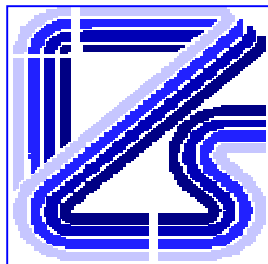


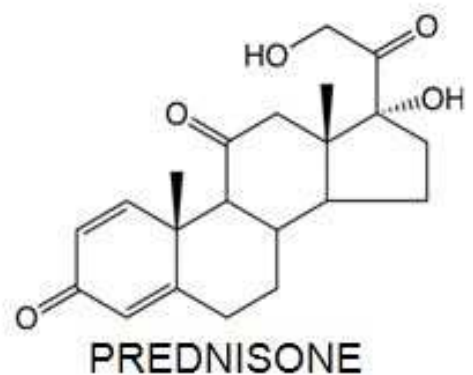
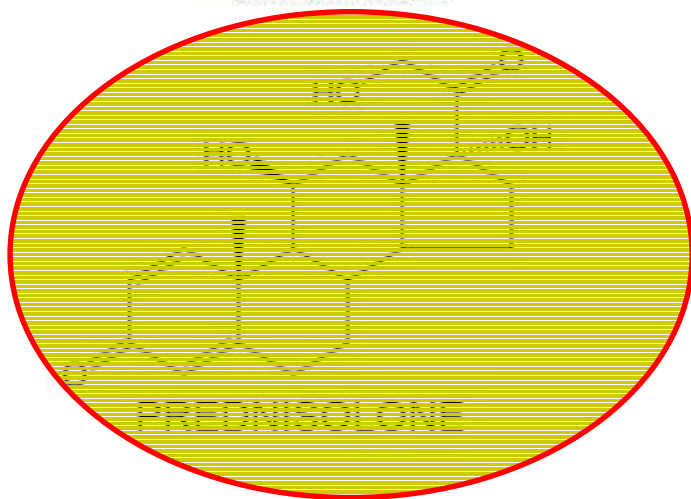
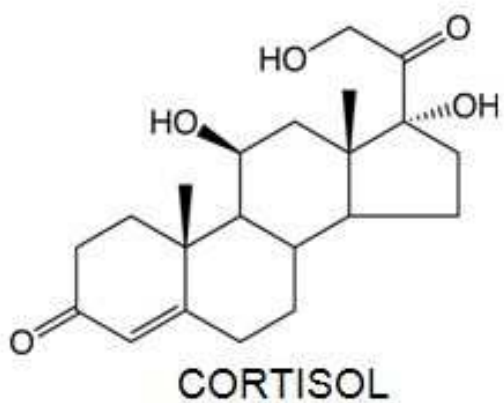
Analytical investigations about the presence of prednisolone in cow urine

Roma 12.10.2012 ore 14.30 / 14.50



Guglielmo Dusi – Reparto Chimica degli alimenti di Origine animale

CORTICOSTEROIDS



Exact Mass: 360.193675

Dr. G. DUSI - IZSLER

LIMITS

REGOLAMENTO (UE) N. 37/2010 DELLA COMMISSIONE
del 22 dicembre 2009

Prednisolone	Bovine	4 µg/kg	Muscle
		4 µg/kg	Fat
		10 µg/kg	Liver
		10 µg/kg	Kidney
		6 µg/kg	Milk

