

Supporting Information to

Doping control analysis for small peptides in human urine using LC-HRMS with parallel reaction monitoring mode: screening and confirmation

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Table S1. The HRMS acquisition time frame and the number of data points over the ion peak for each peptide analyte in the PRM mode

Analytes	Start Time (min)	End Time (min)	Number of Data Points over the Targeted Ion Peak (Ion Transitions)
Alexamorelin	6.50	8.00	5 (209.10310)
Alexamorelin(3-6)FA	8.20	10.20	14 (159.09154)
Anamorelin	9.00	11.00	20 (202.12242)
AOD-9604	7.00	8.40	5 (136.07564)
AOD-9604 (7-16)FA	4.50	6.50	6 (166.08612)
Buserelin	8.00	11.00	5 (249.09781)

Table S1. The HRMS acquisition time frame and the number of data points over the ion peak for each peptide analyte in the PRM mode

Analytes	Start Time (min)	End Time (min)	Number of Data Points over the Targeted Ion Peak (Ion Transitions)
Deslorelin	7.50	9.00	6 (249.09785)
Fertirelin	6.00	8.00	4 (249.09785)
GHRP-1	7.60	8.60	4 (209.10323)
GHRP-1(2-4) FA	6.20	7.50	4 (170.09628)
GHRP-1(3-7)	7.30	9.30	5 (159.09164)
GHRP-1(3-6)FA	9.00	11.00	20 (159.09164)
GHRP-2	8.00	9.50	4 (170.09630)
GHRP-2(1-3)FA	7.20	8.50	4 (170.09639)
GHRP-3	5.80	7.00	20 (183.14911)
GHRP-3 FA	6.20	8.20	9 (183.14893)
GHRP-4	8.50	9.50	5 (159.09149)
GHRP-5	8.60	9.90	6 (159.09151)
GHRP-6	6.80	8.00	5 (159.09154)
GHRP-6 FA	7.40	8.70	5 (159.09154)
GHRP-6(2-5)FA	8.50	10.00	5 (159.09149)
GHRP-6(2-6)	7.00	8.50	4 (159.09154)
GHRP-6(2-6)FA	8.10	9.30	6 (159.09154)
Goserelin	8.00	9.00	4 (249.09782)
Hexarelin	7.00	8.00	6 (110.07115)
Hexarelin FA	7.20	9.20	5 (110.07118)
Hexarelin(1-3)FA	4.80	6.00	7 (110.07121)
Hexarelin(2-6)FA	7.50	9.50	5 (159.09148)
Hexarelin(4-6)	5.50	7.50	6 (129.10196)
Histrelin	7.00	9.00	5 (249.09782)
Ibutamoren	8.60	9.90	14 (267.11575)
Ipamorelin	6.00	7.00	4 (129.10213)
Ipamorelin FA	6.50	8.50	4 (170.09644)
Leuprolide	7.80	9.00	5 (249.09785)
Leuprolide(5-9)	7.20	8.40	5 (136.07565)
LHRH	6.00	7.50	5 (249.09782)
LHRH(2-10)	5.50	7.00	6 (159.09172)
Nafarelin	8.90	10.00	7 (249.09773)
Nafarelin(5-10)	8.00	10.00	6 (170.09613)
Peforelin	5.00	7.00	8 (249.09776)
Tabimorelin	9.20	10.40	39 (184.11188)
Triptorelin	7.50	8.60	4 (249.09776)
Desmopressin	6.40	7.50	6 (328.20880)
Felypressin	6.00	7.50	5 (120.08073)

