### Cannabis adulterated with the synthetic cannabinoid receptor agonist MDMB-4en-PINACA and the role of European drug checking services

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# Keywords

SCRAs, MDMB-4en-PINACA, cannabis, adulteration, drug checking.

# Highlights

* Low-THC (Δ9-tetrahydrocannabinol) cannabis adulterated with synthetic cannabinoid receptor agonists (SCRAs) emerged in early 2020
* The most commonly seen adulterant was the highly potent MDMB-4en-PINACA
* By April 2020, 9 drug checking services in 8 countries identified this adulteration
* Effective monitoring and warning require international cooperation

# Abstract

## Background

European drug checking services exchange information on drug trends within the Trans European Drug Information (TEDI) network, allowing monitoring and coordination of responses. Starting in Spring 2020, several services detected the synthetic cannabinoid receptor agonist MDMB-4en-PINACA in adulterated low-THC cannabis products.

## Methods

Cannabis products suspected of adulteration were analyzed for the presence of MDMB-4en-PINACA by 9 services in 8 countries within the TEDI network. If available, phytocannabinoid analysis was also performed.

## Results

1142 samples sold as cannabis in herbal, resin and e-liquid form were analyzed, of which 270 were found to contain MDMB-4en-PINACA. All cannabis samples contained low THC (<1%), except the e-liquids which contained no phytocannabinoids. Three serious health incidents requiring hospitalization after use of an adulterated cannabis sample were reported.

## Conclusion

Adulteration of cannabis with synthetic cannabinoid receptor agonists is a new phenomenon that carries risk for people who use it. Given that cannabis consumers are not a usual target group for drug checking services, services and associated harm reduction interventions could be reconfigured to include them.

# Introduction

Drug checking services (DCS) are a proven harm reduction strategy, providing chemical analysis of substances of concern submitted by the public combined with direct engagement with people who use drugs (PWUD) through ‘brief interventions’ or consultations (Koning et al., 2021; Measham & Turnbull, 2021; Valente et al., 2019). DCS also may use anonymously gathered data to monitor drug markets and issue alerts when potentially dangerous substances are detected.

Drug market trends and associated risks often transcend national borders. Therefore, in Europe, DCS exchange information and alerts through formal and informal communications. In addition to the European Union (EU) Early Warning System (EWS) of the European Monitoring Centre for Drugs and Drug Addiction (EMCDDA) which exchanges information regarding threats caused by New Psychoactive Substances (NPS) within the EU, they do so via the Trans European Drug Information (TEDI) network (Brunt et al., 2017). This is a network of European DCS that share their expertise and data. Thanks to a collaboration with the EMCDDA, TEDI created an electronic database in 2018 to collect data from TEDI members. This allows systematic identification and monitoring of general trends in European illicit drug markets. Furthermore, TEDI is able to act as a sentinel, providing data biannually and on request to evaluate a specific trend. TEDI represents 18 services from 12 different European countries (Austria, Belgium, France, Germany, Italy, Luxembourg, Portugal, Slovenia, Spain, Switzerland, the Netherlands and the United Kingdom).

Low-THC cannabis – also known as “CBD weed” – is hemp grown for industrial purposes. Its sale is usually not prohibited by drug laws, although most countries have an upper limit of THC that is allowed. In most countries in the EU, this upper limit is 0.2%. However, Switzerland has an upper limit of 1%. Low THC cannabis products are available as herb, resin, extracts, oils or other derivatives that similarly have very low THC levels (EMCDDA, 2020d). Recently, several European countries issued alerts after low-THC cannabis products were found to be adulterated with the synthetic cannabinoid receptor agonist (SCRA) methyl (*S*)-3,3-dimethyl-2-(1-(pent-4-en-1-yl)-1*H*-indazole-3-carboxamido)butanoate, or MDMB-4en-PINACA (EMCDDA, 2020a). This NPS functions as a full, potent agonist on the CB1-receptor. Its *in vitro* activity on the CB1-receptor is several orders of magnitude greater than that of the partial agonist Δ9-tetrahydrocannabinol (THC), the phytocannabinoid primarily responsible for the psychoactive effects of cannabis (De Morais et al., 2020). Comparable to many other SCRAs, these pharmacodynamic properties are accompanied by psychological and behavioural effects not dissimilar to those caused by cannabis – relaxation, euphoria, confusion, anxiety – albeit far more intense (De Morais et al., 2020). Moreover, MDMB-4en-PINACA appears to readily give rise to negative effects, *e.g.* vomiting, paranoia, depersonalization, panic attacks, hallucinations, agitation, seizures, chest pains and arrythmia (EMCDDA, 2020a). Multiple non-fatal incidents involving MDMB-4en-PINACA have been described, with clinical features such as loss of consciousness, seizures, aggressive behaviour and cardiovascular symptoms. Fatal incidents have also been reported, yet in all cases other psychoactive drugs were detected in the body alongside MDMB-4en-PINACA (De Morais et al., 2020; EMCDDA, 2020a).