URINE → MATRIX NOT CONSIDERED

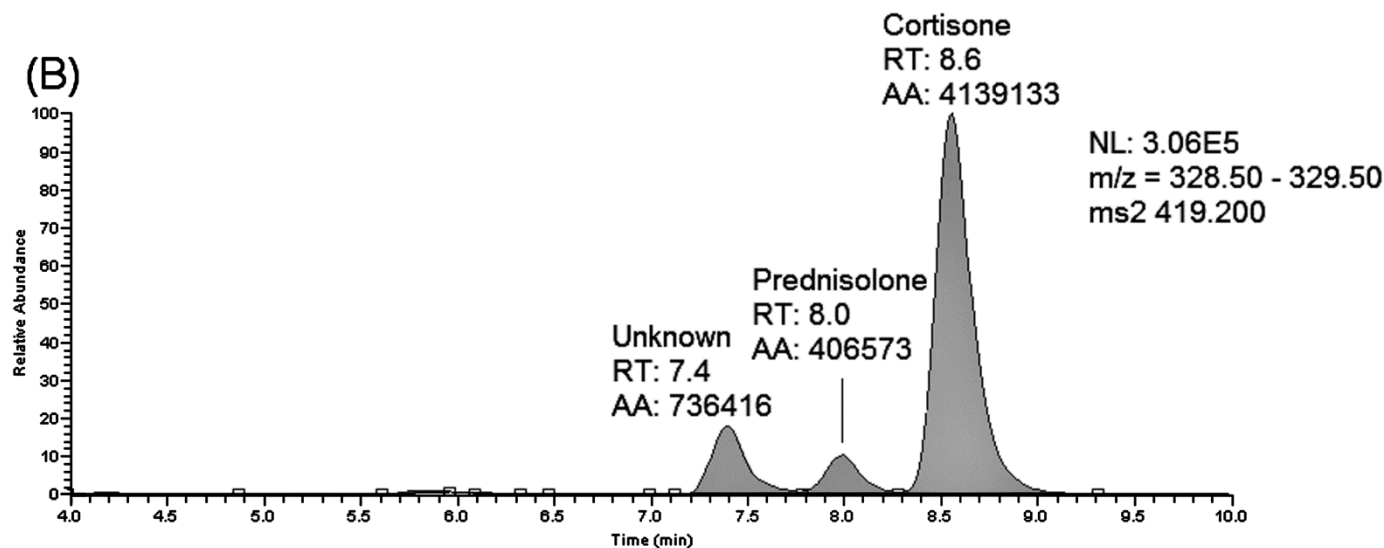
- LC - (LR)MS/MS \rightarrow QqQ
- LC - (HR)MSⁿ \rightarrow LT-Orbitrap

LOW RESOLUTION MASS SPECTROMETRY

TRIPLE QUADRUPOLE

- sensitivity → +++
- qualitative analysis (> 4 IP) → ++
- quantitative analysis → +++

LC-(LR)MS/MS: QqQ



Unknown

#1722-1747 RT: 7.35-7.43 AV: 6

ms2 419.200 [279.500-280.500, 294.500-295.500, 328.500-329.500]

m/z= 279.50-419.70

m/z	Intensity	Relative
279.94	16696.5	29.79
294.98	36318.9	64.79
329.02	56053.3	100.00

Prednisolone

#1902-1927 RT: 7.95-8.03 AV: 6

ms2 419.200 [279.500-280.500, 294.500-295.500, 328.500-329.500]

m/z= 279.50-419.70

m/z	Intensity	Relative
279.97	11709.2	35.85
294.98	12070.8	36.96
329.01	32657.9	100.00

