

Analysis of CBD Products (2022-23)

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FSA Research and Evidence

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There is the potential for residues of chemicals to be present in CBD products as a result of their natural occurrence in the raw material or arising from the manufacturing process, for example, mycotoxins, metals, pesticides, dioxins and the residues of solvents used to extract CBD. This study informs the FSA's understanding of the type and levels of contaminants that may arise in CBD products.

A wide range of analysis on CBD products was undertaken using accredited methods, for heavy metals, Polycyclic Aromatic Hydrocarbons (PAHs), dioxins, pesticides, mycotoxins, CBD content and cannabinoid profiles. Analysis for residual solvents, furans and additional mycotoxins was also carried out, but these were not accredited.

1. Executive summary

The Food Standards Agency (FSA) commissioned Fera Science Ltd. to carry out a survey of cannabidiol (CBD) products on sale in England and Wales in order to inform FSA risk assessment of CBD products. The survey commenced in October 2022 and was completed in March 2023. 100 CBD products on the FSA public list, at the time of the survey, were purchased from a range of online sellers from England and Wales. Samples comprised of oils and sprays, capsules, confectionary, beverages and a range of miscellaneous products that included other foods and dog treats. The sampling followed a scheme suggested by FSA.

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pesticides, dioxins and the residues of solvents used to extract CBD. This study informs the FSA's understanding of the type and levels of contaminants that may arise in CBD products.

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The results of testing found the following:

- For 54 samples, heavy metals (cadmium, mercury & lead and arsenic) were not detected, meaning levels were below the limits of quantification of the method. 41 samples contained lead, 22 samples contained arsenic, 17 samples contained cadmium and mercury was detected in three samples. There is uncertainty on whether products would be classed as an oil or food supplement, if an oil, lead levels would be above the regulatory maximum for nine samples but would be below if classed as a food supplement. For all other metals, all samples were compliant or there was no specific regulatory limit for the product in regulation.
- Of the 100 products tested, 49 matched the CBD claim with eight exceeding the label claim of CBD content. The remaining 43 samples had less CBD than claimed. Notably, only two drink samples and one tea solid matched CBD claims, with all other 17 drink samples being below the claimed CBD level.
- One sample exceeded the Maximum Residue Limit (MRL) for both benzo[a]pyrene (BAP) and the sum of PAH4 (benzo[a]pyrene, benzo[a]anthracene, benzo[b]fluoranthene and chrysene). Another sample exceeded the MRL for BAP only and a third sample exceeded the MRL for the sum of PAH4. 39 samples contained no detectable level of any PAH tested.
- 27 of the 100 samples were found to contain controlled substances above 1 mg sum of controlled substances per container threshold.
- A total of 35 pesticides were found across all products (each product was tested for over 400 pesticides). There are currently no specific MRLs for CBD products.
- Methanol was detected above the MRL in 25 samples.
- Four samples were found to contain Propan-2-ol above the MRL.

- Mycotoxins were detected in 24 samples. There are no maximum permitted levels for aflatoxins or ochratoxin A in these products.
- For the seven polychlorinated biphenyl (PCB) International Council for the Exploration of the Sea (ICES) (sum of (PCB's) 28, 52, 101, 138, 153 and 180) did not exceed 1.30 µg/kg in any sample. The sum of the combination of PCDD/F and PCB World Health Organization (WHO) toxic equivalents (TEQ) (PCB's 77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167 and 189) ranged between 0.32 and 0.60 ng/kg in all 10 samples.

2. Introduction

2.1. Background to the study

At the time of the survey the FSA has had over 12,000 CBD products linked to novel food applications for CBD food products and these are held on a public list, although many of these are derived from the same CBD base oil and consist of the same product but at different concentrations. To inform FSA risk assessment of applications, FSA commissioned this survey to provide a snapshot of products on the FSA public list. The survey will provide useful information on the quality of the applications, their supporting information and composition of the products.

A tailored sampling study of 100 CBD products selected by the FSA from the public list was carried out. These products were selected to provide coverage of both the base oils and the types and quantity of products on the FSA public list. Most samples were focused on oils and tinctures, given their expected market share, with the remaining products covering capsules, gummies, beverages and miscellaneous products. Products that do not have an associated relevant application are non-compliant and should not be on sale. More information is available on the FSA's webpage on CBD (FSA, 2022). A similar study on 30 samples was completed by Fera Science Ltd for the FSA and published on 27th October 2022.

2.2. CBD products – safety considerations

There is potential for certain chemical contaminants to be present in CBD products as a result of their natural occurrence in the raw material or from the manufacturing process. Hemp and cannabis plants are widely reported to be effective at bioremediation and can remediate heavy metals and PAHs from soil, with these contaminants accumulating in the plants (Campbell et al., 2002; Galić et al., 2019). Thus, CBD products could have the potential to contain these chemical contaminants. As the hemp-based CBD products are derived from plant sources there is also the potential for the occurrence of other contaminants such as mycotoxins. Aflatoxins and ochratoxin A are the two most commonly tested for as these are regulated

in other countries where CBD and cannabis products are legal. e.g. Canada and some US states (Wilcox et al., 2019). More recently there have also been reports of some *Fusarium* mycotoxins in some CBD products of botanical origin (Narváez et al., 2020).

Other contaminants or residues that can occur in these products as a result of their growth or production include pesticides and residual solvents. Solvents are used to extract the CBD and other cannabinoids, and can range from approved food solvents such as ethanol or isopropyl alcohol to more harmful ones (petroleum-ether, naphtha), or chemicals used in super-critical fluid extraction (butane, CO₂) (Hazekamp, 2018).

In 2019, CBD was confirmed as a novel food product at a European level and was added to the Novel Food Catalogue (European Commission, n.d.). In February 2020 the FSA announced a deadline of 31 March 2021 for CBD producers in England and Wales to submit valid novel food authorisation applications. The authorisation process ensures novel foods meet legal standards, including safety (FSA, 2021).

