## **APPLICATION NOTE**



Check for updates

Air monitoring for synthetic cannabinoids in a UK prison: Application of personal air sampling and fixed sequential sampling with thermal desorption two-dimensional gas chromatography coupled to time-of-flight mass spectrometry

Richard Paul<sup>1</sup> | Steve Smith<sup>2</sup> | Luke Gent<sup>1</sup> | Ryan Sutherill<sup>3</sup>

#### Correspondence

Richard Paul, Bournemouth University, Department of Life and Environmental Science, Dorset, BH12 5BB, UK. Email: rpaul@bournemouth.ac.uk

#### **Funding information**

The Professional Trades Union for Prison, Correctional & Secure Psychiatric Workers, Grant/Award Number: 11700

# **Abstract**

In recent years, there have been increasing complaints from staff working in UK prisons of secondary exposure to psychoactive drug fumes, often believed to be synthetic cannabinoids. Our pilot study aimed to provide an initial evidence base for this issue and reveal compounds of interest within indoor prison air. Here, we present a new method for the detection of synthetic cannabinoids in air and demonstrate its application in a UK prison. Air sampling was conducted using a fixed sequential sampler, alongside personal air sampling units worn by prison officers within an English prison. Air samples were collected onto thermal desorption (TD) tubes and analysed via comprehensive two-dimensional gas chromatography coupled to time-of-flight mass spectrometry (GC × GC-TOF MS). This study is the first of its kind in a prison setting, and the approach is of importance to analytical scientists, policy makers and public health employees tasked with the health and safety of prison staff. GC × GC-TOF MS analysis was able to separate and identify a range of compounds present in the prison air samples. Analysis of the TD tubes did not reveal any synthetic cannabinoids from the fixed pump air samples or the personal pump samples worn by prison officers. Air monitoring in prisons presents a challenge of logistics and science. Fixed sequential air sampling combined with personal air monitoring devices allowed air from multiple locations within a prison to be collected, providing a comprehensive approach to evaluating the air that prison staff is exposed to during a fixed time period.

# 1 | INTRODUCTION

For several years now, prison staff working in the United Kingdom has consistently expressed concern about the prevalence of psychoactive substance abuse by prisoners.<sup>1</sup> There have been reports (in the media and first-hand from prison officers) of staff illness ranging from symptoms such as headaches and disorientation, to more serious

symptoms, which it is claimed arise from secondary exposure to drug fumes whilst working at prisons. The drugs in question are often reported as new psychoactive substances (NPS). The Psychoactive Substances Act 2016<sup>2</sup> defines a psychoactive substance as something that produces a psychoactive effect in a person by stimulating or depressing the central nervous system and affecting mental functioning or emotional state. To date there is no evidence to support the

This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

© 2021 The Authors. Drug Testing and Analysis published by John Wiley & Sons Ltd.

<sup>&</sup>lt;sup>1</sup>Department of Life and Environmental Science, Bournemouth University, Dorset, UK

 $<sup>^2</sup>$ Markes International, Llantrisant, UK

<sup>&</sup>lt;sup>3</sup>SepSolve Analytical, Peterborough, UK

claims from UK prison staff concerning secondary exposure to these compounds. Here, we report a new method for the detection of synthetic cannabinoids in air and demonstrate its application in a UK prison.

Research to date has largely focused on the sampling of psychoactive substances that are present in particulate matter (PM), typically with the common grain sizes of 2.5 and 10 µm (so-called PM2.5 and PM10, respectively) as these sizes may be inhaled.<sup>3-5</sup> Such particulates may be sampled from air using various volume samplers, low, medium or high volume. Low volume samplers have been used in this manner to collect airborne particulates in a small establishment to minimise microenvironment perturbation, which can occur with larger volume samplers. 6 Polytetrafluoroethylene (PTFE) membrane filters or quartz filters are used for the collection of PM, and such membranes may then be subjected to solvent extraction or similar prior to analysis. Analytical systems require significant separation capabilities to effectively resolve the high number of airborne components that are often present in such samples. Gas or liquid chromatography coupled to mass spectrometry (GCMS or LCMS) have been used in several air sampling studies. 4,7-14 Solid phase microextraction (SPME) with ion mobility spectrometry (IMS) have also been applied in this field. Lai et al. 14 applied SPME and IMS for the headspace sampling and analysis of airborne cocaine, MDMA and marijuana.

