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TECHNICAL NOTE

Criminalistics; Toxicology

Evaluation of illicit drug contaminants in seized cannabis in Arizona and California, 2023–2024

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Abstract

Despite public health concerns, fentanyl, cocaine, and other illicit drug contaminants remain largely understudied in unregulated cannabis. Here, we developed an analytical method to quantify a panel of common illicit drugs in cannabis and tested the law enforcement seizures in Arizona and California in evaluation of the illicit drug contamination in black- and gray-market cannabis. One hundred and eighteen cannabis dried flower samples were collected by the Maricopa County Sheriff's Office and the US Postal Inspection Service between 2023 and 2024. The prepared samples were analyzed using an Agilent 6495 LC–MS–MS for 16 illicit drugs. Twenty-five (21.2%) of the cannabis flower samples had detectable concentrations of illicit drugs. Only three of the 16 analytes tested were detected, including cocaine (20.3%), methamphetamine (MAMP; 3.4%), and 3,4-methylenedioxymethamphetamine (MDMA/Ecstasy; 0.9%). The highest concentration of illicit drugs was cocaine at 12,000 ppm (or 1.2% by weight). This pilot study demonstrates the presence of illicit drugs in black- and gray-market cannabis largely due to the contamination in the unkempt environment where they are being assembled and distributed. Intentional spiking appears to be rare, but the level of spiked cocaine can pose a health risk to consumers of unregulated products. Further studies are needed to survey the chemical contaminants of black- and gray-market cannabis and better understand their public health risk nationwide.

KEYWORDS
cannabis, cocaine, contaminant, ecstasy, fentanyl, illicit market, LC–MS–MS, methamphetamine, polydrug, spiking

Highlights

- This is one of the largest studies of illicit drug contaminants in black- and gray-market cannabis in the US to date.
- One hundred and eighteen dried cannabis flower samples collected by law enforcement in AZ and CA are analyzed for 16 illicit drugs.
- The most commonly detected drug was cocaine (20%), followed by methamphetamine (3%) and ecstasy (1%).

- The highest concentration of illicit drugs was cocaine at 1.2% by weight and no fentanyl was detected.
- The current study provides evidence suggesting the rise of polydrug preparation facilities in the illicit market.

1 | INTRODUCTION

Despite the legalization efforts at the state and federal level, illicit markets of cannabis – including unlicensed production within a legalized state (i.e., black-market cannabis) and products that may be produced legally but sold outside of a legal supply chain (i.e., gray-market cannabis) – account for most of the cannabis sale in the US [1]. Black- and gray-market cannabis, lacking regulatory oversights, is susceptible to contamination and adulteration during cultivation, processing, and transport. Prior studies in the US and Canada have confirmed that black- and gray-market cannabis frequently contains contaminants, including pesticides [2–4], mycotoxins [4, 5], and heavy metals [4]. This presents a potential health risk to consumers who self-treat diagnosed health conditions with unregulated cannabis [6].

Although environmental and microbial contaminants in black- and gray-market cannabis are receiving more attention, the contamination with fentanyl, cocaine, and other illicit drugs remains a largely understudied issue. One of the key challenges is the lack of validated detection methods for illicit drug contaminants in cannabis. For instance, rapid tests such as fentanyl test strips were not validated for plant materials and were unreliable for cannabis flower [7]. While state-licensed cannabis is tested for safety compliance [8], unregulated cannabis is not routinely tested by law enforcement. Despite the public concerns of fentanyl contamination in cannabis, there have only been individual case reports of fentanyl-laced cannabis exposure [9, 10]. No systematic study has examined the prevalence of fentanyl contamination in cannabis in the US illicit markets.

In this study, we tested cannabis seized by law enforcement in Arizona and California and evaluated drug contamination for 16 illicit substances in black- and gray-market cannabis. The 16 illicit substance panel was based on the US Department of Transportation's 5 panel notice for drug testing [11] with the addition of fentanyl, LSD, and psilocybin. Our objectives were to (i) develop an analytical method to quantify a panel of common illicit drugs and (ii) characterize the prevalence and type of illicit drug contaminants in the cannabis. To the authors' knowledge, this is one of the largest studies of illicit drug contaminants in black- and gray-market cannabis in the US to date.