2.3. CBD products – controlled substances

While the FSA has regulatory responsibility for CBD use in foods, products containing the psychoactive substance tetrahydrocannabinol (THC), with limited exemptions, are classed as controlled drugs under the Misuse of Drugs Act. The Home Office have provided a factsheet on Cannabis, CBD and other cannabinoids (Home Office, 2019). It states that the product or preparation should not contain more than one milligram (mg) of the controlled drug. It is important to note it is the Home Office view that the applicable unit of measure for the 1 mg 'threshold' is that of the 'container' (i.e. bottle or packet) and not the 'typical dose' (of any product). Currently, FSA use this 1 mg/container principle as a benchmark of when a CBD product is not considered a food. When FSA become aware of a product exceeding the 1 mg benchmark that product can be removed from the public list. Any product that contains any psychoactive substances is deemed noncompliant. The Government Chemist has published guidance on analytical limits for controlled cannabinoids in products containing CBD. This guidance recommends that the 1 mg threshold be the sum of psychoactive cannabinoids within the container (Walker & Axford, 2021).

The European Food Safety Authority (EFSA) published an opinion in 2015 that estimated an Acute Reference Dose (ARfD) for Δ^9 -THC of 1 $\mu\text{g/kg}$ body weight (EFSA, 2015). In 2020, they also published a report that highlighted that, based on the data available, the EFSA ARfD of 1 $\mu\text{g/kg}$ body weight was exceeded in the adult high consumers of hemp and hemp containing products, including teas, energy drinks and chocolate under the lower-bound (LB) and upper-bound (UB) scenario (EFSA et al., 2020).

In January 2022, the Advisory Council on the Misuse of Drugs (ACMD) published a report recommending a legal framework to control the amounts of phytocannabinoids in consumer CBD products under the Misuse of Drugs Act. This recommended the dose of each controlled phytocannabinoid should not exceed 50 µg per unit of consumption and the total dose of Δ^9 -THC (including Δ^9 -THCA) and all other controlled phytocannabinoids in consumer CBD products be controlled (ACMD, 2021). In October 2023 the Home Office responded to and accepted all of the ACMD recommendations (Home Office, 2023).

2.4. Aims and objectives of the study

The aim of the study was to carry out a tailored sampling study of 100 CBD products selected by the FSA from the public list currently on sale in England and Wales. Samples were analysed for CBD content, other cannabinoids and a range of chemical contaminants.

All samples were purchased on-line. Analyses were carried out to inform the FSA's risk assessment and gain understanding of the composition of CBD products.

3. Methodology

3.1. Samples

The FSA provided a list of suggested product types, which Fera Science Ltd used as a sampling plan for online purchasing. This list represented a snapshot of the main types of CBD products that are available for sale in England and Wales. Where samples were not available these were substituted with products of the same type with a similar declared CBD content and in agreement with the FSA. The broad categories of samples were CBD oils (tinctures, sprays and drops), CBD edibles/confectionary (gummies, sweet and chocolates), ready to drink beverages, capsules (oil or powder filled) and miscellaneous (consisting of peanut butter, protein powder, jams, honeys and baked products).

3.2. Quality control

The quality control criteria and results from all analytical batches of the various methodologies applied in this study will be supplied to the FSA as a separate document.

3.3. Metals analyses

Samples were analysed for arsenic, cadmium, mercury and lead by following in-house methods; FSG 461, FSG 457 and FSG 463. Aliquots of homogenised test sample were digested in a mixture of concentrated

nitric acid and hydrochloric acid using a high-pressure microwave system. Quantification was by inductively coupled plasma-mass spectrometry (ICP-MS) with collision cell. Quality checks included blanks, spikes and certified reference materials.

All data were corrected for reagent blank and spike recovery. The reporting limit was equal to, or exceeded, 10 x standard deviation of reagent blank values adjusted for dilution and sample weight. Reference material results were all satisfactory. Results are UKAS accredited (ISO 17025).

3.4. Polycyclic aromatic hydrocarbons (PAHs) analyses

Samples were analysed for PAHs using in-house method FSG 410 Extraction of Foods for the Determination of PAHs, accredited to ISO 17025. The method can determine 28 PAHs, including the four regulated (marker) PAH compounds BAP, benzo[a]anthracene, benzo[b]fluoranthene and chrysene. The full list of analytes included in the method is:

- acenaphthylene
- acenaphthene
- fluorene
- anthracene
- phenanthrene
- fluoranthene
- benzo[c]fluorene
- pyrene
- benzo[a]anthracene*
- benzo[ghi]fluoranthene
- benzo[b]naphtho[2,1-d]thiophene
- cyclopenta[c,d]pyrene
- chrysene*
- 5-methylchrysene
- benzo-[b]-fluoranthene*
- benzo[j]fluoranthene
- benzo[k]fluoranthene

- benzo[e]pyrene
- benzo[a]pyrene*
- indeno[1,2,3-cd] pyrene
- dibenz[ah]anthracene
- benzo[g,h,i]perylene
- anthanthrene
- dibenzo[a,l]pyrene
- dibenzo[a,e]pyrene
- dibenzo[a,i]pyrene
- dibenzo[a,h]pyrene
- coronene

*These four regulated PAHs are included in the PAH4 SUM.

An aliquot of the homogenised sample was fortified with appropriate ¹³C internal standards and subjected to saponification followed by liquid-liquid extraction. Clean-up was by dimethylformamide/cyclohexane partition followed by elution through a silica gel column. Analysis was by gas chromatography mass spectrometry (GC-MS).

