Title	OPTIMIZATION AND VALIDATION OF HPLC METHOD FOR DETERMINATION UV FILTERS IN SUNSCREEN PRODUCTS
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ABSTRACT

This study was aimed to survey the UV filters commonly used in sunscreen products and develop HPLC systems for simultaneous determination of UV filters commonly used in sunscreen products in a single analysis. The sunscreen survey was done in a local department store in Thailand during August to September 2012. Then, the development of a HPLC method to determine optimum separating conditions for the selected UV filters according to the survey by using an isocratic and gradient methods was performed. The development of a HPLC method was performed on Luna® C18 Column (250 x 4.6 mm, i.d. 5 µm particle sizes) and a UV detection wavelength at 325 nm with an injection volume of 20 µl using an isocratic and gradient methods. After that, the developed method was validated according to the ICH guideline Q2 (R1). The validation parameters were linearity of calibration curve, accuracy, precision, specificity, limit of detection and limit of quantitation. Finally, the developed method was applied for the analysis of UV filters in sunscreen samples. The sunscreen samples tested in this study were standard sunscreens, formulated sunscreens and commercial sunscreens. From the survey, ten UV filters selected in this study were: benzophenone-3, bis-ethylhexyloxyphenol methoxyphenyltriazine, butylmethoxy dibenzoylmethane, ethylhexyl dimethyl PABA, ethylhexyl methoxycinnamate, ethylhexyl salicylate, ethylhexyltriazone, homosalate, methylene bis-benzotriazolyl tetramethylbutylphenol and octocrylene. Using isocratic method, the parameters affecting the separation of UV filters were the mobile phase

composition, the column temperature and the flow rate of the mobile phase. The optimized mobile phase composition was comprising of acetonitrile: ethyl acetate: water (95:5:2 v/v/v). The optimized column temperature and flow rate of mobile phase were 25 °C and 1.0 mL/min, respectively. Since the total run time of the developed isocratic analysis is quite long, i.e. 85 min, the development of a HPLC method by using gradient elution was further investigated. The development was accomplished by the continuous change of acetonitrile, ethyl acetate, water, and methanol, which provided a resolution more than 1.5. The separation was controlled at 25 °C with a flow rate 1.0 ml/min. The total analysis time was 16 min. The developed method was validated which showed a good correlation in calibration curve and the correlation coefficient were more than 0.999 for all UV filters. The limit of detection and limit of quantitation were 5 and 0.05 μg/mL, respectively. The accuracy was in the acceptable range of 95-105%. The applicability of the developed method was performed by analyzing UV filters in sunscreen samples including standard sunscreens, formulated sunscreens and commercial sunscreens. The results could confirm that the method was correctly analyzed when compared with the known amount of UV filters containing in standard and formulated formulations. For the commercial products with a various SPF values, the results showed that the type of UV filters detected was conformed to the list of ingredient which shown on the product labels. In addition, the amount of UV filters detected in all products was within the permitted authorized levels regulated by Thai FDA. This study clearly shows that the developed method was simple, fast, and economic. It can determine ten UV filters simultaneously without any interference by materials used to prepare base formulation as well as physical UV filters which may contain in the primary sunscreen products.