To ascertain broad drug trends in prison populations, drug monitoring research in prisons has more often been accomplished using wastewater analysis, 15,16 as opposed to other matrices. There has been greater success in the detection of volatiles in high-risk areas that face relatively constant exposure to psychoactive substances due to the nature of how they are used. 17-19 Lai et al. 14 screened commercial cargo air for illicit substances using SPME and IMS. Doran et al.<sup>20</sup> used both SPME and charcoal cartridge sampling to determine air quality in police drug safes and storage areas, that is, high-risk drug exposure areas. Various analytical approaches have been used to investigate other high-risk areas. Madireddy et al.<sup>21</sup> investigated the presence of eight drugs on countertop surfaces in a selected drug household; they compared the target recoveries of the SPME fibre to the containers they were in to determine the ageing process of the volatiles on the surface. Variability was low for some of the recoveries after a certain number of hours (15 h), comparing them to van Dyke et al.<sup>22</sup> indicates that wipe sampling is a viable and reproducible method of surface analysis for certain illicit compounds (methamphetamine and related compounds).

Fent et al.<sup>23</sup> (2011) conducted similar research to assess the ventilation and exposure in a Kentucky police station drug vault and adjacent areas. The employees were experiencing relevant health symptoms, and there were concerns it was due to the drug exposure in these areas. Concentrations (nanograms per cubic meter of methamphetamine, oxycodone and THC in the air) were found to be relatively low; however, cocaine ranged from no detection to up to 12,000 ng/m³ (which is relatively low compared to actual recreational doses). Surface sampling revealed quantifiable levels of all the target drugs except for methamphetamines (cocaine was the highest). These combined methods, further information from employees, ventilation

assessments, temperature and humidity data revealed that this workplace needs to improve its health and safety regulations to avoid it becoming a high-risk drug exposure area.

Investigating the issue of secondary exposure to NPS specifically in prisons is a complicated undertaking. Possible approaches include wide-scale investigations to determine which specific NPS and traditional drugs are being abused in prisons, and mandatory drug testing of prisoners assists with gathering this intelligence. In addition, wastewater sampling at prison sites has been shown to provide useful intelligence on drug trends within prisons over specific time periods. <sup>15,24</sup> Intelligence on the drug issues faced by prisons can then be combined with an analytical strategy to investigate the possibility of secondary exposure to such drugs as claimed by prison officers

To address this issue, we have designed a protocol for indoor air sampling in prisons capable of capturing and identifying a wide range of relevant compounds. A combination of air sampling onto TD tubes from a fixed location using a sequential air sampler, with portable air sampling units has been tested in our laboratory (with analysis via GC × GC-TOF-MS) and applied in a pilot study in an English prison in early 2020. Whilst the analysis of synthetic cannabinoids in prison air has not been published before, cigarette smoke and air particulates have been investigated in prisons. Jayes et al.<sup>25</sup> compared the concentrations of airborne particulate matter (PM2.5) in four prisons in England before, and 3 months after, a smoke-free policy was implemented. Using personal aerosol monitors for real-time aerosol mass concentrations, they found a large reduction in PM2.5 concentrations across the four sampled prisons (66% reduction). Smoke-free policies in prisons certainly have improved the level of potential harm due to second-hand exposure to cigarettes. However, second-hand exposure to drug fumes in prisons remains a serious issue requiring investigation. Our study is the first of its kind, and we present here the instrumentation, methodology and data from this first prison trial.

### 2 | INSTRUMENTATION

# 2.1 | MTS-32 sequential sampler and ACTI-VOC low-flow pump air samplers

The multi-tube sequential sampler (MTS-32) is a compact portable sampler used to monitor concentrations of compounds in the air over time periods suited to the researcher. The unit features a constant-flow pump to ensure consistent volumes of air sampled regardless of variations in the ambient air or in tube impedance. The pocket-size ACTI-VOC air pump has a wide range of applications for sampling air and gas for thermal desorption (TD) analysis. The pump has a screw-operated flow adjuster to ensure an easy calibration of flow rate, which automatically compensates for different impedances, meaning constant flow rate is achieved. The ACTI-VOC can be used on its own with a TD tube attached for portable, personal air sampling, and additionally can be used within the MTS-32 sampler.

#### 2.2 | TD and sorbent tubes

TD is a proven 'front-end' technology for GC and GC-MS that is applicable to the analysis of volatile and semivolatile organic compounds (VOCs and SVOCs) in a wide range of samples—gases, liquids and solids. It combines pre-concentration, desorption/extraction and GC injection.

Sorbent tubes, used to trap target analytes emitted from the samples, are thermally desorbed by heating in a flow of inert gas. The released components are then transferred to an electrically cooled, narrower 'focusing' trap within the TD system. After completion of the primary (tube) desorption stage, the focusing trap is desorbed by rapidly heating in a reverse flow of carrier gas ('backflush' operation). This transfers the organic compounds into the capillary GC column for separation. This maximises concentration enhancement and produces narrow chromatographic peaks optimising sensitivity across a broad volatility range.