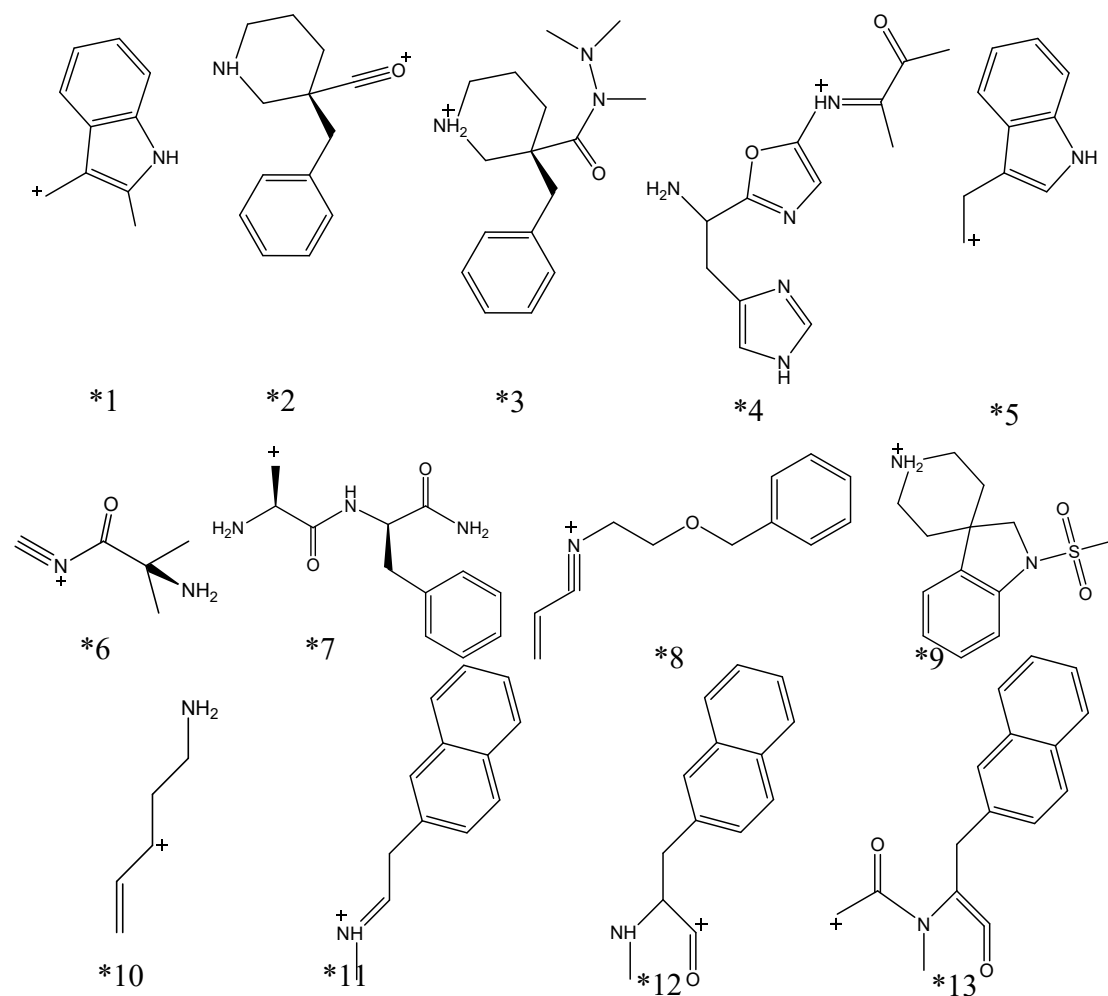
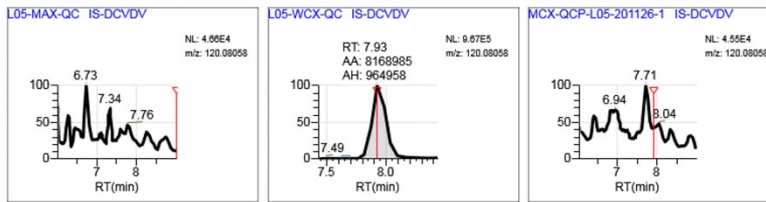
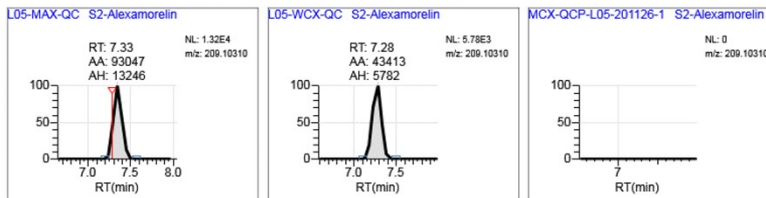


Figure S1. The structures of peptide product ions for alexamorelin(*₁), alexamorelin(3-6)FA(*₁), anamorelin(*₂₋₃), GHRP-1(2-4)FA(*₄), GHRP-1(3-6)FA(*₅), GHRP-3FA(*₆), hexarelin(2-6)FA(*₁), hexarelin(4-6)(*₇), ibutamoren(*₈₋₉), ipamorelinFA(*₁₀) and tabimorelin(*₁₁₋₁₃).

