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# The use of pesticides in Belgian illicit indoor cannabis plantations



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# ABSTRACT

Cannabis (Cannabis spp.) use and cultivation continue to increase in many (European) countries. The illicit indoor cannabis plantations that supply Belgian and European cannabis markets create problems and concerns about health and safety of intervention staff, dismantling companies, the direct environment of cannabis plantations and, eventually, of cannabis users. Main risks may come from pesticide residues on plants, cultivation infrastructure and materials; left-over plant growth-promoting substances; mycotoxins from fungal pathogens on harvested plants; and/or high levels of cannabinoids in cannabis plant parts for consumption. In the present research, we report on pesticides found in illicit indoor cannabis plantations in Belgium. EN15662 QuEChERS extraction method and LC-MS/MS analysis were used to identify pesticides in indoor cannabis plantations and thus to evaluate the hazards associated with the use, cultivation and removal of cannabis plants in plantations as well as with dismantling activities in the cultivation rooms. We found pesticides in 64.3% of 72 cannabis plant samples and in 65.2% of 46 carbon filter cloth samples. Overall, 19 pesticides belonging to different chemical classes were identified. We found o-phenylphenol, bifenazate, cypermethrin, imidacloprid, propamocarb, propiconazole and tebuconazole, which is consistent with the commonly reported pesticides from literature. In only a few cases, pesticides found in bottles with a commercial label, were also identified in plant or stagnant water samples collected from the growth rooms where the bottles had been collected. We further revealed that, even though most pesticides have a low volatility, they could be detected from the carbon filters hanging at the ceiling of cultivation rooms. As a result, it is likely that pesticides also prevail in the plantation atmosphere during and after cultivation. The risk of inhaling the latter pesticides increases when plants sprayed with pesticides are intensively manipulated during dismantling activities. We conclude that pesticides represent an underestimated and under-documented health risk for intervention staff. The standard procedure for dismantling illicit indoor cannabis cultivation sites should be improved by including guidelines for appropriate personal protection equipment and dismantling protocols that take into account all possible hazards.

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## 1. Introduction

Although total size of illicit Belgian indoor cannabis plantations is unknown, official seizure data indicate that cannabis production in Belgium is on the rise. In 2007, police confiscated 466 indoor cannabis plantations in Belgium. By 2010 this number had risen to 979 and by 2015 to 1241 plantations. In 2015, 979 (79%) of the

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confiscated plantations had more than 5 plants and 529 (43%) had more than 50 plants (unpublished data from the Belgian Federal police). Plantations with more than 5 plants are most likely planted for commercial reasons. Spider mites (Fam. Tetranychidae), thrips (Order Thysanoptera), white flies (Fam. Aleyrodidae), aphids (Superfam. Aphidoidea), and fungi such as *Fusarium oxysporum* and rust (Order Pucciniales, several genera and species) can cause a lot of damage to indoor cannabis plants [1–3]. Pesticide applications can prevent or kill most pests and diseases and will increase the likelihood of a successful harvest for the commercial indoor cannabis grower. However, a literature research on the chemical contamination of cannabis did not reveal widespread pesticide use in illicit indoor cannabis plantations [4]. In the USA,

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pesticides (the acaricide dicofol; insecticides chlordane, malathion, chlorpyrifos, fenvalerate, cypermethrin, tetramethrin and permethrin; and the fungicide chlorothalonil) were found in only 5 (12%) out of 40 indoor cannabis plantations studied [5]. Schneider et al. found 7 different pesticides on a total of 50 seized cannabis plants [6]. They comprise the fungicides promocarb, tebuconazole, propiconazole and tolvlfluanid, the neonicotinoid insecticides imidacloprid, bifenthrin and hexythiazox. In the late 1970s and early 1980s, paraquat residues were found on confiscated cannabis samples [7-13]. Recently paraguat, together with other herbicides such as glyphosate and aminomethylphosphonic acid were detected on illicit cannabis samples from unknown origin in Brazil [14]. In the US, Sullivan et al. reported pesticide residues in three cannabis smoking devices (water pipe with filter, water pipe without filter and glass pipe) [15]. Residue recovery was as high as 69.5% of samples depending on the

significant toxicological threat. Pesticide prevalence in cannabis plantations and the risks these products pose to cannabis growers and intervention staff have hardly been investigated. In indoor cannabis plantations, the latter persons can be exposed to pesticides by dermal contact with plants while moving through the plantation and during plant removal, as well as by inhalation of pesticide vapours from the cultivation room atmosphere. Identification of pesticides used in indoor cannabis cultivation as well as data on the frequency and location of their prevalence, is crucial to adequately assess their risks to intervention staff. Martyny et al. and Van Dyke found that most insecticides encountered in indoor cannabis plantations are pyrethroids, which have a low toxicity when inhaled or in case of dermal contact [16,17].

