Analysis of CBD Products

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Report of Analysis of CBD Products for Food Standards Agency by Fera Science Ltd.

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Executive summary

The Food Standards Agency commissioned Fera Science Ltd. to carry out a survey to obtain a snapshot of CBD products on sale in England and Wales in order to inform FSA risk assessment of CBD products. Thirty CBD products were purchased from a range of online sellers from England and Wales. Samples comprised of two broad categories: oils and sprays, and edibles

(including beverages). The sampling followed a scheme suggested by FSA. This is not a statistically representative sample of the market and instead provides a snapshot of the current market, to assist the design of future sampling and surveillance activity.

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, 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), pesticides, mycotoxins, CBD content and cannabinoid profiles. Analysis for residual solvents and additional mycotoxins was also carried out, but these were not accredited.

The results of testing found the following:

- Heavy metals (cadmium, mercury & lead) and arsenic were not detected in the majority of samples, meaning levels were below the limits of quantification of the method. Seven samples contained lead, four samples arsenic and two samples contained cadmium. Mercury was not found in any sample. A definitive statement as to whether products exceed maximum levels cannot be made due to uncertainty as to whether products would be classified as a food (i.e. oil) or a food supplement.
- A low incidence of low levels of mycotoxins, with Fusarium mycotoxins found more frequently than aflatoxins and ochratoxin A, mostly at the methods reporting limit. Three samples were found to contain ochratoxin A at the methods reporting limit.
- A total of seven pesticide residues were found across all of the products (each product was tested for over 400 pesticides). There are no specific Maximum Residue Limits (MRL) for CBD products.
- One oil product was found to have PAHs above the regulated levels, if classed as a product for direct consumption. If classed as a food supplement the PAHs were within regulated levels.
- Three samples contained residual solvents. One product was over the MRL.
- Most products contained CBD close to the declared value. Two oils had substantially different levels than that declared (one higher and one lower). CBD was not detected in one of the drink products. These are potentially non-compliant with compositional and standards requirements.
- Delta 9-THC was detected in 87 % (26) of the samples analysed. Of these 40% (12) were found to have THC+ (the total sum of illicit cannabinoids in the product) above the 1mg threshold outlined in current Home Office guidance.

Introduction

Background to the study

There has been a large increase in the number of hemp-based products, both foods and food supplements on sale in the UK. Many of these products are marketed as containing CBD or cannabidiol, a naturally occurring, non-psychoactive constituent of hemp. In January 2019 CBD food products were confirmed as novel foods, and in February 2020 the Food Standards Agency set a deadline of March 2021 for industry to submit a novel food authorisation application for their products. This authorisation process is still on-going, so while products that have submitted a validated application are allowed to remain on the market they are still not authorised as novel foods. Products that do not have an associated validated application are non-compliant and should not be on sale. More information is available on the FSA's webpage on CBD [1], which

currently includes a public list of about 12,100 products which are linked to validated applications, or applications which are well progressed and for which the FSA is awaiting evidence prior to potential validation.

Cannabidiol (CBD) is one of more than one hundred cannabinoids found naturally in the hemp plant (Cannabis sativa). Unlike the psychoactive cannabinoid delta 9-Tetrahydrocannabinol (?9-THC), CBD does not cause the euphoric "high" that is commonly associated with the consumption of narcotics. There are several varieties of Cannabis plant grown, with low THC varieties, typically those that produce <0.2 % ?9-THC in the plant, selected for use in CBD products.

CBD products: safety

There is not comprehensive data to demonstrate the safety of CBD itself. The toxicity of CBD was considered at the Committee of Toxicity (COT) meeting in January 2020. A discussion paper on CBD was reviewed by the Committee on Mutagenicity (COM) in February 2020. In May 2020 the COT discussed potential risks from topically applied CBD. The COT published a Position paper on the potential risk of CBD in CBD food products summarising these discussions in July 2021 (2). The UK Food Standards Agency and Food Standards Scotland published advice to consumers in February 2020 with recommendations that certain vulnerable groups including breastfeeding women and people taking medication should not consume CBD products unless under medical direction, and that healthy adults should consume no more than 70 mg CBD a day (3).

The European Food Safety Authority (EFSA) published an opinion in 2015 (4) that estimated an Acute Reference Dose (ARfD) for ? 9-THC of 1 μ g/kg body weight. In 2020, they also published a report that highlighted that, based on the data available, the EFSA ARfD of 1 μ 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 (5).

In addition to the presence of THC, there is also a potential for certain chemical contaminants to be present 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 polycyclic aromatic hydrocarbons (PAHs) from soil, with these contaminants accumulating in the plants (6, 7). 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 for example, Canada and some US states (8). More recently there have also been reports of some Fusarium mycotoxins in some CBD products of botanical origin (9).

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) (10).

In 2019 CBD was confirmed as a novel food product at a European level and was added to the Novel Food Catalogue (11). In February 2020 the Food Standards Agency 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 (12).

CBD products: controlled drugs

While the Food Standards Agency 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 (13). It states that the product or preparation covered by the exception in the factsheet should not contain more than one milligram 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). 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 product (14).

In January 2022 the Advisory Council on the Misuse of Drugs 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 micrograms (?g) per unit of consumption and the total dose of ?9-THC (including ?9-THCA) and all other controlled phytocannabinoids in consumer CBD products be controlled. This will have to be implemented by a change to the Misuse of Drugs Regulations 2001 (15).

Aims and objectives of the study

The aim of the study was to carry out a survey to obtain a snapshot of CBD products on sale in England and Wales. Due to the ongoing restrictions because of the COVID-19 pandemic, all samples were purchased on-line. Analysis was carried out to inform the FSA's risk assessment and understanding of the composition of CBD products.

Methodology

Samples

Sample purchase and collection was carried out by Fera Science Ltd. The sampling plan was developed in consultation with the FSA who provided a list of suggested products, this was used to plan purchase of samples. This list was designed to give, as far as possible, a representative snapshot of the main types of CBD products that are available for sale in England and Wales. Due to the UK lockdown restrictions at the time, products were purchased on-line. Where samples were not available these were substituted with products, with agreement of FSA, of the same type with a similar declared CBD content. The broad categories of samples were CBD oils or sprays, CBD edibles, and a sample of CBD isolate. The edibles group included gummies, chocolate and ready to drink beverages.

Metals analyses

Samples were analysed for arsenic, cadmium, mercury and lead using In-house Method FSG 461: Totals method (Batch AXQ and AXR), accredited to ISO 17025. Aliquots of homogenised test sample were digested in a mixture of 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 are corrected for reagent blank and spike recovery. The Reporting Limit was calculated from 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).

Polycyclic aromatic hydrocarbons (PAHs) analyses

Samples were analysed for Polycyclic Aromatic Hydrocarbons (PAHs) using In-House Method FSG 410 Extraction of Foods for the Determination of PAHs, accredited to ISO 17025. The method is able to determine 28 PAHs, including the four regulated (marker) PAH compounds benzo[a]pyrene (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 DMF/cyclohexane partition followed by elution through a silica gel column. Analysis was by gas chromatography mass spectrometry (GCMS).

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 reversed phase HPLC using UV detection, accredited to ISO 17025 for CBD in oils and oil products. The seven cannabinoids included in the method are:

- Cannabichromene (CBC)
- Cannabidiol (CBD)
- Cannabidiolic acid (CBDA)
- Cannabigerol (CBG)
- Cannabinol (CBN)
- ?9-Tetrahydrocannabinol (?9-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 μ m, 25 mm PTFE syringe filter then analysed by reverse phase HPLC using gradient elution and detection by UV at 220nm.

Quality control samples including procedural blanks, in-house reference samples and spiked samples were included in each batch. Results are UKAS accredited (ISO 17025 by flexible scope) for the oil and oil products samples. This method was used to quantify high levels of CBD, and confirm levels of the other cannabinoids listed that were determined by LC-MS/MS.

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 liquid chromatography tandem quadrupole mass spectrometry (LC-MS/MS), accredited to ISO 17025 by Flexible Scope for oil and oil products. 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)
- ?9-Tetrahydrocannabinol (?9-THC)
- ?9-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 diluted with a methanol: water mixture (90 : 10) and THC-d3 ISTD solution. If the extract was cloudy, it was filtered through a 0.2 μ m, 25 mm PTFE syringe filter then analysed by LC-MS/MS.

Quality control samples including procedural blanks, in-house reference samples and spiked samples were included in each batch. Results are UKAS accredited (ISO 17025), following accreditation of the method via Flexible Scope for the oil and oil products samples.

CBD content by NMR

A single sample of CBD isolate was analysed by qNMR to determine purity. The sample was analysed in duplicate, quality control samples (in-house reference material and an internal standard) were included in the analysis.

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 liquid chromatography with mass spectrometric detection (HPLC-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) GCMS – 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.

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. 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 batch as quality control samples.

Analysis for zearalenone was carried out using In-House method FSG 258 Determination of zearalenone using immunoaffinity column clean-up and HPLC. Samples were extracted with a mixture of acetonitrile and water, cleaned-up by immunoaffinity column and analysed using reverse phase HPLC. A blank sample and spiked sample (in the absence of an in-house reference sample) were included in the batch as quality control samples. The reporting limit was 10 µg/kg.

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, zearalenone, 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.

Deoxynivalenol, 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 batch as quality control samples. The reporting limit was 5 μ g/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 inhouse reference sample) were included in the batch as quality control samples. The reporting limit was 1.25 µg/kg for each mycotoxin.

Residual solvent analyses by Headspace GC MS

Samples were analysed using In-House SOP FSG 790. Samples are dissolved in N,N-Dimethylacetamide, sonicated then an aliquot was transferred to a headspace vial containing salt and water. Residual solvents were measured by Headspace GC-MS. The following solvents were measured:

- 1,2 dichloroethane (1,2 DE)
- Acetone
- Acetonitrile (ACN)
- Benzene
- Butane
- Chloroform
- Ethanol
- Ethyl acetate
- Ethyl ether
- Ethylene oxide
- Heptane
- Hexane
- Isopropyl alcohol
- Methanol
- Methylene chloride
- Pentane
- Toluene
- Xylenes (ortho, meta and para) (m p xylene, o xylene)
- Trichloroethylene

Results and discussion

Metals analyses

Metals results are given below in Table 1, and QC results are given in Table 2 (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 Retained EU Law 1881/2006 (16). Results have been compared against relevant legal maximum levels where appropriate.

