

ADDITION OF ETHYLACETATE IN TRIFLUOROACETIC ANHYDRIDE FOR DERIVATIZATION OF *p*-SINEPHRINE

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*Mestranda – Início: 2010/1

Introduction: The *p*-synephrine (SYN) has been used in compounds for weight loss as an alternative to ephedrine. The structural analogy between them provides the same thermogenic mechanism of action: accelerate metabolism and fat burning.^{1,2} However, studies have shown that SYN can induce symptoms like headache, increased blood pressure, arrhythmias, heart attacks and convulsion, such as ephedrine.^{3,4} In Canada, the Health Canada banned the SYN use in dietary products because of 16 suspicion cases.⁵ In 2009, the World Anti-Doping Agency⁶ included SYN in monitoring programs. So, the analysis of SYN is becoming important. To allow GC analysis, SYN is a molecule that requires the derivatization, an important step to improve the chromatographic resolution, analytical efficiency, increase the detectability and make a stable derivative.⁷ So, it is fundamental to study and determine the more adequate derivatizing agent for SYN.

Objective: Test the influence of the addition of ethyl acetate in Trifluoroacetic anhydride (ATFA), and compare this result with Trifluoroacetic anhydride (ATFA) as derivatizing agent to SYN.

Materials and Methods: From a stock solution at 1000 µg/mL of SYN in methanol:isopropanol:NH₄OH, 100 µL was taken to dryness at 40 °C under N₂ flow, and derivatized with 20 µL of ATFA and ATFA+AE. Different times (5, 10, 15, 30, 45 minutes) and temperature (60 °C, 80 °C or 100 °C) reactions were tested. Subsequently, the sample was taken to dryness at 40 °C under N₂ flow and reconstituted with 50 µL methanol. An aliquot of 1 µL was injected into GC-FID. To confirm the peaks, GC/MS were used. Each point was prepared in duplicate.

Results and Discussion: The best derivatization time for ATFA and ATFA+AE is 80 °C for 5 minutes. Though, the chromatographic profile was better for ATFA+AE.

Conclusions: So, due to a good chromatographic profile, ATFA+AE can be considered as a derivatizing agent to SYN at 80 °C for 5 minutes.

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Acknowledgements: Financial support from CAPES/Brazil.