HIGH RESOLUTION MASS SPECTROMETRY

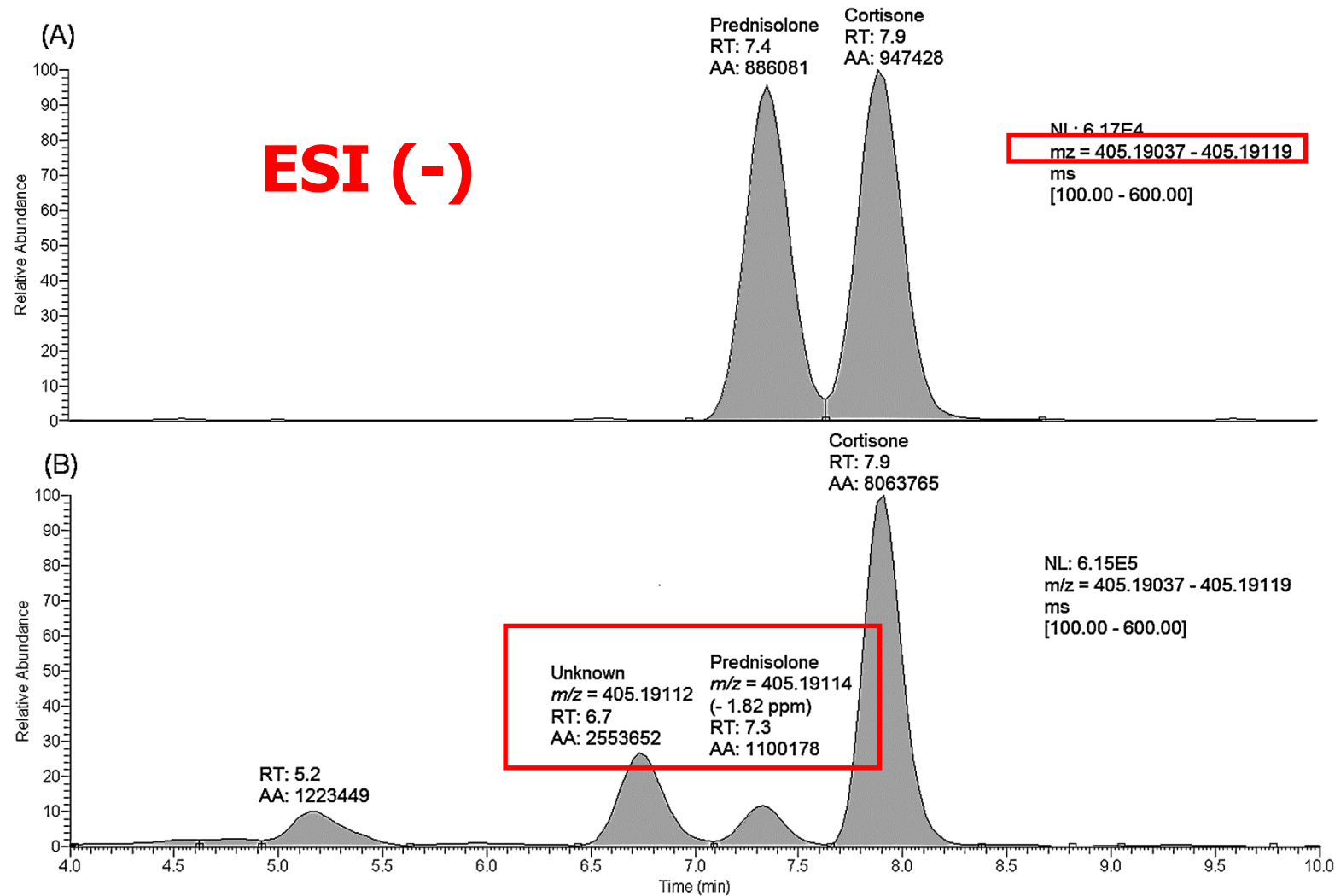
es. Q-Tof, LT-Orbitrap

- sensitivity → ?
- qualitative analysis → +++
- quantitative analysis → ?