Most samples contained levels below the limits of quantification. Lead was the most frequently detected metal, seven samples contained lead above the reporting limit. Levels found ranged from 0.008 to 0.351 mg/kg. The highest level of 0.351 mg/kg was found in sample S21-011617 a Full spectrum CBD oil. Maximum levels for lead in Retained EU Law 1881/2006 are 0.10 mg/kg for fats and oils (including milk fat), and 3 mg /kg for food supplements. The highest level found in this study falls within this range, however due to uncertainty as to whether the product would not be regulated as a food (i.e. oil) or a food supplement it is difficult to make a definitive statement as to whether the product exceeds a maximum level.

Four of the seven samples contained lead only, the other three samples also contained residues of arsenic (S21-011600, at 0.006 mg/kg) or arsenic and cadmium, S21-011609 at 0.021 mg/kg (As) and 0.049 mg/kg (Cd) and S21-011619 at 0.05 mg/kg (As) and 0.006 mg/kg (Cd). Four samples contained arsenic, levels ranged from 0.005 to 0.021 mg/kg, sample S21-011605 contained arsenic only. The highest arsenic level was found in sample S21-011609.

Cadmium was measured in 2 samples at 0.006 and 0.049 mg/kg, samples S21-011619 and S21-011609 respectively, both were chocolate samples. Maximum levels for cadmium in chocolate in Retained Regulation (EC) 1881/2006 range from 0.10 to 0.80 mg/kg depending on the cocoa solids content. Sample S21-011609 was a dark chocolate that contained 70 % cocoa solids, so the limit of 0.80 mg/kg would apply, meaning the cadmium level was compliant. Sample S21-011619 was a 'white' chocolate that contained 0.006 mg/kg cadmium. The maximum level for milk chocolate with <30% cocoa solids is 0.10 mg/kg, so sample S21-011619 was compliant. Mercury was not found in any sample.

PAH analyses: results of BAP and SUM PAH4

The results of the PAH analyses are given below in Table 3. and QC results in Table 4. All results have been corrected for recovery. Results for benz[a]pyrene and the sum of PAH4 (benzo[a]pyrene (BAP), benzo[a]anthracene, benzo[b]fluoranthene and chrysene) are highlighted. These are the compounds for which MPLs are established in legislation. Retained EU Law 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 and also sets maximum levels of 2 µg/kg for BAP and 10 µg/kg for PAH4 in oils and fats intended for direct human consumption or use as an ingredient in food and 5 µg/kg BAP and 30 µg/kg PAH4 fat in cocoa beans and derived products.

The highest level of benzo[a]pyrene found was 3.36 μ g/kg in sample S21-011619. This sample was a chocolate product and contained 13.91 μ g/kg PAH4 Sum. In this case the limit for cocoa beans and derived products would apply and the sample is compliant. The highest BAP level of 3.19 /kg and PAH4 Sum level 27.15 μ g/kg were found in sample S21-011605, this was described as refined hempseed oil. If this sample is classed as an oil for direct consumption then these levels are non-compliant (3.19 ± 0.57 /kg, and 27.15 ± 2.44 μ g/kg) as they exceed the maximum levels of 2 μ g/kg for BAP and 10 μ g/kg for PAH4. If the sample is classed as a food supplement the sample would be deemed compliant. Sample S21-011620 contained the second highest PAH4 sum level of 21.57 μ g/kg, this was labelled as a food supplement, in which case the maximum level of 50 μ g/kg for PAH4 would apply and the product is compliant.

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 3. These results are corrected for recovery.

Sample S21-011620 contained the highest overall PAH content, with residues found for 21 / 28 of the PAHs included in the method. This was a 'full spectrum' CBD oil. Sample S21-011605 contained the second highest level with 22 / 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. Six samples contained no PAH residues above the reporting limit, these were the beverages, a 'liposomal' spray, the gummies and the CBD isolate.

CBD analyses: by HPLC-UV

Results for CBD products are given in Table 5 for oils and liquids (drops & sprays) and Table 6 for other products. A comparison of the labelled or declared CBD content is also given.

The results presented are not corrected for recovery. Recovery values are given in Table 7, recovery was 99 - 111 %. Expanded measurement uncertainty for this analysis was 12%. For the oil and liquid samples the majority of CBD values determined were in good agreement with the stated values. In most cases the concentration measured was within the expected value as stated on the label of the product, particularly when recovery and measurement uncertainty were taken into account. The lowest level measured was 0.5 % in sample S21-011613, but this agreed with the amount claimed on the label (0.6 %). If anything, the values measured tended to be slightly higher than the label claim. The largest difference was seen for sample S21-011615, which was labelled as 10 % CBD, but a concentration of 20.7 % was measured. It is important that the actual content of foods should not deviate substantially from labelled amounts, as the consumer could otherwise be mislead.

The results for the edibles were more variable, the two beverages were both found to contain less CBD than claimed. Sample S21-011607 contained 10 mg per can, rather than the 15 mg on the label, and no CBD was detected in sample S21-011604 although the label stated it contained 5 mg. The CBD levels in the two chocolate bars were close to the labelled values, while the gummy sample contained half the declared amount (10 mg versus 20 mg per gummy on the label).

The CBD mint sample was found to contain 3.51 % CBD. The declared content was 10 mg CBD per mint. Each mint weighed 0.3 g (300 mg), so the measured CBD content of each mint was 10.5 mg, matching the declared content.

The CBD isolate, sample S21-011601 was analysed in duplicate by qNMR to determine purity, as it is not possible to measure the CBD content of this type of sample using LC-UV or LC-MSMS. The purity was determined as 98.9 +/-0.5%. The product was described as 99.9% pure.

Cannabinoid analyses by LC MS/MS

The results of the cannabinoid suite analyses are given in Tables 8 and 10 give results for nonpsychoactive cannabinoids and 9 and 11 for controlled cannabinoids. Products in table 9 and 11 which have results of THC+ above 1mg, exceed the threshold set out in the Home Office's guidance. Fourteen cannabinoids were determined by the LC-MS/MS method, although in effect the method does not measure CBD as the concentration in most samples is outside its working range. 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.

A standard approach for this analysis is to consider the levels of the cannabinoids ? 9-THC, as the main controlled drug, and also ? 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 (17). The sum value was calculated as a lower bound value, i.e. results less than the 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.) the absolute amount, in mg, in each individual item was also calculated.

The data is reported as 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.

The method was accredited to ISO 17025 by Flexible Scope accreditation, applicable for the oil and oil product samples, QC data for the LC-MS/MS analysis in this study is given in Tables 12 and 13. In most cases the recovery values are within the acceptable criteria ranges. Measurement uncertainty (MU) calculations were carried out for all cannabinoids in this method. It was determined that the expanded MU ranged from 17 - 37% depending on the analyte. The expanded MU for ? 9-THC was 17 %.

Pesticide Analysis results

All 30 samples were screened for over 400 pesticide compounds. A full list of all pesticides tested and their reporting limits are given in Annex A. Seven residues above the reporting limit were found in five samples. A summary of the residues found is given in Table 14.

Sample S21-011593, a gummy, contained BAC12 at 0.1 mg/kg. Four samples of CBD oils, contained residues: Sample S21-011595 contained residues of fenpyroximate (0.05 mg/kg), pirimiphos-methyl (0.03 mg/kg), and tebufenpyrad (0.1 mg/kg). Sample S21-011605 contained propamocarb (free base) at 0.01 mg/kg, S21-011606 contained chlorpyrifos at 0.01 mg/kg and S21-011621 contained pirimiphos-methyl at 0.02 mg/kg. There are no specific Maximum Residue Limits (MRLs) for CBD products.

Mycotoxins - HPLC FLD Analysis results

The samples were analysed using immunoaffinity column clean-up, and results are given in Table 15. Aflatoxins were not detected in any sample above the reporting limit of 0.2 μ g/kg. Three samples contained very low levels of 0.2 μ g/kg ochratoxin A, these were samples S21-011601, S21-011609 and S21-011618. Sample S21-011609 also contained trace levels of aflatoxin B1 and G2 (0.1 μ g/kg) but these were below the reporting limit. The recovery values for aflatoxins and ochratoxin A were higher than the usual accepted range, 147-156% for aflatoxins and 137% for ochratoxin A, this was thought to be due to phase separation in the extract causing the analytes to be concentrated. As no significant residues were detected this did not affect the results.

Zearalenone analysis was carried out by HPLC-FLD, all samples were below the reporting limit of this method of 10 μ g/kg. However, 2 samples contained measurable levels below the reporting limit. Sample S21-011609 contained 4.4 μ g/kg and sample S21-011619 contained 7.5 μ g/kg. These findings were confirmed by LC-MS/MS analysis. There are no applicable maximum levels for zearalenone in these types of foods.

Mycotoxins – LC-MS/MS Analysis results

Analysis for deoxynivalenol, T-2 toxin and HT-2 toxin were carried out using immunoaffinity column clean-up and LC-MS/MS, results are given in Table 16, quality control data for LC-MS/MS analysis is given in Table 17. One sample (S21-011611) contained a trace level of deoxynivalenol, but it was below the reporting limit of 5 μ g/kg. The same sample contained the highest level of T-2 toxin (44 μ g/kg) and HT-2 toxin (8.3 μ g/kg). Three other samples contained T-2 toxin above the reporting limit, these were S21-011595 (7.2 μ g/kg), S21-011605 (6.4 μ g/kg) and S21-011621 (5.5 μ g/kg). Two samples contained trace levels of T-2 toxin, these were below the reporting limit but the indicative levels have been reported in Table 16. One of these was the only other sample that contained HT-2 toxin above the reporting limit, sample S21-011606 contained 5.1 μ g/kg. There are no maximum levels in force for T-2 and HT-2 toxin.