analytical device used, suggesting that the danger of pesticide

residues harming cannabis users is substantial and may pose a

Pesticide health hazards are determined by product type and dose, exposure duration and absorption route. Speed of dermal absorption depends on the exposed body part with the slowest absorption rates reported from the lower arm, whereas fastest absorption occurs in the genital area. Oral exposure can lead to severe illness, organ damage and even death. Inhalation is the most dangerous absorption route because pesticides are quickly absorbed by blood vessels through the pulmonary alveoli [18].

There is currently no reliable information on the extent of pesticide use in illicit Belgian indoor cannabis plantations. As a result, cannabis growers, users and intervention staff might be exposed to a great but currently unknown risk. In order to shed more light on the prevalence of pesticides in Belgian indoor cannabis plantations, we investigated the presence of (commercially available) pesticide products as well as of pesticide traces in water tanks, on cannabis plants and on carbon filters sampled from indoor cannabis plantations in Belgium.

## 2. Materials and methods

#### 2.1. Plantation surveys and sampling

Local as well as federal police departments were informed about our study with the express demand to facilitate our research activities in seized plantations. When a plantation had been confiscated by local and federal police staff during the study period (17 July until 3 December 2014), its seizure was immediately signalled to the Central Desk 'Drugs' of the Belgian Federal Police, who then informed researchers when visits were qualified by police as safe and feasible. Descriptive primary data was thus collected from 43 illicit indoor cannabis plantations, spread over 35 Belgian municipalities belonging to 8 out of the 10 Belgian Provinces. Most (35) surveyed plantations were situated in the northern, Flemish part of the country. In 4 plantations, no plants were found. From 38 out of the 43 plantations toxicological product samples were taken. Twenty-six sampling runs were done by academic researchers from Ghent University and the Catholic University of Leuven. The other 17 surveys were performed by police officers in cases where for judicial reasons, plantations had to be dismantled quickly, so that researchers could not visit the plantations in time. During sampling, investigators wore a white Tyvek<sup>®</sup> Expert overall, a 7000 Easylock halfmask with a Moldex<sup>®</sup> P3 R 9030 dust filter and a ABEK1 Easylock<sup>®</sup> 9400 chemical filter, Hazmax<sup>TM</sup> SSSRA HRO CI FO E safety boots and Virtex <sup>TM</sup> 79-700 safety gloves as personal protective equipment (PPE).

To assure accurate and uniform data collection, both researchers and police officers used the same characterization data and sample collection protocol. Primary data concerned (i) a detailed description of the cultivation room's infrastructure; (ii) names of pesticides, growth stimulants and other products, as stated on packages; (iii) description (colour, volume, pH) of samples taken; and (iv) a sketch of the cultivation room compartments. When different cultivation rooms on a same location applied different cultivation techniques (such as substrate, lighting system or plant density), they were considered as different plantations. For each plantation, a photo log was made with pictures of cultivation rooms, equipment and product labels. Data were processed in MS-Excel 2010 and SPSS 22.0.

For all **liquid substances** found in closed containers, a 3 mL sample was collected in a 5 mL Sarstedt CryoPure<sup>®</sup> tube. In cases where puddle water was observed, a 35 mL sample was collected in a 70 mL Sarstedt PP beaker. Liquid samples were immediately transported to and stored at 4 °C at the Catholic University of Leuven, until toxicological analysis.

Per plantation, 3 complete **plants**, cut just above the growth substrate, were collected in a paper bag. When plants were found to be in different development stages, 3 plants from each development stage were collected. Plants were immediately transported to, and stored at -20 °C at the Catholic University of Leuven, prior to toxicological analysis.

Cannabis growth room atmospheres are continuously refreshed by turbines that evacuate air through **carbon filters.** These neutralize the intense cannabis smell [19,20] and can consequently be considered as an archive of all volatile substances that were ever present in the rooms. The latter substances are adsorbed on the active carbon inside the filter, and on the fibres of the filter cloth that is wrapped around the filters. Pesticide residues that can be identified on carbon filter cloth are consequently most likely the same pesticides that have been used to control pests or diseases on cannabis plants cultivated in the same plantations. Filter cloth fibre samples were collected in airtight glass tubes and brought to the Catholic University of Leuven for toxicological analysis.