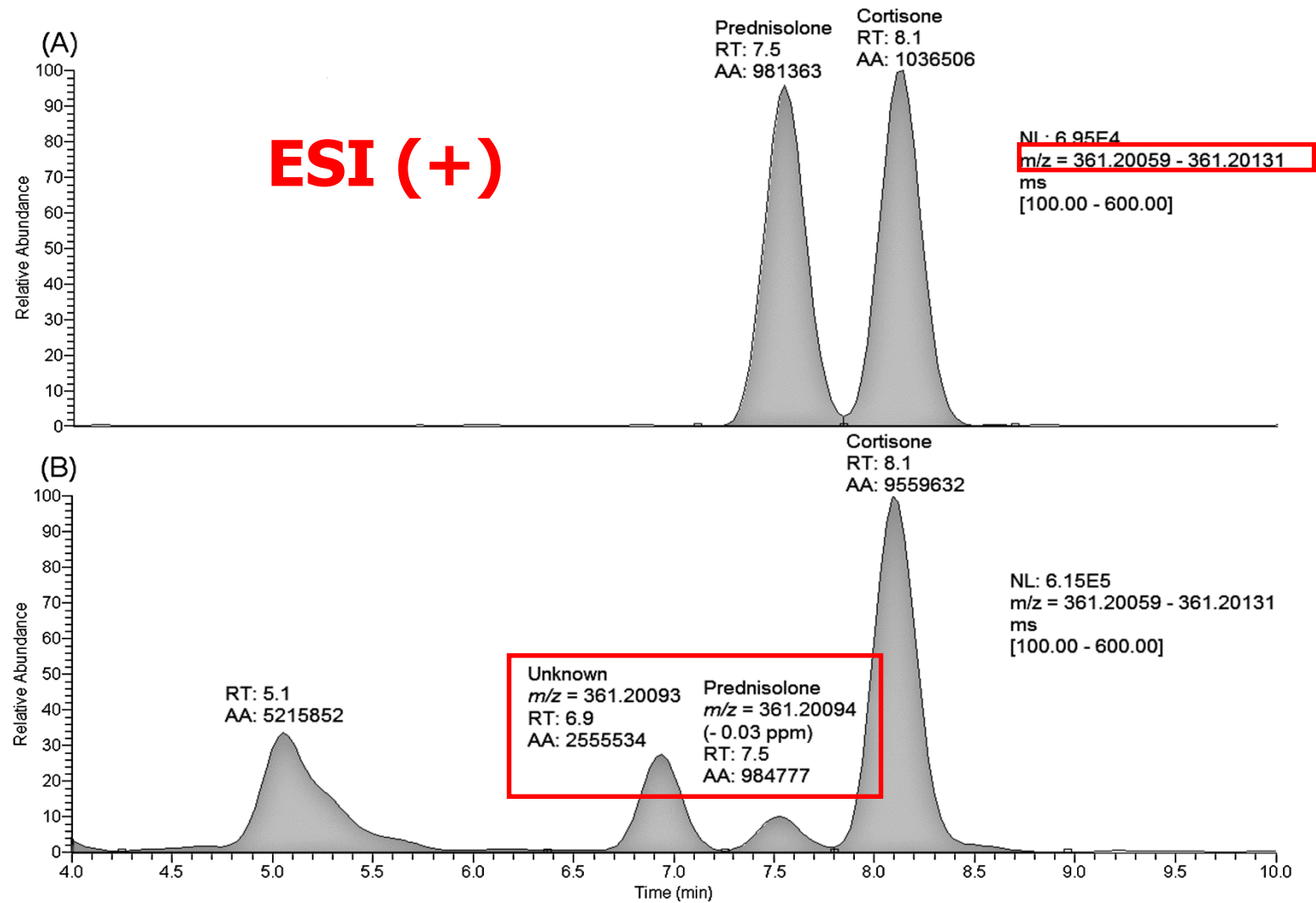
LT-Orbitrap

Source	MS				MS/MS (HCD)				MS/MS/MS (CID)			
	Ionic formula	Exact mass (m/z)	Precur. ion (m/z)	Coll. energy	Ionic formula	Exact mass (m/z)	Precur. ion 1 (m/z)	Coll. energy	Precur. ion 2 (m/z)	Coll. energy	Ionic formula	Exact mass (m/z)
ESI (-)	[C ₂₂ H ₂₉ O ₇] ⁻ [M+HCO ₂] ⁻	406.19188	406.2	96	[C ₂₀ H ₂₅ O ₄] ⁻	329.17683	406.2	26	329.2	36	[C ₁₉ H ₁₆ O ₆] ⁻	286.13397
					[C ₁₉ H ₁₅ O ₅] ⁻	286.13397					[C ₁₈ H ₁₆ O ₅] ⁻	280.11049
					[C ₁₈ H ₁₆ O ₅] ⁻	280.11049					[C ₁₂ H ₁₁ O ₂] ⁻	187.07645
					[C ₁₂ H ₁₁ O ₂] ⁻	187.07645						
					[C ₂₁ H ₂₇ O ₄] ⁺	343.19039					[C ₂₁ H ₂₅ O ₃] ⁺	326.17982
ESI (+)	[C ₂₁ H ₂₉ O ₅] ⁺ [M+H] ⁺	361.20096	361.2	20	[C ₂₁ H ₂₉ O ₅] ⁺	326.17982	361.2	16	343.2	26	[C ₂₁ H ₂₃ O ₂] ⁺	307.16926
					[C ₂₁ H ₂₃ O ₂] ⁺	307.16926					[C ₂₁ H ₂₁ O] ⁺	289.16869
					[C ₂₁ H ₂₁ O] ⁺	289.16869					[C ₂₀ H ₂₃ O] ⁺	279.17434
					[C ₂₀ H ₂₃ O] ⁺	279.17434					[C ₁₀ H ₁₁ O] ⁺	147.08044
					[C ₁₀ H ₁₁ O] ⁺	147.08044						

