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Research Article

DEGRADATION DETERMINATION OF TINOSORB-S IN SUNSCREEN PREPARATIONS

KALLOL JANA*, BEDUIN MAHANTI

Department of School of Pharmacy, Techno India University, Kolkata, West Bengal, India.
***Corresponding author: Kallol Jana; Email: janakallol@gmail.com**

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ABSTRACT

Objectives: The main objective was to determine the Tinosorb-S degradation in the sunscreen preparations with a simple economic linear and specific analytical method.

Methods: The RP-HPLC was achieved with 100% methyl alcohol as the mobile phase, a flow rate of 2.5 mL/min., and an octadecylsilane column (300 × 3.9 cm, 10 µ) at 254 nm.

Results: The developed analytical method of Tinosorb-S was statistically validated for accuracy and linearity of 70–130 µg/mL. The correlation coefficient was 0.996, and the % RSD of precision was 0.957. The recovery percentage was 100.01–105.04, and the excellent thermal and photolytic stability of Tinosorb-S was confirmed by the thermal, dry, and wet chromatograms compared with the standard; no such degradation was developed, and the recovery was 99.52%, 99.44%, and 99.45%, respectively. Acid, oxidative, and alkaline decomposition of Tinosorb-S were established by screening the decomposition peaks, and the percentage recovery was 41.31%, 40.38%, and 40.44%, respectively.

Conclusions: The stress degradation study of Tinosorb-S is important in stability to evaluate the maximum protecting power of skin from harmful UV rays. Conditions like photolytic degradation, exposure to thermal conditions, acid hydrolysis, oxidation, and alkali hydrolysis were studied with a cost-effective, specific, linear, high-performance liquid chromatographic method with a photodiode array detector.

Keywords: Sunscreen, Photolytic and thermal stability, Tinosorb-S, Degradation, Chromatography.

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INTRODUCTION

For protection of the epidermis from skin cancer, skin burns, skin allergies, and skin aging due to UVB and UVA rays of sunlight, sunscreen preparations are used. For the absorption of harmful effects from the sun like UVB (290–320 nm) and UVA (320–400 nm) rays, different sunscreen preparations add Tinosorb-S to the formulation.

The efficiency is increasing due to the fat-soluble Tinosorb-S in the sunscreen preparation and its broad spectrum influence against deleterious rays of sunlight. The Tinosorb-S is chemically 5-(2-ethylhexoxy)-2-[4-[4-(2-ethylhexoxy)-2-hydroxyphenyl]-6-(4-methoxyphenyl)-1,3,5-triazin-2-yl]phenol(Bemotrizinol) (Fig. 1) and absorption peaks at 310 and 340 nm [1,2]. Tinosorb-S melts at 83–85° and has yellowish crystals.

Tinosorb-S is used in the preparation of sunscreen in the European Community, Australia, and other parts of the world, where it is allowed for local application, but in the United States it is not allowed [3-8]. To achieve the highest skin safety from sun light, the degradation study of Tinosorb-S during stability is very significant. It is evaluated by the implementation of different types of stress environments, like exposure to photolytic wet and dry states, oxidation, thermal degradation, and acid and base decomposition of Tinosorb-S. But this type of degradation experiment was not found earlier for the quantification of Tinosorb-S in the sunscreen preparation.

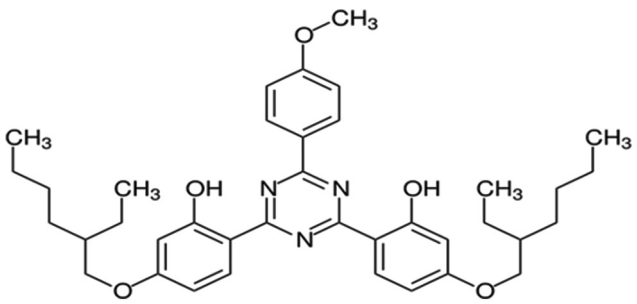

Fig. 1: Tinosorb-S (Bemotrizinol)

In this investigation, we successfully developed an economic, simple, linear analytical method for the quantification of Tinosorb-S and identified its degradations.

METHODS

Instrument

High-performance liquid chromatography, PDA detector, Shimadzu-SPD-M10-AVP with LC solution, binary pump (LC-20-AD and LC10-ATV), column cabinet (CTO10ASV-P), and autosampler (SIL20AH-T).

Chromatographic environment

The Tinosorb-S was eluted isocratically, and methyl alcohol was used at 100% as the mobile phase. Flow was 2.5 mL/min, and octadecylsilane 300 × 3.9 cm, 10 µ was used as the column. The column oven temperature was set at 27°C, and the application volume was 20 µ1. The detection nanometer was set at 254 nm. Trichloromethane and methyl alcohol were used for the preparation of the sample and standard. The chromatograms are shown in Figs. 2 and 3.