Results for enniatins and beauvericin are also reported in Table 16. The majority of the results were below the reporting limit of 1.25 μ g/kg. Only one sample contained beauvericin above the reporting limit, that was sample S21-011609 that contained 1.6 μ g/kg. It also contained low concentrations of enniatins B and B1 at levels of 1.3 and 1.5 μ g/kg respectively. Five other samples contained low levels of enniatin B and B1, these were S21-01603 (1.3 μ g/kg enniatin B1), S21-011605 (4.5 μ g/kg each enniatin B and B1), S21-011606 (enniatin B1, 1.6 μ g/kg), S21-011609 (1.3 and 1.5 μ g/kg enniatin B and B1), S21-011611 (1.3 and 2.3 μ g/kg enniatin B and B1), S21-011613 (5.4 μ g/kg enniatin B1) and S21-011620 (1.2 and 1.3 μ g/kg enniatin B and B1). These results confirm the findings of Narváez et al (9), who reported the occurrence of zearalenone, T-2 toxin, HT-2 toxin, and enniatins in herbal CBD products. It is the first report of beauvericin, albeit at a very low concentration.

Residual Solvents results

Samples were analysed for residual solvents using HS-GC-MS/MS, results are given in Table 18. The limits of quantification varied depending on the solvent. Dichloromethane (DCM), 1,2

Dichloroethane (1,2 DE), chloroform, benzene and trichloroethylene had limits of quantification of 0.6 mg/kg. Acetone, ethanol, ethyl acetate, ethyl ether, heptane, hexane, pentane, isopropyl alcohol (propan-2-ol), toluene, meta, para and ortho xylene had limits of quantification of 6 mg/kg each. The LOQ for methanol was 9 mg/kg and for acetonitrile was 20 mg/kg.

Only permitted extraction solvents as defined in the Food Additives, Flavourings, Enzymes and Extraction Solvent Regulations 2013 (18) can be used in the production of food placed on the market. Across all 30 samples only 3 residues were detected, all of which are permitted extraction solvents. These were ethanol (72 mg/kg) in sample S21-011607, a canned beverage, and sample S21-011613 (1560 mg/kg), a liposomal spray. Propan-2-ol (13 mg/kg) was found in sample S21-011616, a CBD oil, which exceeds the MRL in extracted foodstuff or food ingredient of 10mg/kg, set out in retained EU Directive 2009/32/EC (19). All other results were below the respective reporting limits (or LOQs).

Summary and conclusions

Thirty CBD products were purchased from a range of online sellers from England and Wales following a sampling protocol provided by FSA. Samples comprised 23 oils and sprays, 6 edibles (1 gummy, 2 chocolate bars, 1 mint and 2 ready to drink beverages) and 1 sample of CBD isolate.

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 ? 9-THC. Results reported in this study are UKAS accredited, except for CBD and cannabinoids in confectionary & beverages, pesticides in gummies. Enniatins, beauvericin, ZON, DON, T-2 and HT-2 toxins are out of scope and are not accredited.

Most samples contained heavy metals levels below the limits of quantification. Lead was the most frequently detected metal, seven samples contained lead above the reporting limit. The highest level of lead found was 0.351 mg/kg in a full spectrum CBD oil. Four samples contained arsenic, levels ranged from 0.005 to 0.021 mg/kg. Cadmium was measured in 2 samples at 0.006 and 0.049 mg/kg, both were chocolate samples. Mercury was not found in any sample.

The highest level of benzo[a]pyrene found was $3.36 \ \mu$ g/kg, this was a chocolate product and contained $13.91 \ \mu$ g/kg PAH4 Sum. The highest PAH4 Sum level found was $27.15 \ \mu$ g/kg, this sample was described as refined hempseed oil. If this sample is classed as an oil for direct consumption then these levels are non-compliant when measurement uncertainty is taken into account. Other PAH residues were found, e.g. a 'full spectrum' CBD oil contained 21 / 28 of the PAHs included in the method, while a refined hempseed oil contained 22 / 28 PAHs. The samples that contained the highest levels of BAP and PAH4 also contained the highest levels of the other PAHs. Six samples contained no PAH residues above the reporting limit, these were the beverages, a 'liposomal' spray, the gummies and the CBD isolate.

For oils most samples contained close to the declared value of CBD. Two samples contained higher than the declared amount, with one sample S21-011615, containing double.

The results for the edibles were more variable, the two beverages were both found to contain less CBD than claimed, no CBD was detected in one sample. The CBD levels in the two chocolate bars were close to the labelled values, while the gummies contained half the declared amount. The sample of isolate was found to have a purity of 98.9 +/-0.5%.

Samples were also tested for a range of wider cannabinoids and the results of these can be found in the tables at Annex B.

There was a low incidence of low levels of mycotoxins, with Fusarium mycotoxins found more frequently than aflatoxins and ochratoxin A. This supported recent literature reports, however the levels found were mainly at or close to the method reporting limits.

A low incidence of pesticide residues was determined, only 7 residues were found in 5 of the 30 products.

Three residues of solvents, two of ethanol and one of propan-2-ol, were found across all the samples. These are solvents that are permitted for use in food production; however, the propan-2-ol sample exceeded the MRL. No other solvent residues were found above the LOQ.

Delta 9-THC was detected in 87% (26) of the samples analysed, of these 40% (12) were found to have THC+ (the total sum of illicit cannabinoids in the product) above the legal limit of 1mg threshold outlined in current Home Office guidance.

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Annex A: Pesticides analysed and their reporting limits

Pesticide	RL/mg/kg
2,4-D	<0.01
2,4-DB	<0.01
2-phenylphenol	<0.05
6-benzyl aminopurine	<0.01
abamectin	<0.01
acetamiprid	<0.01
acetochlor	<0.01
acibenzolar-S-methyl	<0.01
aclonifen	<0.01

Pesticide	RL/mg/kg
acrinathrin	<0.01
alachlor	<0.01
aldicarb	<0.01
aldicarb sulfone	<0.01
aldicarb sulfoxide	<0.01
aldrin	<0.01
allethrin	<0.01
ametoctradin	<0.01
amidosulfuron	<0.01
asulam	<0.01
atrazine	<0.01
azinphos-ethyl	<0.01
azinphos-methyl	<0.01
azoxystrobin	<0.01
BAC10	<0.05
BAC12	<0.05
BAC14	<0.05
BAC16	<0.05

Pesticide	RL/mg/kg
benalaxyl	<0.01
bendiocarb	<0.01
benthiavalicarb-isopropyl	<0.01
bifenox	<0.01
bifenthrin	<0.01
bispyribac-sodium	<0.01
biteranol	<0.01
bixafen	<0.01
boscalid	<0.01
bromophos-ethyl	<0.01
bromopropylate	<0.01
bromoxynil	<0.01
bromuconazole	<0.01
bupirimate	<0.01
buprofezin	<0.01
butachlor	<0.01
butocarboxim	<0.01
butocarboxim sulfoxide	<0.01

Pesticide	RL/mg/kg
cadusafos	<0.01
carbaryl	<0.01
carbendazim	<0.01
carbetamide	<0.01
carbofuran	<0.001
carbofuran (3-hydroxy)	<0.001
carboxin	<0.01
chlorantraniliprole	<0.01
chlorbufam	<0.01
chlordane (cis)	<0.01
chlordane (trans)	<0.01
chlorfenapyr	<0.01
chlorfenvinphos	<0.01
chlorfluazuron	<0.01
chloridazon	<0.01
chlorobenzilate	<0.01
chlorothalonil	<0.01
chlorpropham	<0.05

Pesticide	RL/mg/kg
chlorpyrifos	<0.01
chlorpyrifos-methyl	<0.01
chlorthal-dimethyl	<0.01
chlortoluron	<0.01
chlozolinate	<0.01
chromafenozide	<0.01
clethodim	<0.02
clofentezine	<0.01
clomazone	<0.01
clothianidin	<0.01
coumaphos	<0.01
cyanazine	<0.01
cyazofamid	<0.01
cycloate	<0.05
cycloxydim	<0.01
cyflufenamid	<0.01
cyfluthrin	<0.05
cyhalofop butyl	<0.01

Pesticide	RL/mg/kg
cyhalothrin-lambda	<0.02
cymoxanil	<0.01
cypermethrin	<0.05
cyproconazole	<0.01
cyprodinil	<0.01
cyromazine	<0.01
DDAC	<0.1
DDD-pp	<0.02
DDE-pp	<0.01
DDT-op	<0.01
DDT-pp	<0.01
deltamethrin	<0.02
demeton-S-methyl	<0.01
demeton-S-methyl sulfone	<0.01
desmedipham	<0.01
diafenthiuron	<0.01
diazinon	<0.01
dichlobenil	<0.01

Pesticide	RL/mg/kg
dichlofluanid	<0.05
dichlorprop	<0.01
dichlorvos	<0.05
diclobutrazol	<0.01
dicloran	<0.01
dicofol	<0.01
dicrotophos	<0.01
dieldrin	<0.01
diethofencarb	<0.01
difenoconazole	<0.01
diflubenzuron	<0.01
diflufenican	<0.01
dimethenamid	<0.01
dimethoate	<0.01
dimethomorph	<0.01
dimoxystrobin	<0.01
diniconazole	<0.01
dinotefuran	<0.01

Pesticide	RL/mg/kg
diphenylamine	<0.01
disulfoton	<0.02
disulfoton sulfone	<0.01
disulfoton sulfoxide	<0.01
diuron	<0.01
DMF	<0.05
DMPF	<0.05
DMSA	<0.01
dodine	<0.01
emamectin benzoate	<0.01
endosulfan (I)	<0.01
endosulfan (II)	<0.01
endosulfan sulfate	<0.01
endrin	<0.01
EPN	<0.01
epoxiconazole	<0.01
EPTC	<0.05
ethiofencarb	<0.01

Pesticide	RL/mg/kg
ethiofencarb sulfone	<0.01
ethiofencarb sulfoxide	<0.01
ethion	<0.01
ethiprole	<0.01
ethirimol	<0.01
ethofumesate	<0.01
ethoprophos	<0.01
etofenprox	<0.01
etoxazole	<0.01
etridiazole	<0.01
etrimfos	<0.01
famoxadone	<0.01
fenamidone	<0.01
fenamiphos	<0.01
fenamiphos sulfone	<0.01
fenamiphos sulfoxide	<0.01
fenarimol	<0.01
fenbuconazole	<0.01