2 | MATERIALS AND METHODS

2.1 | Cannabis acquisition and extraction

Seized dried cannabis flower was collected by the High-Intensity Drug Trafficking Areas (HIDTA) Task Force of the Maricopa County Sheriff's Office (MCSO) and the US Postal Inspection Service (USPIS) from November 21st, 2023, to June 4th, 2024. Seventy-nine samples were collected in 37 seizures by the HIDTA Task Force in Arizona. In each seizure, cannabis of different sources (e.g., different strain labels) was marked as different batches by law enforcement officers. One dried flower bud was randomly pulled from each batch and stored in a 20 mL scintillation vial on desiccant at 4°C for up to a month. Additionally, the USPIS conducted 24 inspections and collected 39 samples in the same period in California and Arizona. According to the HIDTA Task Force's investigation, 90% of the seized black-market cannabis and marijuana in Arizona in recent years came from California (M. Shay, personal communication).

The cannabis flower samples ($n = 118$) were ground using a mortar and pestle, which was cleaned with 90% ethanol and blow-dried with warm air between samples. A half gram aliquot of each sample was placed into a silylated glass centrifuge tube with 5 mL 80%:20% acetonitrile:water solution and placed on a rotisserie shaker for 5 min. Next, the samples were spun in a centrifuge at 1500 × g for 5 min and the supernatant was transferred to another clean, silylated centrifuge tube. The samples underwent two more 5 mL washes to reach, at most, 15 mL of supernatant extracted.

2.2 | Liquid chromatography sample preparation

The samples were prepared for instrumental analysis by transferring 10 µL of the extract to a LC vial with 25 µL of an internal standard mixture and 1 mL of HPLC grade water (Thermo Scientific). The calibration standards consisted of methamphetamine (MAMP), amphetamine (AMP), benzoylecgonine (BZE, the breakdown product of cocaine), cocaine, codeine, fentanyl, hydrocodone, hydromorphone, LSD, 3,4-methylene dioxymethamphetamine (MDMA/Ecstasy), morphine, oxycodone, oxymorphone, phenylcyclohexyl piperidine (PCP), psilocybin, 3,4-methylenedioxyamphetamine (MDA), and psilocin at 5000 ng/mL each. The internal standards consisted of AMP-d_5, MAMP-d_5, MDA-d_5, and MDMA-d_5 at a concentration of 250 ng/mL in the LC vial.

2.3 | Liquid chromatography analysis

The prepared samples were analyzed for the 16 analytes using an Agilent 6495 LC–MS–MS operated in positive ion mode. Ten microliters of prepared sample were analyzed with an Agilent 1290 LC. The analytical column was an Agilent InfinityLab Poroshell 120

EC-C18 (2.1 × 100 mm 1.9 μm) heated to 50°C. The elution solvents were LC–MS–MS grade water, buffered with 0.1% formic acid, and methanol. The solvent program started with 5% methanol, which was held for half a minute, and then methanol concentration was increased to 98% over 6 min and held for another 4.7 min. The final run time was 11 min. Data from the triple quadrupole MS. was processed using the Agilent MassHunter Quantitative Analysis software (Version 12.0). Analyte identification of cocaine and MAMP was confirmed by three ions in the MS–MS spectrum, while MDMA, MDA, and BZE had two ions (Supporting Information Table S1). The data were manually reviewed to ensure accurate identification of all drugs.

2.4 | Spike sample preparation

Initially, the samples were extracted with a focus on a wider range of analytes, including pesticide residues [12], mycotoxins [5, 12], fungal metabolites [5, 12], and illicit substances. The sample extracts were quickly screened for possible illicit substances. Upon detection of illicit substances, a spiked recovery analysis was conducted to verify the effectiveness of the extraction and analysis procedure for illicit drug contamination in cannabis flowers. The spike recovery trial was conducted on the most commonly detected compounds, namely cocaine, MDMA, and AMP (as a surrogate of MAMP) using certified reference materials. The target concentration for each illicit drug was 1.5 μg/g in dried flower. There were three sets of samples. The first set was three solvent spiked samples consisting of 15 mL acetonitrile–water solution with the spike. The second set was three native samples that were 0.5 g of dried hemp flower without any spike. The last set was three spiked samples that consisted of 0.5 g dried flower spiked with the test chemicals. All nine samples were analyzed with LC–MS–MS. Hemp flower samples from the University of Kentucky Cooperative Extension Service were used in this analysis as surrogates for cannabis flowers. All three sets of samples underwent the same procedure of extraction as stated above (Table 1).

TABLE 1 Spiked recovery analysis of illicit drugs in dried hemp flower ($n = 3$).