## 2.2. Extraction and analysis

#### 2.2.1. Standards

Pestanal<sup>®</sup> pesticide standards (42) and internal standard triphenylphosphate (TPP) were purchased from Sigma-Aldrich, Belgium. A stock solution of 10 mg/mL was prepared. Working solutions of 100 and 500  $\mu$ g/mL were prepared in methanol, ethanol, acetonitrile or dichloromethane, depending on the solubility of the standard. TPP was dissolved in a 10 mg/mL stock solution in ethanol and 10  $\mu$ L of working solution (10.62  $\mu$ g/mL) was used.

#### 2.2.2. Extractions

For every cultivation room of every plantation, the development stage and weight  $(\pm 1 \text{ g})$  of the plant samples were determined (Table 2). 200–300 mg of carbon filter cloth was taken

for extraction (Table 3). Liquid substances (water and pesticides) were diluted in mobile phase and centrifuged before analysis. TPP standard was added to check if the extraction was carried out well.

Extraction was carried out following the EN15662 QuEChERS method [21]. For plant material, dSPE cleanup kit for high pigmented matrices was used because of the high chlorophyll content. QuEChERS extraction Pouches (5982-5650) were purchased from Agilent (Belgium).

# 2.2.3. Analysis

Qualitative analysis on pesticides was carried out using LC–MS/ MS system equipped with ESI probe (Shimadzu UPLC–AB Sciex 3200 QTRAP). A 5  $\mu$ m Restek Allure Propyl column, 50 × 2.1 mm with guard column was used. Mobile phase A (water/2 mM ammoniumformate/0.2% formic acid) started at 10% during 11 min. Mobile phase B (acetonitrile/2 mM ammoniumformate/0.2% formic acid) was decreased to 10% at 11.5 min and kept at 10% for the total run time of 13.5 min. Flow was set at 0.5 mL/min. Oven temperature was 40 °C. Injection volume was set at 30  $\mu$ L. Multiple Reaction Monitoring was carried out to identify all pesticides.

Detailed MS/MS analysis parameters are given in Supplemental information 1, including MRM transitions (with Q1 is the [M +H] + precursor and Q3 is selected fragment ion), retention time of injected standards, ionization mode and the voltage settings Declusterings Potential (DP), Entrance Potential (EP), Collision Cell entrance Potential (CEP), Collision Energy (CE), Collision cell eXit Potential (CXP).

# 3. Results

# 3.1. Liquid substances

At the crime scenes, diverse **labeled** chemical products were found ranging from pH-regulators, plant growth promotors and pesticides. In the present paper, we report only on pesticides. In total, 23 different pesticides were found in liquid formulations; 15 of them were insecticides (Table 1). All these products can be legally purchased either online or from regular (garden) shops, except for Destroyer 480 ec, approved by the European Commission (Reg. (EU) No 540/2011 and Reg. (EU) No 762/2013), but no longer distributed in Belgium, and E605 (parathion, not approved by the European Commission).

From 21 plantations, a total of 41 **liquid samples**, of which 24 samples from water tanks connected to a plant irrigation system, 4 samples from stagnant water in cultivation trays and 13 samples from unlabeled bottles) were analyzed on the presence of pesticides. Pesticides were found in only 7 (17%) liquid samples, originating from 5 different plantations. However, labeled pesticide containers were found in 10 out of 43 plantations (23%). It can therefore be assumed that most pesticides were not applied by mixing them with irrigation water but were sprayed directly on plants. In the liquid samples, 10 different pesticides were identified: bendiocarb, imazalil, tebuconazol, thiacloprid, spiromesifen, demeton-*S*-methyl, metalaxyl, tebufenpyrad, pyrethrin I, and propiconazol.

## 3.2. Plant and carbon filter samples

Qualitative analysis of 118 samples (72 plant and 46 carbon filter cloth samples) revealed 19 different pesticides. On the plant material, 46 out of 72 (64%) samples tested positive on one or more pesticides (Table 3). On 25 (35%) of samples, more than one pesticide was detected (Table 4).

