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Usefulness of a fused-core stationary phase for the development of a fast liquid chromatography method for penicillin determination

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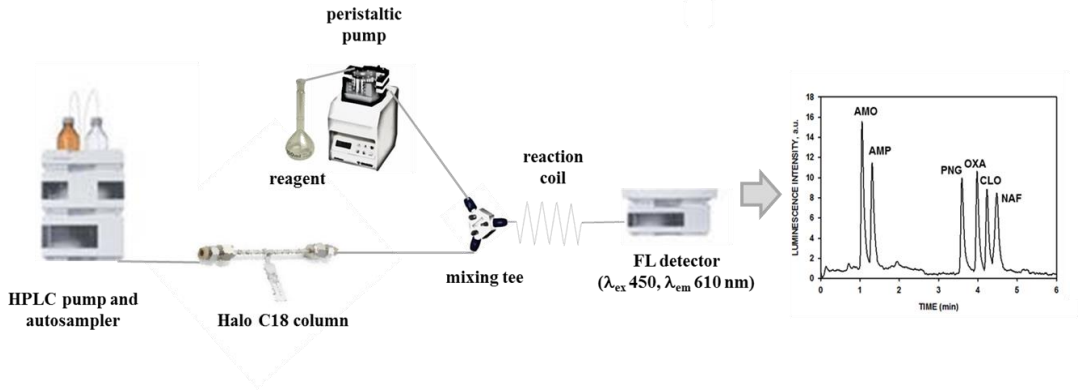
The use of stationary phases based on partially porous microparticles, such as fused-core or core-shell microparticles is attracting much attention owing to their compatibility with conventional HPLC instruments together with the fact that they allow fast and efficient separations to be achieved. Fused-core microparticles are composed of a solid nucleus coated with a porous shell made from nanoparticles. This combination reduces the longitudinal diffusion, so higher peak capacity values are obtained, in comparison with those typical of phases incorporating fully porous microparticles, and hence, narrower peaks. The method presented here demonstrates the usefulness of the combination of these fast LC separations with post-column derivatization procedures in order to achieve proper optical detection of analytes with poor optical properties, but still with adequate chromatographic resolution.

Six penicillin antibiotics used in veterinary practice, such as amoxicillin (AMO), ampicillin (AMP), penicillin G (PNG), oxacillin (OXA), cloxacillin (CLO) and nafcillin (NAF) have been used as model analytes. The tris(2,2'-bipyridyl)ruthenium(II) [$Ru(bpy)_3^{2+}$] - Ce(IV) system has been used as post-column derivatization reagent, obtaining a luminescence signal (λ_{em} 610 nm) proportional to the analyte concentration when the system is excited at 450 nm. The use of a commercial fused-core Halo C18 column provides the fast separation of these antibiotics with retention times lower than 4.5 min. The manifold of the analytical system developed and a chromatogram obtained for the separation of the six penicillins are shown below:



The dynamic ranges of the calibration graphs are 100 - 10000 ng mL^{-1} for all the antibiotics assayed and the limits of detection are in the range of 44 - 51 ng mL^{-1}. The precision, established at two concentration levels of each analyte and expressed as the percentage of the relative standard deviation is in the range of 6.9 - 9.8 %. The method has been satisfactorily applied to the analysis of water and pharmaceutical samples, with recoveries ranging from 88.6 to 108.5%.