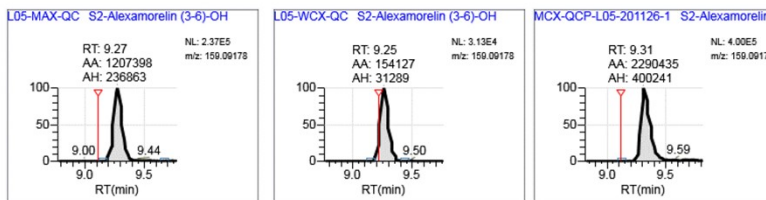
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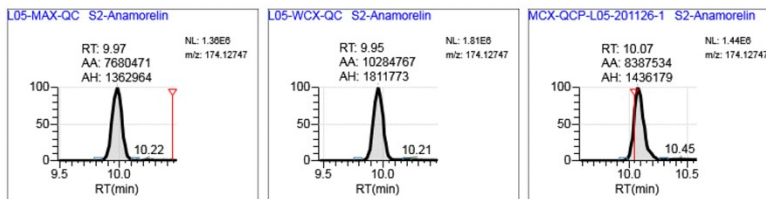
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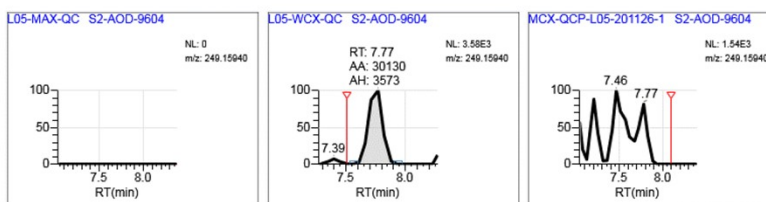
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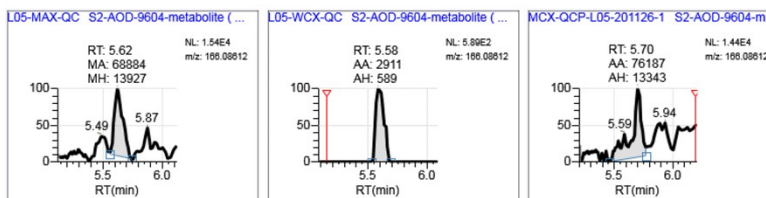
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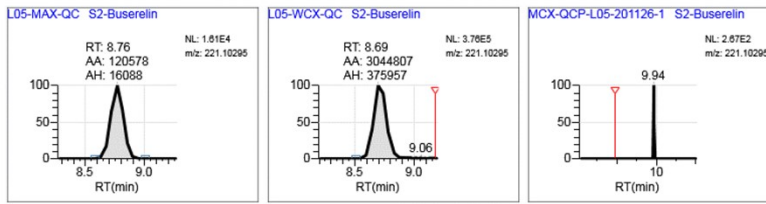
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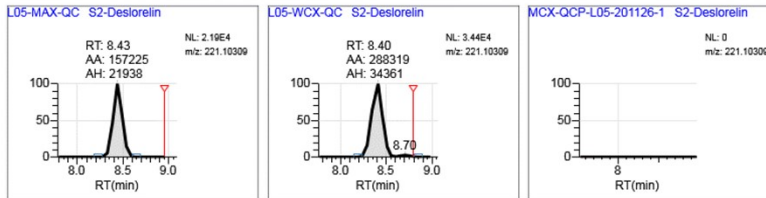
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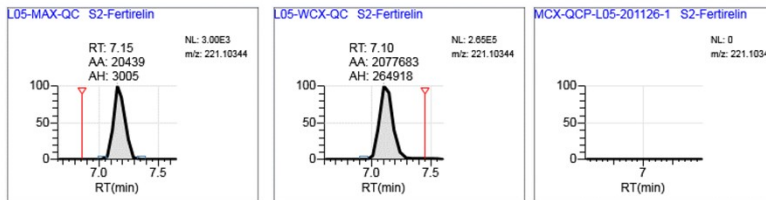
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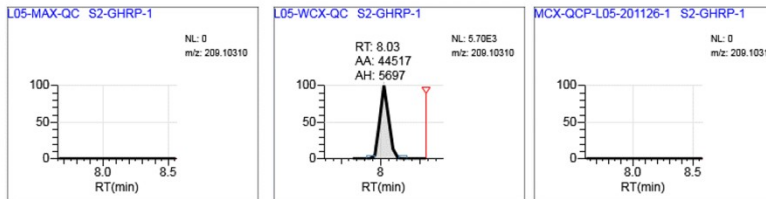
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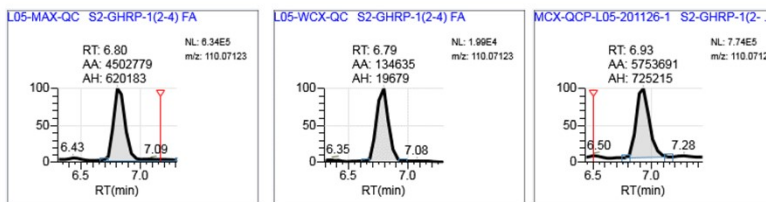