In most cultivation sites, plants were distributed over different cultivation rooms, each with plants in a different plant development stage. However, pesticides found in plant samples from one cultivation room were in most cases also observed in plant samples obtained from other cultivation rooms at the same plantation, indicating that pesticides are most probably used throughout the whole cultivation process, irrespective of development stage.

Pesticides were found in thirty (65%) out of the 46 carbon filter cloth samples. In fifteen samples (33%), more than one pesticide was identified (Table 3).

Twenty-one (55%) out of the 38 plantations where toxicological samples were taken, contained multiple growth rooms, each with cannabis plants in different development stages. In 15 (72%) of these 21 plantations, pesticides were found on plants in each growth room, indicating that plants had probably been sprayed at several development stages during one cultivation cycle. Although pesticide concentrations were not determined, it can therefore be

Table 1

Labeled pesticides found on the crime scenes together with the active compounds mentioned on the labels.

Product	Type pesticide	Active compound	
Floramite 240sc	Insecticide/acaricide	Bifenazate 240 g/L	
Soil Attack Liquid	Bio-insecticide	Terpenoids	
Destroyer 480 ec	Insecticide	Chlorpyrifos 480 g/L	
Crawling Insect Spray	Insecticide	Cypermethrin 0.1%, imiprothrin 0.1%	
K-Othrine	Insecticide	Deltamethrin 0.05%	
SBPI	Bio-insecticide/fungicide	Fe EDTA	
Weedol Ultra	Herbicide	Glyphosate 7.2 g/L, pyraflufen-ethyl 0.02 g/L	
Provado Ultra	Insecticide	Imidacloprid 10 g/L	
Entonem	Bio-insecticide	Steinernema feltia larvae, 3rd phase	
Plant Vitality plus	Bio-insecticide/acaricide	Lactons	
ER II	Bio-insecticide	Maltodextrin	
Dislike	Bio-insecticide/acaricide	Organic oil	
Insectspray	Bio-insecticide	Organic fatty acids	
Sun Spray Garden	Bio-insecticide/acaricide	Paraffin oil	
E605	Insecticide	Parathion	
Permas D	Insecticide	Permethrin 0.1%	
Plant Protection spray	Bio-insecticide	Unknown plant extracts and oils	
Promanal	Bio-insecticide	Pyrethrin	
Bio-insecticide	Bio-insecticide	Pyrethrin 20 g/L, piperonyl butoxide 255 g/	
Rosacur	Insecticide	Tebuconazole 45.9 g/L	
Masai	Acaricide	Tebufenpyrad 25%	
Calypso	Insecticide	Thiacloprid 0.92%	
Exact	Fungicide	Triadimenol 50 g/L	

# Table 2

Confiscated-plant parameters and pesticides dedected on plants.

