

Recent HPLC-UV Approaches for Cannabinoid Analysis: From Extraction to Method Validation and Quantification Compliance

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Table S1 - Extraction methods for the analysis of cannabinoids in different matrices

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^s	Ref.
Plant material						
SE1	Fresh / 0.5 and dried / 0.2 inflorescences	EtOH / 4	SLE with shaking in the dark (500 rpm x 15 min), supernatant (1 mL) centrifugation (12,000 rpm x 4 min)	NR	93	[1]
SE2	Fresh / 0.5 and dried / 0.1 inflorescences	EtOH / 4	SLE with shaking in the dark (500 rpm x 15 min), supernatant (1 mL) centrifugation (12,000 rpm x 4 min)	NR	93	[2,3]
SE3	Inflorescences / 0.5	EtOH / 15	SLE with shaking (15 min), centrifugation (5,000 rpm x 1 min) (repeated 3x)	NR	43	[4]
SE4	Inflorescences / 0.5	EtOH / 15	SLE with shaking (15 min), centrifugation (5,000 rpm x 1 min) (repeated 3x)	NR	43	[5]
SE 5	Inflorescences 130 / 0.1	EtOH / 4	SLE with shaking in the dark (500 rpm x 15 min), supernatant (1 mL) centrifugation (12,000 rpm x 4 min)	NR	93	[6,7]
SE6	Fresh inflorescences / 0.1	EtOH / 4	SLE with shaking in the dark (500 rpm x 15 min), supernatant (1 mL) centrifugation (12,000 rpm x 4 min)	NR	93	[8]
SE7	Inflorescences / 1-2	EtOH / 50	SLE with vigorous shaking (30 min) and centrifugation (4,000 rpm x 5 min)	NR	85	[9]
SE8	Inflorescences / 0.5	EtOH / 10	SLE in a tube drive system (two glass beads 6 mm diameter). Agitation and grinding (6,000 rpm x 4 min), mixture kept at RT (9 min) and centrifugation (10,000 rpm x 3 min)	NR	18	[10]
SE9	Inflorescences / 0.5	EtOH / 20	Vortexing (5 min), horizontal shaking (250 rpm x 10 min) and centrifugation (4,000 rpm x 5 min)	NR	96	[11]
SE10	Inflorescences / 0.3	EtOH / 10	DM (RT, 1 h) under constant stirring (300 rpm) (repeated 3x)	NR	94	[12]
SE11	Inflorescences / 0.25	EtOH / 10	DM (RT, 15 min) using stirring. Repeated 2x using 10 and 5 mL of extraction solvent	NR	28	[13]
SE12	Inflorescences / 0.25	EtOH / 10	DM (RT, 15 min). Repeated 2x using 10 and 5 mL of extraction solvent	NR	14	[14]
SE13	Inflorescences / 50	EtOH / 300	DM (RT, 15 min) using stirring. Repeated 3x with additional 200 mL of extraction solvent	NR	14	[15]
SE14	Inflorescences / 1	EtOH / 8	US-SLE (5 min) with EtOH (repeated 3x)	CBD: 98.8-101.1; 98.8-102.5 Δ^9 -THC:	S20	[16]
SE15	Inflorescences / 0.1	EtOH / 10	US-SLE (50 °C, 30 min)	NR	49	[17]
SE16	Inflorescences (trichomes) / 0.2	EtOH / 10	US-SLE (25 °C, 15 min)	NR	S10	[18]
SE17	Inflorescences (trichomes) / 0.1	EtOH / 20	US-SLE (RT, 30 min), storage in dark room (18 h), vortexing and laid to rest for 10 min before filtration	NR	54	[19]
SE18	Inflorescences / NR	EtOH / NR	US-SLE (30 min, ratio of 1:10 cannabis:EtOH, w/v) and centrifugation (RT, 10,000g x 15 min)	NR	5	[20]