Most SCRAs initially appear on the European market in powder form, after which they are processed into smoking mixtures or e-liquids. Likewise, MDMB-4en-PINACA was first identified in powders seized by the German police in 2017 (De Morais et al., 2020). However, in February 2020 a Swiss DCS based in Zürich first reported low-THC cannabis products adulterated with several SCRAs, including MDMB-4en-PINACA (Saferparty.ch, 2020). In April 2020, low-THC cannabis products were analyzed where MDMB-4en-PINACA was identified as the only SCRA present. The second half of 2020 saw an increase in the number of low-THC cannabis samples containing MDMB-4en-PINACA in Switzerland. Until autumn 2020, it was assumed that this was an exclusively Swiss phenomenon, as so-called “CBD weed”, which is herbal cannabis containing cannabidiol (CBD) and <1% THC, can be legally sold there. However, similarly adulterated cannabis soon appeared at services in the UK, France, the Netherlands and Austria in 2020, and Luxembourg, Germany and Italy in 2021. These samples were submitted by PWUD who were unaware of any adulteration of their cannabis, but experienced serious negative effects after use.

With cannabis being the most widely consumed illicit drug worldwide (UNODC, 2020), and with increased cannabis use reported in some countries during the first lockdown period of the COVID-19 pandemic (Manthey et al., 2021; van Laar et al., 2020), swift action was required. In this short report, we present the results of the chemical analysis of adulterated cannabis samples and subsequent actions as reported by TEDI network members. In conclusion, we use this case study to highlight the value of drug checking and international collaboration between services to monitor potentially dangerous trends when they are detected.

# Materials and Methods

Nine TEDI members from 8 different countries, of which four operate on a nationwide basis, analyzed suspicious cannabis samples for the presence of MDMB-4en-PINACA and other SCRAs. These samples were in herb or resin form and sold as cannabis, or in liquid form sold as THC or CBD e-liquids. A sample was suspicious if the experienced effects deviated from the expected effects, as reported by service users. Samples were usually submitted in person except in the case of the Welsh postal testing service. Various analytical techniques were used (see Table 1). All services performed qualitative analysis of SCRAs, while some also performed (semi-)quantitative analysis of the phytocannabinoids CBD and THC. Data included in this study was collected between April 2020 and April 2021. Samples in which SCRAs other than MDMB-4en-PINACA were detected are excluded from this study.

The results were shared within the TEDI network in an online repository. As the situation progressed and more samples adulterated with MDMB-4en-PINACA were identified, the situation was increasingly monitored. During TEDI meetings, strategies were shared regarding how best to respond to the phenomenon and to share information with the general public. EMCDDA was informed via national EWSs and data were provided to facilitate risk assessment. Data on the self-reported subjective effects of MDMB-4en-PINACA were gathered during brief interventions with DCS users. Furthermore, DCS employed their networks – including hospitals, emergency medical services and poisons centers – to gather information on MDMB-4en-PINACA-related health incidents.

