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Determination of Fluoriquinolones in Soil

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The occurrence of antibiotics and other pharmaceuticals in the environment is of increasing concern. Fluoroquinolones (FQs) represent a very potent class of human and veterinary antibiotics that are regularly used as the second line of defense when other treatments fail, but concern is growing that its frequent use could lead to antibioticresistant bacteria. The U.S. Food and Drug Administration's Center for Veterinary Medicine has proposed banning enrofloxacin, one of the major FQs in the United States, for use in poultry farming because of evidence that shows it contributes to the development of FQ-resistant *Campylobacter* infections in humans [1]. In a 2001 discussion paper, the European Agency for the Evaluation of Medicinal Products proposed that an environmental risk assessment be required for human medicinal products that have predicted concentrations in surface water greater than 0.01 micrograms per liter [2]. The presence of these compounds in soil has scarcely being investigated as compared to the aquatic media [3].

The aims of the present study were: (i) to develop a sensitive analytical protocol to determine norfloxacin, flumequine, ofloxacin, danofloxacin, marbofloxacin, pipemidic acid, levofloxacin, enrofloxacin and ciprofloxacin in sludge-amended soils; (ii) to identify and characterize each FQ by means of electrospray tandem mass spectrometry (LC-ESI-MS/MS) and (iii) to compare environmental concentrations of the nine FQs in soil samples from agricultural fields differing in the type of soil and in the sewage sludge application.

The procedure involves pressurized liquid extraction (PLE) using 100 % methanol and clean-up with C18 material followed by LC-tandem MS detection. Recoveries from soil were higher than 65 % at the limit of quantification (LOQ) level for all the FQs tested with relative standard deviations (RSDs) lower than 19 %. LOQs ranged from

2 to 20 μ g kg⁻¹.

This study shows that antimicrobials are present in soil at concentrations with maxima ranging from 120 μ g kg⁻¹ of enrofloxacin to 800 μ g kg⁻¹ of ofloxacin. Thus, field experiments of sludge-application to agricultural land confirmed the long-term persistence of trace amounts of FQs in sludge-treated soils and indicated a limited mobility of FQs into the subsoil. Sludges prepared for amending soil must receive most attention because of the evidence that these compounds are present in important amounts.

References

[1] Soil Association's reports on the *Use and Misuse of Antibiotics in UK Agriculture* available at: www.soilassociation.org under 'Antibiotics' in the Library section

[2] Committee for medicinal products for human use (CHMP)*draft* "guideline on the environmental risk assessment of medicinal products for human use" European Medicines Agency, *Pre-Authorisation Evaluation of Medicines for Human Use, London, 2001*, CHMP/SWP/4447/00 draft

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