SE19	Inflorescences / 0.5	EtOH / 10	US-SLE (15 min)	NR	16	[21]
SE20	Inflorescences / 1	EtOH / 20	US-SLE (10 min). Extracts cleanup with a mixture of activated carbon (C ₁₈) and Mg ₂ SO ₄ . Sonication (10 min) and centrifugation (5,000 rpm x 10).	NR	59	[22]
SE21	Inflorescences / 100	EtOH / 1000	Reflux (80 °C, 6 h)	NR	37	[23]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE22	Inflorescences / 1) 60; 2) 30	1) EtOH / 360 2) BUT or DME / 500	1) Maceration (48 h) 2) Extraction in a Dexso extractor using a gas flow	NR	6	[24]
SE23	1) Inflorescences / 50 2) Spent inflorescences / 7.5	1) H ₂ O / 500 2) Hex / 75	1) Hydrodistillation (3 h) 2) US-SLE (30 min) of the residue obtained after hydrodistillation (repeated 3x)	NR	25	[25]
SE24	Inflorescences / 0.5	MeOH / 5	Vortexing (10 s), shaking (50 rpm x 5 min) and centrifugation (1,500 rpm x 1 min) (repeated 2x)	NR	38	[26]
SE25	Inflorescences / NR	MeOH / NR	DM with vortexing (15 min)	NR	90	[27]
SE26	Inflorescences / 0.1	MeOH / 10	Horizontal shaking (200 oscillations/min x 90 min) in centrifuge tube light protected and centrifugation (3,000g x 5 min)	Method LRRFM ^d : Δ ⁹ -THC: 98.38-100.76; THCA: 96.07-101.29 Method RRF ^d : Δ ⁹ -THC: 97.55-100.05; THCA: 91.37-99.76	76	[28]
SE27	Inflorescences / 0.01	MeOH / 1	US-SLE (5 min) after vortexing (30 s) and centrifugation (13,000 rpm x 5 min). For recovery determinations, 100 μL of IS (100 μg/mL) was added before extraction	CBDA: 71.2-91.8; THCA-A: 68.6-101.1 Low THC strain THCA-A: 99.8-115.5	23	[29]
SE28	Inflorescences / 0.02	MeOH / 4	US-SLE (10 min) at RT (25 ± 5 °C), centrifugation (3,000 rpm x 5 min), supernatant transfer and residue reextraction	CBD: 91.3-97.4	46	[30]
SE29	Inflorescences / 0.05	MeOH / 5	US-SLE (15 min) and centrifugation (6,000 rpm x 10 min)	NR	45	[31]
SE30	Inflorescences / 0.06	MeOH / 10	US-SLE (10 min)	THC: 94.29-103.44	67	[32]
SE31	Inflorescences / 0.2	MeOH / 20	US-SLE (10 min), vortexing, stored for stabilization in cold (-20 °C, 1 h). Centrifugation (4,200 rpm x 5 min)	NR	31	[33]
SE32	Inflorescences / 0.25	MeOH / 25	US-SLE (30 min), supernatant (2 mL) centrifugation (13,000 rpm x 10 min). For recovery experiences extraction was performed with 30 μg/mL of ACBD in MeOH	Δ ⁹ -THC: 97.0-99.3	102	[34]
SE33	Inflorescences / 0.25	MeOH / 25	US-SLE (30 min), supernatant (2 mL) centrifugation (13,000 rpm x 10 min). For recovery experiences extraction was performed with 30 μg/mL of ACBD in MeOH	ACBD: 84.9-100.8	61	[35]
SE34	Inflorescences / 0.1	MeOH / NR	Sonication (10 min) for homogenization and addition of a ACBD solution in MeOH (75 μg/mL) to afford a final sample concentration of 25 mg/mL. US-SLE (20 min) and centrifugation (13,000 rpm x 10 min)	ACBD: 90-108	86	[36]