Cultivation site	Cultivation room	Plant development stage ( <sup>a</sup> )	Extraction weight (g)	Pesticides
1	1	6	1.0705	O-Phenylphenol, bifenazate, propiconazole
	2	5	1.0123	O-Phenylphenol, bifenazate, propiconazole
	3	5	1.0050	O-Phenylphenol, bifenazate, propamocarb, propiconaz
	4	5	1.0239	O-Phenylphenol, bifenazate
	5	3	1.0213	O-Phenylphenol, bifenazate
2	1	5	1.0191	Myclobutanil
	2	1	1.0186	Eichlorvos, <sup>b</sup> imidacloprid, myclobutanil
3	1	1	1.0319	Eichlorvos <sup>b</sup>
4	1	3	0.9991	Propiconazole
	2	3	1.0086	Abamectin (B1a), propiconazole
	3	3	1.0042	Abamectin (B1a)
	4	1	1.0004	X
5	1	5	1.0223	Chlormequat chloride
,	2	5	1.0322	Chlormequat chloride
6	1	1	0.8956	O-Phenylphenol, bifenazate, etoxazole
J	2	5		
7			1.0005	O-Phenylphenol, bifenazate, propamocarb, propiconaz
7	1	1	1.0143	X
3	1	6	1.0496	x
Ð	1	1	1.0304	Propamocarb
10	1	5	1.0774	Abamectin B1a, Dioxathion NH4 <sup>+</sup> , <sup>b</sup> propiconazole
	2	5	1.0047	Tebufenpyrad, propiconazole
	3	5	1.0086	Abamectin B1a, propiconazole
11	1	4	1.0186	x
12	1	4	1.0054	х
13	1	1	1.0474	Triadimenol
4	1	3	1.0184	Chlormequat chloride
1	2	3	1.0084	Chlormequat chloride, propiconazole
	3	2	1.0146	Chlormequat chloride
-				
5	1	2	1.0275	X
	2	2	1.0030	х
_	3	2	1.0222	X
6	1	5	1.0500	O-Phenylphenol, bifenazate, chlormequat chloride
7	1	1	0.7996	Х
8	1	2	1.0163	Tebufenpyrad
	2	2	1.0536	Tebufenpyrad
19	1	5	1.0350	X
20	1	1	0.6882	Abamectin B1a
21	1	4	1.0064	Bifenazate, propamocarb
	2	4	1.0801	Tebufenpyrad
22	1	1	0.4913	Dicrotophos, <sup>b</sup> imidacloprid, propamocarb, tebufenpyra
	2	1	0.4950	Propamocarb
23	1	3		Propamocarb, tebufenpyrad
25	2		1.0080	
		3	0.9993	Propamocarb, tebufenpyrad
.4	1	6	1.0287	х
.5	1	3	1.0131	Х
	2	5	0.9983	Tebuconazole
26	1	1	х	No plant samples
	2	1	х	No plant samples
7	1	6	1.0180	Bifenazate, imidacloprid, propamocarb
8	1	3	0.9993	Chlorpyrifos, cypermethrin, imidacloprid, propamocarl
	2	3	1.0183	Chlorpyrifos, cypermethrin, propamocarb
9	1	3	0.9993	Abamectin B1a
-	2	3	1.0195	Abamectin B1a, propamocarb
0				Abamectin (B1a + B1b), imidacloprid, propiconazole
0	1	1	1.0065	
	2	4	1.0015	Propiconazole
	3	1	0.4345	Abamectin (B1a), imidacloprid, propiconazole
1	1	3	1.0035	х
	2	3	1.0018	х
	3	3	1.0133	х
2	1	5	1.0127	х
	2	5	1.0075	х
	3	5	1.0610	х
	4	6	1.0467	x
	1	5	1.0859	x
13	2	5	1.0185	
3				x
3			1.0755	Х
	3	5		
	3 1	1	1.0098	х
	3		1.0098 1.0146	x x
	3 1	1		
	3 1 2	1 1	1.0146	x
33	3 1 2 3	1 1 1	1.0146 1.0071	x x

X = No pesticide detected. <sup>a</sup> 1. No flowers; 2. Start of flowering, very small flowers; 3. Start of green flower; 4. Flowers with first resin production 5. Nearly ripe flower buds; 6. Ripe buds with brown pistils. <sup>b</sup> Not approved in the EU.

Table 3
Extraction weight and pesticides identified on carbon filter cloth samples.

Cultivation site	Cultivation room	Extraction weight (g)	Pesticides
7	29	247.4	Propamocarb
	30	246.6	Propamocarb
10	1	267.7	Propamocarb, tebufenpyrad
	2	249.9	Propamocarb, tebufenpyrad
	3	261.1	Propamocarb, tebufenpyrad
11	1	258.7	Х
	2	247.6	Х
	3	239.3	Х
13	1	295.8	Chlorfenvinphos, <sup>a</sup> propamocarb, propoxur, <sup>a</sup> tebuconazole, triadimenol
	2	251.4	Chlorfenvinphos, <sup>a</sup> propamocarb, propoxur, <sup>a</sup> tebuconazole
	3	300.3	Triadimenol
	4	253.1	Chlorfenvinphos, <sup>a</sup> triadimenol
17	1	262.2	X
	2	248.5	Х
	3	273.0	β-Cyfluthrin
	4	268.7	β-Cyfluthrin
	5	262.2	β-Cyfluthrin
	6	263.5	X
18	1	263.7	Tebufenpyrad
20	1	251.4	Bifenazate, propiconazole
21	1	274.8	Propamocarb, tebufenpyrad
	2	160.1	Propamocarb, tebufenpyrad
22	-	257.5	X
24	1	263.2	X
25	1	257.4	X
20	2	259.3	X
26	1	212.4	Propamocarb
20	2	269.2	Propamocarb
28	1	261.2	Chlorpyrifos, imidacloprid, propamocarb
20	2	238.6	Chlorpyrifos, imidacloprid, propamocarb
29	1	263.3	Propamocarb
25	2	248.0	Propamocarb
30	1	250.0	Bifenazate, propiconazole
50	2	255.6	Propiconazole
31	1	228.6	Imidacloprid
51	2	233.6	X
	3	253.0	x
	2	240.8	X
22			
32	1 2	260.1	β-Cyfluthrin
2.4	2	279.9	X
34		253.4	X
25	Cellar 10	278.6	X Chloreno guat chlorida, tabuar e ala
35	10	255.8	Chlormequat chloride, tebuconazole
36	1	271.6	Chlorfenvinphos, <sup>a</sup> propamocarb, tebufenpyrad
37	18	231.3	Chlormequat chloride, propamocarb
38	1	238.4	Propamocarb

