Spectroscopic studies on the stability of selected active pharmaceutical ingrediants

A thesis

Submitted

By

Ahmed Mohamed Ahmed Elewa

(B.Sc in chemistry 2005)

For the master degree in Analytical and Inorganic chemistry

To

Chemistry Department

Faculty of science

Al-Azhar University

(2013)

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APPROVAL SHEET

As members of the dissertation committee, we certify that we have read the dissertation prepared by **Ahmed Mohamed Ahmed Elewa,** entiteled **"Spectroscopic studies on the stability of selected active pharmaceutical ingrediants"** and recommend that it be accepted as fulfilling the dissertation requirement for the degree of **Master** in Inorganic and Analytical chemistry.

Date of defense: 30, December 2013

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ACKNOWLEDGMENTS

*I am deepy thanks to **Allah**, by the grace of whom this work was relized.*

*I wish to express my indebtedness and gratitude to **Prof. Dr. Ibrahim Mohamed Abd-Ellah** proffersore of Inorganic chemistry, chemistry department, faculty of science, Al-Azher University for his sincer help, valuable forsight, stimulating directions and expert guidance during supervision of the hole work. It was a graet honor to work under his supervision and guidance.*

*Sincer thanks and special gratitude to Prof. **Dr. Mohamed Abd-Elsamiaa Elnawawy**, professor of Inorganic chemistry, faculty of science, department of chemistry for his stimulating, directions, contructive criticism and sincer help during the course of this work.*

*My heartily appreciation, special thanks, deep gratitude and appreciation to **Prof. Dr. Fawzy Elayazbi** Professore of analytical chemistry, faculty of pharmacy, Alexandria University for his expert guidance, faithful support, fruitful advice and continous help which gratly facilate many of the diffcults during the course of this work at **Medizen Pharamceutical Industries**.*

My scincer thanks to all the staff mebers and colleagues in ***Pharmed Healthcare*** *especially for* ***Dr. Hammam Habsout*** *(Plant manager),* ***Dr. Mohamed Rafaat*** *(QC manger) and my dear frind* ***Samy Haggag*** *(QC section head) for their continous help and assistance in realizing the present thesis.*

I owe a special aknowlagements to my ***Father****, my* ***Mother****, my* ***Grand father*** *my* ***Brother (Maher)*** *and my* ***Sister (Mona)*** *for their ultimate encouragement, continous support and cordial assistance to me while performing this work.*

A special thanks to my ***Fiancee (Dina Abd-Elhamid Mohamed)*** *and* ***all of her family*** *for their support and standing beside me till the finishing of the present work.*

A special thanks also to my ***Cousin Mahmoud Ali*** *for his great help and support.*

Finally, I am greatfull to all those who might have a hand in realizing this thesis in one way or another but it has not been possible for me to mention them by name.