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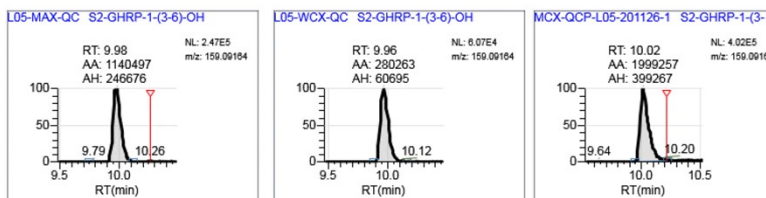
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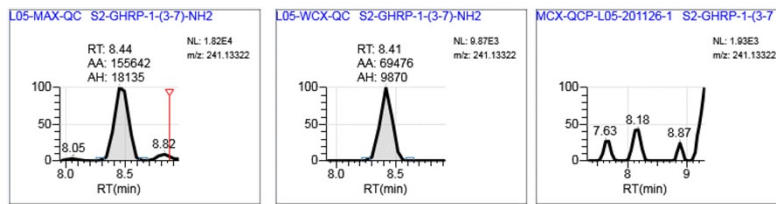
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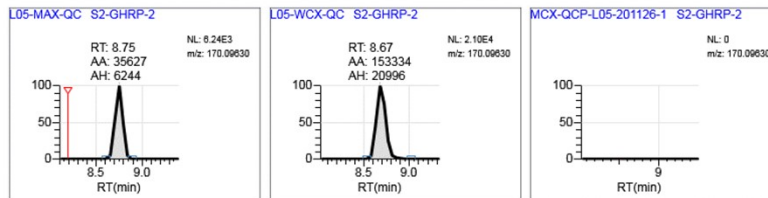
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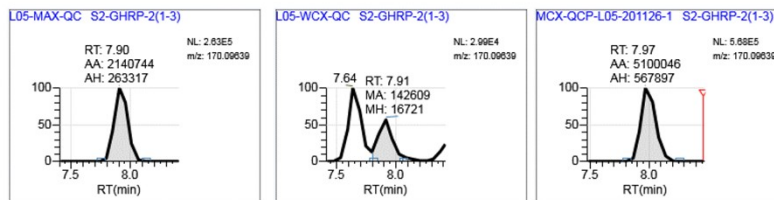
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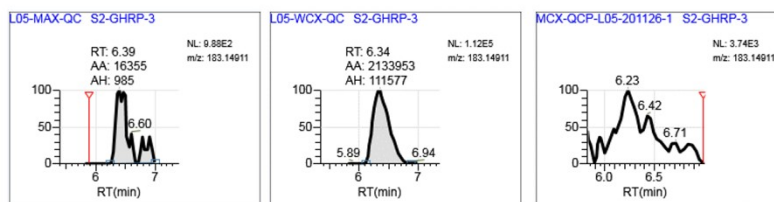
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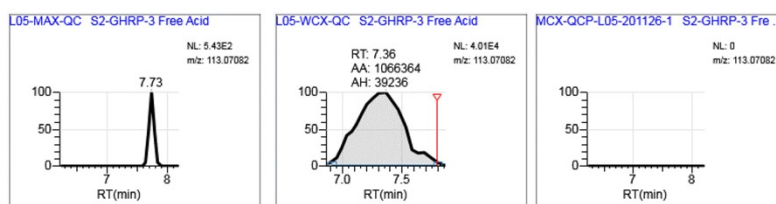
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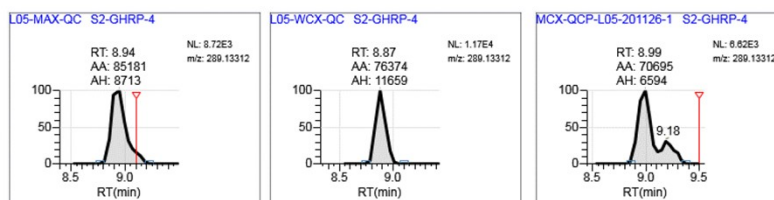
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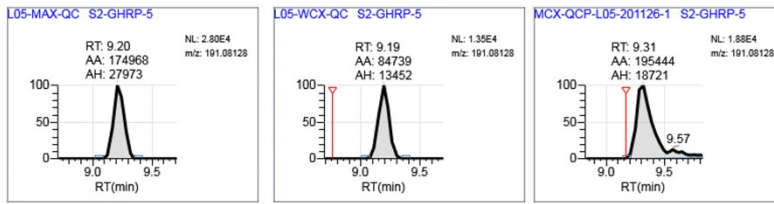