Pesticide	RL/mg/kg
fenbutatin oxide	<0.01
fenhexamid	<0.01
fenitrothion	<0.01
fenoprop	<0.01
fenoxycarb	<0.01
fenpropathrin	<0.02
fenpropidin	<0.01
fenpropimorph	<0.01
fenpyrazamine	<0.01
fenpyroximate	<0.01
fensulfothion	<0.01
fensulfothion sulfone	<0.01
fensulfothion-oxon	<0.01
fensulfothion-oxon-sulfone	<0.01
fenthion	<0.01
fenthion sulfone	<0.01
fenthion sulfoxide	<0.01
fentin acetate	<0.02

Pesticide	RL/mg/kg
fenvalerate	<0.02
fipronil	<0.002
fipronil de-sulfinyl	<0.002
fipronil sulfone	<0.002
flonicamid	<0.01
fluazifop (free acid)	<0.01
fluazifop-p-butyl	<0.01
fluazinam	<0.01
flubendiamide	<0.01
flucythrinate	<0.01
fludioxonil	<0.01
flufenacet	<0.01
flufenoxuron	<0.01
fluometuron	<0.01
fluopicolide	<0.01
fluopyram	<0.01
fluoxastrobin	<0.01
fluquinconazole	<0.01

Pesticide	RL/mg/kg
flurochloridone	<0.01
fluroxypyr	<0.01
flusilazole	<0.01
flutolanil	<0.01
flutriafol	<0.01
fluvalinate	<0.01
fluxapyroxad	<0.01
fonofos	<0.01
formetanate-HCI	<0.01
fosthiazate	<0.01
furalaxyl	<0.02
furathiocarb	<0.001
halofenozide	<0.01
halosulfuron-methyl	<0.01
haloxyfop (free acid)	<0.01
HCH-alpha	<0.01
HCH-beta	<0.01
HCH-gamma	<0.01

Pesticide	RL/mg/kg
heptachlor	<0.01
heptachlor epoxide-cis	<0.01
heptachlor epoxide-trans	<0.01
heptenophos	<0.01
hexachlorobenzene	<0.01
hexaconazole	<0.01
hexazinone	<0.01
hexythiazox	<0.01
imazalil	<0.05
imidacloprid	<0.01
indoxacarb	<0.01
ioxynil	<0.01
iprodione	<0.02
iprovalicarb	<0.01
isazofos	<0.01
isocarbofos	<0.01
isofenphos	<0.05
isofenphos-methyl	<0.01

Pesticide	RL/mg/kg
isoprocarb	<0.01
isoprothiolane	<0.01
isoproturon	<0.01
isopyrazam	<0.01
isoxaben	<0.01
isoxaflutole	<0.01
kresoxim-methyl	<0.01
lenacil	<0.01
linuron	<0.01
lufenuron	<0.01
malaoxon	<0.01
malathion	<0.01
mandipropamid	<0.01
МСРА	<0.01
МСРВ	<0.01
mecarbam	<0.01
mecoprop	<0.01
mepanipyrim	<0.01

Pesticide	RL/mg/kg
pyrethrins	<0.01
pyridaben	<0.02
pyridalyl	<0.01
pyridaphenthion	<0.01
pyrifenox	<0.01
pyrimethanil	<0.01
pyriproxyfen	<0.01
quassia	<0.01
quinalphos	<0.01
quinmerac	<0.01
quinoclamine	<0.1
quinoxyfen	<0.01
quintozene	<0.01
quizalofop P	<0.05
rimsulfuron	<0.01
rotenone	<0.01
simazine	<0.01
spinetroram	<0.01

Pesticide	RL/mg/kg
spinosad	<0.01
spiromesifen	<0.01
spirotetramat	<0.01
spirotetramat enol	<0.01
spiroxamine	<0.01
sulcotrione	<0.01
sulfoxaflor	<0.01
tebuconazole	<0.01
tebufenozide	<0.01
tebufenpyrad	<0.01
tebupirimphos	<0.01
tebuthiuron	<0.01
tecnazene	<0.01
teflubenzuron	<0.01
tefluthrin	<0.01
tepraloxydim	<0.01
terbufos	<0.01
terbufos sulfone	<0.01

Pesticide	RL/mg/kg
terbufos sulfoxide	<0.01
terbuthylazine	<0.01
terbutryn	<0.01
tetrachlorvinphos	<0.01
tetraconazole	<0.01
tetradifon	<0.01
tetrahydrophthalimide	<0.05
tetramethrin	<0.02
TFNA	<0.01
TFNG	<0.01
thiabendazole	<0.01
thiacloprid	<0.01
thiamethoxam	<0.01
thiodicarb	<0.01
thiophanate-methyl	<0.01
tolclofos-methyl	<0.01
tolfenpyrad	<0.01
tolylfluanid	<0.01

Pesticide	RL/mg/kg
triadimefon	<0.01
triadimenol	<0.01
triallate	<0.01
triasulfuron	<0.01
triazamate (free acid)	<0.01
triazophos	<0.01
triclopyr	<0.01
tricyclazole	<0.01
trifloxystrobin	<0.01
triflumizole	<0.01
triflumuron	<0.01
trifluralin	<0.01
triforine	<0.01
triticonazole	<0.01
vinclozolin	<0.01
zoxamide	<0.01

Annex B: Tables

Table 1 Metals results for CBD products (mg/kg) corrected for recovery

Metal element concentration (mg/kg)

Sample ID	Sample description	Arsenic	Cadmium	Mercury	Lead
s21- 011592	Oral drops/spray	<0.005	<0.005	<0.005	<0.005
s21- 011593	CBD Gummies	<0.005	<0.005	<0.005	<0.005
s21- 011594	CBD Oil	<0.005	<0.005	<0.005	0.020 (a)
s21- 011595	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011596	CBD Oil	<0.005	0.021 (a)	<0.005	0.183 (a)
s21- 011597	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011598	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011599	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011600	CBD Edible mint sweet	0.006 (a)	<0.005	<0.005	0.010 (a)
s21- 011601	CBD Isolate	<0.005	<0.005	<0.005	<0.005
s21- 011602	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011603	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011604	Beverage	<0.005	<0.005	<0.005	<0.005

Sample ID	Sample description	Arsenic	Cadmium	Mercury	Lead
s21- 011605	CBD Oil	0.018 (a)	<0.005	<0.005	<0.005
s21- 011606	CBD Oil	<0.005	<0.005	<0.005	<0.005

Results above the reporting limit are marked with an A in brackets.

Table 1 continued metals results for CBD products (mg/kg) corrected for recovery

Metal element concentration (mg/kg)

Sample ID	Sample description	Arsenic	Cadmium	Mercury	Lead
s21- 011607	Beverage	<0.005	<0.005	<0.005	<0.005
s21- 011608	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011609	CBD Edible (chocolate)	0.021 (a)	0.049 (a)	<0.005	0.033 (a)
s21- 011610	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011611	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011612	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011613	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011614	CBD Oil	<0.005	<0.005	<0.005	<0.005

Sample ID	Sample description	Arsenic	Cadmium	Mercury	Lead
s21- 011615	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011616	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011617	CBD Oil	<0.005	<0.005	<0.005	0.351
s21- 011618	CBD Oil	<0.005	<0.005	<0.005	<0.005
s21- 011619	CBD Edible (chocolate)	0.005 (a)	0.006 (a)	<0.005	0.012 (a)
s21- 011620	CBD Oil	<0.005	<0.005	<0.005	0.008 (a)
s21- 011621	CBD Oil	<0.005	<0.005	<0.005	<0.005

Results above the reporting limit are marked with an A in brackets.

Table 2 Metals Analysis QC data

Recovery percentages:

- Arsenic: 99%
- Cadmium: 103%
- Mercury: 98%
- Lead: 97%

Recovery (%)	Analysis	Type of result	Arsenic	Cadmium	Mercury	Lead
LoD	-	-	0.005	0.005	0.005	0.005
BVL-LVU	BVL Liver	Lab result	0.716	0.148	0.147	0.492

Recovery (%)	Analysis	Type of result	Arsenic	Cadmium	Mercury	Lead
Ref.	-	Reference value	0.764	0.159	0.129	0.459
NIST 1548a	Typical Diet	Lab result	0.174	0.03	0.003	0.047
Ref.	-	Reference value	0.200	0.035	-0.005	0.044
TNRL03	Wheat flour	Lab result	0.321	0.151	0.207	0.248
Ref.	-	Reference value	0.320	0.164	0.172	0.270

Table 3: Results of PAHs in CBD products

Sample	S21- 011592	S21- 011593	S21- 011594	S21- 011595	S21- 011596
acenaphthylene	0.29	<0.19	4.20	0.15	2.42
acenaphthene	<1.33	<1.1	1.81	<1.15	1.87
fluorene	<1.32	<1.09	5.58	<1.37	4.54
phenanthrene	<1.97	<1.64	40.12	3.93	15.58
anthracene	0.12	<0.09	4.78	0.97	1.84
fluoranthene	<1.24	<1.03	19.11	<1.52	5.85
benzo(c)fluorene	<0.02	<0.02	0.29	<0.03	0.11
pyrene	<1.31	<1.08	12.75	<1.95	3.78

Sample	S21- 011592	S21- 011593	S21- 011594	S21- 011595	S21- 011596
benzo[ghi]fluoranthene	<0.03	<0.03	1.66	<0.15	0.42
benzo[a]anthracene	<0.04	<0.03	2.76	<0.07	0.97
benzo[b] napthol [2,1, dithiophene]	<0.04	<0.03	0.62	<0.04	0.31
cyclopenta[cd]pyrene	<0.02	<0.02	0.24	<0.03	0.04
chrysene	<0.13	<0.11	2.92	0.22	1.18
5-methylchrysene	<0.01	<0.01	0.07	<0.01	<0.06
benzo[b]fluoranthene	<0.15	<0.12	2.36	<0.23	0.83
benzo[j]fluoranthene	<0.03	<0.02	1.32	<0.06	0.37
benzo[k]fluoranthene	<0.04	<0.03	1.19	<0.08	0.38
benzo[e]fluoranthene	<0.07	<0.06	2.21	<0.12	0.88
benzo[a]pyrene	<0.36	<0.3	2.14	<0.20	0.63
indeno[1,2,3-cd]pyrene	<0.3	<0.25	1.68	<0.33	0.56
dibenzo[ah]anthracene	<0.34	<0.28	0.42	<0.39	<0.4
benzo[ghi]perylene	<0.14	<0.12	1.52	<0.15	0.40
anthanthrene	<0.16	<0.13	<0.18	<0.17	<0.17
dibenzo[a,l]pyrene	<0.69	<0.57	<0.59	<0.58	<0.6
dibenzo[a,e]pyrene	<0.94	<0.78	<1.05	<1.03	<1.07
dibenzo[a,i]pyrene	<1.08	<0.9	<1.18	<1.16	<1.2

