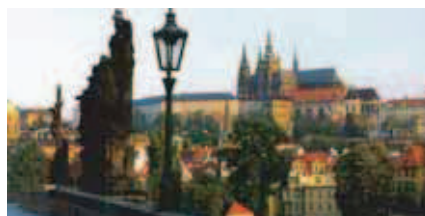


BOOK OF ABSTRACTS

5th International Symposium on
**RECENT ADVANCES IN
FOOD ANALYSIS**

November 1–4, 2011
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Jana Pulkrabová and Monika Tomaniová
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O-23 QUANTIFICATION OF ENDOCRINE DISRUPTORS AND PESTICIDES IN WATER USING WEIGHTED LINEAR REGRESSION SCHEMES

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The presence of pesticides in environmental matrices can have serious consequences such as financial, human health and environmental, being aquatic environment is particularly susceptible to this type of pollution. Unequivocal analytical data require a specific set of validation criteria and verification of method performance. The importance of validation, at least of routine analytical methods, has been hardly overestimated. In recent years this is particularly true in the context of quality management and accreditation, which has become subject of increased importance in analytical chemistry. Therefore, the present trend towards standardization of practices between countries has been at the genesis of the current need of international acceptance of analytical results and accurate validation of methods, which become increasingly important for ensuring a common level of quality. The aim of the present work was the validation of a multi-residue methodology based on a solid phase extraction followed by gas chromatography– tandem mass spectrometry for trace analysis of 31 compounds in water matrices. Heteroscedasticity of results has been overtaken by a weighted least squares linear regression (WLSLR) model application. As part of the assay validation a WLSLR model was used to obtain a calibration scheme once the assumption of homoscedasticity was not met for analytical data. WLSLR is an efficient method that provides unbiased estimative for prediction, calibration and optimization when standard deviation of the data random errors are not constant across all levels of the explanatory variables. It works by incorporating extra nonnegative constants, or weights, associated with each data point, into the fitting criterion and the size of the weight indicates the precision of the information contained in the associated observation. The other analytical performance and characteristics of the method were also studied and validated. The unweighted model overestimates the concentrations in the lower range of the calibration curve, near the limit of quantification. The weighted model presents a best %RE distribution scatter, a lower sum of %RE and, finally, none of the studied compounds showed relative errors greater than the acceptable limits of 15% and 20% for the different calibration line standards and the first pattern, respectively. Although weighted least squares regression is more complex and laborious than ordinary linear regression, involving the use of additional statistical tests and mathematical operations, it should be performed in order to obtain more realistic results and lower limits of quantification.

Keywords: "Endocrine disruptors" "Pesticides" "Water" "Weighted linear regression schemes" "Validation"

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O-24 A NEW COMPLETE SOLUTION FOR AUTOMATED, COMPREHENSIVE ESI– (Q–)TOF FULL SCAN ACCURATE MASS SCREENING OF PESTICIDES IN FOOD WITH HIGH CONFIDENCE

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Fast and comprehensive full scan accurate mass screening for hundreds of pesticides simultaneously has meanwhile found its way into routine use taking advantage of the high number of possible targets and additionally allowing for unknown evaluation and retrospective analysis. The screening procedure relies on full scan accurate mass data and a target compound database, which basically only needs information about name (-> identifier in the result table) and sum formula (-> accurate mass information) of a target. However, the use of comprehensive databases containing hundreds or even thousands of sum formula/name entries only is anything else than advisable. In the case of pesticide screening in food low signal intensities in highly complex matrix samples have to be evaluated to achieve the required reporting levels, thus potentially leading to a meaningless high number of false positive results. Inclusion of additional information and knowledge therefore is essential to obtain reliable results. The screening solution presented here makes use of multiple levels of confirmation and result rating for maximum confidence in the results and is characterized by: – A screening platform consisting of a defined UHPLC and ESI-(Q-)-TOF system, a dedicated screening software (TargetAnalysis) and a high quality/information rich pesticide database. – The ESI-(Q-)-TOF system provides data with high isotopic fidelity and excellent mass accuracy over a wide dynamic range. – Methods and conditions are used giving robust and reliable system performance, allowing to work with known and stable retention times. – TargetAnalysis uses retention time, mass accuracy and isopic pattern information for a first level confirmation and result rating according to customizable settings. – Certainty of results is increased with additional evaluation of known adducts and fragment ions as qualifier ions, also enabling unambiguous compound assignment. – A further level of confirmation to avoid false positive results is obtained by combination of full scan and ISCID (Internal Source Collision Induced Dissociation) data in the same analytical run. The presence of (up to three) qualifier ions is evaluated together with their intensity ratios. Random or matrix related results will not pass this test and are thus filtered out. Number and quality of detected qualifier ions add a fourth rating criterion. – Various sorting options in the TargetAnalysis result table (e.g. name, retention time, registry number) help for easy result grouping (fragments, metabolites) and reviewing. – In case of ambiguous results detailed result evaluation is possible via easy linking to the standard data evaluation software (DataAnalysis). – If calibration data for detected compounds is available a quantitative result can be displayed in TargetAnalysis as well. – A fully automated setup and workflow is possible. Examples for the workflow and system performance will be given.

Keywords: pesticides, multi-target screening, ESI-(Q)TOF, result confirmation