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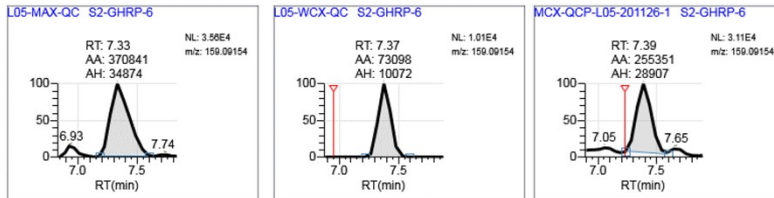
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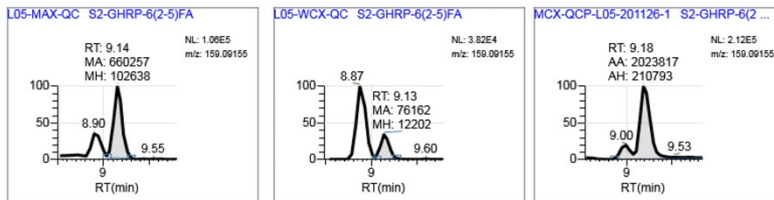
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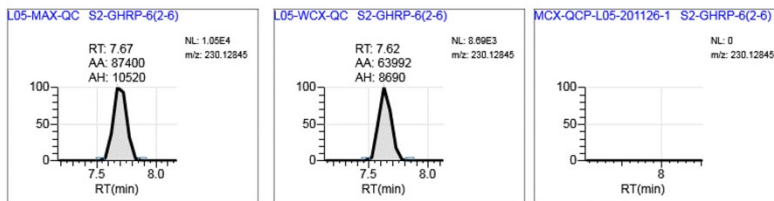
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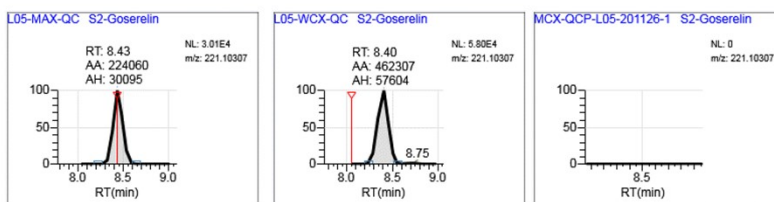
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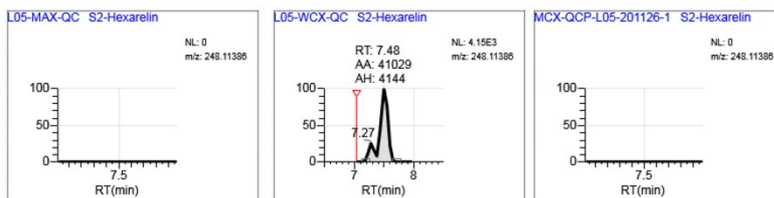
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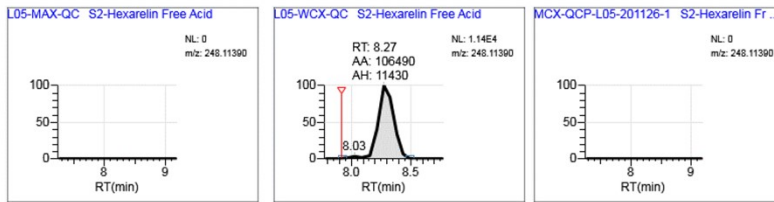
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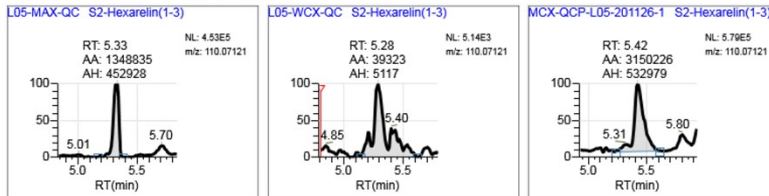
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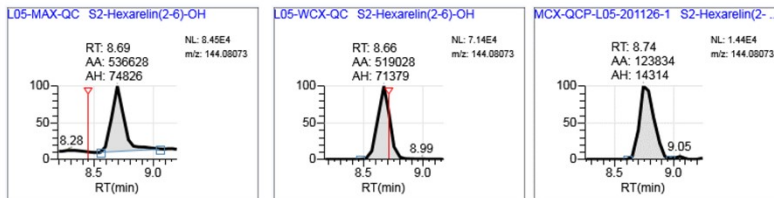