<sup>a</sup> Not approved in the EU.

expected that pesticide concentrations of full-grown plants are higher than those of young plants in earlier development stages, because the former plants probably have received multiple pesticide sprayings.

# 4. Discussion

The most frequently found pesticides in the analyzed samples have a relatively low toxicity. They are particularly harmful when ingested or inhaled (irritation). Dermal contact with pesticides found in our study can cause skin irritations and/or allergic reactions. However, some samples contained pesticides with a high toxicity. Tebuconazole and myclobutanil may be harmful to the unborn child [22]. Allowable tolerances for tebuconazole on food are ranging from 0.05 to 5 ppm. The lowest-observed-adverse-effect-level (LOAEL) is 8.8 mg/kg/day. For myclobutanil, food tolerances are ranging from 0.02 to 5 ppm, with a LOAEL of 10 mg/kg/day [23]. Abamectin (food tolerance: 0.005–1 ppm; LOAEL: 0.5 mg/kg/day), beta-cyfluthrin (food tolerance: 0.05–30 ppm), chlorfenvinphos (LOAEL: 0.0005 mg/kg/day), chlorpyrifos (food tolerance: 0.01–12 ppm; LOAEL: 10 mg/kg/day),

dichlorvos (food tolerance: 0.01-8 ppm), dioxathion (food tolerance: 0.1–5 ppm), parathion (food tolerance: 0.1–5 ppm; LOAEL: 0.09 mg/kg/day) and propoxur (no food tolerance limits found) can be lethal when ingested at a high dose [3,24-28]. Organophosphates (food tolerance dichlorvos: 0.02-2 ppm) and carbamates (food tolerance aldicarb: 0.02-1 ppm carry the highest risks, because they influence the nerve system through inhibition of acetylcholinesterase. Symptoms include dizziness, nausea, headache, abdominal cramps, diarrhea, trembling of (surface) muscles. Organophosphate pesticides intoxication can induce cholinergic syndrome with clinical features including salivations, urinary and fecal incontinence, GI cramping, sweating and pulmonary edema. Pyrethroids (food tolerance permethrin: 0.05-30 ppm) also affect the nerve system, but have a low oral and dermal toxicity. Dermal contact with pyrethroids often leads to skin irritation or allergy. For intervention staff, the highest risk is exposure through dermal contact with pesticides on cannabis plants and inhalation of pesticide residues that were still present in the growth room atmosphere at the time of intervention. Cannabis users face the highest risk from pesticides through inhalation of contaminated cannabis smoke, oral exposure to pesticides when cannabis is

# Table 4

Number of plant and filter cloth samples on which pesticides were identified.

Pesticide	#Positive plant samples	#Positive filter cloth samples	Total positive samples
o-Phenylphenol	8	0	8
Abamectin B1a	9	0	9
Abamectin B1b	1	0	1
β-Cyfluthrin	0	4	4
Bifenazate	12	2	14
Chlorfenvinphos <sup>a</sup>	0	4	4
Chlormetquat chloride	6	2	8
Chlorpyrifos	2	2	4
Cypermethrin	2	0	2
Dichlorvos <sup>a</sup>	2	0	2
Dioxathion <sup>a</sup>	1	0	1
Etoxazole	1	0	1
Imidacloprid	6	3	9
Myclobutanil	3	0	3
Propamocarb	12	17	29
Propiconazole	18	3	21
Propoxur <sup>a</sup>	0	2	2
Tebuconazole	1	2	3
Tebufenpyrad	10	7	17
Triadimenol	1	4	5

Full detail (number of respective cultivation rooms, plant development stage and detected pesticides) of all plantations and the pesticide analysis results can be found in Supplemental information 2.

<sup>a</sup> Not approved in the EU.

processed in food preparations, and dermal exposure to pesticides when cannabis plants are manipulated during cultivation or drug use.