The use of TD sorbent tubes offers some advantages over alternative sampling techniques such as SPME. SPME typically contains a very small amount of phase and is a competitive equilibration technique. With TD sorbent tubes as you pass the gas through multiple beds of sorbent material the material adsorbs volatiles from the gas so enriching the sample rather than simply reaching an equilibration level with the exposed atmosphere. The TD technique is simple to employ as a dynamic technique (pumped air) rather than passive, so many litres of air sample can be drawn over the sorbent material in the TD tube, vastly enhancing the contact area of the sorbent with the surrounding air.

TD tubes may utilise a multi sorbent bed tube containing sorbents of different service area and strength. The first bed that the air sample is exposed to is often a weaker sorbent such as Tenax; this has been shown to have high retention for compounds in the range C6+ enabling it to be used to retain compounds from many litres of sampled air. The Tenax is then backed with a carbon base sorbent with a much higher surface area; this is good for enriching compounds in the range C3+. The reason for placing a weaker sorbent in front of the stronger sorbent is not due to the sampling stage but the TD stage. Tenax will readily release compounds in the range C6-C35 if heated to around 220-280°C with a reverse flow, however if semivolatiles reached the stronger sorbent, in the case a dual bed sorbent was not used, then some of the semi volatile compounds could become irreversibly retained. Hence, using tubes with multiple beds is common practice. In comparison, SPME uses an equilibration approach, and there is competitive sorption from components in the sample and because the phase is very small in comparison to TD sorbent tubes, it can easily become saturated with one chemical at the expense of reduced enrichment of other chemicals.

# 2.3 | Comprehensive two-dimensional gas chromatography and time-of-flight mass spectrometry

Comprehensive two-dimensional gas chromatography (GC  $\times$  GC) has become the technique of choice for the separation of complex

mixtures. The enhanced separation capacity offered by the coupling of two columns of different selectivity provides greater insight in to sample composition. Time-of-flight mass spectrometry is the detector of choice for GC  $\times$  GC, due to high-speed spectral acquisition ensuring compatibility with the narrow peak widths observed in GC  $\times$  GC analyses. Furthermore, time-of-flight instruments are not mass filters, so we simultaneously analyse all ions with high sensitivity, making them well suited to untargeted screening of indoor air.

 $\mathsf{GC} \times \mathsf{GC}\text{-}\mathsf{MS}$  offers some advantages over LCMS-MS in the context of drug analysis from indoor air. GC as a technique offers improved analysis of compounds in air samples as the sample can be concentrated from many litres of air using TD tubes and then conveniently thermally desorbed directly into the GC (GC × GC) MS system to transfer the compounds enriched from many litres into the GC in a few hundred microlitres of gas. Whereas with LC, any collection device would need to be extracted with an organic solvent, potentially several millilitres of solvent would be need, yet only a few microlitres could be injected, diluting the sample and reducing the amount injected for detection. Furthermore, GC × GC is a comprehensive separation technique which can be utilised to separate many thousands of components in a complex sample, so greatly improving the analytical resolution, achieved by the use of two orthogonal column phases, which are applied in series, modulating small sections of the first separation to the second.

## 3 | EXPERIMENT

#### 3.1 | Laboratory trials

Initial trials were conducted with synthetic cannabinoids: AB-FUBINACA, UR144, MDMB 4en Pinaca, MDMB CHMCA. Liquid standards of the synthetic cannabinoids (10 ppb) were directly spiked onto material emission TD tubes prior to TD and analysis via  $GC \times GC$ -TOF-MS as described in Section 3.6. Air sampling trials were conducted by volatilising the synthetic cannabinoids (10 ppb) and collecting on to material emission TD tubes, again followed by  $GC \times GC$ -TOF-MS analysis. Recovery experiments were performed by volatilising the synthetic cannabinoids at their limit of detection (LoD) in 1 L of air. Data from these experiments were then used to calculate LoD in 1 m<sup>3</sup> of air and in 24 L of air (the volume of air sampled during one 8-h prison officer shift using a portable air sampler).

### 3.2 | Prison officer volunteers

Prison officers (n=15) were recruited with assistance from Her Majesty's Prison and Probation Service (HMPPS) and The Professional Trades Union for Prison, Correctional & Secure Psychiatric Workers (POA). Officers taking part in this study agreed to wear a personal air sampling device (ACTI-VOC unit, Markes International) for the duration of one shift (8 or 12 h) during the study period and to record their whereabouts on site at regular intervals. HMPPS, prison staff and

POA representatives were briefed about the experiment parameters, objectives and what was involved in taking part. Full training for officers was provided on the operation of personal air sampling pumps and protocols for TD tube handling, storage and logging.

