron(II)sulfate ($FeSO_4$, 7 H_2O) in 5 ml of water, add 0.3 ml of glacial acetic acid and dilute with methanol to 50 ml (= "iron reagent").
Dissolve 20 mg of Octopirox Standard Substance, accurately weighed, precisely in 100 ml of 80 % acetic acid (= "standard solution").
Dilute about 2.0 g of shampoo, accurately weighed, in a volumetric flask with 80 % acetic acid precisely to 50 ml (= "test sample solution").
Pipet 10.0 ml of the "test sample solution" into a 25 ml volumetric flask and 10.0 ml of the "standard solution" into a second 25 ml flask. A third 25 ml flask is used for the blank. To each flask add 10 ml of 80 % acetic acid and 1.0 ml of "iron reagent" and then fill to the mark with 80 % acetic acid. After storage for one hour in the dark measure the absorbance of the solution containing the "test sample solution" (A_T) and that of the solution containing the "standard solution" (A_S) against the blank at 440 nm.

Content of Octopirox in shampoo in % (w/w) =

$$\frac{1}{20} \times \frac{A_T}{A_S} \times \frac{\text{weight of Octopirox Standard Substance (mg)}}{\text{weight of shampoo (g)}}$$

Remark: The measurement of the absorbance was performed by using the Zeiss Spectrophotometer Model DM4.

3 Results

The stability test results are specified in Appendices 1 - 3.

Appendix 4 shows the Photos No. 1 - 3 of TLC spots of Octopirox shampoo
Lot No. 207-U-051)
Lot No. 207-U-052) Initial Value
Lot No. 207-U-053)

Appendix 5 shows the Photos No. 4 - 6 of TLC spots of Octopirox shampoo
Lot No. 207-U-051)
Lot No. 207-U-052) after 3 years' storage
Lot No. 207-U-053) at room temperature

4 Conclusion

Shampoos containing 0.5 % (w/w) Octopirox in white pigmented opaque plastic bottles (PE) with plastic screw cap did not show any evidence of instability or decomposition over a period of three years' storage at room temperature (18 - 26 ^{o}C) and 50 to 60 % relative humidity.

Frankfurt (Main),
Sept. 25, 1981

CONSUMER PRODUCTS DIVISION
Research

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(Dr. E. Futterer)

Appendices 1 - 5

Stability Test Results of Octopirox Shampoo (0.5 % (w/w)) at Room Temperature

Lot No. 207-U-051

Test Items \ Interval		No.	Initial	6 months	12 months	18 months	24 months	30 months	36 months
Appearance		1	Clear, light yellow	no change	no change	no change	no change	no change	no change
		2	Clear, light yellow	no change	no change	no change	no change	no change	no change
		3	Clear, light yellow	no change	no change	no change	no change	no change	no change
Smell		1	fresh, pleasant	no change	no change	no change	no change	no change	no change
		2	fresh, pleasant	no change	no change	no change	no change	no change	no change
		3	fresh, pleasant	no change	no change	no change	no change	no change	no change
pH (10 % soln.)		1	7.2	7.2	7.3	7.3	7.3	7.3	7.4
		2	7.2	7.3	7.3	7.3	7.4	7.4	7.4
		3	7.1	7.2	7.2	7.3	7.3	7.4	7.4
TLC	Mobile phase No. 1	1	One spot Photo No. 1	no change	no change	no change	no change	no change	One spot Photo No. 4
	Mobile phase No. 2	1	One spot Photo No. 2	no change	no change	no change	no change	no change	One spot Photo No. 5
	Mobile phase No. 3	1	One spot Photo No. 3	no change	no change	no change	no change	no change	One spot Photo No. 6
Assay (%) Colorimetric determination		1	0.50	0.51	0.50	0.49	0.50	0.49	0.48
		2	0.51	0.51	0.49	0.50	0.49	0.48	0.49
		3	0.50	0.50	0.50	0.49	0.49	0.48	0.48