Pesticides identified by the labels on bottles at the crime scenes corresponded in only a few cases to the pesticides found in samples. Since 15 (35%) of the 43 studied plantations were situated in a residential building house in which a few rooms were refurbished for cannabis cultivation, it is not sure that all products found are actually used in cannabis cultivation. Nevertheless, it can be assumed that pesticides identified in plants or on carbon filters (in the cultivation rooms) had indeed been used in cannabis cultivation. It can also be assumed that these pesticides have been sprayed directly on the plants because pesticides were detected in the irrigation tanks of only 5 plantations. Although we did not determine pesticide concentrations as such, the present study shows that pesticides are extensively used in illicit indoor cannabis plantations. Earlier research mainly focused on paraguat residues in confiscated cannabis samples [7-13]. Research on pesticide prevalence and use in liquids and on material other than plants in indoor cannabis plantations is scarce.

Our results not only raise health concerns for cannabis users, but also for intervention staff who is exposed for several hours to pesticides when dismantling indoor cannabis plantations. We not only found pesticide contamination of cannabis plants but also of the plantation atmosphere, as evidenced by carbon filter cloth analysis. As a result, during dismantling, air quality should be carefully monitored. Although most pesticides are not very volatile, we could detect them on the carbon filters found in the growth rooms, indicating that during or after cannabis cultivation, pesticide residues remain in the plantation atmosphere. The risk of inhaling these pesticides increases when plants sprayed with pesticides are manipulated during dismantling since small contaminated plant particles will be released in the air during manipulation. For example, lowest-observed-adverse-effect levels of chlorfenvinphos, which was found in carbon filters and can thus be expected to be inhaled, is very low (0.0005 mg/kg/day). This indicates the potential serious risk for intervention staff. Besides inhaling, pesticides can also partly be absorbed through the skin.

Notwithstanding international allowable food tolerances are described by the European Commission, the so-called Maximum Residue Levels (MRLs) for pesticides to be found in or on food and animal feed, where a MRL is the highest level of a pesticide residue that is legally tolerated in or on food or feed when pesticides are applied correctly (Good Agricultural Practice), there is no regulation regarding their levels on cannabis, even not in countries were cannabis is legalized. Nevertheless, the exposure (inhaled or orally taken) to pesticides for cannabis users can induce severe risks.

# 5. Conclusion

The use of pesticides in illicit indoor cannabis plantations is seriously underestimated. Although earlier research did not reveal alarmingly high frequencies of pesticide use in indoor cannabis cultivation, our study shows that pesticide use in Belgian indoor cannabis plantations is a common practice. As a result, both growers and intervention staff face serious health risks because of pesticide use in indoor cannabis plantations. Furthermore, serious health issues can be expected for chronic cannabis users who most likely inhale substantial amounts of pesticides such as those found in our study.

Illegal cannabis cultivation should thus be considered as a serious health risk for intervention staff and chronic cannabis users. Our results warrant strict safety rules and appropriate personal protection equipment, including at least gloves and a protection mask type P2, to be worn by intervention staff when entering indoor cannabis plantations.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j. forsciint.2017.05.016.