Staff log forms were created for the TD tubes and a daily checklist for the personal air sampling ACTI-VOC units required information specific to the prison officers ID, date, shift time and the TD tube serial number.

Ethical permission was granted for the study by Bournemouth University, Faculty of Science and Technology Ethics Committee (ID 27716).

# 3.3 | MTS-32 fixed sequential air sampling protocol

Air samples were collected over a 24-h period using a field-portable multi-tube sequential sampler (MTS-32, Markes International) unit mounted in a fixed location within the prison. The MTS-32 unit was placed at the head of a first floor landing on a wing within the prison, sited in a secure location approximately 2.4 m above first floor level.

Prior to the start of the sampling period, the TD tubes chosen for this study were conditioned, capped and kept in refrigerated conditions until the day of the experiment. One extra TD tube was capped and stored with the other samples as a 'trip blank' to monitor background levels and assess potential contamination. The TD tubes employed in this study are known as material emissions/indoor air stainless steel sorbent tubes from Markes International. They comply with ISO 16000-6 for simultaneous monitoring of very volatile organic compounds (VVOCs), VOCs and SVOCs due to the sorbents

contained within (plug of quartz wool, Tenax and Carbograph 5TD); suitable for monitoring indoor air quality and for retaining compounds in the C4–C32 analytical range.

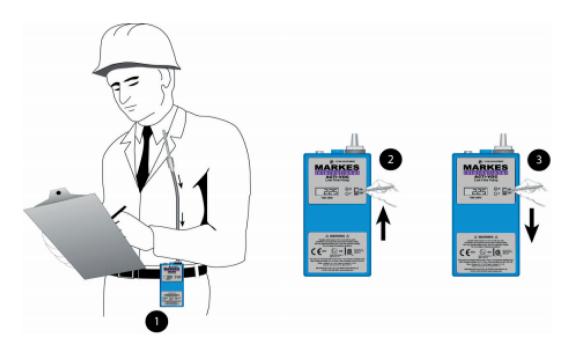
When designing air sampling experiments of this nature using TD tubes, it is important to consider breakthrough volumes. If very volatile analytes are expected then the use of serially coupled TD tubes may be employed. For this application, serial coupling of tubes was not employed; instead, a multibed sorbent packed in the tubes was utilised, because the expected target components were not very volatile then it was deemed the likelihood of breakthrough of the trap for these particular components would be highly unlikely.

Seven TD tubes were inserted into the tube manifold block of the MTS-32, the first two tubes were trial tubes and the remaining five collected air for analysis during the sampling period. Each TD tube sampled air for 4.8 h at a flow rate of 50 ml/min for a total of five tubes over 24 h (excluding the initial trial tubes).

A checklist was prepared for the static sampler and was completed at the end of the allocated sampling time. Following the sampling period TD tubes were removed from the unit, capped and stored in a refrigerator.

#### 3.4 | ACTI-VOC air sampling protocol

Two ACTI-VOC portable air pumps were provided to the 15 staff volunteers to be used during a 8- or 12-h shift during the study period. They are designed to use one TD tube at a time, at a set flow rate, whilst recording the sampling time. Figure 1 shows the setup of the ACTI-VOC units. The pump flow rate was set to 50 ml/min. The TD tubes were stored in an on-site, secure refrigerator and separated



**FIGURE 1** Diagram used to instruct volunteers on how to wear and attach the ACTI-VOC pumps [Colour figure can be viewed at wileyonlinelibrary.com]

between tubes to be used and those tubes that had been used in a shift by the ACTI-VOC pumps.

Volunteers were instructed to follow a daily checklist during their shift to assist in correct sample collection. Volunteers were instructed to record if they left the prison site, and if so, the pump would be switched off and removed, noting the time, and then switched back on when resuming their shift inside the prison. A radio check was performed every 30 min to log the position of volunteers with the ACTI-VOC within the prison. Body cameras were worn to corroborate staff movements further if necessary. At the end of the shift, the pump was turned off and sampling time and shift time was recorded. The TD tube was removed, capped and stored in the refrigerator.

Following the static and personal air sampling protocols, the TD tubes were transported to the laboratory to be analysed using the TD-GC  $\times$  GC-TOF MS analytical system.