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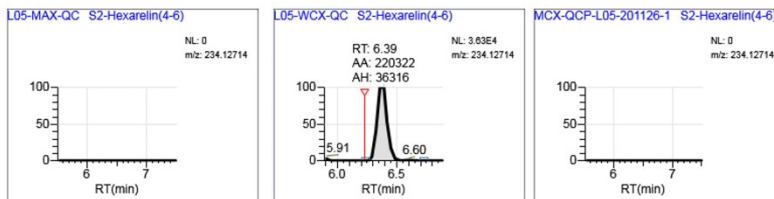
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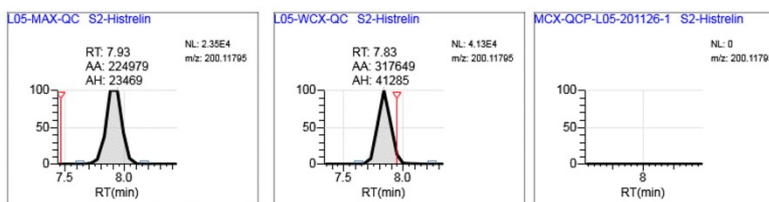
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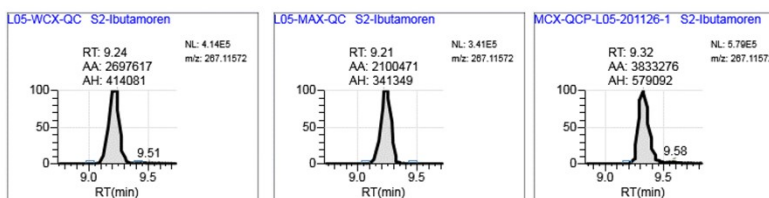
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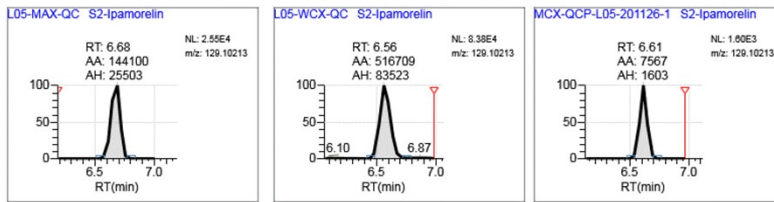
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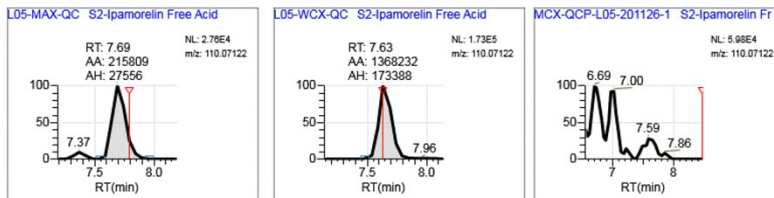
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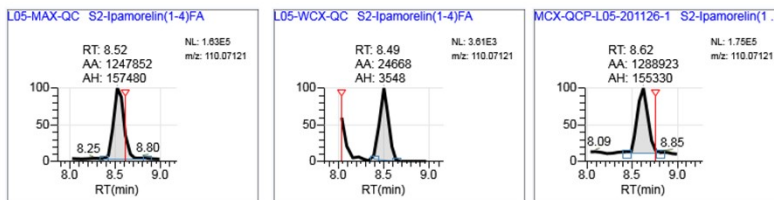
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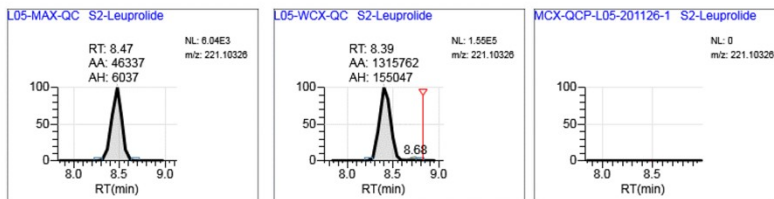
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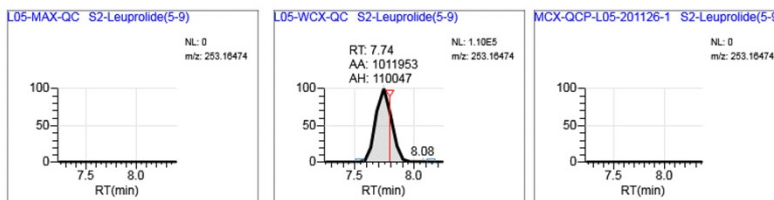
Ipamorelin(1-4)FA



