Rapid Screening of Drug Paraphernalia by DART-HRMS Using the Seized Drug Suite

Juergen Kempf[1], Ilona Nordhorn[2], Carsten Baessmann[2], Eva Niehaus[2], Artem Filipenko[3]

[1]Institute of Forensic Medicine, Medical Center - University of Freiburg, Freiburg, Germany
[2]Bruker Daltonics GmbH & Co. KG, Bremen, Germany
[3]Bruker Scientific LLC, Billerica, MA, USA

Introduction

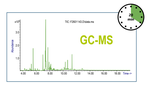
The ESCAPE Project

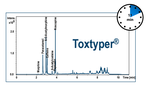
The documentation of injected substances relies primarily on self-reported data from treatment registries and ad hoc surveys, which are often delayed and lack analytical validation. In addition, information on individuals who inject drugs outside the healthcare system remains limited.

The ESCAPE (European Syringe Collection and Analysis Project Enterprise) project of the European Union Drugs Agency (EUDA) aims to close these gaps through timely, local chemical analysis of residual contents of used syringes. It enables monitoring local trends as well as polydrug use and adulterants, that can lead to elevated risks of adverse health effects.

Analytical Bottle Neck in Screening

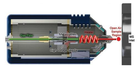
In forensic toxicology, screening for drug of abuse – whether in body fluids, drug paraphernalia, or substance samples – is typically performed using gas or liquid chromatography coupled with mass spectrometry. Depending on the method, analysis usually takes 10 to 25 minutes with additional time for data analysis and reporting. For quick and easy screening, these steps should be highly automated and completed as efficiently as possible.





Direct Analysis in Real Time (DART) is an ionization technique that allows solid, liquid, and gaseous samples to be analyzed directly on a wide variety of surfaces at atmospheric pressure without any special preparation.

In combination with high resolution mass spectrometry this chromatography-free approach provides spectral data for targeted and untargeted screening within a very short analysis time of 20 to 60 seconds.



Aim of the Project

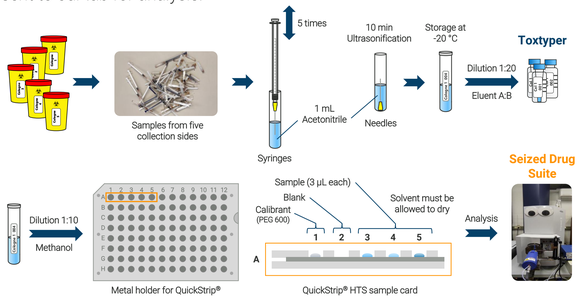
The aim of the project was to evaluate a rapid screening workflow for drugs of abuse and adulterants in used syringes using DART-HRMS.

Workflow:

Methods

Sample Collection and Preparation

Samples of this project were collected at five different harm-reduction services and drug consumptions rooms in the city of Cologne, Germany and sent to our lab for analysis.



Analytical Methods

Bruker Seized Drug Suite

MS-System:	Bruker QTOF
Ion source:	DART JumpShot® pos. mode
Acquisition:	Scanning Linear @ 0.5 mm/s
	Scan Range: 4.5 mm
Gas:	Run Gas: He; Standby Gas: N_2
Temperature:	275 °C
Scan mode:	AutoMS/MS @ 9 Hz
Scan range:	*m/z* 30 -1,000

Routine Toxtyper Screening

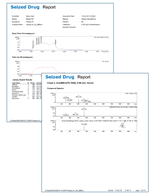
LC-System:	Dionex UltiMate 3000
Eluent A:	H_2O, 2 mM HCO_2NH_4, 0.1% HCOOH, 1% acetonitrile
Eluent B:	Acetonitrile, 2 mM HCO_2NH_4, 0.1% HCOOH, 1% H_2O
Column:	Acclaim® RSLC 120 C18 2,2 µm 120A 2.1x100 mm
Gradient:	11 min gradient elution
MS-System:	Bruker amaZon speed™
Ion source:	Apollo ESI source, pos. mode
Scan mode:	UltraScan (70-800 Da @ 32,500 Da/s)
MSn mode:	AutoMSn (n = 3) with SPL

Data Analysis

For both methods, data evaluation and reporting were performed fully automatically in the DataAnalysis software (Bruker, Germany).

The **Seized Drug Suite** identifies features found via the exact mass of the molecular ion and comparison of MS2 spectra with the associated spectral library (400 entries) and the MMHW LC-HR-MS/MS Library of Drugs, Poisons, and Their Metabolites (Wiley-VCH; 5,000 entries).

With **Toxtyper**, identification is performed by comparing retention times and MS2/MS3 spectra with an in-house spectrum library currently containing 1,475 entries.