# Results

A summary of the results is provided in Table 1. Between April 2020 and April 2021, a total of 1142 suspicious cannabis samples were analyzed. The samples were tested in Germany (n=439), Switzerland (Zürich (338), Wales (230), France (Paris (15), Lyon (29), the Netherlands (45), Austria (27), Luxembourg (18), and Italy (2). Of these, 270 (23.6%) contained MDMB-4en-PINACA. After the first identification of MDMB 4en-PINACA in Zürich in April 2020, the substance appeared in other DCS after several months (Cardiff, June 2020; Paris, October 2020). By March 2021, another six DCS had detected MDMB-4en-PINACA in adulterated cannabis products. Dates of first identification can be found in the Table. Despite regular testing of cannabis products, MDMB-4en-PINACA was not detected by Spanish DCS.

Most samples that were found to contain MDMB-4en-PINACA had been sold as herbal cannabis. Three services also found MDMB-4en-PINACA in resin samples and in the Netherlands, it was also identified in a submitted joint. A breakdown of product types can be found in the Table. The service in Wales detected it only in products sold as THC or CBD e-liquids. In some of the samples, other SCRAs such as ADB-BINACA, AB-PINACA, 4F-MDMB-BINACA and 5F-MDMB-PICA were also found. In Zürich, one sample was analyzed which contained four other SCRAs in addition to MDMB-4en-PINACA. In the Netherlands, MDMB-4en-PINACA was also identified in cannabis submitted to the DCS after being sold in coffeeshops (cannabis dispensaries). All DCS that quantified phytocannabinoids in the MDMB-4en-PINACA-positive samples found that they contained CBD (if quantified, samples were reported to contain ≥4% CBD, with some resin samples up to 18% CBD) and less than 1% THC. None of the e-liquid samples contained any detectable amounts of THC or CBD.

Users reported adverse effects including nausea, vomiting, tremors, paralysis, paresthesia, headaches, paranoia, anxiety, insomnia, hallucinations, aggressiveness, heart palpitations and loss of consciousness. In three reported cases in Germany, emergency hospital treatment was required. These incidents were characterized by excessive emesis, perspiration, panic attacks, tachycardia, amnesia and seizures. To date, no deaths involving MDMB-4en-PINACA-adulterated cannabis have been reported to medical services. Depending on local circumstances, different strategies have been used by DCS to alert the general public to this phenomenon, including via media and social media and directly with PWUD during brief interventions. In some countries, regional and national authorities (medical services, Reitox focal points, ministries of health, etc.) were also informed. In the Netherlands, a so-called restricted ‘Red Alert’ was issued targeted specifically at cannabis users and coffeeshops. Similar alerts were issued in Austria and Luxembourg.

# Discussion

Over the course of one year, nine services in eight countries detected MDMB-4en-PINACA in products sold as cannabis or THC/CBD e-liquids. All MDMB-4en-PINACA-positive non-liquid cannabis products also contained THC, albeit in much lower concentrations (<1%) than normally found in European cannabis products. For example, in 2016, average THC concentrations in European resin and herbal cannabis were 17.2% and 10.2% respectively (Freeman et al., 2018). Levels of CBD in adulterated samples were often normal or high: for example, a recent study found CBD levels to be 0-1% in herbal cannabis and 0-8% in resin (Freeman et al., 2020). This indicates that the source of the plant material may have been industrial hemp which contains low levels of THC. Thus, SCRAs may have been added to increase the strength or psychoactivity of the industrial hemp. The samples were submitted to the services by people who experienced effects that deviated from what was expected. This means that it is likely we are underreporting this phenomenon, as some people might not have been able to discern MDMB-4en-PINACA’s effects from those of cannabis or unable to access a DCS. Therefore, it is difficult to accurately portray the extent of the problem, as the TEDI network does not have the resources to analyze a large random sample of low-THC cannabis products.