Procedure

Preparation of standard

Weight accurately 0.130 g of Tinosorb-S into a 100 mL graduated flask. Add 10 mL of trichloromethane and make the volume 100 mL with methyl alcohol.

Sample preparation

Weight the 25 mg equivalent of the Tinosorb-S sample into a 50 mL graduated flask, add 10ml of trichloromethane, and make the volume 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL.

Study of sunscreen formulation

The formulation was quantified by the difference between the standard and sample responses. The outcome was found to be 99.23% in the Photoderm MAX ^{SPF}50+ sunscreen preparation, which is exhibited in Table 1. The assay result recommended that the developed method is free from interference with pharmaceutical bases and very selective for the estimation of Tinosorb-S from sunscreen formulation.

Method validation

System suitability

The system suitability parameters were used to establish the chromatographic condition and column performance. This was acceptable for methodical application. The RSD percentages of peak areas were not more than 2.0% in this chromatographic estimation of Tinosorb-S. The system suitability parameters are exhibited in Table 2.

Linearity

For the linearity study, Tinosorb-S was examined at a concentration of 70–130 µg/mL. The standard curve was confirmed by the comparability of the peak area response and the concentration of the analyte. In the linear regression of the least squares equation, the slope and y-intercept were documented. The graphically formatted least squares equation in the form of mathematical equitation was Y = 7715X–15320. The correlation coefficient (R^2) value was 0.996, which is exhibited graphically in Fig. 4.

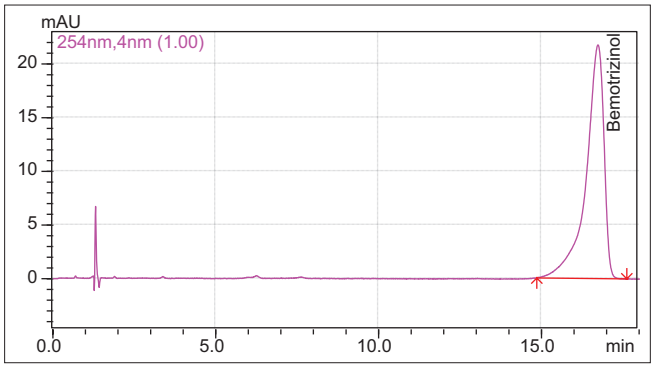

Fig. 2: Chromatogram of standard

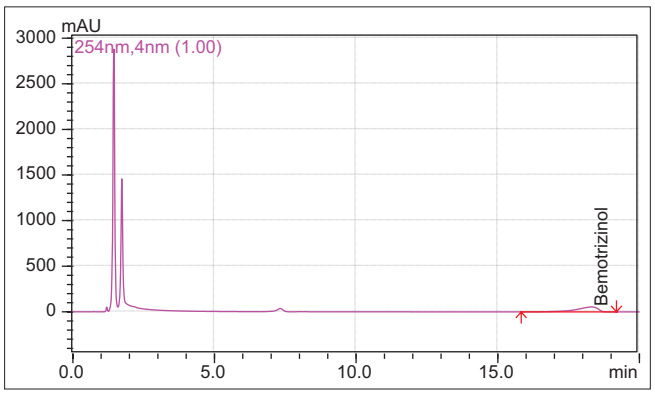

Fig. 3: Chromatogram of sample

Limit of detection (LOD) and limit of quantification (LOQ)

The LOD and LOQ were calculated in the form of the standard deviation of response (σ) and slope (S) from the least squares equation of Tinosorb-S, as demonstrated in the Q2R1 guideline of ICH. LOD was evaluated on the basis of slope (S) and standard deviation (σ); the equation was 3.3 (σ)/S, and LOQ was computed as 10 (σ)/S. The results were 0.12 µg/mL and 0.38 µg/mL, respectively, which are exhibited in Table 3.

Accuracy

The accuracy was determined with a recovery study of the Tinosorb-S from the matrix of the sunscreen formulation. For this experimental work, a known concentration of active material was spiked into the previously analyzed sample. This arrangement was performed with concentrations of 80%, 100%, and 120% with the new chromatographic assay method, which is exhibited in Table 4.

Specificity/selectivity

In this experiment, the specificity of the analytical condition was committed with blank determination. In this developed chromatographic condition, a sample, standard, and blank were prepared as per the analytical method and injected (Fig. 5).