3.5. Cannabinoid analyses by HPLC-UV

Samples were analysed for cannabinoids using in-house method FSG 785: The determination of a suite of seven cannabinoids in CBD products by reverse phase high performance liquid chromatography (HPLC) using UV detection, accredited to ISO 17025 for CBD in oils/oil products and by flexible scope for matrices other than oil. The seven cannabinoids included in the method are:

- Cannabichromene (CBC)
- Cannabidiol (CBD)
- Cannabidiolic acid (CBDA)
- Cannabigerol (CBG)
- Cannabinol (CBN)
- Δ9-Tetrahydrocannabinol (Δ9-THC)
- Tetrahydrocannabinolic acid (THCA)

Aliquots of sample were weighed into a screw top container and dissolved in propan-2-ol. After sonication, the mixture was allowed to cool and an aliquot was diluted with a methanol: water mixture (90 : 10). This was filtered through a 0.2 µm, 13 mm polytetrafluoroethylene (PTFE) syringe filter and analysed by reverse phase HPLC using gradient elution and detection by UV at 220nm.

Quality control samples - procedural blanks, in-house reference samples and spiked samples - were included in each batch. This method was used to quantify high levels of CBD, and confirm levels of the other cannabinoids listed that were determined by liquid chromatography tandem quadrupole mass spectrometry (LC-MS/MS).

3.6. Cannabinoid analyses by LC-MS/MS

Samples were analysed for cannabinoids using in-house method FSG 788: Analysis of Plant Oil for the determination of 14 Cannabinoids by LC-MS/MS, accredited to ISO 17025 for oil/oil products and flexible scope for matrices other than oil. It must be noted that although it is possible to measure CBD using this method the levels in samples are frequently outside the method working range and CBD is quantified by the HPLC-UV method.

The cannabinoids included in the method are:

- Cannabichromene (CBC)
- Cannabidiol (CBD)
- Cannabidiolic Acid (CBD-A)
- Cannabigerol (CBG)
- Cannabinol (CBN)
- Δ9-Tetrahydrocannabinol (Δ9-THC)
- Δ9-Tetrahydrocannabinolic acid A (THC-A)
- 8-Tetrahydrocannabinol (8-THC)
- 9-Tetrahydrocannabivarin (THCV)
- 9-Tetrahydrocannabivarinic acid (THCV-A)
- Cannabidivarin (CBDV)
- Cannabidivarinic acid (CBDV-A)
- Cannabigerolic acid (CBG-A)
- Cannabichromene acid (CBC-A)

Aliquots of sample were weighed into a screw top container and dissolved in propan-2-ol. After sonication, the mixture was allowed to cool and an aliquot was first diluted with methanol, before being diluted with a methanol: water mixture (90 : 10) and THC-d3 internal standard (ISTD) solution. If the extract was cloudy, it was filtered through a 0.2 µm, 13 mm PTFE syringe filter and analysed by LC-MS/MS.

Quality control samples including procedural blanks, in-house reference samples and spiked samples were included in each batch.

3.7. Pesticide analyses

Samples were analysed for over 400 pesticides using two in-house multi-residue screening methods. For in-house method FSG/167 (09) LCMS - a sub-sample was extracted with acetonitrile, in the presence of salts. Analysis was carried out using LC-MS/MS in selected reaction monitoring mode. The presence of residues was confirmed using the same technique in multiple reaction monitoring mode.

For in-house method FSG/167 (09) GC-MS - a sub-sample was extracted with acetonitrile, in the presence of salts. After clean-up using dispersive SPE, analysis was carried out using gas chromatography with mass spectrometric detection (GC-MS/MS) in selected reaction monitoring mode.

Results for all samples except the CBD gummies are UKAS accredited (ISO 17025).

A full list of the analytes included in the methods and their reporting limits is given in Annex A.

3.8. Dioxins, PCBs, Polychlorinated dibenzofurans (PCDFs) analyses by High-resolution mass spectrometry (HRMS)

In-house methods FSG 403 to 408 were used for the detection of dioxins and PCBs.

The methods are UKAS accredited to the ISO/IEC 17025 Standard (animal feeds, beverages, foods and human tissues, crops, soils, pollutants and effluents (solid)).

An aliquot of each sample was fortified with known amounts of surrogate ($^{13}\text{C}_{12}$ -labelled) analogues of target analytes and exhaustively extracted using mixed organic solvents. The extract was cleaned-up using adsorption chromatography. Ortho-PCBs, non-ortho-PCBs and PCDDs/PCDFs were segregated into two separate fractions. Each fraction was then

concentrated and further cleaned-up before the inclusion of additional surrogate standards. Final determination was conducted by high resolution gas chromatography with either low resolution mass spectrometric detection (ortho-PCBs) or HRMS (non-ortho-PCBs and PCDDs/PCDFs).

3.9. Mycotoxin analyses by HPLC-FLD

Analysis for aflatoxins B₁, B₂, G₁ and G₂ and ochratoxin A was carried out using in-house SOP FSG 261 Simultaneous determination of ochratoxin A and aflatoxins B₁, B₂, G₁ and G₂ using immunoaffinity column clean-up and HPLC with fluorescence detection (HPLC-FLD). The reporting limit for each analyte is 0.2 µg/kg, the analysis is accredited to ISO17025.

Samples were extracted with a mixture of acetonitrile and water, cleaned-up by immunoaffinity column and analysed using reverse phase HPLC, with a gradient elution and fluorescence detector programmed to detect aflatoxins and ochratoxin A. A blank sample and two spiked samples (in the absence of an in-house reference sample) were included in the batches as quality control samples.

3.10. Mycotoxin analyses by LC-MS/MS

Two separate LC-MS/MS methods were used to determine other mycotoxins. In-house method FSG 820 – Determination of deoxynivalenol (DON), zearalenone (ZON), T-2 and HT-2 toxins using immunoaffinity column clean-up and LC-MS/MS and in-house method - Analysis of Mycotoxins using LC-MS/MS was used for the determination of enniatins A, A₁, B and B₁ and beauvericin.