Sample	S21- 011592	S21- 011593	S21- 011594	S21- 011595	S21- 011596
dibenzo[a,h]pyrene	<1.14	<0.95	<1.13	<1.11	<1.15
coronene	<0.22	<0.18	<0.21	<0.21	<0.22
PAH 4 SUM Upper µg/kg	0.68	0.56	10.18	0.72	3.61
PAH 4 SUM Lower µg/kg	<0.01	<0.01	10.18	0.22	3.61

Table 3: Continued results of PAHs in CBD products

Sample	S21- 011597	S21- 011598	S21- 011599	S21- 011600	S21- 011601
acenaphthylene	0.10	1.03	0.70	0.42	<0.22
acenaphthene	<0.13	<1.16	<1.18	<1.47	<1.32
fluorene	<0.13	<1.37	<1.39	<1.46	<1.31
phenanthrene	<0.2	2.08	4.56	<2.18	<1.96
anthracene	<0.01	0.13	0.37	0.15	<0.11
fluoranthene	<0.12	<1.53	<1.55	<1.37	<1.23
benzo(c)fluorene	<0.01	<0.06	0.08	<0.02	<0.02
pyrene	0.31	<1.97	2.26	<1.44	<1.3
benzo[ghi]fluoranthene	0.03	<0.15	0.19	<0.03	<0.03
benzo[a]anthracene	<0.01	0.09	0.29	0.05	<0.05
benzo[b] napthol [2,1, dithiophene]	<0.01	<0.03	0.25	<0.04	<0.04

Sample	S21- 011597	S21- 011598	S21- 011599	S21- 011600	S21- 011601
cyclopenta[cd]pyrene	<0.03	<0.03	0.06	<0.03	<0.02
chrysene	0.02	0.29	0.90	<0.14	<0.13
5-methylchrysene	<0.01	<0.01	<0.02	<0.01	<0.01
benzo[b]fluoranthene	0.02	0.27	0.69	<0.16	<0.15
benzo[j]fluoranthene	<0.01	<0.06	0.12	<0.03	<0.04
benzo[k]fluoranthene	<0.01	<0.08	0.10	<0.04	<0.04
benzo[e]fluoranthene	0.02	<0.12	0.65	<0.08	<0.07
benzo[a]pyrene	<0.04	<0.23	<0.31	<0.39	<0.35
indeno[1,2,3-cd]pyrene	<0.03	<0.33	<0.34	<0.33	<0.29
dibenzo[ah]anthracene	<0.03	<0.39	<0.39	<0.37	<0.33
benzo[ghi]perylene	<0.01	<0.15	0.30	<0.15	<0.14
anthanthrene	<0.10	<0.17	<0.17	<0.18	<0.16
dibenzo[a,l]pyrene	<0.10	<0.58	<0.59	<0.76	<0.69
dibenzo[a,e]pyrene	<0.10	<1.04	<1.05	<1.04	<0.93
dibenzo[a,i]pyrene	<0.11	<1.17	<1.18	<1.20	<1.07
dibenzo[a,h]pyrene	<0.11	<1.17	<1.18	<1.20	<1.07
coronene	<0.10	<0.21	<0.44	<0.24	<0.22
PAH 4 SUM Upper µg/kg	0.09	0.88	2.19	0.74	0.68

Sample	S21-	S21-	S21-	S21-	S21-
	011597	011598	011599	011600	011601
PAH 4 SUM Lower µg/kg	0.04	0.65	1.88	0.05	<0.01

Sample	S21- 011602	S21- 011603	S21- 011604	S21- 011605	S21- 011606
acenaphthylene	1.41	1.42	<0.03	6.67	4.38
acenaphthene	2.16	1.57	<0.12	4.38	2.88
fluorene	2.71	2.46	<0.14	6.04	4.77
phenanthrene	7.61	4.45	<0.21	32.54	26.02
anthracene	1.17	0.37	<0.01	4.83	1.82
fluoranthene	2.09	<1.55	<0.16	32.75	7.23
benzo(c)fluorene	<0.49	0.04	<0.01	2.11	0.28
pyrene	<1.96	<2.0	<0.20	31.36	6.66
benzo[ghi]fluoranthene	<0.15	<0.15	<0.02	6.72	0.64
benzo[a]anthracene	<0.33	<0.17	<0.01	6.85	1.10
benzo[b] napthol [2,1, dithiophene]	<0.2	0.04	<0.01	1.33	<0.47
cyclopenta[cd]pyrene	<0.11	0.18	<0.01	5.38	0.10
chrysene	0.39	0.32	<0.02	12.00	2.24

Sample	S21- 011602	S21- 011603	S21- 011604	S21- 011605	S21- 011606
5-methylchrysene	<0.09	<0.01	<0.01	0.52	<0.25
benzo[b]fluoranthene	<0.30	0.32	<0.02	5.11	0.74
benzo[j]fluoranthene	<0.1	<0.06	<0.01	3.02	0.33
benzo[k]fluoranthene	<0.08	<0.08	<0.01	2.26	0.34
benzo[e]fluoranthene	<0.12	0.14	<0.01	4.16	1.43
benzo[a]pyrene	<0.23	<0.26	<0.02	3.19	0.83
indeno[1,2,3-cd]pyrene	1.64	<0.34	<0.03	1.72	0.39
dibenzo[ah]anthracene	<0.39	<0.39	<0.04	0.51	<0.39
benzo[ghi]perylene	<0.15	<0.15	<0.01	1.92	0.55
anthanthrene	<0.17	<0.17	<0.10	<0.27	<0.17
dibenzo[a,l]pyrene	<0.58	<0.59	<0.10	<0.59	<0.58
dibenzo[a,e]pyrene	<1.04	<1.06	<0.11	<1.06	<1.04
dibenzo[a,i]pyrene	<1.16	<1.18	<0.12	<1.19	<1.17
dibenzo[a,h]pyrene	<1.11	<1.14	<0.11	<1.14	<1.12
coronene	<0.21	<0.21	<0.10	<0.34	<0.21
PAH 4 SUM Upper µg/kg	1.25	1.07	0.07	27.15	4.91
PAH 4 SUM Lower µg/kg	0.39	0.64	<0.01	27.15	4.91

Sample	S21- 011607	S21-011608	S21-011609	S21- 011610
acenaphthylene	<0.02	0.30	0.76	0.23
acenaphthene	<0.13	<1.29	0.65	<1.34
fluorene	<0.13	1.36	1.32	<1.33
phenanthrene	<0.2	2.51	16.03	<1.99
anthracene	<0.01	0.15	1.42	0.28
fluoranthene	<0.12	<1.21	6.65	<1.25
benzo(c)fluorene	<0.01	0.15	1.42	0.28
pyrene	<0.13	1.49	5.35	<1.32
benzo[ghi]fluoranthene	<0.01	0.15	0.78	0.03
benzo[a]anthracene	<0.01	<0.12	0.66	0.18
benzo[b] napthol [2,1, dithiophene]	<0.01	0.07	0.09	0.05
cyclopenta[cd]pyrene	<0.01	<0.03	0.09	<0.02
chrysene	<0.01	0.30	1.03	0.39
5-methylchrysene	<0.01	<0.01	<0.02	<0.01
benzo[b]fluoranthene	<0.01	0.17	0.68	0.36
benzo[j]fluoranthene	<0.01	<0.04	0.45	0.07
benzo[k]fluoranthene	<0.01	<0.04	0.28	<0.04

Sample	S21- 011607	S21-011608	S21-011609	S21- 011610
benzo[e]fluoranthene	<0.01	0.13	0.55	0.22
benzo[a]pyrene	<0.04	<0.35	0.47	<0.36
indeno[1,2,3- cd]pyrene	<0.03	<0.29	0.41	<0.3
dibenzo[ah]anthracene	<0.03	<0.33	<0.14	<0.34
benzo[ghi]perylene	<0.01	<0.14	0.32	<0.14
anthanthrene	<0.10	<0.16	<0.10	<0.16
dibenzo[a,l]pyrene	<0.10	<0.67	<0.28	<0.7
dibenzo[a,e]pyrene	<0.10	<0.91	<0.38	<0.95
dibenzo[a,i]pyrene	<1.11	<1.05	<0.44	<1.09
dibenzo[a,h]pyrene	<0.11	<1.12	<0.46	<1.16
coronene	<0.10	<0.22	<0.10	<0.22
PAH 4 SUM Upper µg/kg	0.07	0.94	2.84	1.29
PAH 4 SUM Lower µg/kg	<0.01	0.47	2.84	0.93

Sample	S21- 0116012	S21-011613	S21-011614	S21- 01161
acenaphthylene	0.61	<0.19	0.47	0.40

Sample	S21- 0116012	S21-011613	S21-011614	S21- 01161
acenaphthene	<1.21	<1.3	<1.22	<1.28
fluorene	<1.33	<1.42	<1.34	1.71
phenanthrene	<1.87	<2.01	<1.89	15.89
anthracene	<0.11	<0.12	<0.11	0.26
fluoranthene	<1.26	<1.35	<1.27	<1.33
benzo(c)fluorene	<0.01	<0.01	<0.01	<0.03
pyrene	<1.66	<1.78	<1.67	3.86
benzo[ghi]fluoranthene	<0.12	<0.13	<0.12	0.24
benzo[a]anthracene	<0.03	0.07	<0.07	<0.08
benzo[b] napthol [2,1, dithiophene]	<0.02	<0.04	<0.03	<0.08
cyclopenta[cd]pyrene	<0.04	<0.04	<0.04	0.14
chrysene	<0.15	0.17	0.17	<0.15
5-methylchrysene	<0.01	<0.01	<0.01	<0.01
benzo[b]fluoranthene	<0.12	0.13	0.16	0.14
benzo[j]fluoranthene	<0.01	0.02	0.03	<0.01
benzo[k]fluoranthene	<0.03	<0.04	<0.06	<0.03
benzo[e]fluoranthene	<0.02	0.06	0.08	0.19

