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Development of an analytical method by Liquid Chromatography–High Resolution Mass Spectrometry for the analysis of saxitoxin and related Paralytic Shellfish Poisoning Toxins.

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Statement of the Problem: Saxitoxin and its analogues are very potent neurotoxins produced by marine dinoflagellates and cyanobacteria and have been described as paralytic shellfish poisoning toxins (PSP). Saxitoxin hydrate (free base) is listed in the CWC (1.A.07). Therefore, DGA CBRN Defence must have the capability to identify this compound according to criteria established by OPCW.

Methodology & Theoretical Orientation: To carry out this development, LC-HRMS analyses have been achieved on an Ultimate 3000 liquid chromatography coupled with quadrupole-time-of-flight (MaXis II BRUKER). Three columns have been assessed to promote the separation of saxitoxin and its analogues in a mixture (TSKGel Amide-80T (Tosoh), Atlantis HILIC Silica (Waters) and ZIC HILIC (SeQuant)). A mixture of PSP toxins [saxitoxin (STX), neosaxitoxin (NEO), decarbamoyl saxitoxin (dcSTX), decarbamoyl neosaxitoxin (dcNEO), gonyautoxins 1–6 (GTX1, GTX2, GTX3, GTX4, GTX5, GTX6) and decarbamoyl gonyautoxins (dcGTX2, dcGTX3)] (Novakits) has been used to optimize the method

Findings: The three different columns have been compared with the same MS conditions (positive ESI ionization), in terms of PSP analogues separation and peak resolution. Several LC parameters were tested such as elution gradient, column temperature and flow rate. Increasing the temperature from 30°C to 40°C contributed to the refinement of the chromatographic peaks. Moreover, switching from a flow rate of 0.2 mL/min to 0.4 mL/min reduced by half the analysis time without drawbacks of the PSP analogues separation. The mobile phase was consisted of water containing 2 mM ammonium formate and 3.6 mM formic acid in channel A and acetonitrile/water (95/5, v/v) containing 2 mM ammonium formate and 3.6 mM formic acid in channel B. The gradient elution takes 30 min with 15 min reconditioning.

Conclusion & Significance: The ZIC HILIC column gave the best results due to better separation of PSP. Further work will focus on transferring the method to LC-TQMS.

Biography

Salomé Chaumier graduated from a Master's degrees in Strategy and Quality in Analytical Chemistry in September 2022. After completing her engineering degree, she was hired by DGA CBRN Defence as engineer in the LCMS laboratory of the French OPCW Designated Laboratory and works in collaboration with two experts. She uses four devices with different MS, which allows her to provide expertise on chemical warfare agents and related chemicals in unknown environmental, material and biomedical matrices.

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