Optimization of solid phase extraction clean up and validation of quantitative determination of corticosteroids in urine by liquid chromatography-tandem mass spectrometry

A solid phase extraction (SPE) method for extraction and clean up of 9 synthetic corticosteroids was optimized for quantification by reversed-phase high-performance liquid chromatography/negative electrospray ionisation mass spectrometry. Clean up was accomplished using a mixed mode polymeric strong anion exchange SPE column. The final method was validated according to EU regulations for determination of residues of veterinarian drugs in products of animal origin. Initial results showed a large difference in ion suppression between samples of porcine and bovine urine. The aim of optimisation was to design a procedure that minimised this difference while using a single SPE procedure and a fast HPLC method that enabled sufficient separation of the epimers beta- and dexamethason. To include conjugated corticosteroids in the analysis, the sample was hydrolysed with Helix Pomatia β-glucuronidase/aryl sulfatase. For the final method, which included fluocinolone acetonid, triamcinolone acetonid, beclomethasone, flumethasone, dexamethasone, betamethasone, 6α-methylprednisolone, prednisone and prednisolone, a quantification based on spiked samples carried through the entire analytical procedure was used. For quantification of triamcinolone acetonid an internal standard (triamcinolone acetonid-D6) was used. Relative average recoveries from 96 to 103% were found, except for beclomethason (113%). Absolute average recoveries were 81–99%. Quantification limits (decision limits, CCα) were demonstrated to be not higher than 1 μg L−1 (3 μg L−1 for prednisone and prednisolone). The internal reproducibility, determined by triplicates from spiking at three different levels in six analytical series was 7–19% (at 2–4 μg L−1) except for prednisone and prednisolone (26–27% at 3–6 μg L−1).

General information
State: Published
Organisations: Division of Food Chemistry, National Food Institute
Contributors: Andersen, J. H., Hansen, L. G., Pedersen, M.
Pages: 216-224
Publication date: 2008
Peer-reviewed: Yes

Publication information
Journal: Analytica Chimica Acta
Volume: 617
Issue number: 1-2
ISSN (Print): 0003-2670
Ratings:
BFI (2018): BFI-level 2
Web of Science (2018): Indexed yes
BFI (2017): BFI-level 2
Scopus rating (2017): CiteScore 5.06
Web of Science (2017): Impact factor 1.363
Web of Science (2017): Indexed yes
BFI (2016): BFI-level 2
Scopus rating (2016): CiteScore 5.01
Web of Science (2016): Impact factor 1.74
Web of Science (2016): Indexed yes
BFI (2015): BFI-level 2
Scopus rating (2015): CiteScore 4.94
Web of Science (2015): Impact factor 1.682
Web of Science (2015): Indexed yes
BFI (2014): BFI-level 2
Scopus rating (2014): CiteScore 4.64
Web of Science (2014): Impact factor 2.003
Web of Science (2014): Indexed yes
BFI (2013): BFI-level 1
Scopus rating (2013): CiteScore 4.74
Web of Science (2013): Impact factor 1.547
ISI indexed (2013): ISI indexed yes
Web of Science (2013): Indexed yes