Ambient MS Round Robin

1. Aim of the Study

Over recent years a number of ambient ionisation techniques have been developed, and have become a key tool for the mass spectrometrist, allowing the analysis of samples without the need for sample pre-treatment in a wide variety of application areas, however, to date, no systematic comparison has been done across these techniques with a range of analytes from different application areas.

This inter-laboratory study aims to evaluate the applicability of the different ambient ionisation techniques across a range of analytes and to evaluate which parameters are being used by different laboratories to obtain their spectra. The study will also aim to evaluate typical analytical chemistry metrics such as sensitivity, reproducibility, linearity and the effect of matrix interferences. The resulting data will be used to understand the optimum parameters for each technique and to evaluate the differences in performance between each technique.

2. Samples

A series of sample solutions will be provided for analysis.

- Paracetamol with Internal Standard, 0.01 mg/mL, +ve
- HMX and TNT solution, 0.01 mg/mL, -ve
- Benylin Mucus cough and cold solution, +ve
- Aldrin, 0.1 mg/mL, +ve
- PEG 1500, 0.1 mg/mL, +ve
- Cholesterol, 0.01 mg/mL, +ve
- Diesel/BioDiesel solutions, +ve

3. Experimental

All samples should be analysed directly by ionisation technique(s) of choice. The entire study should take a maximum of 2 hours to complete (Our experience using ASAP)

MS Conditions: All samples should, in the first instance, be analysed in full scan mode using the suggested mass range (*m/z* 100-1500). For some instruments it may be desirable to acquire data in SIM or SRM mode, these experiments should be carried out in addition to the full scan experiments. The collaborator should determine which scan settings are most appropriate for their experimental set-up. The data presented should be 'raw' e.g. background subtraction should not be used. Samples should be analysed in the suggested ionisation mode but can be analysed in both modes if desired. The conditions used for each sample should be recorded on the appropriate spreadsheet.

1. Optional Quantitation Positive Mode:

   Samples: Paracetamol sample set (PARA S0, PARA S1, PARA S2, PARA S3, PARA S4, PARA S5) (*m/z* 50-500)

Each sample should be acquired sequentially in positive mode, full scan N≥3 times. The blank sample (PARA S0) should be run at the start and end of the run. For scanning instruments this calibration curve can also be acquired in SIM or SRM mode providing SIM ions or SRM transitions are included for the internal standard (paracetamol-d4, mass 155.2 u). The conditions used for analysis should be recorded in the spreadsheet.

2. Optional Quantitation Negative Mode:

   Samples: TNT sample set (TNT S0, TNT S1, TNT S2, TNT S3, TNT S4, TNT S5); (*m/z* 100-500)

   Each sample should be acquired in negative mode, full scan N≥3 times within a single run. The blank sample (TNT S0) should be run at the start and end of the run. For scanning instruments this calibration curve can also be acquired in SIM or SRM mode. The conditions used for analysis should be recorded in the spreadsheet.

4. Data Analysis and Reporting

Where possible we would appreciate being sent the full unprocessed data file for each experiment. We will provide an online location for data submission but can also provide memory sticks if desired, please indicate preference on the sign-up sheet

Where it is not possible to send raw data the collaborator should produce representative spectra, total ion chromatograms and appropriate extracted ion chromatograms for each analyte along with peak areas where appropriate (i.e. for the two calibration curves), to be reported in the attached excel sheets. Any data processing prior to peak integration should be noted when reporting the data.

The results of the round robin will be written up and disseminated among the collaborators. We will also aim to present this work to the wider scientific community through publication and presentations.