Determination Of Methanol In Alcoholic Drinks By HPLC-FLD Using Pyrene Sulfonyl Chloride As Fluorescence Probe

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A method for the determination of methanol in alcoholic beverages by liquid chromatography and two detectors in series (HPLC-UV-FLD) after derivatization with pyrene sulfonyl chloride (PSCl) was developed. Generally, methanol is determined by HPLC after appropriate derivatization [1-5]. Methanol is a highly toxic compound whose ingestion or inhalation can cause blindness or death. In the alcoholic beverages methanol is formed from the demethoxylation of esterified methoxyl groups of the pectin polymers.

The separation is carried out using reversed phase column C18 (Merck LiChrosphere 100 RP-18, 4.0×250 mm, particle size 5 μm) and a gradient elution program using as mobile phase water and acetonitrile at a flow rate of 2.0 mL min⁻¹. After derivatization, the derivatives were detected with UV, set at 350 nm, and FLD at λₐₑₓ=350 nm and λₑₐₐₗ=460 nm. Several parameters affecting the derivatization process of methanol were investigated and optimized. These parameters are the temperature and the duration of the derivatization, the concentration of KOH or N₂CO₃ aqueous solution and the quantity of the pyrene sulfonyl chloride on the derivatization process. After optimization, the method was validated.

The instrumental detection limits were 0.93 μmol mL⁻¹ for the UV detector and 0.41 μmol mL⁻¹ for the FLD. The method LOD was 2.55 μmol mL⁻¹ for the UV detector and 0.62 μmol mL⁻¹ for the FLD. The method was applied in traditional Greek distillates ‘ouzo’ and ‘tsipouro’, which are produced by the distillation of the residual of wine making, mostly the marc. The average recoveries of methanol for spiking levels of 12.5-75.0 μmol mL⁻¹ were between 94.4%-108 % for ouzo and 60.2%-97.7 % for tsipouro. Finally, the method was applied to commercial and homemade alcoholic beverages and the concentrations of methanol were found in the range of 64.7 – 193 μmol mL⁻¹, below the legislation limit of 312.5 μmol mL⁻¹. The results showed that the homemade drinks present higher concentrations of methanol than the commercial ones.

References