ASSESSING ACCURACY AND PRECISION USING QUALITY CONTROLS FOR NON-TARGETED ANALYSIS

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ABSTRACT

The benchmarks to assess reproducibility are not well defined for non-targeted analysis. Performance evaluation of analytical methodologies such as accuracy, precision and selectivity are well defined for target analysis, but remain novel and elusive topic for non-targeted screening analysis. In this study, quality control (QC) guidelines implemented in our laboratory are proposed with the aim to assess accuracy of the data in non-targeted screening methodologies using a simple set of standards. Workflow reproducibility was assessed using an in-house QC mixture containing selected compounds with a wide range of polarity that can be detected either by electrospray ionization (ESI) in positive or negative mode. The analysis was done by online solid phase extraction (SPE) liquid chromatography coupled to high resolution mass spectrometry (LC-HRMS). Data processing was done by a commercially available software, Compound Discoverer. In this study, method specificity, precision, accuracy and reproducibility was evaluated in terms of peak area and retention time variability, true positive detection rate, intra-day and inter-day variations. Accuracy was found to be consistent between intra-day and inter-day analysis, with a detection rate of ≥70% for most of the QC compounds. Intraday and interday precision estimated based on peak area relative standard deviation (RSD) ranged between 30 to 50% for most of the compounds. Overall, RSDs varied largely depending on the compounds, with sulfamethoxazole, atrazine and carbamazepine exhibiting a RSD ≤30%, while lincomycin, gemfibrozil and mfenamic acid showed a RSD ≥70%. Retention time precision for both intra- and inter-day analysis showed great repeatability and reproducibility, with all the detected compounds having a retention time RSD ≤5%.

OBJECTIVES

- The main objective of this study was to introduce simple preliminary quality control guidelines to be followed in non-targeted screening methodologies.
- Workflow specificity, precision, accuracy, repeatability and reproducibility were assessed using an in-house QC mixture that could be easily implemented in a typical analytical lab and customized containing a wide range of compounds that can be detected in both electrospray ionization (ESI) positive as well as ESI negative.

MATERIALS AND METHODS

Non-targeted Analysis Workflow for environmental analysis adopted from Hollender et al.1

Conclusions

- The intraday accuracy of the NTA workflow was greater than 75% detection rate for majority of the detected QC compounds except for trimethoprim, diphenhydramine which were detected 60% and 40% respectively; and 3 compounds that were not detected or correctly identified (clofazimine, sulfa and hydrochlorothiazide).
- The interday accuracy of the NTA workflow was consistent with that of the intraday study, in which the majority of the detected QC compounds had a detection rate greater than 75%.
- Intraday precision in terms of peak area for the detected compounds varied by compound, ranging from a RSD of 8.2% for sulfamethoxazole to 106.5% for gemfibrozil. Sulfamethoxazole, atrazine and carbamazepine exhibiting a RSD less than 30%, four compounds, diphenhydramine, lincomycin, gemfibrozil and mfenamic acid showing a RSD greater than 70% and the other compounds having a RSD between 30 to 50%.
- Interday precision in terms of peak area for the detected compounds were consistent with that of the intraday. Intraday and interday precision in terms of RT for all the detected compounds were ≤5%, showing a very good reproducibility and repeatability in terms of retention time.

REFERENCES


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