DON, ZON, T-2 and HT-2 toxins were analysed using FSG 820. Samples were extracted with a mixture of acetonitrile and water, cleaned-up by immunoaffinity column and analysed using LC-MS/MS. A blank sample and spiked samples (in the absence of an in-house reference sample) were included in the batches as quality control samples. The reporting limit was 5 µg/kg for each mycotoxin.

Enniatins A, A₁, B and B₁ and beauvericin were extracted with a mixture of acetonitrile and water and analysed using LC-MS/MS. A blank sample and spiked samples (in the absence of an in-house reference sample) were included in the batches as quality control samples. The reporting limit was 1.25 µg/kg for each mycotoxin.

This methodology is not covered by ISO17025 accreditation.

3.11. Residual solvent analyses by headspace GC-MS

Analysis was undertaken using modified methodology based on “Analysing residual solvents in cannabis concentrates via HS-GC” by Restek Corporation.¹

All samples and standards were prepared in dimethyl acetamide (DMA) in a laminar flow cabinet, procedural blanks were prepared in the same manner.

Analyses were carried out in split mode using scan methods to accommodate the range of reporting levels and sample analyte concentrations. A DB-624 UI Gas Chromatograph column was used to separate the analytes of interest before determination by Mass Spectrometry.

This methodology is not covered by ISO17025 accreditation.

3.12. Furans by headspace GC-MS

Analysis for furans (furan, 2-methylfuran, 3-methylfuran, ethyl furan, 2,5-dimethyl furan, propyl furan, butyl furan and pentyl furan) was carried out using in-house SOP FSG 824 - Determination of furans in foodstuffs using headspace GC-MS. A blank sample and spiked samples (in the absence of an in-house reference sample) were included in the batches as quality control samples. The reporting limit for each analyte was 5 µg/kg.

This methodology is not covered by ISO17025 accreditation.

4. Results and discussion

4.1. Metal analyses

Metals results are given below in Table 3 of Annex B. Samples were analysed for arsenic, cadmium, mercury and lead as the four main potential contaminant heavy metals and for which MPLs are set in Assimilated Regulation (EC) No 1881/2006 (Assimilated Regulation (EC) No 1881/2006, 2006). Results have been compared against relevant legal maximum levels where appropriate.

A total of 54 samples did not contain any metal levels above the limits of quantification.

¹ Harper, et al. 2020 [Novel Residual Solvents Analysis of Cannabinoid Products with the Agilent Headspace-GC/MS System \(agilent.com\)](#)

Lead was the most frequently detected metal, 41 samples contained lead above the reporting limit. Levels found ranged from 0.005 to 0.878 mg/kg. The highest level of 0.878 mg/kg was found in sample S22-047113 a broad-spectrum CBD oil. Maximum levels for lead in Assimilated Regulation (EC) No 1881/2006 are 0.10 mg/kg for fats and oils (including milk fat), and 3 mg/kg for food supplements. The lead level in nine samples were above the maximum level for fats and oils but are below 3 mg/kg. However, due to uncertainty as to whether the product would be classified as a food supplement or other form of food it is difficult to make a definitive statement as to whether the products exceed a maximum lead level. 18 samples contained only lead and a further 23 contain lead plus other metals.

Three of the samples were found to contain traces of mercury above the reporting limit (S22-047096 at 0.007 mg/kg, S22-047128 at 0.007 mg/kg and S22-047132 at 0.01 mg/kg). All three of these samples are powder filled capsules. There are no regulatory limits for mercury in any of these types of products. The three samples found to contain mercury also contained traces of arsenic and lead. Two of the three also contained traces of cadmium.

Arsenic was found above the limits of quantification in 22 samples. Levels of arsenic found ranged from 0.006 to 0.08 mg/kg. The highest level was found in sample S22-047128, a powder filled capsule. There are no regulatory limits for arsenic in any of these types of products. Arsenic was the only metal found in samples S22-047043 (oil), S22-047066 (gummies), S22-047092 (oil) and S22-047114 (beverage).

17 samples were found to contain cadmium above the reporting limit. The levels of cadmium ranged from 0.009 to 0.364 mg/kg. The highest levels were measured in samples S22-047071 (0.364 mg/kg) and S22-047100 (0.211 mg/kg). Both are samples of chocolate at 70% and 64% cocoa solids respectively. Maximum levels for cadmium in chocolate in Assimilated Regulation (EC) No 1881/2006 range from 0.10 to 0.80 mg/kg depending on the cocoa solids content. For chocolate with ≥ 50 % total dry cocoa solids the maximum level of cadmium should not exceed 0.8 mg/kg. Therefore, both samples are compliant. The other samples found to contain cadmium above the reporting limit are chocolates (n=2), oils (n=2), baked goods/products containing chocolate (n=4), confectionary (n=1), tea/coffees (n=3), powder capsules (n=2) and nut butter (n=1). All samples were either compliant with the regulatory limit or were not categorised by the regulation.

4.2. PAH analyses – results of BAP and SUM PAH4

The results of the PAH analyses are given below in Table 4 of Annex B. All results have been corrected for recovery. Results for BAP and the sum of PAH4 (BAP, benzo[a]anthracene, benzo[b]fluoranthene and chrysene) are highlighted. These are the compounds for which MPLs are established in legislation. Assimilated Regulation (EC) No 1881/2006 sets maximum levels of 10 µg/kg for BAP and 50 µg/kg for PAH4 in food supplements containing botanicals and their preparations. Powders of food of plant origin for the preparation of beverages in food also have maximum levels set at 10 µg/kg BAP and 50 µg/kg PAH4.