Appendix 1

Stability Test Results of Octopirox Shampoo (0.5 % (w/w)) at Room Temperature

Lot No. 207-U-052

Test Items \ Interval		No.	Initial	6 months	12 months	18 months	24 months	30 months	36 months
Appearance		1	Clear, light yellow	no change	no change	no change	no change	no change	no change
		2	Clear, light yellow	no change	no change	no change	no change	no change	no change
		3	Clear, light yellow	no change	no change	no change	no change	no change	no change
Smell		1	fresh, pleasant	no change	no change	no change	no change	no change	no change
		2	fresh, pleasant	no change	no change	no change	no change	no change	no change
		3	fresh, pleasant	no change	no change	no change	no change	no change	no change
pH (10 % soln.)		1	6.9	6.9	6.9	7.0	7.0	7.1	7.1
		2	6.9	6.9	7.0	7.0	7.1	7.1	7.2
		3	6.9	7.0	6.9	6.9	7.0	7.1	7.1
TLC	Mobile phase No. 1	1	One spot Photo No. 1	no change	no change	no change	no change	no change	One spot Photo No. 4
	Mobile phase No. 2	1	One spot Photo No. 2	no change	no change	no change	no change	no change	One spot Photo No. 5
	Mobile phase No. 3	1	One spot Photo No. 3	no change	no change	no change	no change	no change	One spot Photo No. 6
Assay (%) Colorimetric determination		1	0.51	0.50	0.51	0.50	0.50	0.49	0.50
		2	0.51	0.51	0.51	0.51	0.51	0.50	0.49
		3	0.50	0.50	0.50	0.50	0.49	0.50	0.49

Appendix 2

Stability Test Results of Octopirox Shampoo (0.5 % (w/w)) at Room Temperature

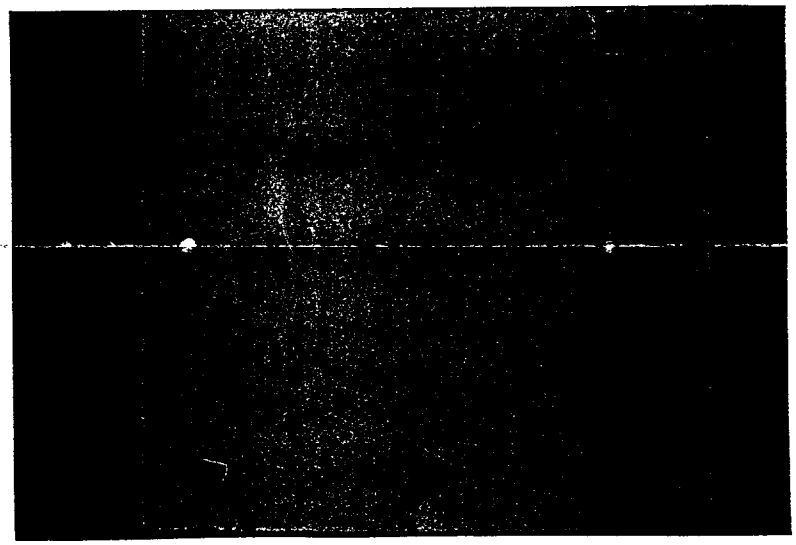
Lot No. 207-U-053

Test Items \ Interval	No.	Initial	6 months	12 months	18 months	24 months	30 months	36 months
Appearance	1	Clear, light yellow	no change	no change	no change	no change	no change	no change
	2	Clear, light yellow	no change	no change	no change	no change	no change	no change
	3	Clear, light yellow	no change	no change	no change	no change	no change	no change
Smell	1	fresh, pleasant	no change	no change	no change	no change	no change	no change
	2	fresh, pleasant	no change	no change	no change	no change	no change	no change
	3	fresh, pleasant	no change	no change	no change	no change	no change	no change
pH (10 % soln.)	1	7.0	7.0	7.1	7.1	7.1	7.2	7.2
	2	7.0	7.0	7.1	7.1	7.2	7.1	7.2
	3	7.0	7.1	7.0	7.1	7.1	7.1	7.1
TLC Mobile phase No. 1	1	One spot Photo No. 1	no change	no change	no change	no change	no change	One spot Photo No. 4
TLC Mobile phase No. 2	1	One spot Photo No. 2	no change	no change	no change	no change	no change	One spot Photo No. 5
TLC Mobile phase No. 3	1	One spot Photo No. 3	no change	no change	no chagne	no change	no change	One spot Photo No. 6
Assay (%) Colorimetric determination	1	0.51	0.51	0.50	0.51	0.51	0.51	0.50
	2	0.50	0.50	0.51	0.50	0.50	0.49	0.50
	3	0.51	0.51	0.50	0.50	0.50	0.50	0.49