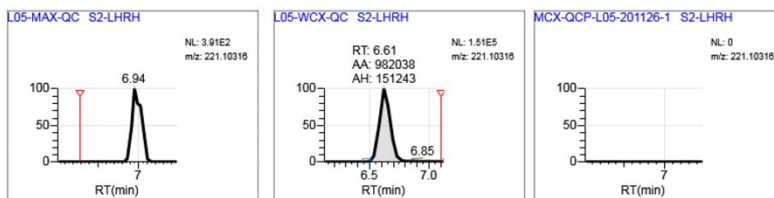
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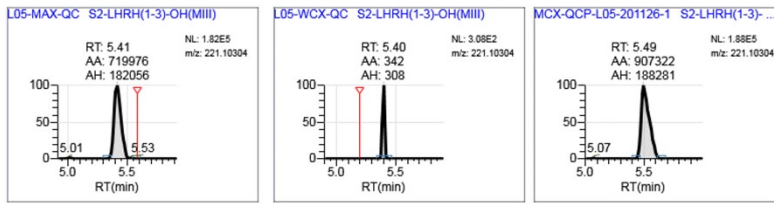
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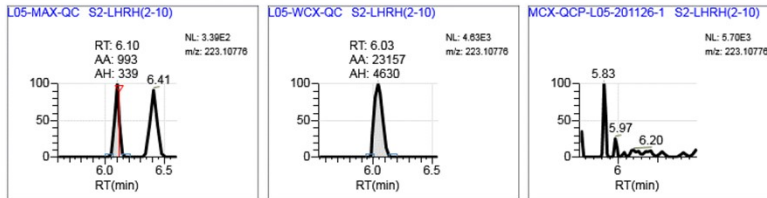
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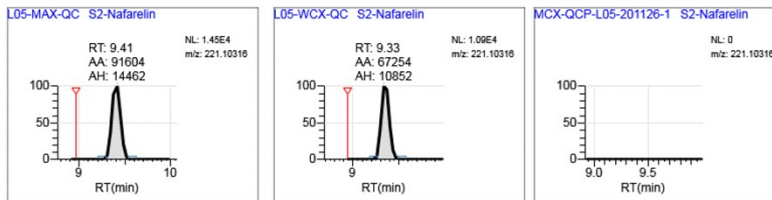
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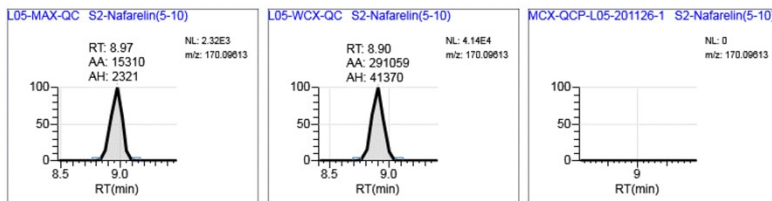
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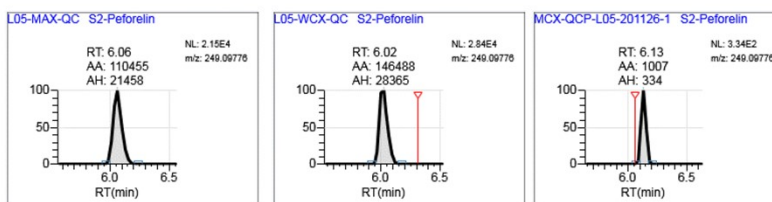
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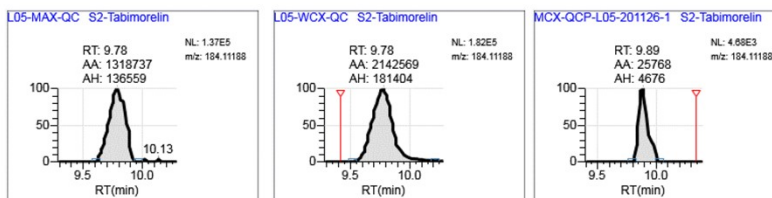
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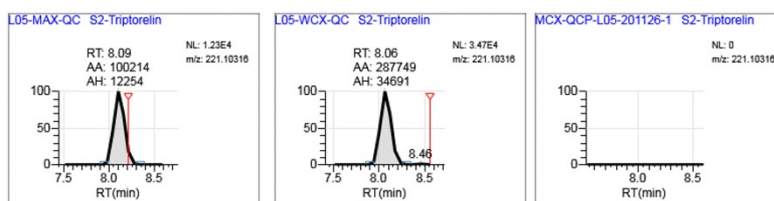
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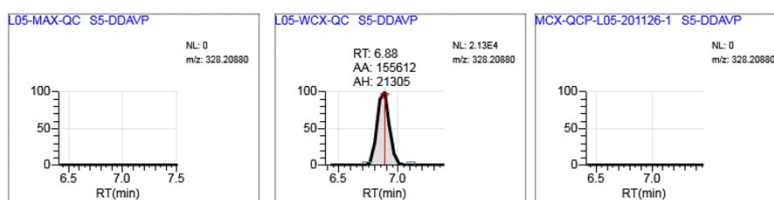
Tabimorelin