Sample	S21- 0116012	S21-011613	S21-011614	S21- 01161
benzo[a]pyrene	<0.31	<0.33	<0.31	<0.33
indeno[1,2,3- cd]pyrene	<0.22	<0.24	<0.22	<0.24
dibenzo[ah]anthracene	<0.27	<0.29	<0.27	<0.29
benzo[ghi]perylene	<0.14	<0.15	<0.14	<0.14
anthanthrene	<0.14	<0.15	<0.14	0.27
dibenzo[a,l]pyrene	<0.48	<0.52	<0.49	<0.51
dibenzo[a,e]pyrene	<0.67	<0.72	<0.67	<0.71
dibenzo[a,i]pyrene	<0.27	<0.29	<0.27	<0.28
dibenzo[a,h]pyrene	<0.70	<0.75	<0.70	<0.74
coronene	<0.18	<0.19	<0.18	<0.19
PAH 4 SUM Upper µg/kg	0.61	0.70	0.71	0.70
PAH 4 SUM Lower µg/kg	<0.01	0.37	0.33	0.14

Sample	S21- 0116016	S21-011618	S21-011619	S21- 01162
acenaphthylene	0.69	0.46	3.17	3.27
acenaphthene	<1.07	<1.08	<1.09	4.50

Sample	S21- 0116016	S21-011618	S21-011619	S21- 01162
fluorene	<1.21	<1.22	2.29	6.76
phenanthrene	21.88	<1.97	19.78	50.56
anthracene	0.67	<0.1	2.45	8.17
fluoranthene	3.00	<1.29	14.44	49.51
benzo(c)fluorene	0.05	<0.02	0.53	0.48
pyrene	2.99	<1.54	16.52	30.48
benzo[ghi]fluoranthene	0.34	<0.12	3.57	1.38
benzo[a]anthracene	0.85	0.05	3.68	5.51
benzo[b] napthol [2,1, dithiophene]	0.37	<0.04	0.08	3.22
cyclopenta[cd]pyrene	<0.05	<0.02	1.63	0.24
chrysene	1.26	0.18	3.93	9.46
5-methylchrysene	0.08	<0.03	<0.05	0.47
benzo[b]fluoranthene	1.08	0.18	2.94	4.40
benzo[j]fluoranthene	0.37	<0.02	2.15	1.28
benzo[k]fluoranthene	0.40	<0.04	1.51	1.53
benzo[e]fluoranthene	1.58	0.04	2.75	4.68
benzo[a]pyrene	1.02	<0.37	3.36	2.20

Sample	S21- 0116016	S21-011618	S21-011619	S21- 01162
indeno[1,2,3- cd]pyrene	0.53	<0.30	3.13	0.96
dibenzo[ah]anthracene	<0.3	<0.31	<0.37	<0.39
benzo[ghi]perylene	0.82	<0.14	3.40	1.19
anthanthrene	<0.15	<0.15	0.31	<0.15
dibenzo[a,l]pyrene	<0.57	<0.57	<0.58	<0.58
dibenzo[a,e]pyrene	<0.79	<0.80	<0.81	<0.81
dibenzo[a,i]pyrene	<0.30	<0.30	<0.30	<0.30
dibenzo[a,h]pyrene	<0.86	<0.87	<0.88	<0.88
coronene	<0.18	<0.18	1.12	<0.19
PAH 4 SUM Upper µg/kg	4.21	0.78	13.91	21.57
PAH 4 SUM Lower µg/kg	4.21	0.41	13.91	21.57

Table 4 QC Data for PAH analysis - T0658 Cocoa butter reference materialacceptance criteria

Compound	Assigned value from consensus data (µg/kg)	Target standard deviation ± μg/kg	Batch PAH1203	Batch PAH1205	Batch PAH1206	Ba PA
benzo[a]anthracene	3.22	0.708	3.51	3.40	3.41	3.3
benzo[b]fluoranthene	2.22	0.488	2.28	2.29	2.24	2.2

Compound	Assigned value from consensus data (µg/kg)	Target standard deviation ± μg/kg	Batch PAH1203	Batch PAH1205	Batch PAH1206	Ba PA
benzo[a]pyrene	2.00	0.440	2.07	2.10	2.05	2.0
indeno[1,2,3- cd]pyrene	1.20	0.264	1.35	1.22	1.56	1.2
benzo[g,h,i]perylene	1.55	0.341	1.45	1.42	1.42	1.4
Chrysene	4.60	1.010	4.32	4.18	4.33	4.0
PAH4 (sum)	12.60	2.770	12.18	11.97	12.03	11.

Table 5 CBD results by HPLC-UV Oils

Sample ID	Description	CBD content stated	Weight/volume as received	Measured Conc. CBD (%)*	CBD claimed as % (or calculated from unit weight)
S21- 011592	CBD spray	4800 mg	30	17.4	16
S21- 011594	CBD oil	1800 mg	10	19.0	18
S21- 011595	CBD oil	1000 mg (10%)	10	9.4	10
S21- 011596	CBD oil	500 mg	10	5.9	5
S21- 011597	CBD oil	10000 mg	30	42.0	33.3

Sample ID	Description	CBD content stated	Weight/volume as received	Measured Conc. CBD (%)*	CBD claimed as % (or calculated from unit weight)
S21- 011598	CBD oil	2500 mg (25%)	10	24.2	25
S21- 011599	CBD oil	1000 mg (10%)	10	10.1	10
S21- 011602	CBD oil	600mg	12	5.5	5
S21- 011603	CBD oil	250 mg	10	3.0	2.5
S21- 011605	CBD oil	500 mg	10	5.0	5
S21- 011606	CBD oil	500 mg	10	6.4	5
S21- 011608	CBD oil	2000 mg (20%)	10	24.2	20
S21- 011610	CBD oil	1000 mg	15	6.6	6.7
S21- 011611	CBD oil	Approx 500 mg	10	5.0	5
S21- 011612	CBD oil/drops	1200 mg	30	4.7	4
S21- 011613	Liposomal oil	6 mg/ml (360 mg in bottle)	60	0.5	0.6

Sample ID	Description	CBD content stated	Weight/volume as received	Measured Conc. CBD (%)*	CBD claimed as % (or calculated from unit weight)
S21- 011614	CBD oil	500 mg (5.6%)	10	5.7	5.6
S21- 011615	CBD oil	1000 mg (10%)	10	20.7	10
S21- 011616	CBD oil	1000 mg (10%)	10	10.9	10
S21- 011617	CBD oil	500 mg (5%)	10	4.9	5
S21- 011618	CBD drops	6000 mg (20%)	30	21.8	20
S21- 011620	CBD spray	750 mg (5%)	15	5.6	5
S21- 011621	CBD oil	1000 mg	30	5.2	3.33

*Results are not corrected for recovery, the recovery for this analysis was 107 to 112%.

Expanded measurement uncertainty for this analysis is 12%.

Table 6 CBD Results by HPLC-UV edibles

Sample ID	Description	CBD content/stated	Weight/volume as received	Weight of unit (grams)	CBD claimed per unit (mg)	Measured Conc CBD (%)
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S21- 011593	CBD gummies	600 mg	30 gummies	5	20 mg per gummie	0.2
S21- 011600	CBD mint	40 times 10 mg	40 mints	0.3	10 mg per mint	3.51
S21- 011609	CBD chocolate	20 mg	45 g	45	20 mg per bar	0.051
S21- 011619	CBD chocolate	10 mg	30 g	30	10 mg per bar	0.024
S21- 011604	botanical drink sour cherry and hibiscus	5 mg	250 ml	250	5 mg	<0.001
S21- 011607	sparkling driink with 15 mg CBD	15 mg	250 ml	250	15 mg	0.004

*Results are not corrected for recovery. Expanded measurement uncertainty for this analysis is 28%. nd - not detected.

Table 7 Summary of QC data for CBD LC-UV analysis

Quality control	Spike (1 or 2%)	IHR 0.5%	IHR 5%	IHR 11%
Recovery (%)	106	111	111	99
Number of analysis	7	6	6	6

Table 8 Cannabinoid results by LCMS - oils and sprays

Cannabinoid results (mg/kg), LCMS or (LCUV) not recovery corrected

Sample Description	СВС	CBC- A	CBD- A	CBD- V	CBDV- A	CBG	CBG- A	TH A
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S21- 011592	CBD spray	10.3	<1	5.4	1521#	<1.75	<20	<1.75	<1.
S21- 011594	CBD oil	27.6	<2.5	35.3	239	<1.75	<50	<2.5	<1.
S21- 011595	CBD oil	135	<2.5	27.2	84.7	<2.5	<50	<1.75	<1.
S21- 011596	CBD oil	8.8	2.9	71	74.3	<2.5	<20	3.9	<1.
S21- 011597	CBD oil	35.7	<1	<2.5	894#	<1.75	<50	<1.75	<1.
S21- 011598	CBD oil	21.2	<1	<2.5	427	<1.75	<50	<1.75	<1.
S21- 011599	CBD oil	35.2	<1	<2.5	1572#	<1.75	3944*	<1.75	<1.
S21- 011602	CBD oil	<6	<1	2.5	99.7	<1.75	<20	<1.75	<2.
S21- 011603	CBD oil	49.8	9.8	359	1199#	5.6	<100	8.0	<1.
S21- 011605	CBD oil	936*	107	1923*	192	32.9	<150	14.6	<1.
S21- 011606	CBD oil	109	21.1	498*	161	10.2	<50	19.5	<1.
S21- 011608	CBD oil	296	<1.75	<1.75	1564#	<1.75	12243*	<1.75	<1
S21- 011610	CBD oil	61.4	<1.75	<1.75	962#	<1.75	2874*	<1.75	<1
S21- 011611	CBD oil	207	4.7	71.2	123	<2.5	<100	<2.5	<1

S21- 011612	CBD oil/drops	<10	<1.75	<1.75	215	<1.75	<20	<1.75	<1
S21- 011613	Liposomal oil	2.9	<1.75	<1.75	157	<1.75	<100	<1.75	<1
S21- 011614	CBD oil	7.1	<2.5	25.3	132	<2.5	<20	<1.75	<1
S21- 011615	CBD oil	11.0	<1.75	<1.75	453	<1.75	<50	<1.75	<1
S21- 011616	CBD oil	92.4	<2.5	22.4	581#	4.0	4636*	<2.5	<1.
S21- 011617	CBD oil	12.5	8.4	161	662#	<2.5	<50	5.6	<1.
S21- 011618	CBD drops	49.7	<2.5	<10	1144#	<2.5	<50	<2.5	<1.
S21- 011620	CBD spray	72.4	54.8	274	430#	4.8	1338*	3.4	<1.
S21- 011621	CBD oil	<5	3.2	36.8	154	<2.5	5012*	<2.5	<1.

