

**Studies on Novel Calix[4]arene based
Sun-Protection Agents**

**By
SARIKA MRIG
Department of Chemistry**

**Submitted
*In fulfillment of the requirements of the degree of***

Doctor of Philosophy

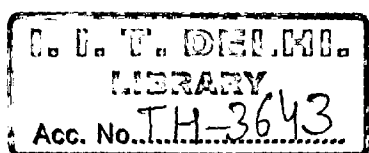
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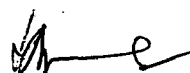
*DEDICATED
TO
MY FAMILY*

CERTIFICATE

This is to certify that the thesis entitled, “**Studies on novel calix[4]arene based sun-protection agents**” being submitted by **Ms. Sarika Mrig** to the Department of Chemistry, Indian Institute of Technology, Delhi, for the award of degree of **Doctor of Philosophy** is a record of bonafide research work carried out by her.

Ms. Sarika Mrig has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis, which to my knowledge has reached the requisite standard.

The results contained in this thesis are original and have not been submitted, in part or full, to any other University or Institute for the award of any other degree or diploma.



Prof. H. M. Chawla

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Journey is easier when you travel together. Interdependence is certainly more valuable than independence. This thesis is the result of five and half years of work whereby I have been accompanied and supported by many people. It is a pleasant aspect that I have now the opportunity to express my gratitude for all of them.

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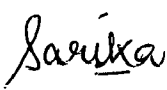
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SARIKA MRIG

ABSTRACT

Primary protection from ultra-violet radiations of sun involves application of sunscreens that usually contain a combination of organic ultra-violet filters and physical blockers to achieve protection against the entire range of ultraviolet radiation (UVR). The thesis titled “*Studies on novel calix[4]arene based sun-protection agents*”, describes the research work carried out on the evaluation of novel calix[4]arene derivatives as sun-protectants through a series of protocols which include determination of the sun-protection factor, UVA protection potential and assessment of *in-vitro* percutaneous permeation as well as preparation of different types of sunscreen formulations through modification of procedures available in the literature. The thesis also presents the work carried out to establish suitable spectrophotometric and liquid chromatographic assay protocols for the calixarene analogs and widely used UV filters available in the market.

Chapter I of the thesis highlights some of the general aspects of sunscreens with special emphasis on organic sunscreens and their mode of action. Salient features of recent research work carried out on the development of broad spectrum UVB and UVA filters and the latest US food and Drug Administration guidelines for their regulation to provide a modern perspective required for sunscreen development have been discussed in this account.

Chapter II of the thesis describes the synthesis of novel calix[4]arenes with extended conjugation and functional groups that have been examined to act as efficient sun-protectants. While structure of synthesized derivatives have been determined by spectroscopic methods (IR, NMR and Mass spectroscopy), the sun protection ability has been determined by measurement of absorption/extinction in the UV region (280-400 nm).

A comparison with prominent sunscreen agents in use today (oxybenzone, octyl methoxycinnamate and avobenzone) has been made. This chapter also embodies the research work accomplished to develop quantitative UV spectrophotometric and HPLC procedures for these sun-protectants. Since sun protection usually involves radicals, these calixarene based sun-protectants have been analyzed for their radical scavenging potential by the well established literature protocols and the results have been discussed in **Chapter III** of the thesis. This chapter also discusses the sun protection factor as segregated through UVA and UVB protection in the light of prevalent protocols. The sun protection potential of the novel calixarene derivatives has been compared with currently employed UVA and UVB sun-protectants. **Chapter IV** of the thesis describes our efforts to develop three different types of sunscreen formulations (gels, creams and lotions). The formulations developed contained new sun-protectant as well as widely used UV filters (octyl methoxycinnamate, oxybenzone and avobenzone) and physical sunscreens (titanium dioxide and zinc oxide) for augmentation of the SPF. The basis for selection of solubilizers, emollient/cosmetic oils, emulsifiers/surfactants and preservatives and other excipients has been discussed in this chapter. **CHAPTER V** of the thesis describes our preliminary results on *in vitro* permeation studies on the selected calixarene derivative by using Modified Franz diffusion cell technique and artificial membrane from various cosmetic vehicles. Permeation studies have also been performed by using an ideal human skin model (pig ear skin membrane) to determine the penetration of calixarene based sun-protectant molecule from the skin membrane into the receptor fluid. Other widely used UV filters have also been examined under similar experimental conditions.

CONTENTS

Title	Page No.
Certificate	i
Acknowledgements	ii
Abstract of the Thesis	iv
List of Figures and Schemes	vi
Notes	xvii
CHAPTER I	
Ultraviolet radiation, skin damage and sun protectants	1
CHAPTER II	
Synthesis and characterization of calix[4]arene based sun-protection agents	50
CHAPTER III	
Evaluation of synthesized sun protection agents	100
CHAPTER IV	
Development of oil in water type formulations for sun-protection	153
CHAPTER V	
Assessment of dermal penetration of developed sun protectants <i>in vitro</i>	194
Brief Bio-Data of the Author	241