#### References

- G. Green, The Cannabis Grow Bible. The Definitive Guide to Growing Marijuana for Recreational and Medicinal Use, Green Candy Press, San Francisco, CA, USA, 2001.
- [2] L.I. Johnson, J.D. Miller, Consequences of large-scale production of marijuana in residential buildings, Indoor Built Environ. 21 (4) (2011) 595–600.
- [3] D. Stone, Cannabis, pesticides and conflicting laws: the dilemma for legalized States and implications for public health, Regul. Toxicol. Pharmacol. 69 (3) (2014) 284–288.
- [4] J. McLaren, W. Swift, P. Dillon, S. Allsop, Cannabis potency and contamination: a review of the literature, Addiction 103 (7) (2008) 1100–1109.
- [5] T.D. Koch, C.-L. Chambers, S. Bucherl, J. Martyny, J. Cotner, S. Thomas, Clandestine Indoor Marijuana Grow Operations—Recognition Assessment and Remediation Guidance, American Industrial Hygiene Association (AIHA), Fairfax, VA, USA, 2010.
- [6] S. Schneider, R. Bebing, C. Dauberschmidt, Detection of pesticides in seized illegal cannabis plants, Anal. Methods 6 (2014) 515–520.
- [7] K.K. Kaistha, R. Tadrus, D. Wojtulewicz, Paraquat in marijuana, J. Pharm. Sci. 67 (8) (1978) IV.
- [8] K.E. Powell, A summary of pertinent medical information about paraquat in marijuana, Bull. Natl. Clgh. Poison Control Cent. (1978) 1–4.
- [9] C.E. Turner, P.C. Cheng, L.M. Torres, M.A. Elsohly, Detection and analysis of paraquat in confiscated marijuana samples, Bull. Narc. 30 (4) (1978) 47–56.
- [10] C.E. Turner, M.A. Elsohly, F.P. Cheng, L.M. Torres, Marijuana and paraquat, JAMA 240 (17) (1978) 1857.
- [11] L. Needham, D. Paschal, Z.J. Rollen, J. Liddle, D. Bayse, Determination of paraquat in marijuana by reversed-phase paired-ion high performance liquid chromatography, J. Chromatogr. Sci. 17 (2) (1979) 87–90.
- [12] J.A. Liddle, L.L. Needham, Z.J. Rollen, B.R. Roark, D.D. Bayse, Characterization of the contamination of marijuana with paraquat, Bull. Environ. Contam. Toxicol. 24 (1) (1980) 49–53.
- [13] P.J. Landrigan, K.E. Powell, L.M. James, P.R. Taylor, Paraquat and marijuana: epidemiologic risk assessment, Am. J. Public Health 73 (7) (1983) 784–788.
- [14] R. Lanaro, J.L. Costa, S.O. Cazenave, L.A. Zanolli-Filho, M.F. Tavares, A.A. Chasin, Determination of herbicides paraquat, glyphosate, and aminomethylphos-

phonic acid in marijuana samples by capillary electrophoresis, J. Forensic Sci. 60 (Suppl. 1) (2015) S241–S247.

- [15] N. Sullivan, S. Elzinga, J.C. Raber, Determination of pesticide residues in cannabis smoke, J. Toxicol. 2013 (2013) 378168.
- [16] J.W. Martyny, K.A. Serrano, J.W. Schaeffer, M.V. Van Dyke, Potential exposures associated with indoor marijuana growing operations, J. Occup. Environ. Hyg. 10 (11) (2013) 622–639.
- [17] M.V. Van Dyke, Potential Exposures in Illegal Marijuana Grow Operations, Colorado Department of Public Health and Environment, CO, USA, 2013.
- [18] C. Ogg, J. Hygnstrom, E. Bauer, P. Hansen, Managing the Risk of Pesticide Poisoning and Understanding the Signs and Symptoms, University of Nebraska Extension, USA, 2012.
- [19] P. Adams, Marijuana Indoor Growing, Positive Publishers b.v.b.a., Amsterdam, The Netherlands, 2007.
- [20] W. Vanhove, P. Van Damme, N. Meert, Factors determining yield and quality of illicit indoor cannabis (*Cannabis* spp.) production, Forensic Sci. Int. 212 (1–3) (2011) 158–163.
- [21] J. Cochran, J. Kowalski, S. Lupo, M. Misselwitz, A. Rigdon, F. Dorman, High quality analysis of pesticides in marijuana for medicine using QuEChERS, cartridge SPE cleanup, and GCxGC-TOFMS, in: R. Corporation (Ed.), Pittcon 2013, 2013.
- [22] RIVM/SEC, CLH Report Proposal for Harmonised Classification and Labeling Tebuconazole, RIVM/SEC, The Netherlands, 2008.
- [23] U.S.N.L.o.M. (NIH), TOXNET Toxicology Data Network, (2017) (Accessed 13 April 2017).
- [24] K.H. Kim, E. Kabir, S.A. Jahan, Exposure to pesticides and the associated human health effects, Sci. Total Environ. 575 (2017) 525–535.
- [25] Hazards of pesticides to the environment and human health, J. Environ. Sci. Health B 15 (6) (1980) 611–1114.
- [26] H.B. Weiss, Human exposures to pesticides. A 1979–1980 report of the Wisconsin Division of Health, Wis. Med. J. 80 (12) (1981) 12–15.
- [27] I. Batora, D. Grellneth, Pesticides and human health, Bratisl. Lek. Listy 109 (10) (2008) 472.
- [28] P. Nicolopoulou-Stamati, S. Maipas, C. Kotampasi, P. Stamatis, L. Hens, Chemical pesticides and human health: the urgent need for a new concept in agriculture, Front. Public Health 4 (2016) 148.