Refinement and validation of the AOAC method (2005.06) to improve the determination of toxins in scallops

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Background

The HPLC official method AOAC 2005.06 has been previously refined and validated for the determination and quantitation of PSP toxins (PSTs) in a range of species. Work conducted for scallops showed the method behaved acceptably for the non-N-hydroxylated toxins but, with poor method performance for N-hydroxylated toxins, would not have been fit for purpose to implement for official testing without further refinement.

Research Approach

This work comprised investigations to improve the method performance for the N-hydroxylated toxins. Once improved, work was conducted to validate the modified method for the determination of PSP toxins in whole king and queen scallops. Work conducted followed the protocol used for other species, involving the assessment of selectivity, linearity, sensitivity, recovery, precision, ruggedness and uncertainty of measurement. Testing was also conducted on contaminated king and queen scallops using both HPLC and MBA in order to compare the results obtained by both methods.

Results

A small inter-laboratory study highlighted that the issues with the method performance for AOAC 2005.06 in scallops were not restricted to just one laboratory. Refinement work focussed on the potential effects of varying key method parameters. Successful refinement was obtained through use of higher analytical injection volumes, larger volumes of extract for clean-up, use of a modified periodate oxidant and a novel matrix modifier comprising king scallop extract.

Validation results of the refined method showed acceptable selectivity, linearity, recovery, precision, sensitivity and ruggedness in both scallop species for the N-hydroxylated toxins, with the refined method providing superior method performance as compared to the official AOAC 2005.06 method. Method parameters were not changed for the non-N-hydroxylated toxins oxidised by peroxide prior to HPLC quantitation. Method performance characteristics obtained were subsequently used to generate values for the size of the uncertainty of measurement for each PSP toxin in both scallop matrices. These were found to compare well with those generated from the method performance characteristics of AOAC 2005.06 in other non-scallop matrices.

Results from the HPLC analysis of PSP-positive scallops were compared with those generated by the official MBA reference method. Results from PSP-negative scallops showed no indications of

any false negatives. 19 scallop samples found positive by MBA showed a good overall agreement in total sample toxicities determined using both methodologies. Previous poor agreement between the methods in queen scallops using the official method was improved through use of the refined method.

Overall, the results show the modified AOAC 2005.06 HPLC method for scallops provides an improved level of performance for the determination of PSP toxins in whole king and queen scallops, with method performance characteristics similar to the official method in mussels, cockles, oysters, razors and clams. Consequently, the recommendation is to implement the modified method into the official monitoring programme for the analysis of PSP toxins in both whole king and queen scallops.

Published Papers

1. Turner, A.D., & Hatfield, R.G. (2012). Refinement of AOAC 2005.06 LC-FLD method to improve the method performance characteristics for the analysis of king and queen scallops for paralytic shellfish toxins. Journal of AOAC International, 95(1), 129-142 doi: doi.org/10.5740/jaoacint.11-184

Research report

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PDF
Gweld Research report FS235002A (ZB1807): In-house validation of the AOAC HPLC method (2005.06): the determination of paralytic shellf as PDF(Open in a new window) (2.8 MB)