### 3.5 | TD parameters

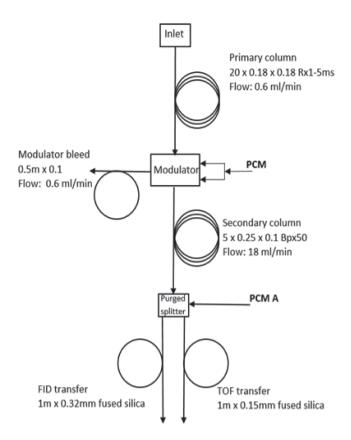
The TD instrument was a UNITY-xr, utilising a materials emissions (U-T12ME-2S) cold trap. Pre-purge was set for 1 min at 50 ml/min. Tube desorption ran for 10 min at 280°C, 50 ml/min. Trap puge was 1 min at 50 ml/min with a low temperature of 30°C and high of 300°C set to maximum heating rate. Trap hold time was 2 min, with outlet split at 4 ml/min and flow path temperature of 180°C.

## 3.6 | $GC \times GC$ method and TOF conditions

The GC was an Agilent 7890B GC using helium carrier gas. Oven ramp was set to 40°C for 2 min, ramped at 5.5°C/min to 250°C and held for 15 min. An INSIGHT<sup>®</sup> flow modulator (SepSolve Analytical) was used Modulation period was 3.5 s with a flush time of 160 ms. Figure 2 shows the column set configuration used in the GC × GC setup. The BenchTOF-Select<sup>™</sup> mass spectrometer (Markes International) used in the study had an ion source temperature of 325°C with a transfer line temperature of 300°C. Mass range was set to 35–500 amu. Data rate was 50 Hz with ionisation energies set to −70 and −14 eV.

# 3.7 | Spectral libraries and chromatographic searching

Chromatography arising from indoor ambient air monitoring is often complex, and the GC  $\times$  GC technology employed in this study has the ability to resolve thousands of individual compounds that may be present in each sample. Peak identifications were made using a combination of three libraries: (1) NIST mass spectral library, (2) Cayman Spectral Library and (3) a bespoke target library of 134 compounds comprising psychoactive substances and known thermal degradation products.



**FIGURE 2** Column set configuration employed for  $GC \times GC$ . Primary column: Rx1-5ms. Secondary column: Bpx50

#### 4 | RESULTS

#### 4.1 | Laboratory trial results

Analysis of direct TD tube spiking experiments with AB-FUBINACA, UR144, MDMB 4en Pinaca, MDMB CHMCA showed that the synthetic cannabinoids could be retained by the TD tubes and accurately resolved and identified with our GC × GC-TOF-MS method. The compounds were correctly identified by the NIST library as the top hit.  $GC \times GC$ -TOF-MS results from the air sampling experiments with volatilised standards showed a complex chromatogram with a large number of peaks present. The enhanced separation provided by flow modulated GC  $\times$  GC was vital for the detection of the synthetic cannabinoids within the air sample. UR144 when volatilised breaks down to a compound known as UR144 degradant, and both the original compound and the degradant were resolved as separate peaks. UR144 degradant was present at approximately twice the signal of UR144, but due to availability of reference standards for the degradant, this compound was not assessed for LoD and recovery. The LoD on-tube, LoD in 24-L air (volume sampled during an 8-h prison officer shift using portable sampler), LoD in 1-m3 air and calculated percentage recoveries for the synthetic cannabinoids are presented in Table 1.

TABLE 1 Limits of detection and recovery for AB-FUBINACA, UR144, MDMB 4en Pinaca and MDMB CHMCA

	LoD (nanogram on tube)	LoD in 24 L air (ng/L)	LoD in 1-m <sup>3</sup> air (pg/m <sup>3</sup> )	Percentage recovery in 1-L air (%)
AB-FUBINACA	0.15	0.02	0.5	30
UR144	1.7	0.07	1.7	50
MDMB 4en Pinaca	4.3	0.18	4.3	39
MDMB CHMCA	3.8	0.16	3.8	21

Abbreviation: LoD, limit of detection.

# 4.2 | Prison study results overview

On average the GC  $\times$  GC-TOF-MS system resolved 1000 to 2000 peaks in each sample of air sampled within the prison (fixed or personal pumps). Chromatography was searched against the three libraries as discussed in Section 3.7. Any peak reported as matching a compound of interest (psychoactive substance or thermal degradation product) was manually examined to compare the mass spectrum. There was no evidence of synthetic cannabinoids detected in the MTS-32 air samples or ACTI-VOC personal pump air samples.

A possible explanation for the negative finding could be that no synthetic cannabinoids were being used during the study period or that the level of substance use was very low. The increased restrictions on prison visitation and prisoner movement during the study period as a result of Covid-19 may have contributed to this. It is also a possibility that synthetic cannabinoids were present in the air, but at levels too low to detect. Whilst our study did not reveal detectable drug concentrations in the prison air, there may be alternative explanations to attempt to explain the serious symptoms experienced by prison officers in the past. Drug residues may be transferred to work surfaces and handles in a variety of settings<sup>19</sup> and such contamination could pose a risk. In our opinion though, a contaminated surface mechanism of secondary drug exposure seems unlikely as a cause of prison officer symptoms. Some UK prisons have described guite high numbers of officers claiming exposure to psychoactive substances within short periods of time, which does not tally well with a surface transfer mechanism.