Forced degradation

The stress decomposition of Tinosorb-S was demonstrated through the investigation of the sample for acidic, alkali, oxidation, and photolytic stress degradations. The samples were explored with this condition, and the peaks were studied. This investigation successfully

Table 1: Evaluation of Tinosorb-S in sunscreen formulation

Trade name	Assay %
PhotodermMAX^{SPF}50+(Bioderma)	99.23

Table 2: System suitability

System suitability parameters	Tinosorb-S
Retention time	17.599
Theoretical plates	4680.915
Linear dynamic range	70–130 microgram per ml
R^2 value	0.996
LOD	0.12 (µg/mL)
LOQ	0.38 (µg/mL)
Tailing factor	0.958
Specificity	Specific
Recovery	100.01–105.04%
Inter day	100.125%
Intra day	100.248%
RSD %	0.33

Table 3: LOD and LOQ

y-intercepts	Slope S	LOD and LOQ	
15320	7715		
14910	7686	3.3σ	10 σ
SD: 289.91378	SD: 20.50609665	LOD=------=0.12	LOQ=-------=0.38
Mean: 15115	Mean: 7700.5	S	S

Table 4: Recovery of Tinosorb-S

Concentration	Results (%)
80	100.52
100	105.04
120	100.01

differentiates the degraded peaks from the principal peak (Fig. 6). This was established by its recovery, as shown in Table 5.

Acid degradation

Weight accurately 0.025 g Tinosorb-S in a reflux condenser and reflux at 70° for 5 h with 1 mL 5(N)HCl and transmit into a 50 mL graduated flask. Add 10 mL of trichloromethane and make the volume up to 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL. The chromatogram was obtained after injecting the prepared sample solution into the chromatographic system (Fig. 7).

Alkaline degradation

Weight accurately 0.025 g Tinosorb-S in a reflux condenser and reflux at 70° for 5 h with 1 mL 5(N) NaOH and transmit into a 50 mL graduated flask. Add 10 mL of trichloromethane and make the volume up to 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL. The chromatogram was obtained after injecting the prepared sample solution into the chromatographic system (Fig. 8).

Oxidative degradation

Weight accurately 0.025 g Tinosorb-S in a reflux condenser and reflux at 70° for 2 h with 1 mL of 30% H_2O_2 and transmit into a 50 mL graduated flask. Add 10 mL of trichloromethane and make the volume up to 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL. The chromatogram was obtained after injecting the prepared sample solution into the chromatographic system (Fig. 9).

Table 5: Forced degradation recovery

Test	Recovery %	Degradation %
Degradation in acid	41.31	58.69
Degradation in Alkali	40.44	59.56
Degradation in oxidation	40.38	59.62
Degradation in photolysis (dry stage)	99.44	0.56
Degradation in photolysis (wet stage)	99.45	0.55
Thermal degradation	99.52	0.48

