

Supplementary Information

Figure S1. EDX measurement of the isolated skeletons of *Aplysina cavernicola* before MeOH extraction.

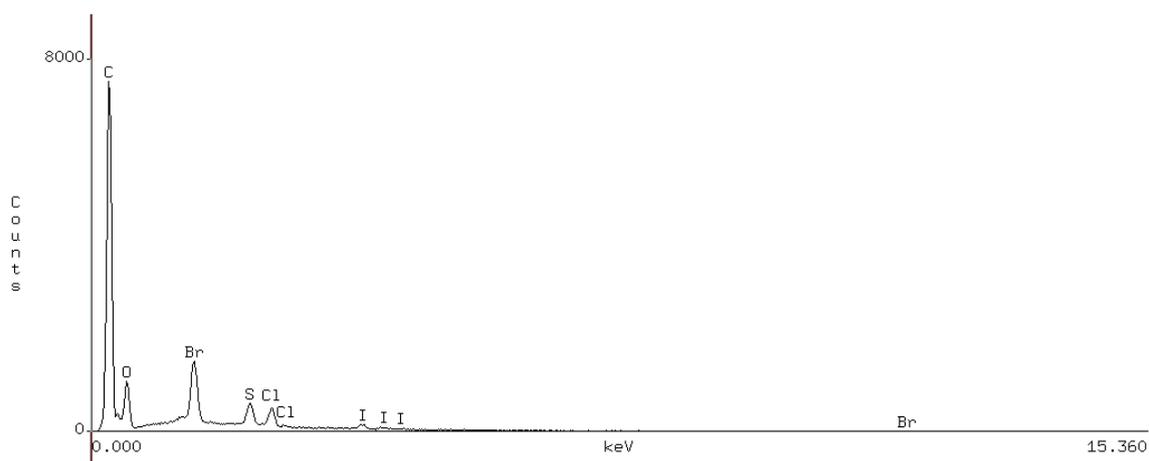


Figure S2. EDX measurement of the isolated skeletons of *A. cavernicola* after MeOH extraction.

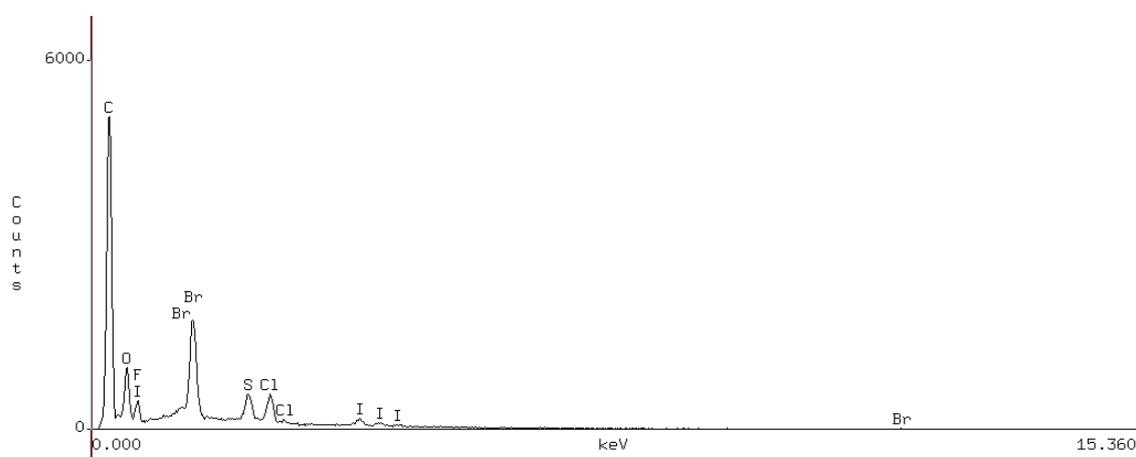


Figure S3. EI-MS data of TBDMS-derivative of the arothionin standard.