	Recovery	Standard deviation	Limit of detection (ppm)	Limit of quantification (ppm)
MDMA	77.5%	20.0%	0.0170	0.84
AMP	71.1%	17.1%	0.0567	2.67
cocaine	79.8%	12.5%	0.0034	0.19

Note: Hemp flower samples from the University of Kentucky Cooperative Extension Service were used in this analysis as surrogates for cannabis flowers. The limit of quantification was defined as the mean blank plus six standard deviations of the blank. It is presented as ppm of dried cannabis flower.
Abbreviations: AMP, amphetamine, as a surrogate of methamphetamine; MDMA, 3,4-methylenedioxymethamphetamine (Ecstasy).

3 | RESULT AND DISCUSSION

3.1 | Spike recovery

The results from the spike recovery trial demonstrated that the extraction and analysis procedure was effective for the detected illicit drugs. First, none of the blank samples had detectable concentrations of the analytes. Acceptable recoveries, defined as recoveries between 70% and 130%, were obtained for cocaine (79.8%), AMP (71.1%), and MDMA (77.5%). The relative standard deviations for cocaine, AMP, and MDMA were 12.5%, 17.1%, and 20.0%, respectively, in the triplicate spiked sample set. The current extraction method was designed to quantify a diverse set of analytes, including cannabinoids, pesticides, mycotoxins, and illicit drugs. As such, it is a generalist extraction method that is not optimized for any particular class of chemicals. Improved recovery of spikes could be obtained if the procedure was optimized for a specific group of analytes such as amine-containing illicit drugs. Figures S1A–F and S2A–D show the calibration curves for MAMP, cocaine, MDMA, and BZE and the chromatograms for the calibration standards using cocaine as an example, respectively.

3.2 | Detections of illicit drugs in seized cannabis samples

Twenty-five out of 118 cannabis flower samples (21.2%) had detectable concentrations of illicit drugs (Table 2 and Figure S3). Illicit drugs were detected in samples from different seizures. Only three of the 16 analytes tested were detected. The most commonly detected drug was cocaine (20.3%), followed by MAMP (3.4%) and MDMA (0.9%). Benzoylecgonine, the breakdown product of cocaine, was also found in four samples containing cocaine. Three MAMP and one MDMA samples were also found to contain cocaine. The highest concentration of illicit drugs was cocaine at 12,000 ppm (or 1.2% by weight). Only one positive seized sample (2.6%) was collected by the USPIS, while the remaining 24 positive samples were all collected by the MCSO at a detection rate of 27.8%. Notably, fentanyl was not detected in any of the

TABLE 2 Concentration of illicit drugs in seized dried cannabis flower samples ($n = 118$). Twenty-five samples (21.2%) had a detectable level of illicit drugs. Benzoylecgonine is a breakdown product of cocaine.

	Median (ppm)	Number of detections	Range of detections (ppm)
MAMP	20.77	4 (3.4%)	4.48–131
MDMA	4.75	1 (0.9%)	4.75
cocaine	133	24 (20.3%)	3.38–12,000
benzoylecgonine	2.65	4 (3.4%)	2.17–267

Abbreviations: MDMA, 3,4-methylenedioxymethamphetamine (Ecstasy); MAMP, methamphetamine.

118 samples. This is consistent with a 2021 study in Connecticut that found fentanyl in illicit cannabis as a standalone case of unintentional contamination [13, 14].

The results from this research demonstrate widespread contamination of black and gray-market cannabis products with other illicit drugs. Approximately, 20% of the samples were contaminated with cocaine, which often exists in powder form. The contamination by cocaine most likely arose from poorly cleaned preparation areas and equipment used to weigh and package the cannabis products. This was illustrated in Figure 1 where the unclean conditions in product preparation areas in clandestine cannabis growing and processing facilities could give rise to cross-contamination of products. MDMA is often in pill or powder form (M. Shay, personal communication). The latter form can contaminate the scale used in cannabis preparation, therefore resulting in the contamination of illicit cannabis.

