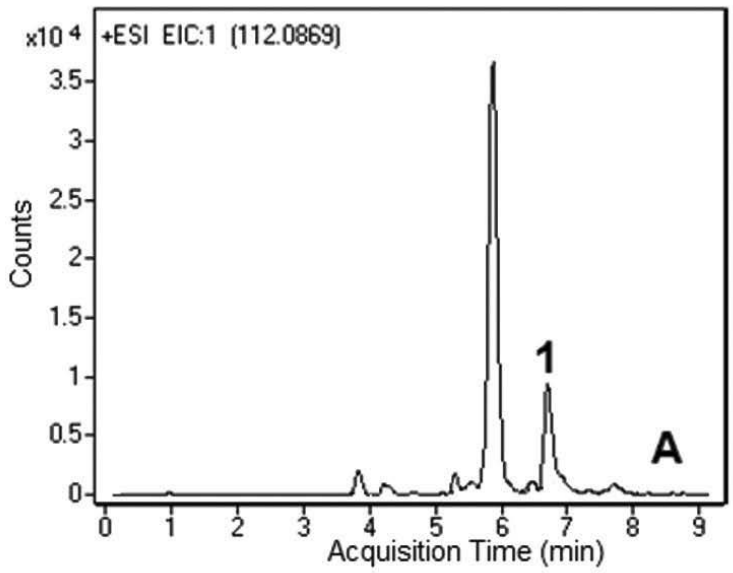
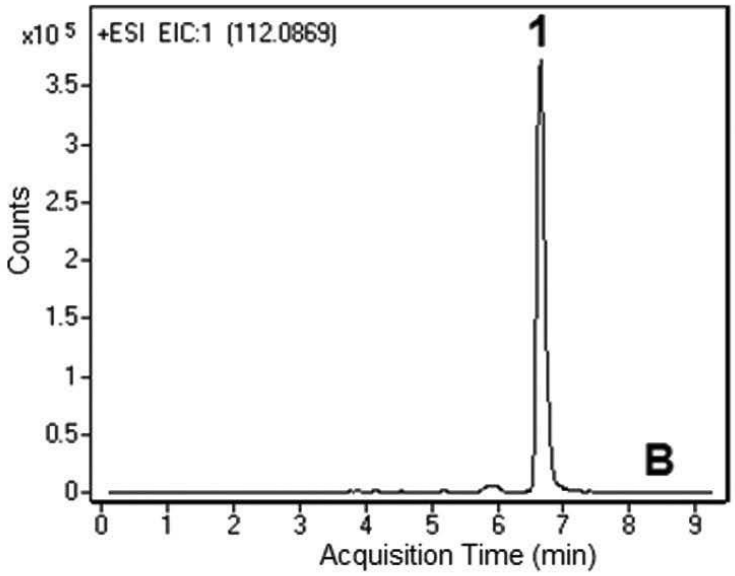
Histamine Analysis in Tuna by LCMS - AppNote

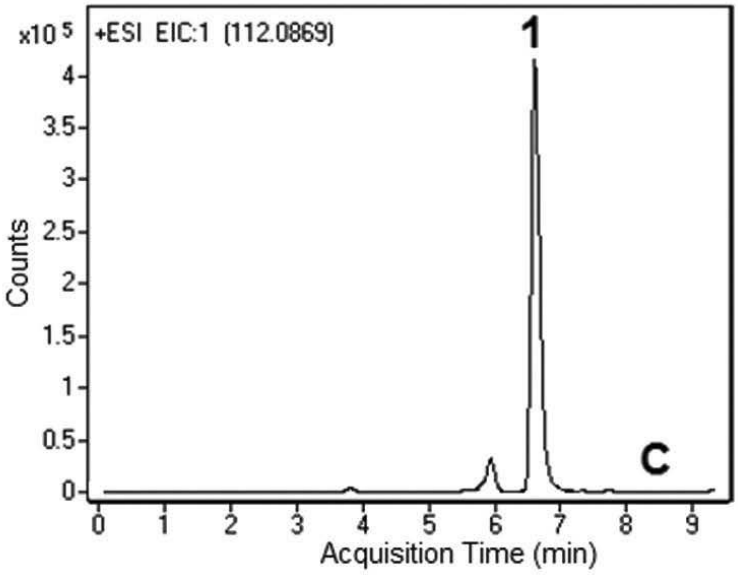
LC-MS method without derivatization

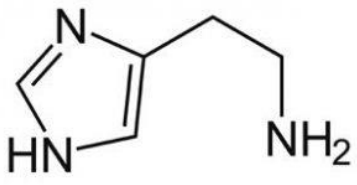
A small amount of Histamine was found in a Tuna sample (Figure A) after an extraction procedure and analysis using the Cogent Diamond Hydride Column and MS detection. In Figure B, a Tuna sample was spiked before the extraction procedure at a level of 0.5 mg/L and Figure C shows the Histamine peak in a spiked extract from the Tuna sample at a level of 1.0 mg/L.

The figures show that the identification of Histamine by mass or retention time is not affected by the Tuna matrix or extracted material. The Histamine content in the Tuna sample was determined based on the calibration curve and it was determined to be 320 ± 4 ng/grams of Tuna (with a %RSD of 0.2 for n=5). The developed protocol after validation can be used for the analysis of this polar compound in a variety of food matrices.


x10 4
+ESI EIC:1 (112.0869)
Counts
1
A
Acquisition Time (min)


x10 5
+ESI EIC:1 (112.0869)
Counts
1
B
Acquisition Time (min)


x10 5
+ESI EIC:1 (112.0869)
Counts
1
C
Acquisition Time (min)


N
HN
NH2

Histamine

Peak:

Histamine 112.0869 m/z

Method Conditions

Column: Cogent Diamond Hydride™, 4µm, 100Å
Catalog No.: 70000-15P-2
Dimensions: 2.1 x 150mm
Mobile Phase:
A: 50% DI Water / 50% 2-Propanol / 0.1% Formic Acid
B: Acetonitrile / 0.1% Formic Acid
Gradient:

Time (Minutes)	%B
0	80
5	10
7	10
8	80

Post Time: 3 minutes
Flow rate: 0.4 mL/minute
Detection: ESI – POSG - Agilent 6210 MSD TOF Mass Spectrometer
Injection vol.: 1µL
Sample Preparation:
Canned Tuna was purchased from a local supermarket. Three Tuna samples were prepared. The unspiked SPE sample was prepared by homogenizing 5 g of Tuna and 50 mL of DI Water / 0.1% FA in a Waring blender for 10 min at 13,500 rpm. The mixture was then centrifuged at 4000 g for 20 minutes. The supernatant was refrigerated (20 °C) for 10 min, treated by adding dropwise 3 M Ammonia to a pH of 11.0, then centrifuged at 1000 g for 5 min. The resulting supernatant was purified by solid phase extraction (SPE) on a conditioned C18 sorbent and eluted with 2 mL of methanol.

After removal of the Methanol by Nitrogen gas, the extracted sample was re-dissolved in 2.0 mL of DI Water / 0.1 % FA for direct analyses. The spiked SPE samples were prepared by homogenizing 5.0 g of Tuna, 50 mL of DI Water / 0.1% FA, and appropriate amount of 1 mg/mL Histamine stock solution in a Waring blender for 10 minutes at 13,500 rpm. Afterwards, the sample preparation was completed by following procedures for the unspiked SPE samples (i.e. centrifuge, SPE, etc.).

t_0: 0.9 minutes

COGENT HPLC Columns™

Attachment

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