The BAP level in two products exceeded the MRL, one product (S22-047078 an oil filled capsule containing 11.8 µg/kg) was within the MRL when taking into account the measurement uncertainty and all other products tested were within the limits. The products in exceedance for BAP levels were sample S22-047079 (a CBD infused ground coffee containing 16.7 µg/kg) and S22-047132 (a powder filled capsule containing 74.1 µg/kg). The sum of PAH4 level in coffee sample, S22-047079, was 31.1 µg/kg which is compliant with the MRL of 50 µg/kg. However, samples S22-047078 and S22-047132 (oil and powder filled capsules) contained PAH4 levels at 64.6 and 309 µg/kg respectively. If classified as food supplements they are both non-compliant against the limit of 50 µg/kg. Due to the very high levels of BAP and the sum of PAH4 in sample S22-047132 the product was re-purchased and re-analysed (new sample number S23-017195, same batch and best before date). The results from sample S23-017195 were again non-compliant with a BAP level of 46 µg/kg and sum of PAH4 of 221 µg/kg.

4.3. PAH analyses – other PAH compounds

In addition to the marker compounds that are controlled by legislation the method used can also determine other PAHs. The results for the other compounds are also given in Table 4 of Annex B. These results are corrected for recovery.

Sample S22-047132 contained the highest overall PAH content, with residues found for 27 out of 28 of the PAHs included in the method. This was a powder containing capsule. Sample S22-047078 (an oil capsule) contained the second highest level with 25 out of 28 PAHs detected above the reporting limit.

The samples that contained the highest levels of BAP and PAH4 also contained the highest levels of the other PAHs. Low levels of other PAHs were also detected in several other samples. 40 samples contained no PAH residues above the reporting limit, these were split between confectionary (n=14), drinks (n=13), oils (n=6), capsules (n=4) and miscellaneous (n=3).

4.4. Cannabinoid profiling

All 100 samples were analysed for CBD and 13 cannabinoids of which 4 are controlled substances (CBN, Δ 8-THC, Δ 9-THC, THCV) and THC-A. We categorised the samples into three classes of products: oil-based (n = 46; oils, tinctures, sprays, capsules), edibles (n = 34; powder capsules, baked goods, sugary spreads - honey/jam, confectionary – gummies, boiled sweets, chewing gum, chocolate/peanut butter) and drinks (n = 20; soft, alcoholic, tea/coffee solids).

4.4.1. CBD analyses – by HPLC-UV

Results for CBD products are given in Annex B, Tables 6 to 6.2 for oil-based products and Tables 6.3 to 6.9 for edibles and drinks respectively. A comparison of the labelled or declared CBD content is also given. The results are not corrected for recovery.

Claimed CBD concentrations ranged from 0.0024 % for a soft drink to 40 % in an oil.

We took the expanded relative measurement uncertainty of 12.2 % for this method into account for any CBD level claims.

Of the 100 samples analysed, 49 matched their CBD claim, with 8 samples containing more, but 43 samples containing less CBD than claimed.

It is important that the actual CBD content of foods should not deviate substantially from labelled amounts, as the consumer could otherwise be misled or accidentally exceed guidance levels.

Five samples (4 soft drinks - S22-047114, S22-047091, S22-047123, S22-04790 and peanut butter - S22-047116) and the brewed versions of the tea/coffee solids did not contain any measurable amounts of CBD using our standard extraction procedure.

4.4.1.1. Oils

For the 46 oil-based samples (including 6 oil filled capsules), 65% of samples matched their CBD level claim, whilst 24% fell below and 11% above the CBD claim.

4.4.1.2. Edibles

One of the chocolate samples (S22-047071) matched the CBD level, while the other four fell below. 50 % of the baked goods matched their CBD claim. For the six powdered capsules 1 sample fell below and 1 above the labelling claim. The CBD contents in sugary spreads (n=3) were by a factor 10 or 5 lower than the label claim.

For the confectionary, four of the gummies contained 60% or less of their CBD claim (S22-047070, S22-047105, S22-047067, S22-047074), whilst two gummies (S22-047119, S22-047066) and a chewing gum (S22-047112) contained around 25% more.

The CBD levels measured in one gummy (S22-047065), one marshmallow (S22-047065) and one boiled sweet (S22-047110) product fell just below the CBD claim level.

4.4.1.3. Drinks

Only two of the drink samples, which were in aluminium and glass bottles (S22-04075 and S22-0471526), and one of the tea solids (S22-04793) matched the CBD claims, while the other 17 drinks samples were below the claimed CBD level. It appears to be a re-occurring phenomenon that CBD sticks to plastic or plastic lining of cans. This is not likely to be a food safety issue but means the CBD level could be lower than advertised to consumers.

4.4.2. Cannabinoid analyses by LC-MS/MS

The results of the cannabinoid suite analyses are given in Annex B, Tables 7 to 7.9 for non-psychoactive cannabinoids and Tables 8 to 8.9 for controlled cannabinoids. The working range for this method is 2.5 mg/kg to 500 mg/kg. In nearly all cases the CBD levels in the samples were too high to quantify using this method. In some cases, the levels of other cannabinoids were also over the calibration range of the method. These extracts were diluted and reanalysed to bring them within the working range of the method. In many instances where the levels were too high to quantify using LC-MS/MS it was possible to quantify the cannabinoid using the HPLC-UV results. These values are indicated in the tables.

In some cases, HPLC-UV data for CBG and CBC was used instead, due to the occurring mass interferences from CBD itself for samples high in CBD.

Products in tables 8 to 8.9 of Annex B which have results of THC+ above 1 mg per container, exceed the threshold set out in the Home Office's guidance and would be deemed non-compliant and are not classed as foods. Samples that exceeded the ACMD recommended daily dose of 50 µg THC per day were reanalysed in duplicate for confirmation. Separate reports (n=15 oil-based and 1 powder capsule) were issued to the FSA.