SE35	Inflorescences / 0.1	MeOH / 25	US-SLE (40 min) and centrifugation (3,000 rpm x 5 min)	NR	22	[37]
SE36	Inflorescences / 0.05	ACN / 5	US-SLE (20 min) in a sealed glass test tube, vortexing and centrifugation (6,000 rpm x 2 min)	NR	101	[38]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE37	Inflorescences / 0.204	EtOH or MeOH or ACN or <i>n</i> -Hex or IPA or EtOAc or or combination with different polarity index (n= 14)/ 20	US-SLE (40 °C, 15 min) after vortexing (10 s), sample cooling to RT and centrifugation (1930g x 5 min)	NR	15	[39,40]
SE38	Inflorescences / 0.2	MeOH:CH ₃ Cl (9:1, v/v) / 20	US-SLE (30 min), centrifugation (6,000 rpm x 10 min), supernatant transfer and residue reextraction (10 mL)	CBD: 94.4, 91.1; CBDA: 97.5, 93.8; CBN: 87.2, 89.0; Δ ⁹ - THC: 94.0, 95.0; THCA: 90.0, 106.6	7	[41]
SE39	Inflorescences / 0.5	MeOH:CHCl ₃ (9:1, v/v) / 20	DM (RT, 30 min) and sonication (15 min)	NR	27	[42]
SE40	Inflorescences / 1	MeOH:CHCl ₃ (9:1, v/v) / NR	DM (20 min) and filtration	NR	36	[43]
SE41	Inflorescences / 0.05	MeOH:CHCl ₃ (9:1, v/v) / 2	US-SLE (40 min) and centrifugation (10 °C, 10,000 rpm x 15 min)	NR	S32	[44]
SE42	Inflorescences / 0.1	MeOH:CHCl ₃ (9:1, v/v) / 5	US-SLE (15 min, RT) (repeated 2x)	CBD: 98; CBDA: 115; CBN: 98; CBC: 98; CBG: 97; Δ ⁹ - THC: 94; THCA: 99	40	[45]
SE43	Inflorescences / 1	MeOH:CHCl ₃ (9:1, v/v) / 10	SLE (1 h) with an IS (4-androstene-3,17-dione) solution at 1 mg/mL in extraction solvent	NR	79	[46]
SE44	Inflorescences / 0.05 and cannabis- derived extracellular vesicles / 0.005	MeOH:CHCl ₃ (9:1, v/v) / 1.5	US-SLE (40 min) after vortexing (5 min). Vortexing was used every 20 min. Centrifugation (10,000 rpm x 15 min)	NR	S32	[47]
SE45	Inflorescences / 0.05, 0.1, and 0.2	MeOH: <i>n</i> -Hex (9:1, v/v) / 10	1) DM with stirring (10 min), centrifugation (2,007g) and collection of supernatants. Reextraction 2x (10 and 5 mL) 2) US-SLE (10 min), centrifugation (2,007g) and collection of supernatants. Reextraction 2x (10 and 5 mL)	CBD ^b : 94.77-99.45; CBDA ^b : 88.32-94.71; CBN ^b : 113.21- 116.85; Δ ⁹ -THC ^b : 109.76- 119.28; THCA ^b : 76.92-82.07 (used for method validation)	56	[48]
SE46	Inflorescences / 5	Olive Oil Ph. Eur. / 50	DM of grinded sample homogenised in olive oil using a Tolotto Gear [®] extraction (TGE) method or a TGE preceded by a pre-extraction procedure	NR	51	[49]
SE47	Inflorescences / 2	Refined olive Oil Ph. Eur. / 20	1) DM (100 °C, 40 min) using stirring of grinded sample homogenised in olive oil 2) US-SLE (ratio of 1:10 cannabis:oil, 20 min) equipped with a 2 mm sonotrode (200 W, 26 kHz)	NR	39	[50]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE48	Inflorescences / 5	scCO ₂ / 100-300	SFE using extraction vessel (5 mL) with compacted sample, heating (30-50 °C) without CO ₂ and gradual continuous flow of fluid until reaching a pressure of 3,000-5,000 psi. At 100-300 mL of scCO ₂ and stabilisation, the extract was collected	NR	97	[51]