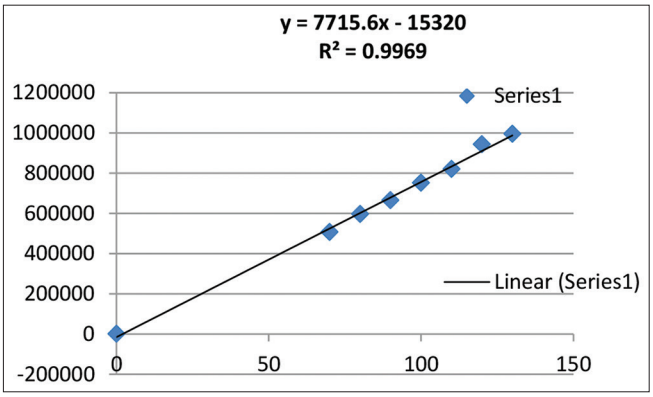

Fig. 4: Standard curve

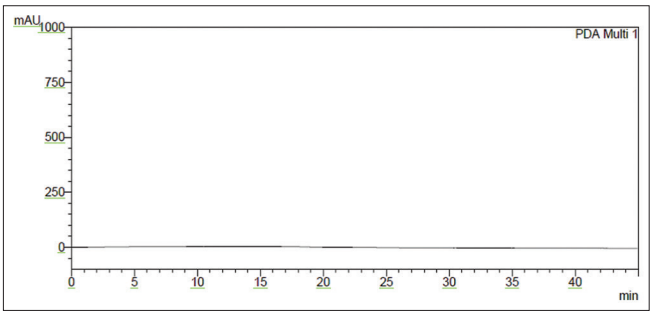

Fig. 5: Blank chromatogram

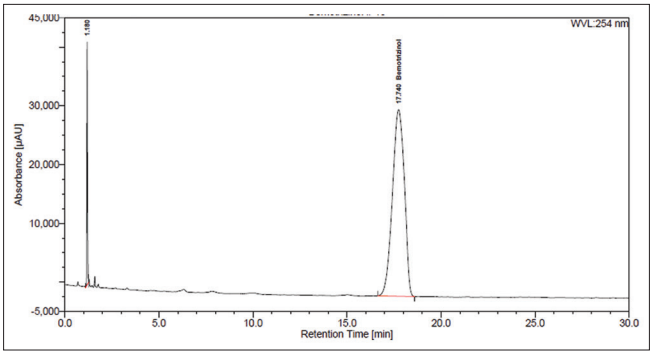

Fig. 6: Tinosorb-S

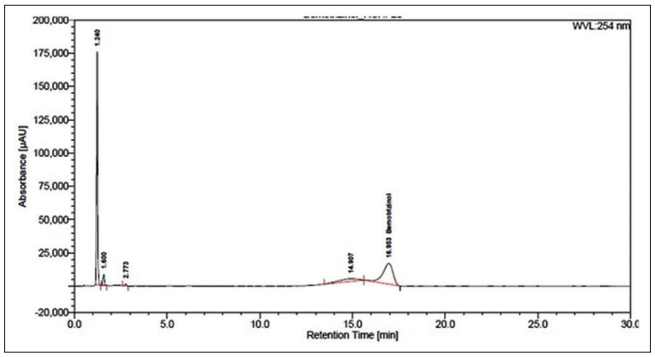

Fig. 7: Acid degradation

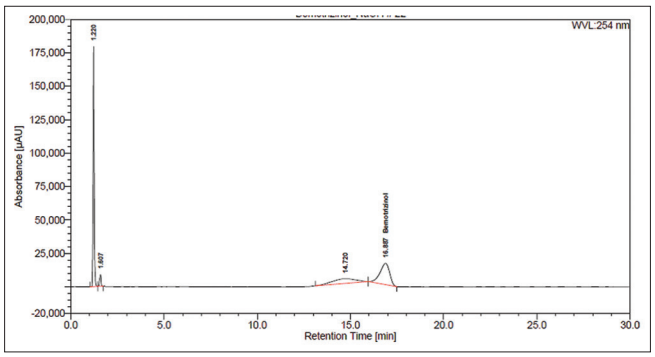

Fig. 8: Alkali degradation

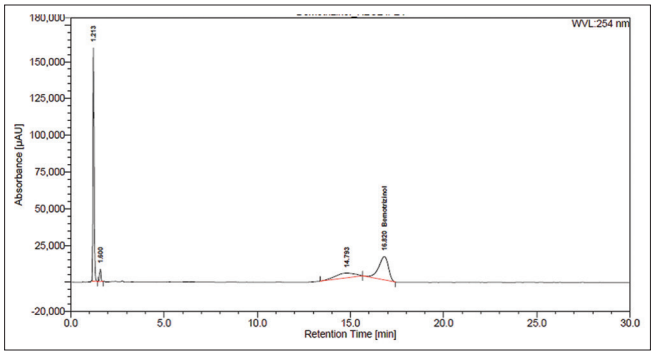

Fig. 9: Oxidative degradation

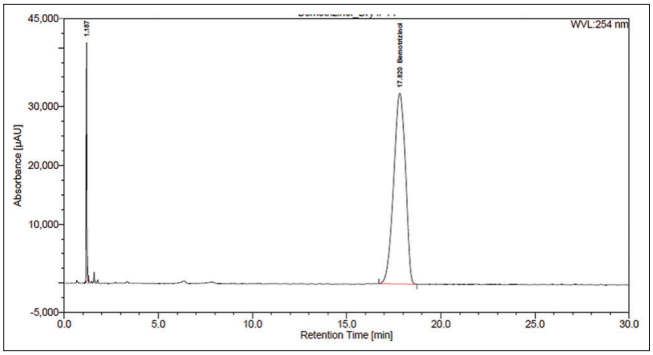

Fig. 10: Dry stage

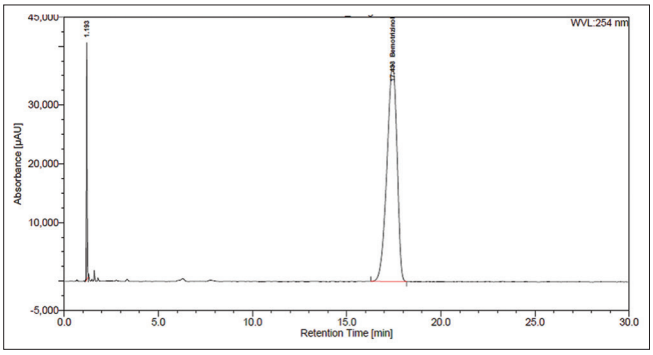

Fig. 11: Wet stage

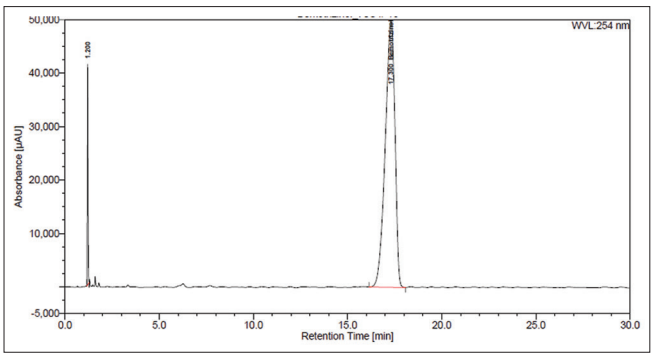

Fig. 12: Thermal degradation

Photolysis

Weight accurately 0.025 g Tinosorb-S and store in a UV chamber at 254 nm in dry and wet conditions for 2 h and transmit into a 50 mL graduated flask. Add 10 mL of trichloromethane and make the volume up to 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL. The chromatograms were obtained after injecting the prepared sample solutions into the chromatographic system (Figs. 10 and 11).

Thermal degradation

Weight accurately 0.025 g Tinosorb-S and kept for 6 h in an oven at 70° and transmitted into a 50 mL graduated flask. Add 10 mL of trichloromethane and make the volume up to 50 mL with methyl alcohol. Dilute 5.0 mL of this solution with methyl alcohol to 20 mL. The chromatogram was obtained after injecting the prepared sample solution into the chromatographic system (Fig. 12).

RESULTS AND DISCUSSION

A high-performance reversed-phase liquid chromatographic analytical technique was successfully developed to estimate Tinosorb-S at 254 nm with an octadecylsilane C18 column (300 × 3.9 mm, 10 micron). For the symmetrical peak, methyl alcohol 100% was applicable as the mobile phase in this chromatographic method. The flow was expanded from 2.0 mL to 2.5 mL/min to obtain a retention time of 17.599 min. The enhancement of the life time of both the chromatographic pump and column was achieved with the use of methyl alcohol as the mobile phase, which reduced the total cost of the analysis. The total run time was 30 min by using the developed chromatographic system, and this acknowledges that a large number of analyses should be done with this short run time. The trade sunscreen preparation was examined, and the recovery of Tinosorb-S from the