Acoustic Ejection Mass Spectrometry (AEMS) for ultra-fast quantification of 17 drugs in sewage

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ABSTRACT

The Echo® MS system performs sample analysis as quickly as 1 sample/second, enabling the testing of hundreds or thousands of samples in a few hours using standard 384- or 1536-well plates. The Echo MS system uses a flow injection input and therefore does not require additional chromatographic supplies (Figure 1). Here, a method using magnetic bead adsorption for pretreatment was used to simplify the preparation process for sewage samples before AEMS analysis.

INTRODUCTION

Measurement of drugs and their metabolites in sewage is typically performed using LC-MS due to its high sensitivity and specificity. Samples of domestic sewage can be tested and the concentration of the drugs present in sewage discharge can be accurately determined, providing insight into the drug consumption trends in a particular area.

MATERIALS AND METHODS

Sample preparation:
A 50 mL sample was dispersed and adsorbed by magnetic beads for 20-30 min. After the magnetic beads and sewage were separated, beads were incubated in 3 mL organic solvent for 15 minutes to elute the compounds before removing the magnetic beads. After drying with nitrogen, the sample was redissolved in 200 µL 20:80, methanol/water.

Echo MS system conditions:
The Echo MS system operates using deionized water for the coupling fluid for acoustic analysis of the sample plate. The carrier solvent was methanol with 0.1% formic acid and a flow rate of 360 µL/min was used. The ejection volume was a single 2.5 nL droplet and SP mode for aqueous/organic samples was used. MS source conditions were optimized for the established flow rate.

MS conditions:
The Echo MS system has an ultra-fast sampling speed of up to 1 sample/second. A method was developed using a magnetic bead sample preparation that allowed sewage samples to be cleaned up and analyzed by a flow injection workflow. The sample ejection parameters, MS conditions and source conditions for this method were optimized. The MS source conditions used were: curtain gas (CUR), 20 psi; collision gas (CAD), 9; nebulizing gas (GS1), 90 psi; heater gas (GS2), 45 psi; ion spray voltage (IS), 5500 V in positive mode and source temperature, 300ºC.

Figure 1. The Echo® MS system used for Acoustic Ejection Mass Spectrometry (AEMS).

RESULTS

The Echo MS system operating on the SCIEX Triple Quad 6500+ mass spectrometer demonstrated strong quantification capability. The method provided lower limits of quantification (LLOQs) for 17 drugs as low as 0.004-0.008 ng/mL in sewage matrix (Table 1 and Figure 2). This sensitivity level was sufficient to detect and quantify the low concentration levels of these drugs in sewage.

Table 1. Linear range and LLOQ of 17 drugs in sewage.

NO.	Compound name	Linear range (ng/mL)	LLOQ (ng/mL)
1	Benzedrine	0.008-2	0.008
2	Methamphetamine	0.004-1	0.004
3	O^6-monoacetylmorphine	0.008-2	0.008
4	Morphine	0.008-2	0.008
5	Ketamine	0.004-2	0.004
6	Norketamine	0.008-2	0.008
7	Cocaine	0.004-2	0.004
8	Benzoylecgonine	0.004-2	0.004
9	MDA	0.008-2	0.008
10	MDMA	0.008-2	0.008
11	Cathinone	0.004-2	0.004
12	Methcathinone	0.004-2	0.004
13	Fentanyl citrate	0.004-2	0.004
14	Diazepam	0.004-2	0.004
15	Estazolam	0.004-2	0.004
16	Methadone	0.004-2	0.004
17	5F-MDMB-PICA	0.004-2	0.004

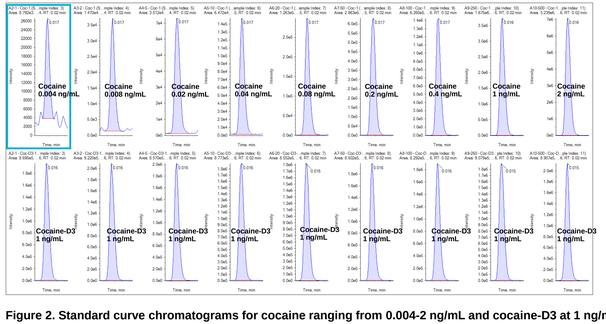

Figure 2. Standard curve chromatograms for cocaine ranging from 0.004-2 ng/mL and cocaine-D3 at 1 ng/mL.