SE49	Inflorescences / 100	scCO ₂ / 250	SFE with a mass flow rate of scCO ₂ of 1.4 kg/h at 15 bar and 25 °C for 30 min	NR	50	[52]
SE50	Inflorescences / 1) 3.7-5.1; 2) 5	1) scCO ₂ / 100-300 2) EtOH / 50	1) SFE with extraction vessel (25 mL) with sample loaded with CO ₂ and maintained in static conditions (15 min). Continuous flow of CO ₂ to an average value of 0.50 L/min (NTP). EtOH was used as co-solvent in some experiments (10 wt% to CO ₂ mass). Extraction finishing at 100 L of CO ₂ at NTP 2) Manual stirring at RT (40 s)	(CBD+CBDA): 95.4±1.4 (THC+THCA): 97.2±0.5	27	[53]
SE51	Inflorescences 1) 1; 2) NR	1) scCO ₂ / NR 2) EtOH / NR	1) SFE with extraction vessel (100 mL) with scCO ₂ for 180 min under a pressure of 250 bar at 45 °C (repeated 2x). Reconstitution of solvent-free extract in MeOH (2 mL), sonication (15 min) and centrifugation (10,000 rpm x 10 min) 2) Ratio of 1:10 (w/w) for extraction. Sonication (15 min) and stirring (magnetic stirrer) in a dark room (4 °C, 24 h). Evaporation using N ₂ , reconstitution in MeOH (2 mL) and centrifugation (10,00 rpm x 15 min)	NR	57	[54]
SE52	Inflorescences / 1) and 2) 5; 3) 10; 4) 2	1) hot and 2) cold H ₂ O / 100 3) EtOH / H ₂ O (20, 40, and 80% v/v) 4) EtOH / 10	1) The extractant was added hot. Heating and boiling (3 min) 2) Shaking (rt, 100 rpm x 24 h) 3) DM by stirring (RT, 100 rpm x 3 days) 4) US-SLE (40 kHz x 30 min)	NR	4	[55]
SE53	Inflorescences and leaves / 6.5	scCO ₂ / 250	SFE with scCO ₂ under a pressure of 2,000 and 6,000 psi and heating at 50 °C	NR	97	[56]
SE54	Inflorescences and leaves / 0.3	EtOH / 10	DM with constant stirring (RT, 300 rpm x 1 h) (repeated 3x)	NR	94	[57]
SE55	Inflorescences and leaves / 1) 10; 2) depending on solvent-to-solid ratio	1) EtOH / 200 and EtOAc / 200 2) EtOH / 750	1) Soxhlet extractions (2 cycles) under vacuum: 1 st cycle with EtOH at reflux (38 °C, 57.4 kPa, 6 h), removal of extract and 2 nd cycle with EtOAc at reflux (31 °C, 57.4 kPa, 6 h) 2) Cryogenic extraction using a rotary tumbler operating at different temperatures (-80-20 °C, 32 rpm x 30 min)	NR	77	[58]
SE56	Inflorescences or leaves / 0.04	MeOH (80%) / 2	Vortexing (30 s) and US-SLE (5 min) (repeated 3x)	NR	S22	[59]
SE57	Inflorescences and leaves / 0.05	MeOH / 10	US-SLE (30 °C, 20 min) with a 100 µg/mL solution of IS (4-androstene-3,17-dione) in MeOH (10 mL)	91.6-111.7	81	[60]
SE58	Inflorescences and leaves / 0.1	ACN / 1	US-SLE (35 °C, 30 min) and centrifugation (10,000 rpm x 10 min)	NR	103	[61]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^s	Ref.