*LC_UB data as outside LC/<S/Ms calibration range. #LC-MS/MS 10 fold diluted sample to bring into calibration range of method.

Table 9 Controlled Cannabinoid results by LC-MS - oils and sprays

Cannabinoid results (mg/kg), LCMS not recovery corrected

Sample ID	Description	CBN	Delta8- THC	тнс	THC-A	тнс v
S21- 011592	CBD spray	3.0	8.0	16.3	<1	<2.5
S21- 011594	CBD oil	30.6	102	28.5	<2.5	<2.5

Sample ID	Description	CBN	Delta8- THC	тнс	THC-A	THC V
S21- 011595	CBD oil	3.0	3.1	122	2.8	<2.5
S21- 011596	CBD oil	7.4	20.8	8.1	2.7	<2.5
S21- 011597	CBD oil	3.6	8.8	107	<1	<2.5
S21- 011598	CBD oil	2.6	8.1	69.5	<1	<2.5
S21- 011599	CBD oil	29.9	4.3	32.6	<1	7.4
S21- 011602	CBD oil	<1	6.7	5.7	<2.5	<2.5
S21- 011603	CBD oil	6.6	<5	45.9	<2.5	146
S21- 011605	CBD oil	46.5	<10	357	20.4	6.1
S21- 011606	CBD oil	14.6	<5	75.8	4.5	<2.5
S21- 011608	CBD oil	168	<1.75	111	<1.75	<2.5
S21- 011610	CBD oil	38.9	<1.75	46.4	<1.75	9.8
S21- 011611	CBD oil	39.6	<1.75	268	<5	5.5
S21- 011612	CBD oil/drops	<2.5	<1.75	8.2	<1.75	<1.7

Sample ID	Description	CBN	Delta8- THC	тнс	THC-A	тнс v
S21- 011613	Liposomal oil	8.4	<1.75	2.7	<1.75	<1.7
S21- 011614	CBD oil	<2.5	<1.75	7.7	2.8	<1.7
S21- 011615	CBD oil	<2.5	<1.75	28.8	<1.75	<1.7
S21- 011616	CBD oil	4.4	<1.75	51.8	<1.75	63.5
S21- 011617	CBD oil	<1.75	<1.75	22.6	5.9	<2.5
S21- 011618	CBD drops	6.8	<1.75	20.5	<1.75	<2.5
S21- 011620	CBD spray	27.4	<1.75	47.7	4.6	1.0
S21- 011621	CBD oil	<1.75	<1.75	10.0	<2.5	<1.7

Controlled cannabinoid per unit

Sample ID	Description	THC + (sum CBN, D8-THC, D9- THC,THCV, 0.877*THCA) [mg/kg]	mg THC per container	mg THC+ per container*
S21- 011592	CBD spray	27	0.49	0.82
S21- 011594	CBD oil	161	0.29	1.61

Sample ID	Description	THC + (sum CBN, D8-THC, D9- THC,THCV, 0.877*THCA) [mg/kg]	mg THC per container	mg THC+ per container*
S21- 011595	CBD oil	130	1.22	1.30
S21- 011596	CBD oil	39	0.08	0.39
S21- 011597	CBD oil	119	3.20	3.57
S21- 011598	CBD oil	80	0.69	0.80
S21- 011599	CBD oil	74	0.33	0.74
S21- 011602	CBD oil	12	0.07	0.15
S21- 011603	CBD oil	198	0.46	1.98
S21- 011605	CBD oil	427	3.57	4.27
S21- 011606	CBD oil	94	0.76	0.94
S21- 011608	CBD oil	278	1.11	2.78
S21- 011610	CBD oil	95	0.70	1.43
S21- 011611	CBD oil	313	2.68	3.13

Sample ID	Description	THC + (sum CBN, D8-THC, D9- THC,THCV, 0.877*THCA) [mg/kg]	mg THC per container	mg THC+ per container*
S21- 011612	CBD oil/drops	8	0.25	0.25
S21- 011613	Liposomal oil	11	0.16	0.66
S21- 011614	CBD oil	10	0.08	0.10
S21- 011615	CBD oil	29	0.29	0.29
S21- 011616	CBD oil	120	0.52	1.20
S21- 011617	CBD oil	28	0.23	0.28
S21- 011618	CBD drops	27	0.61	0.82
S21- 011620	CBD spray	96	0.72	1.44
S21- 011621	CBD oil	10	0.30	0.30

Recovery values are in Table 12, results are not corrected for recovery. Expanded measurement uncertainty was 17-37% for individual cannabinoids, 17% for THC, 34% for THCA, 37% for delta 8THC and 23% for CBN. Products exceeding 1mg are above the current Home Office guidance threshold and are non-compliant and are not classed as foods.

Table 10 Cannabinoid results by LCMS - edibles and isolates

Sample Description CBC	CBC- A	CBD- A	CBD- V	CBDV- A	CBG	CBG- A	THC\ A
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S21- 0011593	CBD gummies	<1	<2.5	<2.5	14.3	<1	32.4	<1	<1
S21- 011600	CBD mint	3.9	<2.5	<2.5	92	<1	<20	<1	<1
S21- 011609	CBD chocolate	21.1	<2.5	4.8	<5	<1	<20	<1	<1
S21- 011619	CBD chocolate	7.5	8.8	259	<5	4.2	<20	8.9	<1
S21- 011604	Beverage	<1	<1	<2.5	<1	<1	<1	<1	<1
S21- 011607	Beverage	<1	<1	<2.5	<1	<1	<1	<1	<1
S21- 011601	CBD isolate	81	<1.75	<5	<1	<1	<20	<1.75	<1

Table 11 Controlled Cannabinoid results by LCMS - edibles and isolates

Cannabinoid results (mg/kg(, LCMS not recovery corrected

Sample ID	Description	CBN	Delta8-THC	THC	THC-A	THC-V
S21-0011593	CBD gummies	<1	<1	<1	<1	<1
S21-011600	CBD mint	<2.5	<1	<2.5	<1	<1
S21-011609	CBD chocolate	4.5	<1	9.9	<1.75	<2.5
S21-011619	CBD chocolate	<2.5	<1	6.4	4.6	<2.5
S21-011604	Beverage	<1	<1	<1	<1	<1
S21-011607	Beverage	<1	<1	<1	<1	<1

Sample ID	Description	CBN	Delta8-THC	THC	THC-A	THC-V
S21-011601	CBD isolate	15.1	<1	281	<1	<1

Controlled cannabinoid per unit

Sample ID	Description	THC + (sum CBN, D8-THC, THC, 0.877*THCA)	mg THC per container	mg THC+ per container*	
S21- 0011593	CBD gummies	0	0	0	
S21- 011600	CBD mint	0	0	0	
S21- 011609	CBD chocolate	14.4	0.4	0.6	
S21- 011619	CBD chocolate	10.4	0.2	0.3	
S21- 011604	Beverage	0	0	0	
S21- 011607	Beverage	0	0	0	
S21- 011601	CBD isolate	296	14.1	14.8	

Recovery values are in Table 13, results are not corrected for recovery. Expanded measurement uncertainty was 17-37% for individual cannabinoids, 17% for THC, 34% for THCA, 37% for delta 8THC and 23% for CBN. Products exceeding 1mg are above the current Home Office guidance threshold and are non-compliant and are not classed as foods.

Table 12 QC data for Cannabinoids by LCMS

Matrix	Cannabinoid	CBC	CBC- A	CBD- A	CBD- V	CBDV- A	CBG	CBG- A
oil	Low spiking level (mg/kg)	5	5	5	5	5	20	5
oil	Recovery (%)	105	99	114	122	109	94	103
oil	High spiking level (mg/kg)	200	200	200	200	200	200	200
oil	Recovery (%)	100	99	100	99	100	101	98
oil	Number of analysis	3	3	3	3	3	3	3
isolate starch	Low spiking level (mg/kg)	5	5	5	5	5	20	5
isolate starch	Recovery (%)	101	87	91	98	86	109	91
isolate starch	High spiking level (mg/kg)	200	200	200	200	200	200	200
isolate starch	Recovery (%)	100	102	106	95	103	98	103
isolate starch	Number of analysis	1	1	1	1	1	1	1
confectionary	Low spiking level (mg/kg)	5	5	5	5	5	20	5
confectionary	Recovery (%)	98	103	95	91	101	94	92
confectionary	High spiking level (mg/kg)	200	200	200	200	200	200	200
confectionary	Recovery (%)	116	109	113	108	112	105	109

Matrix	Cannabinoid	CBC	CBC- A	CBD- A	CBD- V	CBDV- A	CBG	CBG- A
confectionary	Number of analysis	2	2	2	2	2	2	2
drinks	Low spiking level (mg/kg)	5	5	5	5	5	20	5
drinks	Recovery (%)	83	77	81	78	76	88	81
drinks	High spiking level (mg/kg)	200	200	200	200	200	200	200
drinks	Recovery (%)	88	95	96	87	95	89	95
drinks	Number of analysis	2	2	2	2	2	2	2
overall	Low spiking level (mg/kg)	5	5	5	5	5	20	5
overall	Recovery (%)	97	91	95	97	93	96	92
overall	High spiking level (mg/kg)	200	200	200	200	200	200	200
overall	Recovery (%)	101	101	104	97	102	98	101
overall	Number of analysis	8	8	8	8	8	8	8

Table 13 QC data for controlled Cannabinoids by LCMS

Matrix	Cannabinoid	CBN	Delta 8-THC	Delta 9-THC	THC- A	THC- V
oil	Low spiking level (mg/kg)	5	5	5	5	5