Proper analysis of cannabis samples is performed in a “wet” lab: more easily accessible analysis methods such as IR spectroscopy have been shown to have serious limitations when it comes to samples other than powders or pills (Goncalves et al., 2021). However, when the main psychoactive phytocannabinoid, THC, is virtually absent in plant material that contains MDMB-4en-PINACA, it effectively turns the adulterated cannabis into an SCRA. Analysis of this and similar compounds requires powerful techniques (EMCDDA, 2021). Therefore, all services used at least gas chromatography coupled with mass spectrometry to detect MDMB-4en-PINACA, except two, who used liquid chromatography coupled with mass spectrometry (Brunt et al., 2017). Analysis was further complicated by the fact that MDMB-4en-PINACA is usually present in much lower concentrations than native phytocannabinoids. This is mostly due to the high potency of the SCRA compared to THC: a significantly smaller quantity of SCRA is needed to achieve a similar pharmacological effect (De Morais et al., 2020). Moreover, SCRAs are often applied unevenly to the smoking material they are distributed on, resulting in areas of very high or low SCRA concentration with consequently variable effects (EMCDDA, 2017). However, although several hundred MDMB-4en-PINACA-positive cannabis samples were identified by the TEDI network, very few major incidents involving the use of these products were reported.

The fact that cannabis is a popular drug with relatively low harm potential adds to the risk of PWUD becoming exposed to potentially dangerous amounts of MDMB-4en-PINACA. Prior to this case study, adulteration of cannabis was not frequently observed, especially in countries with a relatively tolerant cannabis legislative framework. Furthermore, the two drug groups usually submitted to DCS are “party drugs” (*e.g.* stimulants, empathogens and dissociatives) and “problem drugs” associated with injecting and dependence (*e.g.* opioids), but rarely cannabis. Thus, the lack of familiarity of cannabis consumers with DCS might be a barrier preventing them from coming forward and having samples analyzed. Moreover, the current COVID-19 pandemic and associated measures (*e.g.* lockdown) have caused DCS to become harder to access (EMCDDA, 2020b) and also appear to have caused changes in drug markets and use, with shifts away from party drugs, towards depressant and dissociative drugs consumed at home, including cannabis and alcohol (EMCDDA, 2020c). In the Netherlands, this is reflected in a reported increase in cannabis use during the first lockdown of the pandemic (van Laar et al., 2020). Although this cannot be excluded based on our data, the emergence of adulterated cannabis products is itself likely not directly related to lockdown measures, as the first adulterated samples were identified before any such measures were in place.

Following the identification of SCRA-adulterated cannabis products in Europe, the TEDI network has met regularly to discuss the developments. Information has also been received from national focal points notifying the EMCDDA’s Reitox system. This exchange facilitated the identification of similarities in adulterated samples, including the presence of MDMB-4en-PINACA, CBD and low amounts of THC, resulting in systematic monitoring of this trend throughout Europe. It allowed DCS to act appropriately in response to signs of adulterated cannabis, which, as mentioned above, is not something DCS are normally focused on. The value of market monitoring through drug checking has been established before: it allows assessment of the gap between expected and actual substance composition, while also functioning as an effective harm reduction strategy (Brunt et al., 2017; Measham and Turnbull, 2021). However, as trends in illicit drug markets and adulteration do not conform to national borders, DCS are also well placed to initiate joint communication and risk assessment strategies between countries and cities. Alongside a review of international collaboration between DCS, a critical assessment of the barriers that currently prevent cannabis users from having their drugs analyzed is also required. The importance of this is further underlined by the huge array of possible chemical variations on the SCRA theme, allowing covert manufacturers to switch production once individual SCRAs leave the stage – either for legal reasons, or because of manufacturing issues (Banister & Connor, 2018). Outside of this study, cannabis containing the SCRA ADB-BUTINACA was recently reported in Germany, Italy, Austria and the Netherlands, indicating that adulterated cannabis might be proliferating. The toxicity profile of most novel SCRAs is unknown, making the emergence of adulterated cannabis in Europe especially dangerous. This could be exacerbated by the recent Chinese ban of all SCRAs (Reuters, 2021), which could motivate clandestine chemists to venture even further from the more well-known compounds.