Plant material						
SE59	Inflorescences and leaves / 0.1	ACN / 1	US-SLE (30 min)	NR	103	[62]
SE60	Inflorescences and leaves / 1	pentane / 5	US-SLE (RT, 30 min)	NR	13	[63]
SE61	Inflorescences and leaves / 0.1-0.2	H ₂ O, MeOH or H ₂ O:MeOH/ 1 or 15	Mixture of sample and extractant was left standing for 15 min, centrifugation (3,000 rpm x 10 s)	NR	95	[64]
SE62	Inflorescences, leaves, stems / 0.15	EtOH / 5	DM (60 min) (repeated 3x)	NR	94	[65]
SE63	Inflorescences, leaves, stems / 0.5	MeOH:CHCl ₃ (9:1, v/v) / 5	US-SLE (15 min) and vortexing at the beginning and every 5 min. Centrifugation (4,500 rpm x 15 min)	NR	55	[66]
SE64	Inflorescences / 0.4	ACN:MeOH (4:1, v/v) / 25	US-SLE (RT, 30 min, low power)	NR	65	[67]
SE65	1) Inflorescences / a) 0.02 b) 100 2) Cannabis crude extract / 0.01	1a) MeOH (80%) / 5 1b) EtOH / 500 2) MeOH / 1	1a) After decarboxylation: US-SLE (25 °C, 15 min) 1b) DM with shaking in an orbital shaker (24 h) to obtain the crude extract 2) After decarboxylation: vortexing and dilution	NR	35	[68]
SE66	1) Aerial parts (including leaves and stems) / 0.1 2) Inflorescences / 0.02	1) H ₂ O 30% (w/w) KBr / 2 and ES (100 µL) 2) EtOH / 40 µL; H ₂ O 30% (w/w) KBr / 2 and ES (100 µL)	1) DSLME. Vortexing (30 s) and sonication (40 KHz, 25 °C, 10 min). Vortexing (30 s) and centrifugation (2,200g x 5 min). The supernatant was transferred and centrifuged (2,200g x 5 min). ES-rich phase (upper layer) was collected 2) same as 1) but sample was mixed with EtOH prior to the addition of the KBr aqueous solution and ES	NR	S04,S05	[69]
SE67	Inflorescences, stalks / 1) 15; 2) 1; 3) 25	1) EtOH / 400 2) EtOH / 40 3) scCO ₂ / NR	1) Soxhlet extraction (78 °C, 8 h) of sample in a thimble filter 2) DM (RT, 15 min) under magnetic stirring (repeated 2x using 40 and 20 mL of extractant) 3) SFE (40 °C, 360 bar), 30 min in static mode interposed between dynamic phases of 15 min (total extraction time: 140 min)	NR	S06	[70]
SE68	Inflorescences, EtOH and CO ₂ crude extract, distillate, distillation mother liquors, distil-lation bottoms / 0.1	H ₂ O / 10	DM with vortexing	NR	42	[71]
SE69	1) Inflorescences / 20	1) H ₂ O / 6000	1) Sample allowed to soak (30 min). Hydrodistillation (5 h) to obtain essential oil (EO).	NR	63	[72]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE69 (cont.)	2) Inflorescences, lyophilized extracts and deterpenated material / 0.2	2) MeOH:H ₂ O (70:30, v/v) / 25	1a) Aqueous residue was collected and kept at -20 °C after filtration. Freeze-dried (-54 °C, 0.05 mbar) to obtain lyophilized extracts. 1b) Plant biomass (deterpenated material) was dried at 60 °C for 24 h. 2) US-SLE (15 min) and centrifugation	NR	63	[72]
SE70	Inflorescences, cannabis samples / 0.1	MeOH / NR ^{c)}	US-SLE (5 min) and vortexing (repeated 4x). Centrifugation (13,000 rpm, 10 min) of supernatant (2 mL)	ACBD: 93.6-106.1	84	[73]
SE71	Cannabis samples (aerial parts) / 0.1	MeOH / 10	SLE by shaking horizontally (200 oscillations/min, 90 min) light protected and centrifugation (3000g x 5 min)	NR	76	[74]