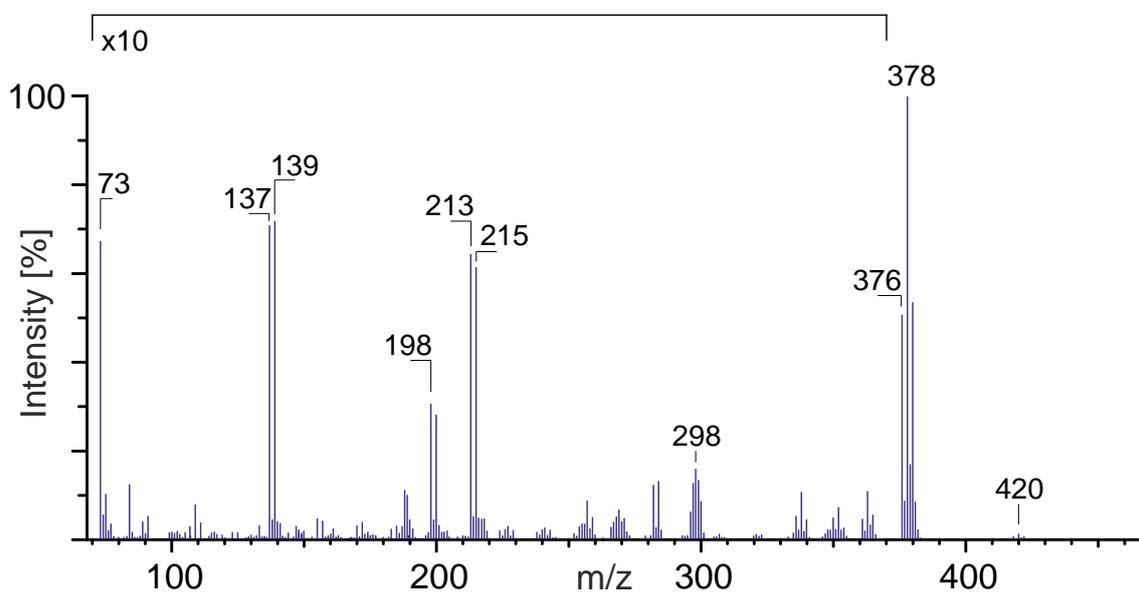


Figure S4. EI-MS data of TBDMS-derivative of the aerothionin fragment found in the MeOH extract.

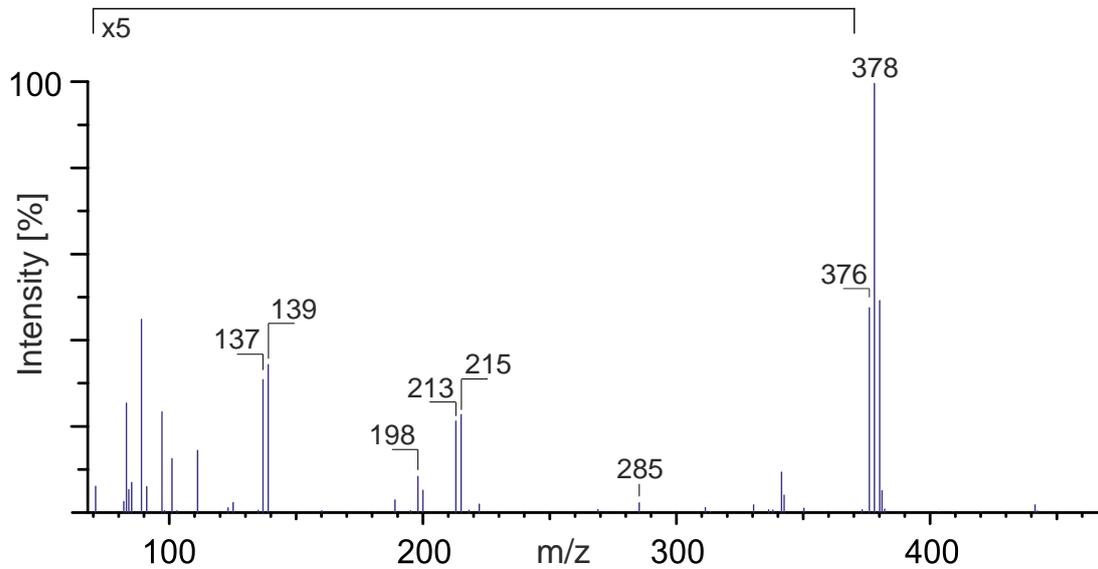


Table S1. Experimental Errors of six amino acids. * The experimental error is determined for these amino acids. For each amino acid, the experimental error is calculated from three measurements of the same sample. The average value is about 15%.