Doran et al.<sup>20</sup> investigated air quality inside police drug safes and storage areas and also found no evidence of drug residues in air samples. Surface drug residues were found on handles and shelving units; however, no residues (22 illicit compounds and 2 metabolites) were detected using carbon traps and analysis via LC-MS-MS. The authors reported that chemical odours emanating from drug safes may not be a result of the drugs themselves but are likely due to VOCs arising from chemicals used in drug manufacture amongst other potential sources. The preparation of synthetic cannabinoids by drugs users may involve bulk drug powders, dissolved in organic solvents, which are sprayed onto herbs; residual solvents may cause toxic effects.<sup>26</sup>

We have demonstrated in our volatilisation studies for the four synthetic cannabinoids that they are detectable in air samples following volatilisation. Volatility of the synthetic cannabinoids will vary due to chemical class and structure, though it has been demonstrated in other studies that synthetic cannabinoids are also detectable in air

following smoking experiments. Naqi et al.<sup>27</sup> investigated the thermal degradation of synthetic cannabinoids through a smoking simulation experiment. They found that the target analytes (AB-CHMINACA, AM-694, 5F-ADB, MDMB-CHMICA, MMB-2201, and 5F-PB-22) could be detected after combustion in the first smoke trap outside of the combustion zone, indicating a very good volatility. The volatility of synthetic cannabinoids has not though been investigated heavily, and will range based on structure and class of the specific compounds.

#### 4.3 | MTS-32 results

The chromatography revealed a wide range of compounds present in the prison air, and these fall into several categories: flavours, fragrances, pharmaceutical preparation, chemical reagents/intermediates, industrial manufacturing or refining of commercial products and pollutant/contaminants. These categories overlap one another for several compounds as they are multi-functional for their compound class; for example, several alcohol-based compounds serve different potential functions, so determining their origin in the current context is not possible.

There was a range of compounds found in the MTS-32 samples. The largest proportion of the compounds detected were alkenes (13.45%), alkanes (11.21%), alcohols (10.31%), benzene derivatives (9.87%) and aldehydes (9.87%). The remaining types range from toluenes, ketones, to furans, terpenes and others in smaller proportions (0.45%-6.8%). Our analysis of the data has suggested that many of these compounds would routinely be expected in indoor air samples, particularly in sites with heavy human activity or close to vehicular traffic.

#### 4.4 | ACTI-VOC personal pump results

A wider range of compounds was detected in the ACTI-VOC personal air samples when compared to the MTS-32 samples overall, which is expected due to there being a larger number of samples collected over several days, coupled with the portability of the personal pumps sampling air from several areas of the prison site. Similarly, alkanes (16.42%), alkenes (10.58%), aldehydes (10.22%) and alcohols (8.02%) make up a significant proportion of this sample group. Personal air samples displayed a similar range of less frequent chemical classes

ranging from benzene derivatives, esters and terpenes to ketones, naphthalenes, toluenes and others (0.36%–7.66%). Our analysis of the data has suggested that many of these compounds would routinely be expected in indoor air samples, particularly in sites with heavy human activity or close to vehicular traffic. MTS32 samples and ACTI-VOC samples both displayed a very large number of resolved peaks, often 1000 to 2000 peaks. We have presented the top 50 compounds detected from each sampling device in Table S1 in the supporting information.

## 4.5 | Trip blank TD tube

One TD tube was designated as a 'trip blank' and set aside to be kept with the other TD tubes during transportation and storage at the prison site to compare with the analytical tubes used for the ambient air via the MTS-32 and ACTI-VOC sampling devices. The purpose of this tube is to act as a control for potential contamination events occurring during storage of tubes on site or transportation to and from site.

Analysis of the trip blank revealed a small number of compounds at very low levels (in most cases approximately 1000 times lower than levels found in the real samples). These compounds present at such levels do not suggest any contamination.

## 5 | CONCLUSION

This study represents the first trial of the combination of fixed location sequential air sampling with portable, body-worn active air sampling devices for the purpose of air monitoring for synthetic cannabinoids in a public sector prison. The methodology for air sampling using this combined approach allowed a significant range within the prison to be sampled over the study period, collecting air from a prison wing (MTS-32) and from all areas patrolled by participating prison officers (ACTI-VOC). An effective methodology for GC  $\times$  GC-TOF-MS analysis of the TD tubes was created, which enabled a wide range of compounds to be detected, with excellent resolution.

