

Six years of interlaboratory ring-test exercises for the analysis of illicit drugs in wastewater – What have we learnt ?

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One of the cornerstones of wastewater-based epidemiology (WBE) is the accurate quantification of the excretion products of illicit drugs in wastewater. Methods of analysis must, therefore, be fully validated before they are brought into routine use. Participation in quality control schemes is also strongly advised for laboratories carrying out such analysis. In 2011, SCORE started organising interlaboratory exercises to provide quality assurance for the reporting of WBE data.

Interlaboratory ring-test exercises were performed annually from 2011 to 2016 for the following illicit drugs and metabolites: benzoylecgonine (BE), cocaine (COC), methylenedioxymethamphetamine (MDMA), amphetamine (AMP), methamphetamine (METH), 11-nor-9-carboxytetrahydrocannabinol (THC-COOH), and 6-monoacetylmorphine (6-MAM). Different matrices containing the analytes at different concentrations were included: standard solution in methanol, spiked tap water samples and/or (spiked) wastewater. Over the period 2011-2016, 37 laboratories from 25 countries participated in the exercises. Most laboratories (81%) were located in Europe, while the other 19% were spread over different continents (North-America, Asia and Oceania). Participating laboratories were asked to analyse the samples according to their in-house validated analytical procedure.

Data analysis was performed in multiple steps: (i) a Grubbs' test was performed to identify outliers; these were excluded from the dataset and not taken into account in further evaluations; (ii) the group's mean and the group's standard deviation were calculated; (iii) z-scores were determined for each laboratory, for each sample and for each analyte based on the group's mean and standard deviation (1,2).

The results showed satisfactory accuracy for the majority of the seven analytes and generally good performance by the majority of participating laboratories. Not surprisingly, precision of results in the standard spiking solution was better than that observed in the more complex wastewater samples. Results for THC-COOH did however highlight some particular problems with this compound. Accuracy and precision were poor. Subsequent investigation has identified the most likely pre-analytical cause of this poor performance, and guidelines for the analysis of this compound have been amended accordingly.

This presentation will discuss the results of this six-years interlaboratory testing scheme, will evaluate the improvements made during the process and will formulate recommendations for future inter-laboratory exercises.

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References:

- (1) ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO, 2015, Geneva, Switzerland
- (2) M. Thompson, S.L. Ellison, R. Wood. The international harmonized protocol for the proficiency testing of analytical chemistry laboratories. Pure Appl. Chem. 2006, 78, 1, 145-196