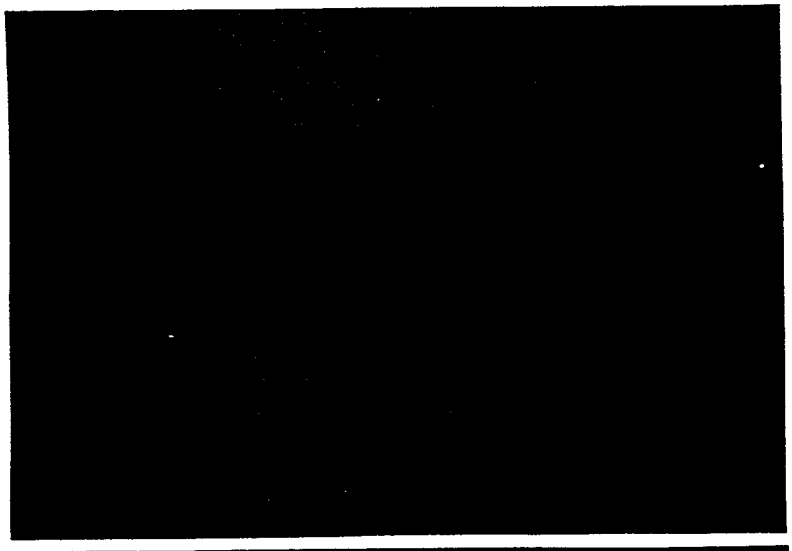
Appendix 3

Photos of TLC spots of Octopirox shampoo (0.5 %)
/ Initial Value

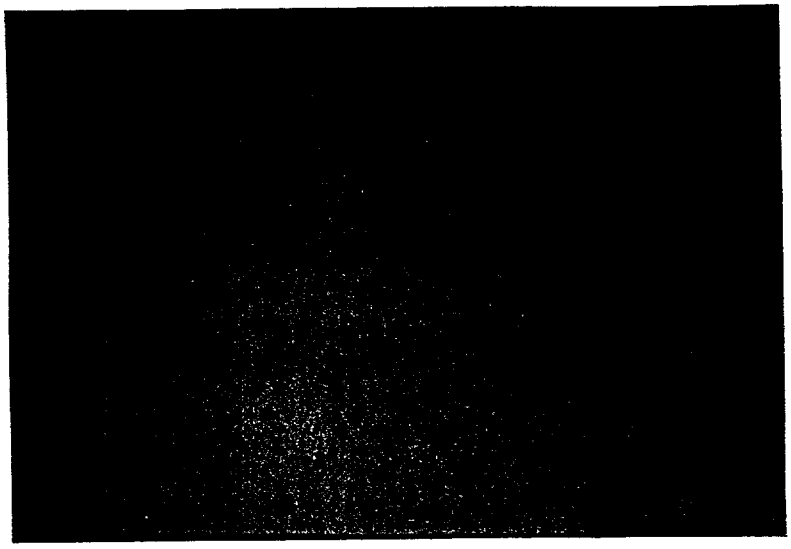
Mobile Phase No. 1
(Photo No. 1)



Mobile Phase No. 2
(Photo No. 2)



Mobile Phase No. 3
(Photo No. 3)



