

Summary of methods used by participants (samples of urine).

Nr	Methods	Technique	Internal stand.	CC α and CC β (ppb)
15	Hydrolysis (Suc d'helix pomatia) 15 hours 52 °C Liquid extraction diethylether SPE ENVI Chrom P. LLE with Diethylether SPE SiOH/NH ₂	GCMS/MS	17 β -Estradiol-d ₃	Screening: CC α = 0.01 CC β = 0.03 Confirmation: CC α = 0.04 CC β = 0.08
16	Hydrolysis (β -glucuronidase)(Suc d'helix pomatia) (2 hours, 50 °C) SPE extraction Oasis / NH ₂ Coupled-column HPLC	LCMS – TSQ Quantum	17 β -Estradiol-d ₃	Information not available
17	Hydrolysis (Suc d'helix pomatia) (16 hour, 37 °C) SPE- extraction C18/NH ₂	LCMS/MS- ESI	17 β -Estradiol-d ₃	Information not available
19	Liquid extraction with acetate buffer (pH 5) SPE- extraction C ₁₈	LC-APCI- MS/MS	17 β -Estradiol-d ₃	17 α -E: CC α = 0.06 CC β = 0.08 17 β -E: CC α = 0.03 CC β = 0.04
20	Hydrolysis (β -glucuronidase/arylsulfat.) pH=4.8 (16 hours, 37 °C) SPE- extraction C ₁₈ /NH ₂ HPLC fraction Derivatisation MSTFA ⁺⁺	GCMS-MS	17 β -Estradiol-d ₃	Information not available
21	Hydrolysis (overnight, 37 °C) (pH=5.2) (β -glucur. from Suc d'helix pomatia) SPE- extraction C ₁₈ /NH ₂ HPLC fraction Derivatisation MSTFA/TMIS/DTE	GCMSD	17 β - Nortestosterone- d ₃	17 α -E: CC α = 0.5 CC β = 1.0 17 β -E: CC α = 0.5 CC β = 1.0
22	Hydrolysis (β -glucuronidase/arylsulfat.) (3 hours, 40 °C) SPE- extraction Oasis LLE with diethyl ether-petr.benzine Derivatisation MSTFA ⁺⁺	GCMS-MS Ion-trap	Dienestrol	CC α = 0.5 CC β = 1.0
Nr	Methods	Technique	Internal stand.	CC α and CC β (ppb)
26	Hydrolysis (β -glucuronidase/sulfatase) (2 hours, 62 °C) LLE extraction with diethylether HPLC fraction Derivatisation MSTFA ⁺⁺	GCMS-MS	Equilinine	CC β = 0.5
28	Hydrolysis (β -glucuronidase/arylsulfatase) (2 hours, 55 °C) SPE-Oasis Trisbuffer/Hexane Trisbuffer/TBME MEOH/NaOH (40:60)	GCMS	17 β -Estradiol-d ₃	CC α = 0.5 CC β = 0.55

29	Hydrolysis (β -glucuronidase) (pH 5.2) (2 hours, 55 °C) SPE- extraction C18/Oasis HLB Derivatisation	GCMS	Testosterone-d2	CC α = 0.4
31	Hydrolysis (β -glucuronidase) (pH 5.0) (2 hours, 60 °C) SPE- extraction C18/NH2	GCMS-MS	Ostriol	Information not available.
32	Hydrolysis (β -glucuronidase) (2 hours, 62 °C) Liquid extraction diethyl ether HPLC fraction Derivatisation MSTFA++	GCMS-MS	Equilinine	CC α = 0.66 CC β \leq 2 ppb
34	Hydrolysis (β -glucuronidase/arylsulfatase) (3 hours, 52 °C) SPE- extraction C18/NH2 HPLC fraction Derivatisation BSA:TMCS (10:1)	GCMS	Information not available	Information not available.
38	Hydrolysis (β -glucuronidase) (overnight room temp.) Extrelut NT (Merck) Derivatisation HFBA	GCMS	17 β -Estradiol	CC α = 0.2 / 0.3 CC β = 0.3 / 0.4
43	SPE extraction C18/NH2 LLE with hexane/dichlormethane	GCMS- NCI	Testosterone-d2	CC α = 1.0 CC β = 1.2