A standard approach for this analysis is to consider the levels of the cannabinoids Δ 9-THC, as the main controlled drug, and also Δ 8-THC, CBN, THC-V and THC-A. Results have been calculated to determine the amount of these compounds in the sample in mg/kg, the sum of these five compounds (using a factor of 0.877xTHC-A to account for the decarboxylation reaction for transformation of THC-A into THC through the loss of a carbon dioxide molecule (CO₂)) was also calculated (Fundacion Canna, n.d.). The sum value was calculated as a lower bound value, i.e. results less than the limit of quantification (LOQ) were presumed to be zero. Using these values, the absolute amount of THC and the sum of the five compounds (THC+), in mg, in each container was calculated. In the case of edibles (gummies, chocolate bars etc.) and drinks the absolute amount, in mg, in each individual item was also calculated.

The data is not corrected for recovery; it was decided to report in this way to report the data in the most conservative way. There is currently no established convention for reporting and correction for recovery, as there is for example for PAHs or mycotoxins.

Of the 100 samples analysed, 27 were above the threshold of the sum of 1 mg controlled substances/container.

4.4.2.1. Oils

Only two (S22-047059 and S22-047101) out of the 46 oil-based samples contained no controlled substances. Nearly 50 % (21 out of 46) possessed Δ 9-THC level > 1 mg/container, of which 6 products contained between 10 and 117 mg Δ 9-THC per container, and an additional three samples failed compliance, when taken the sum of controlled substance into account.

4.4.2.2. Edibles

No controlled substances were detected in sugary spreads, baked goods, boiled sweets/chewing gum and chocolate/peanut butter. One gummy sample (S22-04766) exceeded 1 mg Δ 9-THC per bag, whilst another (S22-047061) contained 1.8 mg of the controlled substance CBN in the bag.

4.4.2.3. Drinks

The liquid beverages were all compliant with the level of Δ 9-THC, as expected due to the low CBD concentrations measured. For the tea/coffee solids one coffee sample (S22-047081) - based on the bigger packs size (340 g vs 60 mL max) compared to oil-based products – contained 2.5 mg of Δ 9-THC per bag.

4.5. Pesticide analysis results

All 100 samples were screened for over 400 pesticide compounds. A full list of all pesticides tested, and their reporting limits are given in Annex A, Table 1. 35 residues above the reporting limit were found in 21 samples. A summary of the residues found is given in Annex B, Table 5. Levels of pesticides above the reporting limit ranged from 0.01 to 0.93 mg/kg. The highest concentration was fenhexamide found in sample S22-047057 which is an oil.

There are no specific MRLs for CBD products.

4.6. Mycotoxins – HPLC-FLD analysis results

The samples were analysed using immunoaffinity column clean-up, and results are given in Table 9 of Annex B. Aflatoxins were detected in 10 samples above the reporting limit of 0.2 µg/kg. 13 samples contained ochratoxin A above the reporting limit of 0.2 µg/kg, five samples contained both aflatoxins and ochratoxin A. The maximum level of total aflatoxins found was 3.0 µg/kg. The highest level of ochratoxin A found was 4.6 µg/kg in sample S22-047093, a turmeric and ginger infusion. This sample also contained 2.6 µg/kg aflatoxins. There are no maximum permitted levels for aflatoxins or ochratoxin A in these products.

4.7. Mycotoxins – LC-MS/MS analysis results - (deoxynivalenol, zearalenone, T2 toxin, HT-2 toxin) (DZT)

Analysis for deoxynivalenol, zearalenone, T-2 toxin and HT-2 toxin (DZT) were carried out using immunoaffinity column clean-up and LC-MS/MS, results are given in Table 10 of Annex B. Five samples contained DON above the reporting limit, the highest level found was 137 µg/kg in S22-047083, dog treats. Six samples contained T-2 toxin, or HT-2 toxin or both. The highest levels found were 40.4 µg/kg T-2 and 16.8 µg/kg HT-2 in sample S22-047041 (a raw hemp oil) and 40.6 µg/kg T-2 and 10.5 µg/kg HT-2 in sample S22-047127 (a CBD tincture).

Four samples contained ZON above the reporting limit of 5 µg/kg. The maximum level found was 47.6 µg/kg in sample S22-047071, a CBD infused chocolate. There are no maximum levels for ZON in CBD products or chocolate products.

4.8. Mycotoxins – LC-MS/MS analysis results - Enniatins and Beauvericin

Results are given in Table 11 of Annex B. Most of the results were below the reporting limit of 5 µg/kg. Only one sample contained beauvericin above the reporting limit, that was sample S22-047093 (CBD, turmeric and ginger teabag) that contained 68.2 µg/kg. Enniatin B (Enn B) and enniatin B1 (Enn B1) were also detected in this sample at 13.7 and 8.5 µg/kg respectively. This sample also contained aflatoxins and ochratoxin A. Only eight further samples contained levels of enniatins above the reporting limit. These consisted of oils (n=3), capsules (n=3), dog treats (n=1) and cookies (n=1). The levels ranged between 5.3 and 97.8 µg/kg. There are no maximum levels for enniatins and beauvericin in CBD products.

4.9. Residual solvents results

All 100 samples were screened for 18 residual solvents by headspace GC-MS/MS. A full list of the solvents, and their reporting limits are given in Annex A, Table 2. Nine of the solvents (ethyl ether, hexane, chloroform, benzene, 1,2 Dichloroethane (1,2 DE), trichloroethylene, toluene, m&p-xylene and o-xylene) were not detected in any of the samples above the reporting limit. Nine solvents (methanol, pentane, ethanol, acetone, propan-2-ol, acetonitrile, dichloromethane, ethyl acetate and heptane) residues above the reporting limit were found across 69 samples. A summary of the residues found is given in Table 12 of Annex B. Methanol was the most frequently detected solvent in 41 samples. Levels of methanol ranged between 6 and 801 mg/kg. The highest concentration of methanol was measured in sample S22-047132 which is a capsule. The MRL for both methanol and propan-2-ol in extracted foodstuff or food ingredient is 10 mg/kg, as set out in The Food Additives, Flavourings, Enzymes and Extraction Solvents (England) Regulations (2013). 25 of the samples had levels of methanol above the MRL of 10 mg/kg. Four of the samples had levels of propan-2-ol above the MRL of 10 mg/kg.

