Rapid analysis of Sedatives, basic and ACIDIC NSAIDs in Kidney and Muscle by LC-MS/MS

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Introduction

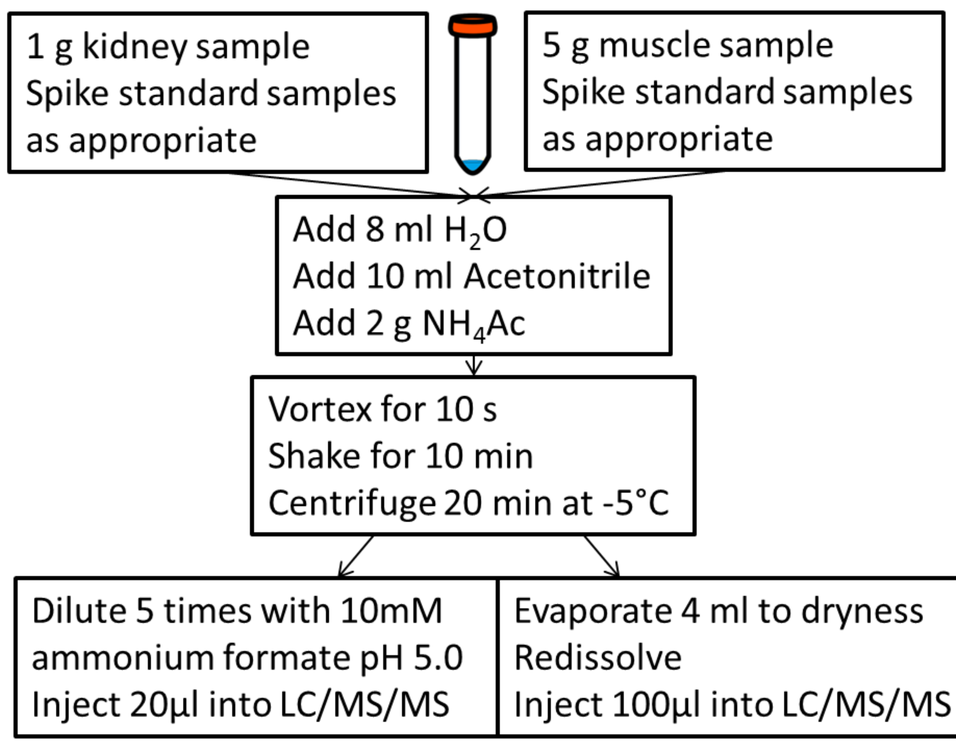
A rapid method for quantitative and confirmative analysis of sedatives, basic and acidic NSAIDs (Non-Steroidal Anti-Inflammatory Drugs) in mammal kidney and basic and acidic NSAIDs in poultry muscle using liquid chroma-tography-tandem mass spectrometry (LC/MS/MS) was developed and validated. 7 sedatives in kidney and 19 NSAIDs in kidney and 20 NSAIDs in muscle were included in the method.

Validation

The validation was performed according to Commission Decision 2002/657/EC. In order to compensate for matrix effects, a six point matrix matched standard curve (0.25 to 4.0 of the target concentration) was used.

The target concentration was either the MRL, or chosen in accordance with concentrations proposed in the guidance-paper published by the European Community Reference Laboratories (EURLs), or as low as possible, shown in Table 1.

Sample preperation



UPLC

Waters Acquity

Column: Waters ACQUITY UPLC BEH C18

Flow rate: 0.45 ml/min

Temperature: 40°C

Gradient

A: Metanol/acetonitril 80/20

B: 10 mM ammoniumformiat (aq), pH 5,0

Tid (min)	%A	%B
Initial	5	95
1	5	95
1.05	35	65
7	95	5
10	95	5
10.1	5	95
12	5	95

Detection

Waters Xevo TQS Electrospray ionisation

ESI+ and ESI-

Table 1

Analyte	Target conc. Kidney (µg/kg)	CC_α (µg/kg)	CC_β (µg/kg)	Target conc. Muscle (µg/kg)	CC_α (µg/kg)	CC_β (µg/kg)
MAA	100[a,b]	125	150	5	0.9	1.5
AA	100	165	229	5	1.7	3.0
FAA	100	180	260	5	2.2	4.0
AcAA	100	181	262	5	1.9	3.3
Carprofen	1000[a]	1211	1421	5	1.0	1.7
Celecoxib	10	4.5	7.9	5	1.4	2.3
Diclofenac	10[a]	12	14	5	0.6	1.0
Firocoxib	10[a]	11	13	5	0.7	1.1
Flunixin (b/p/e)	100/30/200[a]	111/33/222	122/37/244	5	0.7	1.1
Ibuprofen*	10	-	-	5	-	-
Ketoprofen	10	3.3	5.4	5	1.3	2.1
Mefenamic acid	10[c]	4.1	6.8	5	1.7	2.7
Meloxicam	65[a]	72	80	5	0.6	0.9
Naproxen	10[c]	3.1	5.3	5	0.8	1.4
Oxyphenbutazone	5[c]	3.2	5.4	5	2.0	2.6
Phenylbutazone[a]	5[b,c]	3.3	5.8	5	0.6	1.0
Rofecoxib	10	2.2	3.8	5	1.4	2.2
Salicylic acid	-	-	-	400[a]	470	533
Tolfenamic acid	100[a]	116	133	5	0.7	1.1
Vedaprofen	1000[a]	1297	1594	5	0.7	1.2
Acepromazine	50[c]	6.2	11	-	-	-
Azaperol	100[a]	109	119	-	-	-
Azaperone	100[a]	113	125	-	-	-
HEPS*	10	-	-	-	-	-
Karazolol (b/p)	15/25[a]	19/ 32	23/ 38	-	-	-
Propionylpromazine	50[c]	7.8	13	-	-	-
Xylazine	10	1.9	3.3	-	-	-

[a)] MRL

[b)] Marker residue

[c)] Recommended concentration (EURL)

*) Only screening

Summary

The method fulfils the criteria for confirmatory analysis according to Commission Decision 2002/657/EC, except for Ibuprofen and HEPS, and can also be used for screening.

The proposed method is simple and specific, allowing a single analyst to easily prepare a batch of 40 samples in less than 4 hours.

Livsmedelsverket

National Food Agency