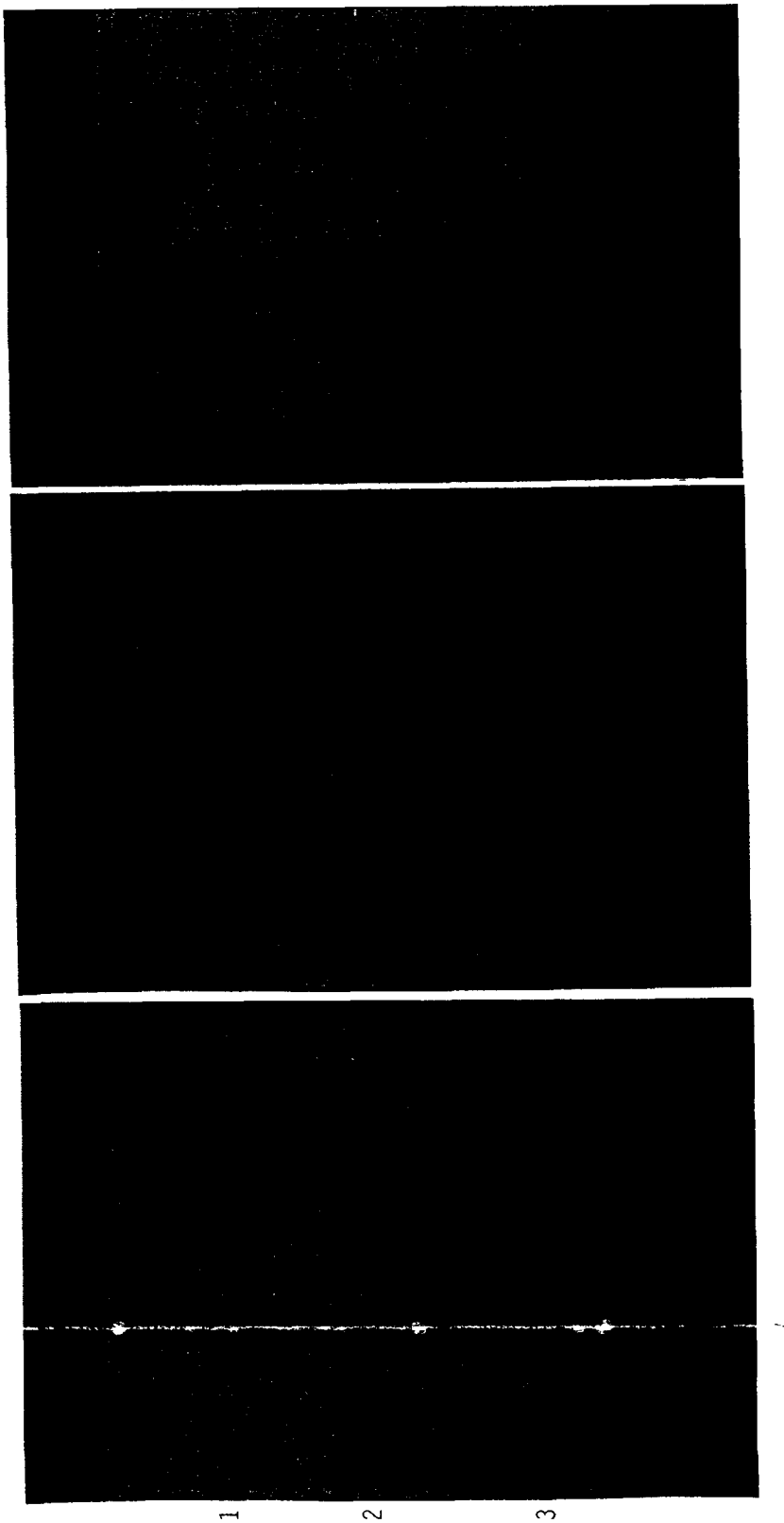
Lot No. 207-U-051 | Lot No. 207-U-052 | Lot No. 207-U-053

Photos of TLC spots of Octopirox shampoo (0.5 %)
after 3 years' storage at room temperature

Mobile Phase No. 1
(Photo No. 4)

Mobile Phase No. 2
(Photo No. 5)

Mobile Phase No. 3
(Photo No. 6)



Lot No. 207-U-051 | Lot No. 207-U-052 | Lot No. 207-U-053