In conclusion, the emergence of adulterated cannabis widens the remit of DCS and associated harm reduction messaging from the two usual target groups (people who use ‘party’ or ‘problem’ drugs) to cannabis consumers too. However, analysis of SCRAs and THC is technically challenging, and cannabis users might not be as familiar with DCS as other PWUD groups. This indicates a need for a coordinated and adequately funded approach for cannabis drug checking and harm reduction services.

# Acknowledgements

The TEDI-network receives financial support from the EMCDDA under the grant “DATA COLLECTION OF RESULTS FROM ANALYSES ON DRUG SAMPLES FROM DRUG CHECKING SERVICES IN EUROPE”. Contract code: CT.19.HEA.0090.1.0.

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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Organisation | Country | City | Samples collected (nr) | MDMB-4en-PINACA positive (nr) | First detection (date) | Analytical technique used | Forms containing MDMB-4en-PINACA | Phytocannabinoids (THC, CBD) | Reaction of the organisation |
| **THC** | **CBD** |
| 4motion asbl. / PIPAPO | LU | nationwide | 18 | 8 | 11-1-2021 | GC-MS + HPLC | Herbal | yes (<0.3%, most samples no THC)  | yes | Consumer Alert through social media, communication to the Ministry of Health, the National Drug Coordinator, the National Focal Point, Suchtverband Lëtzebuert, TEDI network and EMCDDA |
| Neutravel | IT | Turin | 2 | 1 | 25-03-2021 | GC-MS + UPLC-QTOF | Herbal | yes (<0.5%) | yes | Alerts to social media and communication to the National Early Warning System |
| LHI | DE | nationwide | 438 | 96 | 7-2-2021 | GC-MS | Herbal (79) and resin (17) | yes | yes | Warnings in social media, publish interactive map, warning over vocal point |
| DIMS | NL | nationwide | 45 | 18 | 5-11-2020 | GC-MS | Herbal (15),resin (2), joint (1) | yes (all under 1.0%, average 0.6%) | yes | Initially only participants of the DIMS network were informed. On December 18 2020, a selective warning campaign via the Red Alert procedure was started (including push message Red Alert app). Notification of this was sent to the EWS EMCDDA and TEDI network. In January 2021 all coffeeshops in the Netherlands received a letter. |
| Analyse ton prod IDF | FR | Paris | 15 | 11 | 29-10-2020 | TLC, GC-MS, UPLC-QTOF | Herbal | yes | yes | Alert sent to regional health organisation |
| Pause diabolo | FR | Lyon | 29 | 19 | 22-12-2020 | TLC, GC-MS, UPLC-QTOF | Herbal | yes | yes |  |
| Checkit! | AT | Vienna | 27 | 3 | 16-11-2020 | UPLC-MS³, UPLC-DAD, MALDI-HRMS, FTIR | Herbal | yes | yes | Consumer alert on social media and our website, communication to the national focal point and EMCDDA Early Warning System.  |
| WEDINOS | WLS | nationwide | 230  | 50 | 18-06-2020 | UPLC-QTOF | Liquid (Vape /e-liquids) | no | no | All results are published on the public facing website www.wedinos.org |
| Saferparty/DIZ | CH | Zürich | 338 | 64 | 28-04-2020 | HPLC, GC-MS | Herbal (60) and resin (4) | yes (all under 1.0%, average 0.6%) | yes | Consultations with clients on the topic in our office, creation of warnings and publication on our homepage saferparty.ch as well as on social media channels (Instagram and Facebook). Exchange with other DCSs in Switzerland. Info events in schools, social institutions etc. and evaluation reports. |

Table 1: Data gathered by various DCSs within the TEDI network following the analysis of potentially adulterated cannabis or e-liquid samples during the period April 2020 – April 2021. GC-MS: gas chromatography-mass spectrometry; HPLC: high-performance liquid chromatography; TLC: thin-layer chromatography; UPLC-QTOF: ultra-performance liquid chromatography-quadruple time-of-flight; MS3: mass spectrometry cubed; DAD: diode-array detector; MALDI-HRMS: matrix-assisted laser desorption/​ionization high-resolution mass spectrometry; FTIR: Fourier-transform infrared spectroscopy.