Matrix	Cannabinoid	CBN	Delta8- THC	Delta9- THC	THC- A	THC- V
oil	Recovery (%)	96	97	98	102	101
oil	High spiking level (mg/kg)	200	200	200	200	200
oil	Recovery (%)	99	103	100	100	97
oil	Number of analysis	3	3	3	3	3
isolate starch	Low spiking level (mg/kg)	5	5	5	5	5
isolate starch	Recovery (%)	101	100	95	85	97
isolate starch	High spiking level (mg/kg)	200	200	200	200	200
isolate starch	Recovery (%)	96	100	101	107	95
isolate starch	Number of analysis	1	1	1	1	1
confectionary	Low spiking level (mg/kg)	5	5	5	5	5
confectionary	Recovery (%)	91	130	97	95	87
confectionary	High spiking level (mg/kg)	200	200	200	200	200
confectionary	Recovery (%)	111	121	116	109	106
confectionary	Number of analysis	2	2	2	2	2
drinks	Low spiking level (mg/kg)	5	5	5	5	5

Matrix	Cannabinoid	CBN	Delta8- THC	Delta9- THC	THC- A	THC- V
drinks	Recovery (%)	77	84	82	82	78
drinks	High spiking level (mg/kg)	200	200	200	200	200
drinks	Recovery (%)	83	89	88	96	87
drinks	Number of analysis	2	2	2	2	2
overall	Low spiking level (mg/kg)	5	5	5	5	5
overall	Recovery (%)	91	103	93	91	91
overall	High spiking level (mg/kg)	200	200	200	200	200
overall	Recovery (%)	97	103	101	103	96
overall	Number of analysis	8	8	8	8	8

Table 14 Summary of pesticide residues found in CBD samples

Sample ID	Sample description	Pesticide	mg/kg
s21-011593	CBD Gummies	BAC12	0.1
s21-011595	CBD oil	fenpyroximate	0.05
-	-	pirimiphos-methyl	0.03
-	-	tebufenpyrad	0.1
s21-011605	CBD oil	propamocarb (free base)	0.01

Sample ID	Sample description	Pesticide	mg/kg
s21-011606	CBD oil	chlorpyrifos	0.01
s21-011621	CBD oil	pirimiphos-methyl	0.02

Table 15 Mycotoxin HPLC FLD results - aflatoxins, ochratoxin A and zearalenone

Corrected results µg/kg

Sample ID	AFB1	AFB2	AFG1	AFG2	ΟΤΑ	ZON
S21-011592	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011593	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011594	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011595	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011596	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011597	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011598	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011599	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011600	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011601	<0.2	<0.2	<0.2	<0.2	0.2	<10
S21-011602	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011603	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011604	<0.2	<0.2	<0.2	<0.2	<0.2	<10

Sample ID	AFB1	AFB2	AFG1	AFG2	ΟΤΑ	ZON
S21-011605	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011606	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011607	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011608	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011609	<0.2	<0.2	<0.2	<0.2	0.2	<10 (4.4)*
S21-011610	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011611	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011612	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011613	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011614	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011615	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011616	<0.2	<0.2	<0.2	<0.2	<0.2<	<10
S21-011617	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011618	<0.2	<0.2	<0.2	<0.2	0.2	<10
S21-011619	<0.2	<0.2	<0.2	<0.2	<0.2	<10 (7.5)*
S21-011620	<0.2	<0.2	<0.2	<0.2	<0.2	<10
S21-011621	<0.2	<0.2	<0.2	<0.2	<0.2	<10

*Zearalenone confirmed by LC-MS/MS

Table 16 Mycotoxin LC-MS/MS results

Corrected results µg/kg

Sample ID	DON	T-2	HT- 2	Bea	Enn-A
S21- 011592	<5	<5	<5	<1.25	<1.25
S21- 011593	<5	<5	<5	<1.25	<1.25
S21- 011594	<5	<5 (3.8)	<5	<1.25	<1.25
S21- 011595	<5	7.2	<5 (4.0)	<1.25	<1.25
S21- 011596	<5	<5	<5	<1.25	<1.25
S21- 011597	<5	<5	<5	<1.25	<1.25
S21- 011598	<5	<5	<5	<1.25	<1.25
S21- 011599	<5	<5	<5	<1.25	<1.25
S21- 011600	<5	<5	<5	<1.25	<1.25
S21- 011601	<5	<5	<5	<1.25	<1.25
S21- 011602	<5	<5	<5	<1.25	<1.25
S21- 011603	<5	<5	<5	<1.25	<1.25
S21- 011604	<5	<5	<5	<1.25	<1.25

Sample ID	DON	T-2	HT- 2	Bea	Enn-A
S21- 011605	<5	6.4	<5 (4.0)	<1.25	<1.25
S21- 011606	<5	<5 (3.2)	5.1	<1.25	<1.25
S21- 011607	<5	<5	<5	<1.25	<1.25
S21- 011608	<5	<5	<5	<1.25	<1.25
S21- 011609	<5	<5	<5	<1.25	<1.25
S21- 011610	<5	<5	<5	<1.25	<1.25
S21- 011611	<5 (3.8)	44.0	8.3	<1.25	<1.25
S21- 011612	<5	<5	<5	<1.25	<1.25
S21- 011613	<5	<5	<5	<1.25	<1.25
S21- 011614	<5	<5	<5	<1.25	<1.25
S21- 011615	<5	<5	<5	<1.25	<1.25
S21- 011616	<5	<5	<5 (2.9)	<1.25	<1.25
S21- 011617	<5	<5	<5	<1.25	<1.25

Sample ID	DON	T-2	HT- 2	Веа	Enn-A
S21- 011618	<5	<5	<5	<1.25	<1.25
S21- 011619	<5	<5	<5	<1.25	<1.25
S21- 011620	<5	<5	<5	<1.25	<1.25
S21- 011621	<5	5.5	<5	<1.25	<1.25

Table Key: DON = Deoxynivalenol, T-2 = T-2 toxin, HT-2 = HT-2 toxin, Bea = Beauvericin, Enn A = Enniatin A, Enn A1 = Enniatin A1, Enn B = Enniatin B, Enn B1 = Enniatin B1

Table 17 Mycotoxin LC-MS/MS QC data

(recovery %)

-	DON	T2	HT2	Bea	Enn-A	Enn-A1	Enn-B	Enn-B1
Spike 1	71	78	80	104	97	100	104	101
Spike 2	92	94	98	105	106	105	108	105
mean	81	86	89	104	102	103	106	103

Table 18 Results of Headspace GC MS/MS analysis for residual solvents

Residual solvent results (mg/kg)

-	Pentane	Methanol	Ethanol	Ethyl ether	Acetone	IPA	ACN	DCM	Hex
S21- 011592	<6	<9	<6	<6	<6	<6	<20	<0.6	<6

-	Pentane	Methanol	Ethanol	Ethyl ether	Acetone	IPA	ACN	DCM	Hex
S21- 011593	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011594	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011595	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011596	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011597	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011598	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011599	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011600	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011601	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011602	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011603	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011604	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011605	<6	<9	<6	<6	<6	<6	<20	<0.6	<6

-	Pentane	Methanol	Ethanol	Ethyl ether	Acetone	IPA	ACN	DCM	Hex
S21- 011606	<6	<9	<6	<6	<6	<6	<20	<0.6	<6

Table 18 continued Results of Headspace GC MS/MS analysis for residual solvents

-	1.2DCE	Ethyl acetate	Chloroform	Benzene	Heptane
S21- 011592	<0.6	<6	<0.6	<0.6	<6
S21- 011593	<0.6	<6	<0.6	<0.6	<6
S21- 011594	<0.6	<6	<0.6	<0.6	<6
S21- 011595	<0.6	<6	<0.6	<0.6	<6
S21- 011596	<0.6	<6	<0.6	<0.6	<6
S21- 011597	<0.6	<6	<0.6	<0.6	<6
S21- 011598	<0.6	<6	<0.6	<0.6	<6
S21- 011599	<0.6	<6	<0.6	<0.6	<6
S21- 011600	<0.6	<6	<0.6	<0.6	<6
S21- 011601	<0.6	<6	<0.6	<0.6	<6

-	1.2DCE	Ethyl acetate	Chloroform	Benzene	Heptane
S21- 011602	<0.6	<6	<0.6	<0.6	<6
S21- 011603	<0.6	<6	<0.6	<0.6	<6
S21- 011604	<0.6	<6	<0.6	<0.6	<6
S21- 011605	<0.6	<6	<0.6	<0.6	<6
S21- 011606	<0.6	<6	<0.6	<0.6	<6

Table 18 continued Results of Headspace GC MS/MS analysis for residual solvents

-	Pentane	Methanol	Ethanol	Ethyl ether	Acetone	IPA	ACN	DCM	Hex
S21- 011607	<6	<9	72	<6	<6	<6	<20	<0.6	<6
S21- 011608	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011609	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011610	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011611	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011612	<6	<9	<6	<6	<6	<6	<20	<0.6	<6

-	Pentane	Methanol	Ethanol	Ethyl ether	Acetone	IPA	ACN	DCM	Hex
S21- 011613	<6	<9	1560	<6	<6	<6	<20	<0.6	<6
S21- 011614	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011615	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011616	<6	<9	<6	<6	<6	13	<20	<0.6	<6
S21- 011617	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
SS21- 011618	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011619	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011620	<6	<9	<6	<6	<6	<6	<20	<0.6	<6
S21- 011621	<6	<9	<6	<6	<6	<6	<20	<0.6	<6

Table 18 continued Results of Headspace GC MS/MS analysis for residual solvents

Residual solvent results (mg/kg)

-	1.2DCE	Ethyl acetate	Chloroform	Benzene	Heptane	Trichloroethylene	Tol
S21- 011607	<0.6	<6	<0.6	<0.6	<6	<0.6	<6

-	1.2DCE	Ethyl acetate	Chloroform	Benzene	Heptane	Trichloroethylene	Tol
S21- 011608	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011609	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011610	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011611	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011612	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011613	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011614	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011615	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011616	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011617	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
SS21- 011618	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011619	<0.6	<6	<0.6	<0.6	<6	<0.6	<6
S21- 011620	<0.6	<6	<0.6	<0.6	<6	<0.6	<6

-	1.2DCE	Ethyl acetate	Chloroform	Benzene	Heptane	Trichloroethylene	Tol
S21- 011621	<0.6	<6	<0.6	<0.6	<6	<0.6	<6

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