Only permitted extraction solvents as defined in the Food Additives, Flavourings, Enzymes and Extraction Solvent Regulations 2013 (*The Food Additives, Flavourings, Enzymes and Extraction Solvents (England) Regulations, 2013*) can be used in the production of food placed on the market. Three solvents not on the permitted solvents list were detected above the reporting limit in 24 samples. Acetonitrile was detected in 13, heptane was detected in seven and pentane was detected in five samples.

4.10. Furans results

All 100 samples were screened for eight furans. 40 samples were found to contain furans above the reporting limit. An additional 14 samples contained furans that could only be tentatively assigned. Samples S22-047079, S22-047081 and S22-047125 all contained levels of each furan above the reporting limit. All three were samples of coffee. A summary of the furan results is given in Table 13 of Annex B.

There are no specific maximum levels for furans in CBD products, or foods.

4.11. Dioxins, Polychlorinated biphenyls (PCB's) and Polychlorinated dibenzofurans (PCDF's) results

A total of 10 oil samples were analysed for dioxins, PCB's and PCDF's. The summary of results is presented in Table 14 of Annex B. The sum of ICES (PCB's 28, 52, 101, 138, 153 and 180) ranged between 0.14 and 1.30 µg/kg. The highest value was detected in sample S22-047045. The sum of the combination of PCDD/F and PCB WHO TEQ (PCB's 77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167 and 189) ranged between 0.32 and 0.60 ng/kg. Again, the highest value was detected in sample S22-047045.

There are no specific maximum levels for PCB's in CBD products.

5. Summary and conclusions

100 CBD products were purchased from a range of online sellers from England and Wales following a sampling protocol provided by FSA. Samples comprised 40 oils and sprays, 20 beverages, 19 confectionaries (gummies, chewing gum, chocolate bars and mints), 12 capsules (oil, gel and powder) and 9 miscellaneous (jams, peanut butter, baked goods and honey).

UKAS (ISO17025) accredited methods were used to analyse the products for heavy metals, PAHs, pesticides, mycotoxins, CBD content and selected cannabinoid profile, including the controlled drug Δ 9-THC. Results reported in this study are UKAS accredited, except for pesticides in gummies. Enniatins, beauvericin, ZON, DON, T-2 and HT-2 toxins, residual solvents and furans are out of scope and are not accredited.

Heavy metals were not detected above the LOQ in 54 samples. Lead was the most frequently detected metal, 41 samples contained lead above the reporting limit. The highest level of lead found was 0.878 mg/kg in a broad-spectrum CBD oil. Arsenic was measured in 22 samples with levels ranging between 0.006 and 0.08 mg/kg. 17 samples were found to contain cadmium at levels between 0.009 and 0.364 mg/kg. The highest levels were

found in two chocolate samples. Mercury was detected in three samples at levels between 0.007 and 0.01 mg/kg. All three samples were powder filled capsules.

The BAP level in three products exceeded the MRL, all other products tested were within the limits. The products in exceedance for BAP levels were an oil filled capsule containing 11.8 µg/kg, a CBD infused ground coffee containing 16.7 µg/kg and a powder filled capsule containing 74.1 µg/kg. The two capsule samples with high levels of BAP also contained the highest PAH4 sum levels. The oil filled capsule contained 64.6 µg/kg and the powder filled capsule contained 309 µg/kg. If classified as food supplements they are both non-compliant against the limit of 50 µg/kg. 39 samples contained no PAH residues above the reporting limit, these were split between confectionary (n=14), drinks (n=13), oils (n=6), capsules (n=4) and miscellaneous (n=3).

Of the 100 samples analysed, 47 matched their CBD claim, 8 samples were higher and 45 samples contained less than the CBD levels claimed. Five samples (4 soft drinks – and a peanut butter) and the brewed versions of the tea/coffee solids did not contain any measurable amounts of CBD using our standard extraction procedure.

Of the 100 samples analysed, 27 were above the 1 mg sum of controlled substances per container threshold (THC+).

When taking into account the manufacturers recommended daily dose the potential consumed level of THC+ was a concern for some products in table 8 to 8.9. These samples were re-analysed in duplicate for confirmation and separate reports issued (n = 15 oil-based products and 1 powder capsule).

35 pesticide residues above the reporting limit were found in 21 samples. Levels of pesticides ranged from 0.01 to 0.93 mg/kg. The highest concentration was fenhexamide found in an oil.

Five samples contained DON above the reporting limit, the highest level found was 137 µg/kg in dog treats. Six samples contained T-2 toxin, or HT-2 toxin or both. The highest levels found were 40.4 µg/kg T-2 and 16.8 µg/kg HT-2 in a raw hemp oil and 40.6 µg/kg T-2 and 10.5 µg/kg HT-2 in a CBD tincture. Four samples contained ZON above the reporting limit. The maximum level found was 47.6 µg/kg in a CBD infused chocolate. Only one sample contained beauvericin above the reporting limit, that was a CBD, turmeric and ginger teabag containing 68.2 µg/kg. Enn B and Enn B1 were also detected in this sample at 13.7 and 8.5 µg/kg respectively. Only eight further samples contained levels of enniatins above the reporting limit.