Ahmed M. Elewa

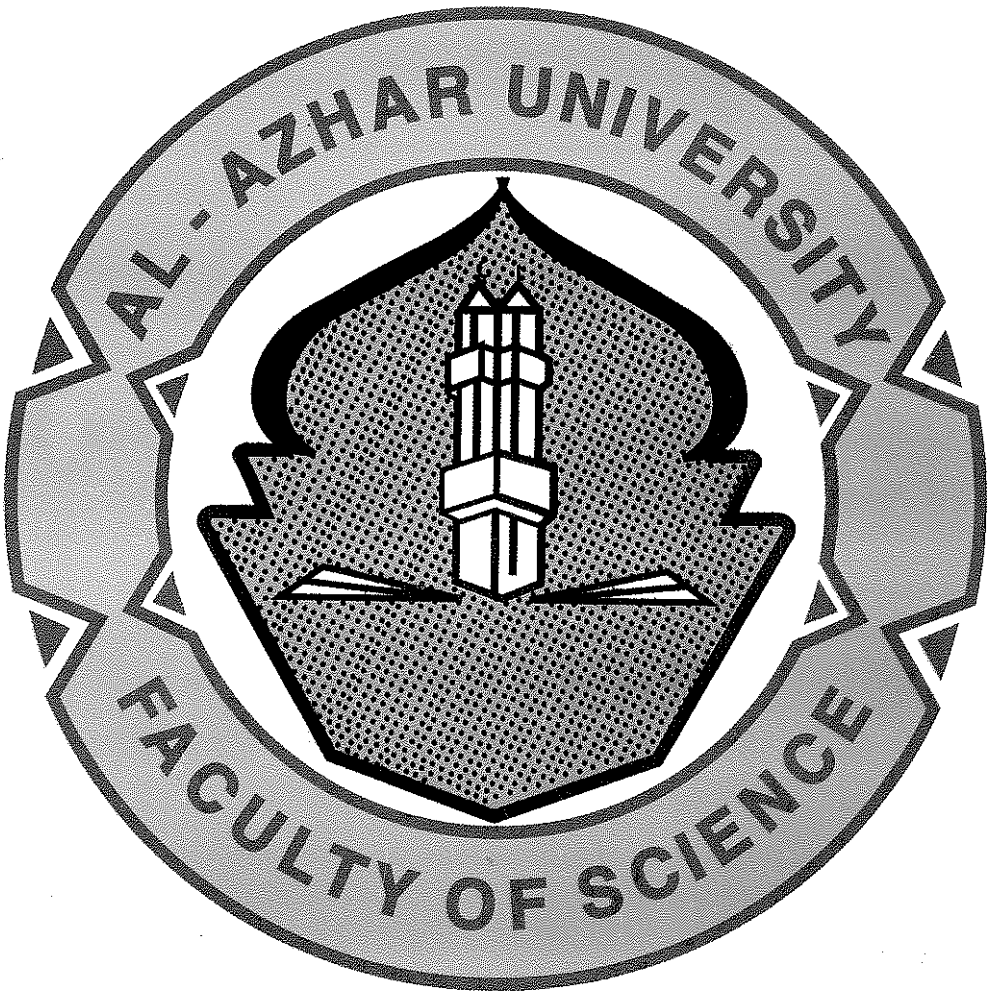
DEDICATION

I WANT TO DEDICATE THIS WORK TO MY FATHER, MOTHER AND MY FIANCÉE

PUBLICATIONS

AL-AZHAR UNIVERSITY

Al-Azhar Bulletin of Science - Basic Science Sector



Vol. 23, No. 2 **(December) 2012**

Al-Azhar Bull. Sci. Vol. 23, No. 1 (Dec.): pp. 45-60, 2012.

VALIDATED STABILITY INDICATED RP-HPLC METHOD FOR SIMULTANEOUS DETERMINATION OF METHYL SALICYLATE, CAMPHOR AND MENTHOL IN CREAM PREPARATIONS

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Abstract

A method for the determination of three active products in pharmaceutical preparation Rheumatizen topical cream was described. The method was based on isocratic elution of Methyl Salicylate, Camphor and Menthol on reversed phase column C_8 Thermo hyperseal 250mm x 4.6mm 5µm - using a mobile phase consisting of mixture of 45% Acetonitrile: 55% (Water+0.2% Triethyl amine adjusted to pH 5 with acetic acid) at a flow rate of 1 ml/min. Quantization was achieved with Refractive index (RI) detector. Linearity of Methyl Salicylate, Camphor and Menthol was found to be from (2.4 mg/ml to 9.6 mg/ml), (0.32 mg/ml to 1.28 mg/ml) and (0.8 mg/ml to 3.2 mg/ml), with variation coefficient 0.9998, 0.997 and 0.998 respectively. The stress testing was carried out by using the solutions that had been treated with [2M NaOH, 2M HCl and 30%H_2O_2] and all of these solutions were leaved for 22 hours at room temperature in tightly closed containers without further heating to avoid the loss via evaporation of the analyets due to their volatility character.

Keywords: Methyl salicylate, Camphor,Menthol., analgesic, anti inflammatory., Reversed phase, refractive index., HPLC, drug analysis

Introduction

Both menthol (MN) and methyl salicylate (MS) are active substances in many medicines commonly used in treatment of rheumatic diseases due its analgesic and anti-inflammatory characteristics[1]. It is difficult to determine these substances in the same preparation due to their similar physical and chemical properties such as volatility and solubility. Another difficulty is a large disproportion (the ratio MS: MN is 8:1 in the medicine under examination). Thus, separation methods are recommended in analysis of these constituents. Among these methods gas chromatography (GC) was used. The gas chromatography methods were used for determining menthol and methyl salicylate in solid and liquid medicines [(2-5)], natural products [(6,7)] and biological material [(8,9)]. Methyl salicylate (MS) is a salicylic acid derivative that is irritant to the skin and is used topically in rubefacient preparations for the relief of pain in musculoskeletal, joint, and soft-tissue disorders. It is also used for minor peripheral vascular disorders such as chilblains and as an ingredient in inhalations for the symptomatic relief of upper respiratory-tract disorders[10]. Camphor