

CON-PRA-041

CONFIRMATORY METHODS

Corticosteroids

• Context

Natural corticosteroid hormones are produced in the surrenal gland cortex. Because of their anti-inflammatory properties, they were used very early for therapeutic purposes and a lot of more active synthetic glucocorticoids have been chemically synthesised for human and veterinary medicines. This utilisation concerns registered compounds and imposes withdrawal periods between treatment and slaughtering when administered to food producing animals. Maximal residue levels (MRL) have also been fixed in Europe for several corticosteroids. Besides this legal use, corticosteroids are known to have growth promoting effect, in low concentration conditions or when associated with other molecules like β -agonists or anabolic steroids. In order to check whether the regulation is fulfilled or not (respect of MRL or antidoping-like approaches), controls have to be organised and performed. As GC-MS is not a suitable technique for these compounds (because of a relative high polarity and thermal lability), LC-MS/MS with electrospray (ESI) or atmospheric pressure chemical ionisation (APCI) appears as a powerful tool for confirmatory analysis.

• General objective(s)

The goal of this practical training is to demonstrate the suitability of LC-MS/MS for the detection and identification of polar and thermolabile molecules such as corticosteroids, to present the various technical parameters to consider and optimise during such developement of a measurement method, and finally to give a chronological and comprehensive practical guideline from the optimisation of ionisation interface and mass analyser parameters through the choice of appropriate diagnostic ions and acquisition parameters.

• Pedagogical objectives

- ✓ Know the basic rules of corticosteroid fragmentation after electrospray ionisation (ESI+/ESI-)
- ✓ Know the main chronological steps for a typical optimisation of a LC/MS/MS measurement method

• Main items

- ✓ Introduction on corticosteroid compounds, interpretation of MS spectra, optimisation of ionisation source parameters, interpretation of MS/MS spectra, optimisation of fragmentation parameters, signal acquisition.

• Pedagogical tools

- ✓ PowerPoint slide show
- ✓ Practical exercise printout (real case study including mass spectra and ion chromatograms to interpret)

• Duration

- ✓ 2 hours

• Pre-requisite

- ✓ Chemical structure (understanding)
- ✓ Basics of mass spectrometry (CON-THE-010, CON-THE-020 and CON-THE-030)