Air sampling at the prison did not reveal any synthetic cannabinoids from either sampling method. It is possible that no synthetic cannabinoids were being used during the study period, or that the level of substance use was very low. The increased restrictions on prison visitation and prisoner movement during the study period as a result of Covid-19 may have contributed to this. It is also a possibility that synthetic cannabinoids were present in the air but at levels too low to detect. Laboratory trials were however successful and the approach for deployment in a prison setting including staff training, MTS-32 and ACTI-VOC sampling protocols has been piloted for the first time in this novel study.

#### **ACKNOWLEDGEMENT**

This work was funded by The Professional Trades Union for Prison, Correctional & Secure Psychiatric Workers (Grant ID 11700).

#### CONFLICT OF INTERESTS

There are no conflicts of interest to disclose.

#### ETHICAL APPROVAL

Ethical permission was granted for the study by Bournemouth University, Faculty of Science and Technology Ethics Committee (ID 27716).

#### **CONSENT STATEMENT**

All prison staff volunteers provided consent through approved ethical procedures as approved by Bournemouth University, Faculty of Science and Technology Ethics Committee (ID 27716). Permission to reproduce material from other sources: not applicable.

#### **ORCID**

Richard Paul https://orcid.org/0000-0001-5545-3582

#### **REFERENCES**

- Public Health England. New psychoactive substances (NPS) in prisons: A toolkit for prison staff 2015. https://assets.publishing. service.gov.uk/government/uploads/system/uploads/attachment\_ data/file/669541/9011-phe-nps-toolkit-update-final.pdf. Accessed 4<sup>th</sup> November 2020.
- The National Archives. Psychoactive Substances Act 2016 C.2 2016. https://www.legislation.gov.uk/ukpga/2016/2/crossheading/ psychoactive-substances/enacted. Accessed 4<sup>th</sup> November 2020
- Ling SH, Eeden SFV. Particulate matter air pollution exposure: role in the development and exacerbation of chronic obstructive pulmonary disease. *Int J Chron Obstruct Pulmon Dis.* 2009;4:233-243. https://doi. org/10.2147/copd.s5098
- Viana M, Postigo C, Querol X, et al. Cocaine and other illicit drugs in airborne particulates in urban environments: a reflection of social conduct and population size. *Environ Pollut*. 2011;159(5):1241-1247. https://doi.org/10.1016/j.envpol.2011.01.032
- Xing Y, Xu Y, Shi M, Lian Y. The impact of PM2.5 on the human respiratory system. J Thorac Dis. 2016;8(1):69-74. https://doi.org/10.3978/j.issn.2072-1439.2016.01.19
- Cecinato A, Balducci C, Romagnoli P, Perilli M. Behaviours of psychotropic substances in indoor and outdoor environments of Rome, Italy. *Environ Sci Pollut Res.* 2014;21:9193-9200. https://doi.org/10.1007/s11356-014-2839-2
- Cecinato A, Balducci C, Mastroianni D, Perilli M. Sampling and analytical methods for assessing the levels of organic pollutants in the atmosphere: PAH, phthalates and psychotropic substances: a short review.
   Environ Sci Pollut Res. 2012;19:1915-1926. https://doi.org/10.1007/s11356-012-0959-0
- Cecinato A, Balducci C, Romagnoli P, Perilli M. Airborne psychotropic substances in eight Italian big cities: burdens and behaviours. *Environ Pollut*. 2012;171:140-147. https://doi.org/10.1016/j.envpol.2012. 07.033
- Balducci C, Green DC, Romagnoli P, et al. Cocaine and cannabinoids in the atmosphere of Northern Europe cities, comparison with Southern Europe and wastewater analysis. *Environ Int*. 2016;97:187-194. https://doi.org/10.1016/j.envint.2016.09.010
- Cecinato A, Romagnoli P, Perilli M, Balducci C. Psychotropic substances in house dusts: a preliminary assessment. *Environ Sci Pollut Res.* 2017;24:21256-21261. https://doi.org/10.1007/s11356-017-9549-5
- Postigo C, de Lopez MJ, Viana M, et al. Determination of drugs of abuse in airborne particles by pressurized liquid extraction and liquid chromatography-electrospray-tandem mass spectrometry. *Anal Chem*. 2009;81(11):4382-4388. https://doi.org/10.1021/ac9002485