SE72	Seeds / 0.6	Acetone/H ₂ O ^{d)} (1:1, v/v)	Defatted seeds mixture with solvent, vortexing (5 min), US-SLE (45 min) in a cold and dark room, centrifugation (4,800 rpm, 10 min) (repeated 2x)	NR	9	[75,76]
SE73	Seeds and biomass / 0.5	MeOH / 3.5 and <i>n</i> -Hex / 3.5	Shaking (800 motion/min, 30-40 min) of samples in MeOH spiked with IS (3,5-dimethylphenol 2.8 mg/1 mL MeOH) and centrifugation (20 °C, 13 000 rpm x 3 min) (repeated 2x). Addition of <i>n</i> -Hex to residue, sonication (30-40 min, 20 °C) and centrifugation (20 °C, 13,000 rpm x 3 min) (repeated 2x)	NR	62	[77]
SE74	Leaves / 25	EtOH (70%) / 250	Reflux (80 °C, 2 h) (repeated 2x) and evaporation under vacuum (rotovap). Further extractions were performed in the residue using petroleum ether, EtOAc, and <i>n</i> -BuOH	NR	S08,S26	[78]
SE75	Leaves / NR	EtOH / NR	Reflux (80 °C) (repeated 2x). The ratio of dried leaves to solvent was 1:15 by weight	NR	92	[79]
SE76	Leaves / 1	MeOH, EtOH, IPA, (1:1, v/v) of MeOH:EtOH, EtOH:IPA, MeOH:IPA	1) DM with shaking (RT, 300 rpm x 24 h) 2) US-SLE (40 °C, 1 h)	NR	97	[80]
SE77	Leaves / 20	H ₂ O / NR	Blended sample was infused in boiling H ₂ O and allowed to stand for 2 h	NR	S03	[81]
SE78	Leaves / 4,000	DCM:MeOH (1:1, v/v) / NR	Extraction at RT, 72 h (method NR)	NR	66	[82]
SE79	Leaves and stems / 0.1	MeOH or Acetone / 5	US-SLE (10 min) (repeated 3x)	NR	10	[83]
SE80	Leaves and vegetative shoots / 0.15	ACN / 1	US-SLE (1 h), incubation at 55 °C (1 h) and centrifugation (13,000g x 10 min)	NR	12	[84]
SE81	Cannabis samples (inflorescences, leaf, stem, and root) / 1	MeOH:CHCl ₃ (9:1, v/v) / 10	US-SLE (30 min) and centrifugation (5,000 rpm x 5 min)	NR	8	[85]
SE82	Cannabis samples / 0.5	MeOH / 20	Vortexing (10 s) and shaking (50 rpm x 30 min), centrifugation (1,000 rpm x 5 min) (repeated 2x)	NR	38	[86]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE83	Cannabis samples / 0.1	MeOH / 0.3	Vortexing (3 min)	NR	26	[87]
SE84	Cannabis sample / 0.05	MeOH / 1.5	US-SLE (15 min) and centrifugation (12100g x 7 min) (repeated 4x)	NR	32	[88]
SE85	Cannabis samples / NR	EtOH / NR	US-SLE (15 min)	NR	16	[89]
SE86	Cannabis samples / 258.6	Petroleum ether / 1000	US-SLE (3 h) (repeated 3x). Filtrate crude is further extracted using an acid-base extraction	NR	92	[90]
SE87	Cannabis samples / 50	EtOH / 200 or 250 g (75% m/m)	Stirring (2 h)	NR	24	[91]
SE88	Cannabis samples / 50	Hex or EtOAc / 1000	Maceration (RT, 72 h)	NR	68	[92]
SE89	Cannabis samples / 1	EtOH:Hex (1:1, v/v) / 10	US-SLE (probe n° 6, 7 min) in ice bath and centrifugation (400 rpm x 20 min)	CBD: 83.43-112.94; CBDA: 77.88-101.52; CBN: NR; CBC: 77.89-97.77; CBG: 81.42-121.41; CBGA: 80.00- 108.62; CBDV: 84.38-113.51; CBL: NR; Δ ⁸ -THC: 77.42- 110.99; Δ ⁹ -THC: 73.78-88.67; THCA: 75.89-93.06; THCV: 83.12-112.93	71	[93]
SE90	Cannabis samples / 5	1) EtOH / 100 2) MeOH:CHCl ₃ (9:1, v/v) / 100	Turbo-extraction (25 °C, 4,000 rpm x 2 min) (repeated 2x)	CBD: 91.2-106.7	48	[94]