pre-analyzed batch was between 100.01% and 105.04% (Table 2). The concentration ranges of 70–130 micrograms per mL obeyed linearity, and the linear line was passing through the origin of the standard curve (Fig. 4). The LOQ and LOD were obtained at 0.38 and 0.12 micrograms per mL, respectively. In terms of robustness, little changes in chromatographic conditions were developed in the assay method, which covered column oven temperature (29 and 25°), wavelengths (256 and 252 nm), and flow rate (2.75 mL/min. and 2.25 mL/min.). The overall RSD percentage for estimation of the primary and robustness samples was <2.0%. It is strongly recommended that the experimental technique be robust, precise, economic, accurate, and simple. It was also established that the continuous use of additives and different excipients in the sunscreen formulations does not obstruct the analytical method for the estimation of Tinosorb-S in pure form and its cosmeceutical formulation.

Decomposition of Tinosorb-S was never found in forced degradation studies like photolytic and thermal exposure except in acidic, alkali hydrolysis, and oxidative conditions (Table 5). Degradation of Tinosorb-S was established in an acidic condition, and the degradation was found at 14.907 min. and 2.773 min. (Fig. 7). During alkali degradation, a peak was built up on at 14.720 min (Fig. 8). In oxidation, the decomposition peak response was found at 14.793 min (Fig. 9). The estimation of Tinosorb-S with this analytical method is uninfluenced by different sunscreen preparations, which validates its stability evaluation power.

CONCLUSION

This high-performance liquid chromatographic method was ideal for the quantification of Tinosorb-S in cosmetic formulations. This stability-indicating experiment is economic, highly sensitive, accurate, scientific, uncomplicated, and very specific. Therefore, in the pharmaceutical industry, this analytical technique can be regularly used for the quantification and stability study of Tinosorb-S in sunscreen preparations.

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CONFLICTS OF INTEREST

The authors have no competing interests to declare that are relevant to the content of this article.

AUTHORS FUNDING

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