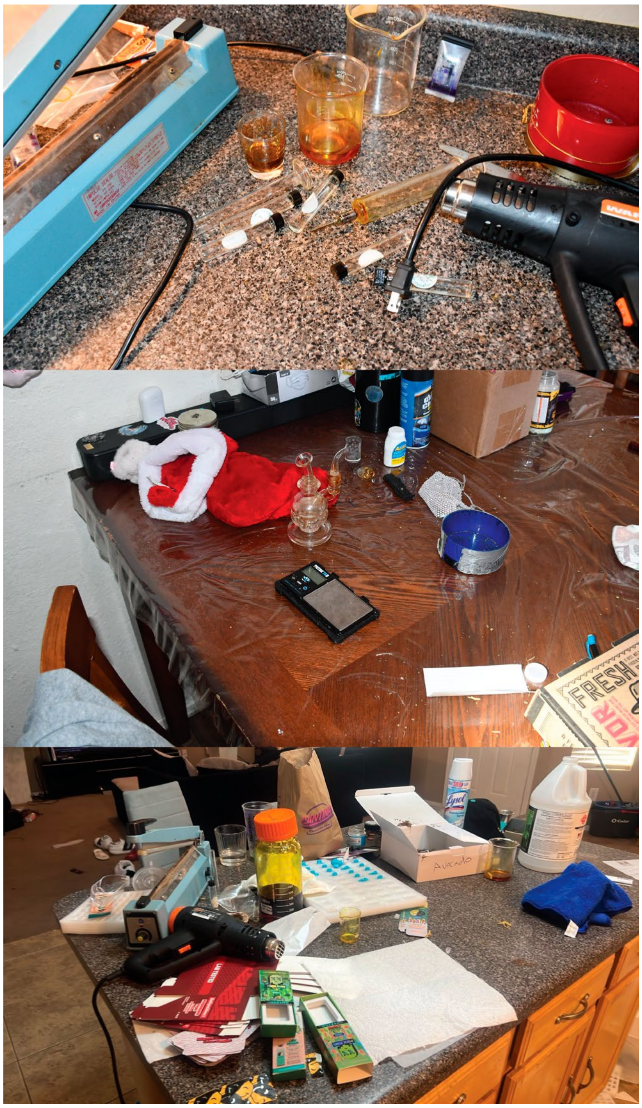

FIGURE 1 Clandestine cannabis processing areas demonstrate poor chemical hygiene and hence the likelihood of cross-contamination, particularly on the scale and benchtops (Photo credits to Maricopa County Sheriff Office).

Methamphetamine is the precursor of the synthesis of MDMA [15] and the two chemicals often appear together.

The median concentration of cocaine when it was detected was 133 ppm (Table 2). This corresponds to a dose of 0.065 mg for a typical 0.5 gram portion of cannabis used. If the average person is assumed to be 60 kg in weight, then this dose becomes 0.0011 mg/kg, which is over two orders of magnitude less than the lowest observed effect level for cocaine at 0.5 mg/kg [16]. This assumes that the illicit drug is not destroyed during consumption, which would likely occur if the cannabis was smoked. One sample had extremely high concentrations of cocaine that corresponded to 1.2% cocaine by weight. This corresponds to a dose of 6 mg for 0.5 gram cannabis, which is a fraction of a typical line of cocaine (approximately 100 mg; Shay 2025, personal communication). It is difficult to envision this amount being the result of cross-contamination, so this sample might have been deliberately spiked with cocaine. Additionally, the dose in this case would be 0.1 mg/kg for a 60 kg person consuming 0.5 gram cannabis, which is approaching the biological effect level for cocaine assuming the cocaine is not destroyed in the consumption process.

Assessing potential fentanyl contamination was one of the primary reasons for this research, but fentanyl was not detected in any of the samples. One possible explanation is that fentanyl is typically pressed into pills, which eliminate the need for the product to be weighed by local distributors. Moreover, the pressed pills would be less likely to shed mass if they were weighed compared with loose products like cocaine. Deliberate spiking of cannabis with fentanyl might occur, but a recent data analysis of the National Forensic Laboratory Information System showed that the co-occurrence of fentanyl and cannabinoids in substance samples is rare (~0.2%) [17].

4 | CONCLUSION

To the authors' knowledge, this is one of the largest studies of illicit drug contaminants in black- and gray-market cannabis in the United States to date. It demonstrates the presence of illicit drugs in black- and gray-market cannabis, which is largely due to the contamination in the unkempt environment where the unregulated cannabis is being assembled and distributed. The results of this study suggest that intentional spiking of illicit drugs on cannabis is rare. If cannabis is intentionally spiked with cocaine, it could pose a health risk to unsuspecting cannabis consumers. While previous studies have suggested fentanyl co-occurrence in other illicit drugs such as heroin, MAMP, and cocaine [17, 18], this study finds no evidence of widespread fentanyl contamination in illicit cannabis. Instead, it provides evidence suggesting the rise of polydrug preparation facilities in the illicit market.

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CONFLICT OF INTEREST STATEMENT

The authors declared no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the Supporting Information of this article.

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SUPPORTING INFORMATION

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

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