Amino Acid	Experimental Error *
Alanine (Peak 1)	4.8%
Phenylalanine (Peak 11)	23.8%
Aspartic Acid (Peak 12)	12.2%
Ornithine (Peak 14)	18.4%
Dichlorotyrosine (Peak 23*)	5.5%
Monobromo-Monochlorotyrosine (Peak 25*)	22.9%

Figure S5. LC-ESI-MS spectra of mono- and dichloro-, monobromo-monochloro-, monochloro-monoiodo- and monobromo-monoiodotyrosine detected in the Ba(OH)₂ skeleton extracts of *A. cavernicola*.

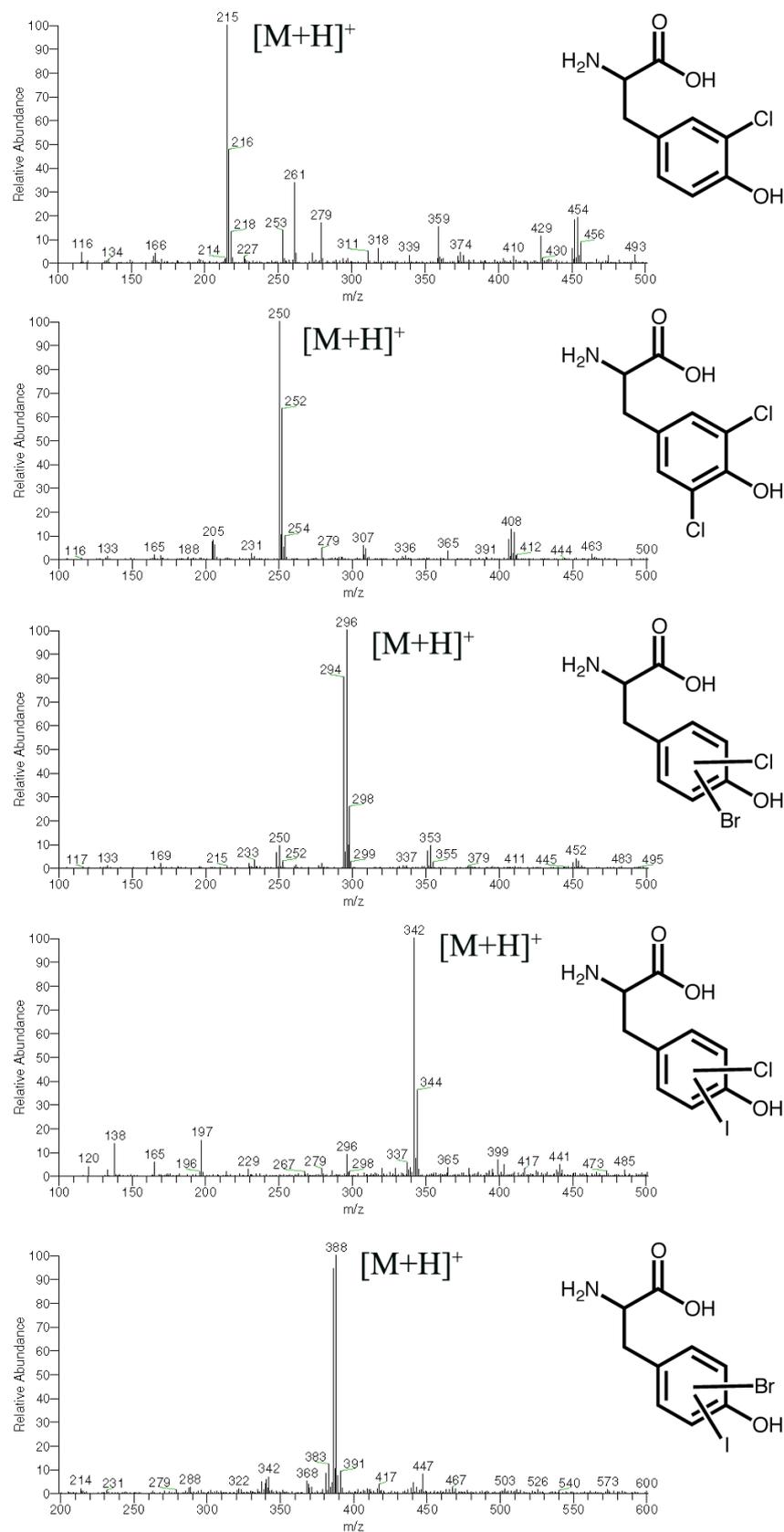


Figure S6. LC-ESI-MS spectra of halogenated tyrosines detected in the $\text{Ba}(\text{OH})_2$ skeleton extract of *A. cavernicola* after MeOH extraction.

