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

Gabriela Salomé Yáñez-Jácome, Maria Paz Aguilar-Caballlos, Agustina Gómez-Hens
Phone number: +34-957218645, Fax: +34-957218644
E-mail: ga1gohea@uco.es, web: http://www.uco.es/investiga/grupos/FQM-303

The potential usefulness of Tb₄O₇ nanoparticles (Tb₄O₇ 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 Tb₄O₇ NPs to give rise to luminescent chelates, and the measurements are performed at λₜₚ 340 and λₑₘ 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, high-pressure injection valve; LPP, low-pressure pump, L₁, mixing reactor, FD, fluorescence detector; PC, personal computer, DR, derivatizing reagent, w₁ and w₂, 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). 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