Triptorelin



Desmopressin



Felypressin

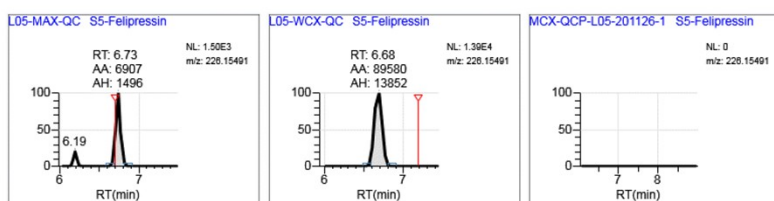


Figure S2. The HRMS extraction ion chromatogram (EIC) of QCP urine samples pre-treated with three kinds of SPE cartridges (Oasis MAX,WCX,MCX cartridges). The concentration of small peptide analytes were at 1ng/mL level , and one diagnostic ion was presented for each analyte.

Table S2. The detection rates at different concentrations of the small peptides of which the LODs were higher than 0.2 ng/mL

Analytes	Concentration (ng/mL)	Detection Rate (%)
AOD-9604	0.2	83.3
	0.5	100
	1.0	100
	2.0	100
AOD-9604 (7-16)FA	0.2	0
	0.5	33
	1.0	100
	2.0	100
GHRP-1	0.2	83.3
	0.5	100
	1.0	100
	2.0	100
GHRP-1(2-4)FA	0.2	66.7
	0.5	100
	1.0	100

Table S2. The detection rates at different concentrations of the small peptides of which the LODs were higher than 0.2 ng/mL

Analytes	Concentration (ng/mL)	Detection Rate (%)
GHRP-6(2-5)FA	2.0	100
	0.2	0
	0.5	50
	1.0	100
Hexarelin	2.0	100
	0.2	66.7
	0.5	83.3
	1.0	100
Hexarelin FA	2.0	100
	0.2	83.3
	0.5	100
	1.0	100
Hexarelin (1-3)FA	2.0	100
	0.2	0
	0.5	50
	1.0	100
	2.0	100

Table S3. The identification rates at different concentrations of the small peptides of which the LOIs were higher than 0.2 ng/mL

Analytes	Concentration (ng/mL)	Detection Rate (%)
Alexamorelin(3-6)FA	0.2	0.0
	0.5	50.0
	1.0	100.0
	2.0	100.0
AOD-9604	0.2	0.0
	0.5	50.0
	1.0	33.3
	2.0	100.0
Deslorelin	0.2	50.0
	0.5	66.7
	1.0	100.0
	2.0	100.0
Fertirelin	0.2	83.3
	0.5	100.0
	1.0	100.0
	2.0	100.0
GHRP-1(2-4)FA	0.2	0.0

Table S3. The identification rates at different concentrations of the small peptides of which the LOIs were higher than 0.2 ng/mL

Analytes	Concentration (ng/mL)	Detection Rate (%)
GHRP-1(3-6)FA	0.5	66.7
	1.0	100.0
	2.0	100.0
	0.2	50.0
	0.5	83.3
GHRP-1(3-7)	1.0	100.0
	2.0	100.0
	0.2	83.3
	0.5	100.0
	1.0	100.0
GHRP-2	2.0	100.0
	0.2	33.3
	0.5	83.3
	1.0	100.0
	2.0	100.0
GHRP-2(1-3)FA	0.2	16.7
	0.5	83.3
	1.0	100
	2.0	100
	0.2	16.7
GHRP-5	0.5	66.7
	1.0	83.3
	2.0	100
	0.2	16.7
	0.5	33.3
GHRP-6	1.0	50.0
	2.0	100.0
	0.2	16.7
	0.5	50.0
	1.0	100.0
GHRP-6FA	2.0	100.0
	0.2	16.7
	0.5	50.0
	1.0	100.0
	2.0	100.0
GHRP-6(2-5)FA	0.2	0.0
	0.5	50.0
	1.0	100.0
	2.0	100.0
	0.2	50.0
GHRP-6(2-6)	0.5	83.3
	1.0	100.0
	2.0	100.0
	0.2	50.0
	0.5	83.3
GHRP-6(2-6)FA	1.0	100.0
	2.0	100.0
	0.2	0.0

Table S3. The identification rates at different concentrations of the small peptides of which the LOIs were higher than 0.2 ng/mL

Analytes	Concentration (ng/mL)	Detection Rate (%)
Hexarelin	0.5	66.7
	1.0	66.7
	2.0	100.0
	0.2	16.7
	0.5	66.7
HexarelinFA	1.0	83.3
	2.0	100.0
	0.2	16.7
	0.5	83.3
	1.0	83.3
Hexarelin(2-6)FA	2.0	100.0
	0.2	83.3
	0.5	100.0
	1.0	100.0
	2.0	100.0
Histrelin	0.2	66.7
	0.5	83.3
	1.0	100.0
	2.0	100.0
	0.2	50.0
Ipamorelin	0.5	100.0
	1.0	100.0
	2.0	100.0
	0.2	33.3
	0.5	33.3
Nafarelin	1.0	66.7
	2.0	100.0
	0.2	66.7
	0.5	100.0
	1.0	100.0
Nafarelin(5-10)	2.0	100.0
	0.2	0.0
	0.5	50.0
	1.0	100.0
	2.0	100.0
Peforelin	0.2	66.7
	0.5	50.0
	1.0	100.0
	2.0	100.0
	0.2	66.7
Triptorelin	0.5	50.0
	1.0	100.0
	2.0	100.0
	0.2	66.7
	0.5	50.0