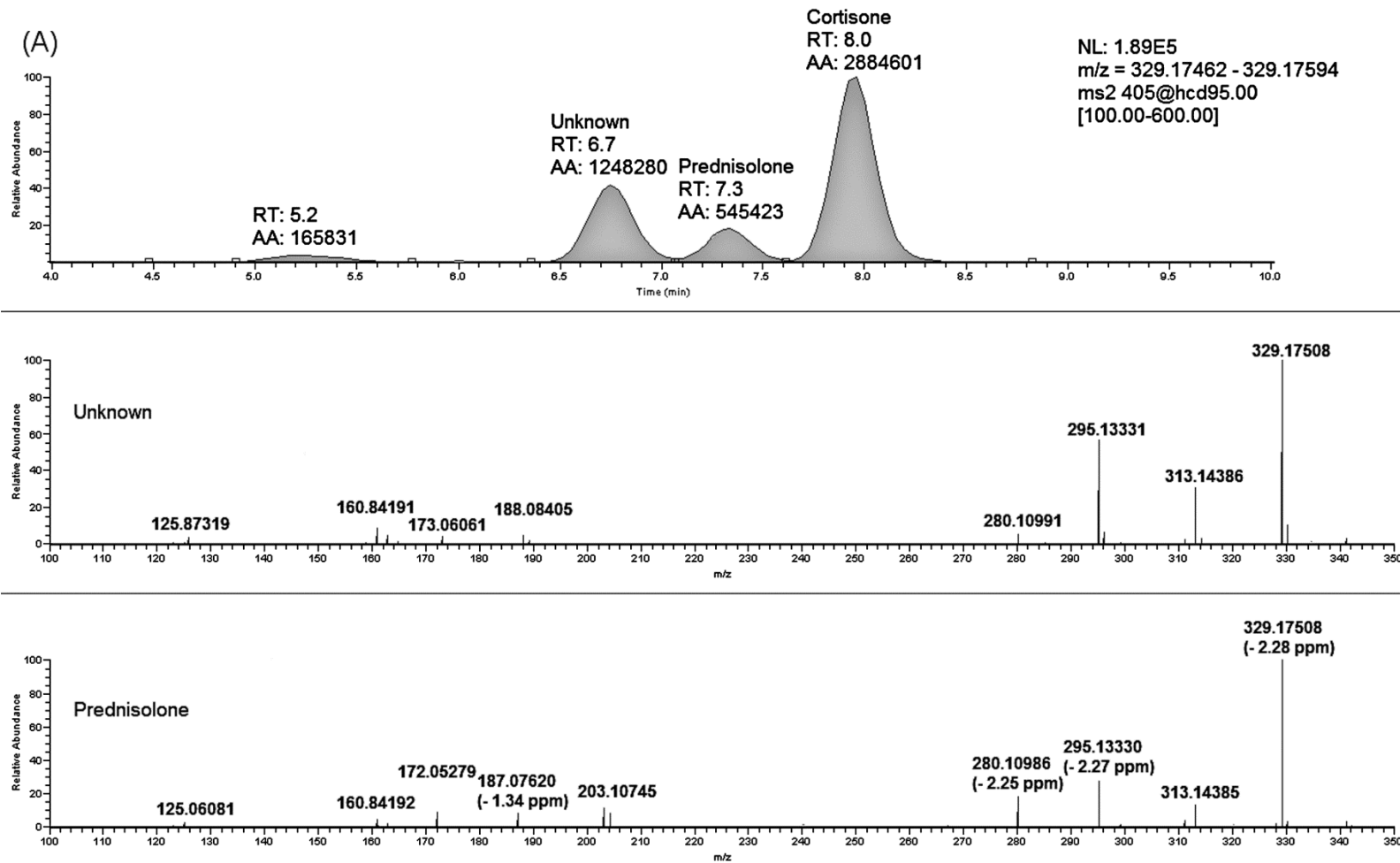
LC-(HR)MS: LT-Orbitrap



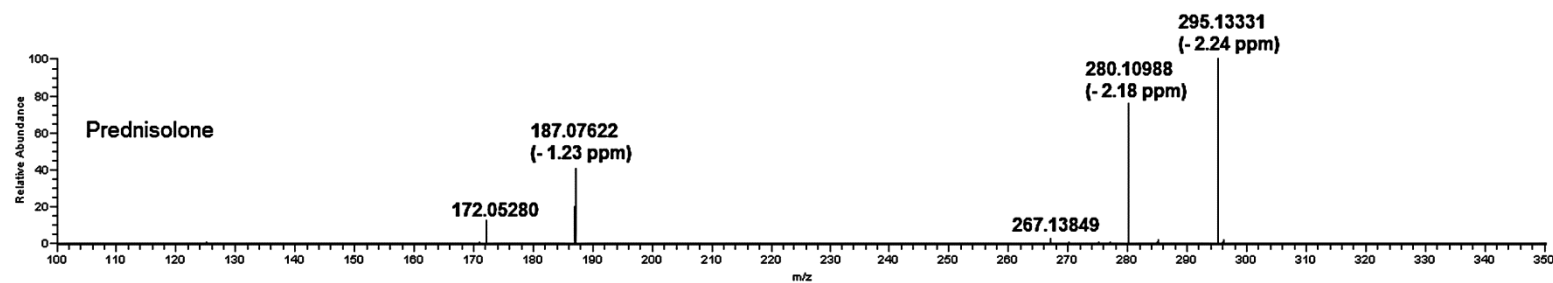
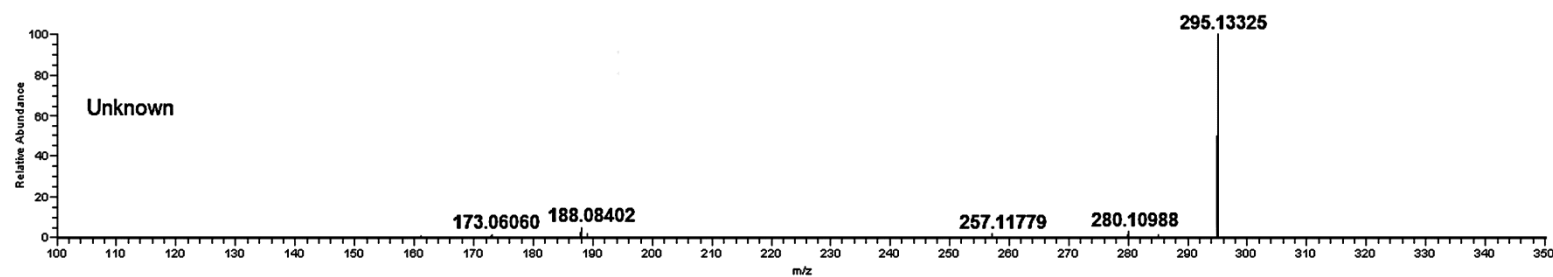
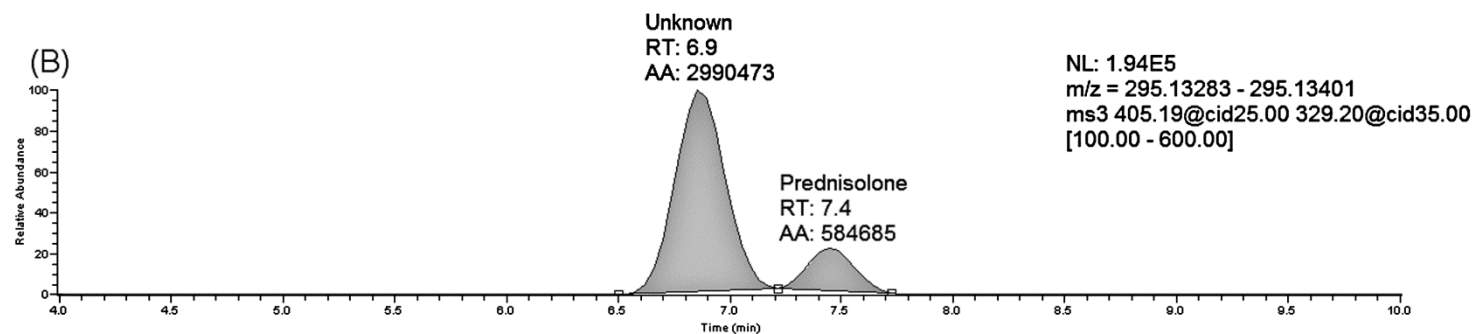
LC-(HR)MS: LT-Orbitrap



LC-(HR)MS/MS: LT-Orbitrap



LC-(HR)MS/MS/MS: LT-Orbitrap



LC-(LR)MS/MS vs LC-(HR)MS

Urine sample	Instrument (acquisition mode)		
	QQQ (MS/MS)	LT Orbitrap (MS)	Q TOF (MS)
1	0.61 µg/L	0.69 µg/L	0.66 µg/L
2	1.18 µg/L	1.42 µg/L	1.60 µg/L
3	2.75 µg/L	3.44 µg/L	3.33 µg/L

Research article



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Guglielmo Dusi, Giorgio Vago, Valentina Ghidelli, Roberto M. Pellegrino, and Roberta Galarini

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Grazie a tutti
per
l'attenzione

