Implementation and verification of an analytical method for the quantification of biogenic amines in seafood products

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•The ***aim*** of the master thesis was the implementation and verification of an HPLC method for the determination and quantification of histamine and other biogenic amines (BAs) in fish and fish meal. The implementation of the method included sample preparation, HPLC analysis and data evaluation. Thereafter, the method performance was verified to demonstrate that the results obtained were consistent, correct and satisfactory for analysis of BAs in fish and fish meal.

•In the ***method*** BAs were extracted with 0,6M perchloric acid. The separation of the BAs was achieved on a C_{18} reverse-phase column (5µm 250X4,6 mm inner diameter) with gradient elution separation with a binary mixture with increasing sodium-acetate and acetonitrile concentration (pH=4,5), with a flow rate of 0.9mL/min, at 50°C. Applying online post-column OPA derivatization with UV detection (Ex: 330, Em: 456 nm

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***Results*:** The method was selective: the biogenic amines (BAs) separated from each other with a good resolution. The analytical method had good ($r^2 \geq 0.994$). Enabled the measurement of BAs in a very low concentration. The method was accurate, repeatable and reproducible; with a recovery of 80-100%. Showed no significant differences in inter-laboratory measurements. The precision of the method showed that the relative standard deviation (RSD) of the peak areas of the replicates were 3-4,2% in fish flesh and RSD= 4,2-5,9% in fish meal, with a relative measurement uncertainty of 3-4% (fish flesh) and 5,86-8,30% (fish meal). The reproducibility also revealed a low variations in the results obtained, the RSD was in the range of 5-9% with a relative uncertainty between 5-7,8% .

***Conclusion*:** The implementation of the new analytical method and the verification study was successful and proved that HPLC system and method served its purposes.. The application of the method was easy, mainly due to the application of post-column derivatization, which shortened the time for sample preparation, resulted in stable derivatives of the analytes and enabled the possibility of continuous measurements applying autosampler and online derivatization. This increased the efficiency and sample turnover rate of the laboratory. Verification parameters were defined and the results confirmed the validity of BA measurement in fish and fish products. The successful implementation and verification enabled Matís laboratory to aquire the official accreditation in this analysis. More official authorities and seafood producers will be able to monitor the occurrence of BAs in Icelandic products and lead to increased food and feed safety.

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