Results

Samples Collected (n = 299)

	Syringes with needle	Syringes w/o needle	Needles	No of samples
Side I	29	4	2	35
Side II	11	6	28	45
Side III	44	7	7	58
Side IV	21	12	28	61
Side V	46	34	20	100

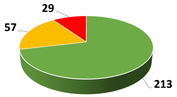
Data Evaluation and Method Comparison

The automatically generated analysis results from the **Seized Drug Suite** and the **Toxtyper** were reviewed and cross-compared. Each of the samples was assigned to one of three categories (see graph on the right):

Consistent results: Both methods detected the sample's main active ingredient; degradation products and accompanying substances were excluded.

Low intensity results: Compounds not detected with DART-MS but low signal intensity in the LC-MSn run.

Inconsistent results: Main active ingredients were not identified by DART-HRMS.



Overall, consistent results were obtained for **71.2 %** of the samples

Exemplary Analytical Results of Seized Drug Suite and Toxtyper

		Seized Drug Suite	Toxtyper	
		Int.	Int.	S/N
Syringe Vision 01	Cocaine	1.0e5	6.5e8	536
	Benzoylecgonine	9.7e4	1.9e8	680
	ADB-4en-PINACA	5.0e3	1.0e8	298
	Paracetamol	8.5e5	1.6e6	2
	Caffeine	3.1e4	-	-
	Methylecgonine	-	6.5e6	39
	Norcocaine	-	2.2e6	8

- Cocaine was detected in the syringe
- The synthetic cannabinoid ADB-4en-PINACA was identified with both methods
- Toxtyper additionally detected Methylecgonine (S/N 38) and Norcocaine (SN 8)
- Paracetamol showed a bad library match with both methods

		Int.	Int.	S/N
Needle DKR 34	Noscapine	8.4e4	5.9e8	1371
	Caffeine	5.6e5	4.9e8	624
	6-Acetylmorphine	1.4e5	3.5e8	632
	Paracetamol	2.1e5	2.2e8	335
	Heroin	6.8e4	9.7e7	348
	Papaverin	-	7.2e7	267
	6-Acetylcodeine	-	5.9e7	176
	ADB-4en-Pinaca	-	1.6e7	41

- Heroin and poppy alkaloids were detected in the needle
- Both methods identified the common cutting agents caffeine and paracetamol
- Toxtyper additionally detected 6-Acetylcodeine an additional heroin marker
- ADB-4en-PINACA could not be identified with DART-MS, probably due to low signal intensity

		Int.	Int.	S/N
Syringe Vision 04	Cocaine	4.0e4	9.9e9	5036
	Benzoylecgonine	5.7e4	2.5e9	5516
	Noscapine	-	1.3e8	431
	Norcocaine	4.2e3	1.2e8	273
	6-Acetylmorphine	-	9.9e7	229
	Methylecgonine	5.0e3	5.3e7	247
	Paracetamol	-	2.2e7	34
	Papaverin	-	2.0e7	64
	Caffeine	7.1e3	1.6e7	35
	6-Acetylcodeine	-	9.5e6	29
	Heroin	-	4.1e6	15
	Cocaethylene	5.6e4	1.6e6	3

- Cocaine and metabolites/degradation products were detected by both methods
- Detection of cocaethylene (low signal-to-noise ratio in LC-MSn) by the Seized Drug Suite
- DART-HRMS did not detect heroin or any of the poppy alkaloids
- Although it is very frequently used as an adulterant, the detection of caffeine alone does not indicate the presence of heroin

Summary

ESCAPE Project

- As expected, heroin and/or cocaine, were the most frequently detected substances in syringe residues. Additional findings included small numbers of amphetamine, MDMA, methylphenidate and methadone.
- A notable result was the detection of the synthetic cannabinoid ADB-4en-PINACA in nine samples from four different collection sites.
- Syringe residue analysis provides a complementary view of the compounds injected within a specific community of drug users

DART-HRMS Analysis of Syringes

- As residual amounts in this sample type are unpredictable, higher initial dilution levels are typically used to avoid contamination, which may initially yield negative results.
- Variability in results for compounds with low signal intensity or low signal-to-noise ratios in LC-MSn analyses highlights the need for a clear strategy to define optimal dilution levels for future studies.
- Due to differences in ionization efficiency and matrix effects, signal intensities can only be compared to a limited extent.
- Although routine LC-MSn analysis provided more detailed sample characterization, the DART-HRMS results show strong concordance with those obtained by LC-MSn.
- Depending on the analytical question, e.g. differentiation of isobaric compounds, strategies for re-analysis and use of a confirmatory analysis (e.g. LC-QTOF-MS) are recommended.

Conclusion

- **Seized Drug Suite** delivered a **>20-fold reduction in overall analysis time** versus conventional LC-MSn screening.
- The integration of DART with HRMS enables high-speed analysis of drug paraphernalia and **confident compound identification** through both targeted and untargeted workflows. Leveraging **third-party spectral libraries and high-resolution data**, this approach delivers reliable identification of unknown substances.
- With its unmatched speed, DART-HRMS is purpose-built for **high-throughput forensic laboratories**, delivering rapid screening either as a stand-alone approach or as an efficient triage tool for targeted follow-up analysis.

Seized Drug Suite