Reproducibility in matrix was tested by spiking blank sewage samples with the mixture of the 17 of drugs at a final concentration of 1 ng/mL. Three parallel samples were tested with 6 replicates. The data showed that the RSD values of the peak area of 17 drugs were all less than 5% (Figure 3).

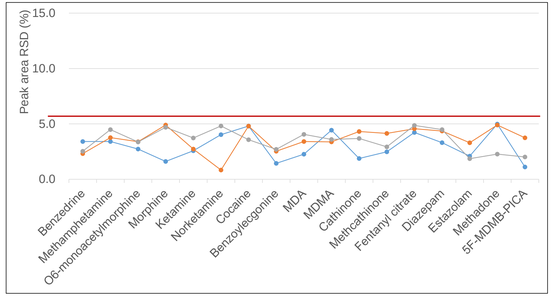

Figure 3. Reproducibility of peak area of 17 drugs spiked in matrix. All drugs targeted in this study showed excellent precision with RSD values less than 5% for all the targeted drugs.

Experiments showed that all 17 drugs were resistant to matrix interference, as matrix interferences were less than 30% (Figure 4). The drugs tested, except for ketamine and cathinone, achieved greater than 80% recovery from sewage (Figure 5).

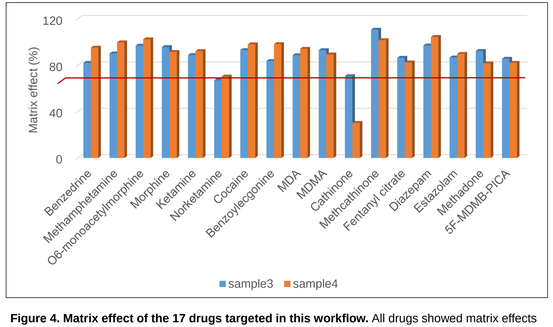

Figure 4. Matrix effect of the 17 drugs targeted in this workflow. All drugs showed matrix effects less than 30% except for norketamine and cathinone.

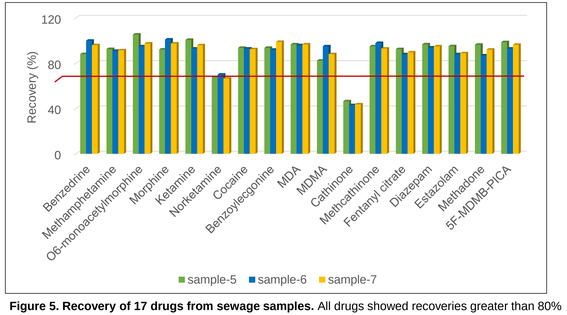

Figure 5. Recovery of 17 drugs from sewage samples. All drugs showed recoveries greater than 80% except for cathinone.

CONCLUSIONS

The Echo MS system has an ultra-fast sampling speed of up to 1 sample/second. 1In this study, 27 sewage samples were tested for the detection of 1 drug in 6.3 min（including pre-scan time). In comparison, the traditional LC-MS method, which needs 11 min per sample, would require more than 23 h to analyze all 127 samples. Moreover, the Echo MS system operating on the SCIEX Triple Quad 6500+ mass spectrometer achieved strong quantification capability, good reproducibility, low matrix effect and high recovery.

This method provided LLOQs for 17 drugs as low as 0.004-0.008 ng/mL in sewage matrix. Reproducibility in matrix was tested by adding 17 drugs at a concentration of 1 ng/mL in sewage. Three parallel samples were tested with 6 replicates. The RSD values of the peak area of 17 drugs were all less than 5%. Furthermore, all 17 drugs were resistant to matrix interference, as matrix interferences were less than 30%. All 17 drugs, except for ketamine and cathinone, achieved recoveries rates greater than 80% recovery from sewage.

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