Luminescent determination of fluoroquinolones in milk samples by liquid chromatography/post-column derivatization with terbium oxide nanoparticles

Gabriela Salomé Yánez-Jácome, **María Paz Aguilar-Caballos**, Agustina Gómez-Hens
Analytical Chemistry Department. Institute of Fine Chemistry and Nanochemistry. Campus of Rabanales. Annex to Marie Curie Building. University of Córdoba. 14071-Córdoba. Spain.Phone number: +34-957218645, Fax: +34-957218644
e-mail: qa1gohea@uco.es, web: http://www.uco.es/investiga/grupos/FQM-303

The potential usefulness of Tb_4O_7 nanoparticles (Tb_4O_7 NPs) for the luminescent detection of fluoroquinolone antibiotic residues in milk samples has been studied by using a liquid chromatography (LC)-post-column derivatization approach. Seven fluoroquinolones of veterinary use were chosen as model analytes to develop this LC method. The derivatization step is based on the post-column reaction between the fluoroquinolones with Tb_4O_7 NPs to give rise to luminescent chelates, and the measurements are performed at λ_{ex} 340 and λ_{em} 545 nm. A modular system has been used to develop this approach (Figure 1).

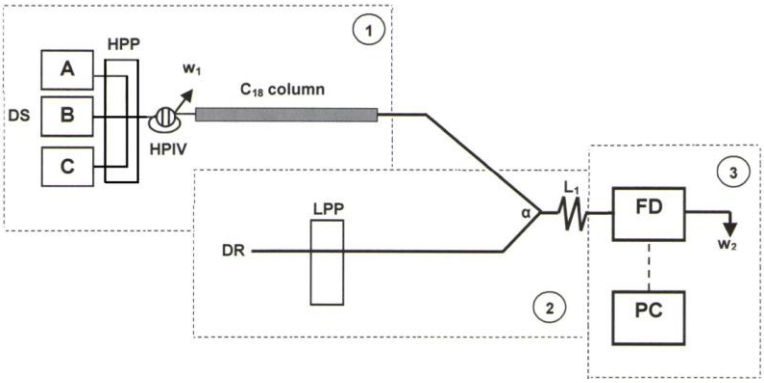

Figure 1. Modular system used to develop the method: 1, 2 and 3: chromatographic, derivatizing and detection subsystems, **A, B and C,** MetOH, ACN and acetic acid, **DS,** delivery system; **HPP,** high-pressure quaternary gradient pump, **HPIV, h**igh-pressure injection valve, **LPP,** low-pressure pump, **L_1,** mixing reactor, **FD,** fluorescence detector, **PC,** personal computer, **DR, d**erivatizing reagent, **w_1 and w_2,** waste

The dynamic ranges of the calibration graphs and limits of detection are, respectively: 65 – 900 and 35 ng mL^{-1} for marbofloxacin, 7.2 – 900 and 2.5 ng mL^{-1} for ciprofloxacin, 6 - 900 and 2 ng mL^{-1} for danofloxacin, 50 – 900 and 20 ng mL^{-1} for enrofloxacin, 35 – 900 and 12 ng mL^{-1} for sarafloxacin, 5 – 900 and 2 ng mL^{-1} for oxolinic acid, and 7 – 900 and 2.5 ng mL^{-1} for flumequine. These features have compared to those provided by previously reported methods using terbium(III)[1]. The precision has been established at two concentration levels of each analyte and expressed as the percentage of the relative standard deviation with values in the range of 1.9-8.1 %.

This method has been applied to the analysis of skimmed, semi-skimmed and whole milk samples, with recoveries ranging from 89.0 to 106.5 %.

References

[1]Rodríguez Díaz, R.C., Fernández Romero, J.M., Aguilar Caballos, M.P., Gómez Hens, A. J. Agric. Food Chem., **54** (2006) 9670-9676.