- Viana M, Querol X, Alastuey A, et al. Drugs of abuse in airborne particulates in urban environments. *Environ Int.* 2010;36(6):527-534. https://doi.org/10.1016/j.envint.2010.04.004
- Mastroianni N, Postigo C, de López M, et al. Comprehensive monitoring of the occurrence of 22 drugs of abuse and transformation products in airborne particulate matter in the city of Barcelona. Sci Total Environ. 2015;532:344-352. https://doi.org/10.1016/j.scitotenv. 2015.06.027
- Lai H, Corbin I, Almirall JR. Headspace sampling and detection of cocaine, MDMA, and marijuana via volatile markers in the presence of potential interferences by solid phase microextraction-ion mobility spectrometry (SPME-IMS). *Anal Bioanal Chem.* 2008;392:105-113. https://doi.org/10.1007/s00216-008-2229-z
- Postigo C, Alda ML, Barceló D. Evaluation of drugs of abuse use and trends in a prison through wastewater analysis. *Environ Int*. 2011; 37(1):49-55. https://doi.org/10.1016/j.envint.2010.06.012
- van Dyken E, Thai P, Lai FY, et al. Monitoring substance use in prisons: assessing the potential value of wastewater analysis. Sci Justice. 2014;54:338-345. https://doi.org/10.1016/j.scijus.2014. 06.006
- McKenzie EJ, Miskelly GM, Butler PAG. Detection of methamphetamine in indoor air using dynamic solid phase microextraction: a supplementary method to surface wipe sampling. *Anal Methods*. 2013;5: 5418-5424. https://doi.org/10.1039/C3AY40537K
- Cecinato A, Romagnoli P, Perilli M, Patriarca C, Balducci C. Psychotropic substances in indoor environments. *Environ Int.* 2014;71:88-93. https://doi.org/10.1016/j.envint.2014.06.008
- Doran GS, Deans RM, de Filippis C, Kostakis C, Howitt JA. Quantification of licit and illicit drugs on typical police station work surfaces using LC MS/MS. *Anal Methods*. 2017;9:198-210. https://doi.org/10.1039/C6AY02668K
- Doran GS, Deans R, De Filippis C, Kostakis C, Howitt JA. Air quality inside police drug safes and drug storage areas. J Anal Toxicol. 2018; 42:360-364. https://doi.org/10.1093/jat/bky011
- Madireddy SB, Bodeddula VR, Mansani SK, Wells MJM, Boles JO. Wipe sampling of amphetamine-type stimulants and recreational drugs on selected household surfaces with analysis by ultraperformance liquid chromatography quadrupole time-of-flight mass spectrometry. J Hazard Mater. 2013;254–255:4-56. https://doi.org/ 10.1016/j.jhazmat.2013.03.012

- 22. van Dyke MV, Serrano KA, Kofford S, Contreras J, Martyny JW. Variability and specificity associated with environmental methamphetamine sampling and analysis. *J Occup Environ Hyg.* 2011;8(11): 636-641. https://doi.org/10.1080/15459624.2011.616820
- Fent KW, Durgam S, West C, Gibbins J, Smith J. Department of Health and Human Services, Centers for Disease Control and Prevention. National Institute for Occupational Safety and Health 2010. https://www.cdc.gov/niosh/hhe/reports/pdfs/2010-0017-3133.pdf
- van Dyken E, Lai FY, Thai PK, et al. Challenges and opportunities in using wastewater analysis to measure drug use in a small prison facility. *Drug Alcohol.* 2016;35(2):138-147. https://doi.org/10.1111/dar. 12156
- Jayes LR, Murray RL, Breton MO, Hill C, Ratschen E, Britton J. Smoke-free prisons in England: indoor air quality before and after implementation of a comprehensive smoke-free policy. *BMJ Open*. 2019;9:e025782. https://doi.org/10.1136/bmjopen-2018-025782
- Choi H, Heo S, Choe S, et al. Simultaneous analysis of synthetic cannabinoids in the materials seized during drug trafficking using GC-MS.
   Anal Bioanal Chem. 2013;405:3937-3944. https://doi.org/10.1007/ s00216-012-6560-z
- Naqi HA, Pudney CR, Husbands SM, Blagbrough IS. Analysis of synthetic cannabinoid agonists and their degradation products after combustion in a smoking simulator. *Anal Methods*. 2019;11:3101-3107. https://doi.org/10.1039/c9ay00722a

#### SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

How to cite this article: Paul R, Smith S, Gent L, Sutherill R. Air monitoring for synthetic cannabinoids in a UK prison: Application of personal air sampling and fixed sequential sampling with thermal desorption two-dimensional gas chromatography coupled to time-of-flight mass spectrometry. *Drug Test Anal.* 2021;13(9):1678–1685. <a href="https://doi.org/10.1002/dta.3101">https://doi.org/10.1002/dta.3101</a>