Nine of the solvents (ethyl ether, hexane, chloroform, benzene, 1,2 Dichloroethane (1,2 DE), trichloroethylene, toluene, m&p-xylene and o-xylene) were not detected in any of the samples above the reporting limit.

Methanol was the most frequently detected solvent in 41 samples. Levels of methanol ranged between 6 and 801 mg/kg. The highest concentration of methanol was measured in a capsule. 25 of the samples had levels of methanol above the MRL of 10 mg/kg. Four of the samples had levels of propan-2-ol above the MRL of 10 mg/kg. Three solvents not on the permitted solvents list were detected above the reporting limit in 24 samples. Acetonitrile was detected in 13, heptane was detected in seven and pentane was detected in five samples.

40 samples did not contain furans above the reporting limit. An additional 14 samples contained furans that could only be tentatively assigned. Three samples of coffee contained levels of each furan above the reporting limit.

The sum of ICES (PCB's 28, 52, 101, 138, 153 and 180) did not exceed 1.30 µg/kg in any sample. The sum of the combination of PCDD/F and PCB WHO TEQ (PCB's 77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167 and 189) ranged between 0.32 and 0.60 ng/kg in all 10 samples.

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References

- ACMD. (2021). *Consumer Cannabidiol (CBD) products*. https://assets.publishing.service.gov.uk/government/uploads/system/uploads/attachment_data/file/1041851/For_publication_-_CBD_products_report_17_December.pdf
- Assimilated Regulation (EC) No 1881/2006. (2006). *Assimilated Regulation (EC) No 1881/2006, of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs*. <https://www.legislation.gov.uk/eur/2006/1881>
- Campbell, S., Paquin, D., Awaya, J. D., & Li, Q. X. (2002). Remediation of Benzo[a]pyrene and Chrysene-Contaminated Soil with Industrial Hemp (*Cannabis sativa*). *International Journal of Phytoremediation*, 4(2), 157–168. <https://doi.org/10.1080/15226510208500080>
- EFSA. (2015). *Scientific Opinion on the risks for human health related to the presence of tetrahydrocannabinol (THC) in milk and other food of animal origin*. <https://doi.org/10.2903/j.efsa.2015.4141>
- EFSA, Arcella, D., Cascio, C., & Mackay, K. (2020). Acute human exposure assessment to tetrahydrocannabinol (D9-THC). *EFSA Journal*, 18(1), 5953. <https://doi.org/10.2903/j.efsa.2020.5953>
- European Commission. (n.d.). *EU Novel Food status Catalogue*. <https://ec.europa.eu/food/food-feed-portal/screen/novel-food-catalogue/search>
- FSA. (2021). *CBD products linked to novel food applications*. <https://www.food.gov.uk/business-guidance/cbd-products-linked-to-novel-food-applications>
- FSA. (2022). *Cannabidiol (CBD) guidance for England and Wales*. <https://www.food.gov.uk/business-guidance/cannabidiol-cbd>
- Fundacion Canna. (n.d.). *Quantification of the concentration of THC, CBD, CBG, THCA, CBDA, CBGA, THCTotal, CBDTTotal and CBGTTotal in HPLC*. <https://www.fundacion-canna.es/en/quantification-concentration-thc>
- Galić, M., Perčin, A., Zgorelec, Ž., & Kisić, I. (2019). Evaluation of heavy metals accumulation potential of hemp (*Cannabis sativa* L.). *Journal of Central European Agriculture*, 20(2). <https://doi.org/10.5513/JCEA01/20.2.2201>
- Hazekamp, A. (2018). The Trouble with CBD Oil. *Medical Cannabis and Cannabinoids*, 65–72. <https://doi.org/10.1159/000489287>
- Home Office. (2019). *Drug Licensing Factsheet-Cannabis, CBD and other cannabinoids*. https://assets.publishing.service.gov.uk/government/uploads/system/uploads/attachment_data/file/825872/factsheet-cannabis-cbd-and-cannabinoids-2019.pdf
- Home Office. (2023). *Government response to the ACMD's advice on consumer CBD products*. <https://www.gov.uk/government/publications/acmd-advice-on-consumer-cannabidiol-cbd-products/government-response-to-the-acmds-advice-on-consumer-cbd-products-accessible-version>

- Narváez, A., Rodríguez-Carrasco, Y., Castaldo, L., Izzo, L., & Ritieni, A. (2020). Ultra-High-Performance Liquid Chromatography Coupled with Quadrupole Orbitrap High-Resolution Mass Spectrometry for Multi-Residue Analysis of Mycotoxins and Pesticides in Botanical Nutraceuticals. *Toxins*, 12(114). <https://doi.org/10.3390/toxins12020114>
- The Food Additives, Flavourings, Enzymes and Extraction Solvents (England) Regulations*. (2013). <https://www.legislation.gov.uk/uksi/2013/2210/contents>
- Walker, M., & Axford, I. (2021). *Government Chemist Guidance: Analytical Limits for Controlled Cannabinoids in Specified Products Containing Cannabidiol (CBD)*. https://assets.publishing.service.gov.uk/government/uploads/system/uploads/attachment_data/file/957590/Government_Chemist_Guidance_on_Analytical_Limits_for_Controlled_Cannabinoids_in_Specified_Products_Containing_Cannabidiol_CBD_22.01.21_.pdf
- Wilcox, J., Pazdanska, M., Milligan, C., Chan, D., MacDonald, S. J., & Donnelly, C. (2019). Analysis of Aflatoxins and Ochratoxin A in Cannabis and Cannabis Products by LC-Fluorescence Detection Using Cleanup with Either Multiantibody Immunoaffinity Columns or an Automated System with In-Line Reusable Immunoaffinity Cartridges. *Journal of AOAC International*, 103. <https://doi.org/10.5740/jaoacint.19-0176>

Supplementary Materials

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