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Portable Quadrupole-Based GCMS Validation and Analysis of Drug Samples

T. R. Fiorentin[1], D. M. Martin[2], T. Browne[2], B. K. Logan[1,3], E. F. Rieders[3]
[1] The Center for Forensic Science Research and Education, 2300 Stratford Ave, Willow Grove, PA 19090
[2]The Colombo Plan, 31 Wijerama Mawatha, Colombo 7, Sri Lanka
[3]NMS Labs, 200 Welsh Road, Horsham, PA 19044

Introduction

Cutting agents, classified as diluents (pharmacologically inactive and readily available substances; e.g. sugars) and adulterants (pharmacologically active, more expensive and less available; e.g. phenacetin) are commonly used to increase profits from street drug sales. They are constantly changing over time increasing the risks to the user's health caused by the compounds interactions. Knowledge about cutting agents is commonly neglected either because they are not detected or not reported. This leads to a lack of information that could be useful for management of intoxications in hospital or other clinical settings, as well in criminal investigations including assistance in the identification of routes of narcotics trafficking.

Objective

To develop, validate and apply a simple procedure using a new portable instrument platform to the analysis of drugs of abuse and adulterants in seized material to elucidate hidden/underlying information of potential great importance to characterizing drug abuse epidemics at a regional, national or international level.

Method

- Samples were prepared by dissolution of powder material in methanol (~0.1 mg/mL) followed by direct injection
- Validation followed the guidelines set forth by the UNODC and SWGDRUG
- In a blind experiment, 38 mock "street drug" samples were prepared via "dilute and shoot" containing varying concentrations and porportions of cocaine, heroin, methamphetamine, and adulterants
- Samples were screened using the FLIR G510 and confirmed by Agilent benchtop GCMS [specify model]
- Analysis performed using Receiver Operating Characteristic (ROC)

Conditions	FLIR G510	GCMS
Acquisition	Full scan 43-425 m/z	Full scan 40-550 m/z
Injection volume	1 µL	1 µL
Injection temperature	275°C (splitless mode)	265°C (splitless mode)
Column	DB-5 (5m x 0.18mm x 0.18µm)	DB-1 (12m x 0.200mm x 0.3µm)
Program temperature	50 to 340°C, 30°C/min, final hold at 340°C for 4 min	50 to 340°C, 30°C/min, final hold at 340°C for 2.33min
Run time	14.6 min	12 min

Acknowledgements

Results/Discussion

Validation

- Method successfully validated for 24 substances
  - Alprazolam, aminopyrine, amphetamine, benzocaine, caffeine, cocaine, codeine, diltiazem, ephedrine, fenethylline, fentanyl, furanylfentanyl, heroin, hydroxyzine, levamisole, lidocaine, methamphetamine, morphine, noramidopyrine (metimazole marker), phenacetin, phencyclidine, procaine, stychnine and xylazine
- Parameters evaluated: interference, precision, limit of detection (LOD), robusteness and carryover
  - Method free from carryover and interferences from 15 commonly encountered analytes
  - LOD varied between 1 and 10% w/w in the drug material
  - Heroin and morphine failed to be detected in 3.33% of the injections, diltiazem and fenethylline in 10%.

Mock samples

FLIR G510 vs. GCMS (%) n=38					
Drug	*Sensitivity*	*Specificity*	*Accuracy*	*PPV*	*NPV*
Alprazolam	100	100	100	100	100
Amphetamine	88.8	100	97.3	100	96.6
Aminopyrine	100	100	100	100	100
Benzocaine	100	100	100	100	100
Caffeine	100	100	100	100	100
Cocaine	100	100	100	100	100
Codeine	100	100	100	100	100
Diltiazem	83.3	100	97.3	100	96.9
Ephedrine	100	100	100	100	100
Fenethylline	100	100	100	100	100
Fentanyl	100	100	100	100	100
Heroin	100	100	100	100	100
Hydroxyzine	100	100	100	100	100
Levamisole	100	100	100	100	100
Lidocaine	100	100	100	100	100
Methamphetamine	100	100	100	100	100
Morphine	100	100	100	100	100
Phenacetin	100	100	100	100	100
Phencyclidine	100	100	100	100	100
Procaine	100	100	100	100	100
Strychnine	100	100	100	100	100
Xylazine	100	100	100	100	100

Conclusion

- Method was successfully validated and proved to be suitable to detect the 24 substances proposed
- Work provides information about accuracy and reliability of new methodology used for on-site seized drug material screening
- Accuracy values were at or close to 100% classifying the FLIR G510 as a suitable tool for field-based screening in seized material analysis
- Further field testing will be performed to continue to evaluate the method's robustness and overall utility in application.

Reference

1. UNODC. Guidance for the validation of analytical methodology and calibration of equipment used for testing if illicit drugs in seized materials and biological specimens: a commitment to quality and continuous improvement. United Nations Office on Drugs and Crime: New York, 2009.
2. SWGDRUG. Scientific working group for the analysis of seized drugs recommendations. 2013.