SE91	Cannabis samples / 0.05	EtOH:ACN (1:1, v/v) / 10	Samples spiked with IS (phemprocoumon, 50 µg/mL). US-SLE of powder (1 min, 40 kHz, 180 W) using the solvent extraction	98.8-105.6 ^c	74	[95]
SE92	Cannabis samples / 0.1	MeOH:CHCl ₃ (9:1, v/v) / 1	Vortexing (10 s) and US-SLE (15 min with shaking every 5 min) and centrifugation	NR	58	[96]
SE93	Cannabis samples / 0.05	MeOH:CHCl ₃ (9:1, v/v) / 10	US-SLE (30 °C, 20 min) with IS solution (4-androstene-3,17-dione, 100 µg/mL)	NR	19	[97]
SE94	Cannabis samples / 0.1	H ₂ O / 2 and ES (100 µL)	DSLME. Vortexing (30 s) and sonication (40 KHz, 25 °C, 10 min). Vortexing (30 s) and centrifugation (4,000 x 5 min). The ES-rich phase (upper layer) was transferred and centrifuged (4,000 rpm x 5 min)	NR	10	[98]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Plant material						
SE95	Cannabis samples / 1) and 2) 5; 3) 60	1) EtOH, petroleum ether (light and heavy), hexane 2) vegetable oils (virgin coconut, sacha inchi, perilla seed, sesame seed, olive and rice bran) 3) scCO ₂ / NR	1) and 2) Maceration (24 h, powder to solvent/oils ratio 1:10) and filtration 3) SFE (225 bar, 55 °C, 4 h)	NR	8	[99]
SE96	Cannabis samples/ 0.5	EtOH / 10	Agitation and grinding in a tube dry system of sample and extractant mixture (3,500 rpm x 4 min). Mixture left to stand at RT for 9 min and centrifugation (3,900 rpm x 3 min)	NR	18	[100]
SE97	Callus cell (calli dried powder) / 0.3	MeOH / 12	US-SLE (37 °C, 20 min) and centrifugation	NR	69	[101]
Oils and resins						
SE98	Oils / 0.02	<i>n</i> -Hex:H ₂ O (1:1, v/v) / 2	US-LLE. Partitioning with a mixture of extraction solvent spiked and IS (benzo[ghi]perylene acetone solution, 1,120 mg/L), vortexing, sonication (15 min) and centrifugation (1,157g x 15 min)	NR	20	[102]
SE99	Oils / 1) 10; 2) 20	1) MeOH or EtOH or MeOH:H ₂ O or EtOH:H ₂ O (8:2, v/v) / 10 2) MeOH / 20	1) Classical LLE by shaking (RT, 1.5 h). Repeated 2x 2) SPE. Washing of the column with MeOH (5 mL) and <i>n</i> -Hex (5 mL). Oil and <i>n</i> -Hex (20 mL) mixture was loaded into column. Washing 3x with <i>n</i> -Hex (2.5 mL) and extraction with MeOH	NR	S02	[103]
SE100	Oils / 1 mL	ACN / 100	Sonication (15 min)	CBD: 1.57-149.18	17	[104]
SE101	Oils / 5x10 ⁻⁶	ACN:MeOH (4:1, v/v) / 25	Oil dilution in CH ₂ Cl ₂ (1 mL) and extraction solvent (total of 25 mL). US-LLE (10 min, low power)	NR	65	[67]
SE102	Oils, resins / 1	EtOH / 100 (resin) and 20 (oil)	Vortexing (10 min) and centrifugation (5,000 rpm x 10). Extracts cleanup with a mixture of activated carbon (C ₁₈) and Mg ₂ SO ₄ . Sonication (10 min) and centrifugation (5,000 rpm x 10)	NR	59	[22]
SE103	Seed oil / 0.5	MeOH / 25	Vortex (10 s) and sonication (15 min)	NR	35	[105]
SE104	Seed oil / 100 µL	IPA / 0.4	Sonication (10 min)	NR	30	[106]
SE105	CBD full-spectrum oils / 1	EtOH / 20	Stirring (30 °C, 120 min) and centrifugation (4,000 rpm x 10 min)	NR	100	[107]
SE106	CBD oils / 1.455	<i>n</i> -Hex:EtOAc:ACN:MeOH (8:2:8:2, v/v) / NR	LLE by partitioning in a biphasic solvent system	> 90	11	[108]
SE107	CBD oils (internet purchase) / 300 µL	MeOH / 10	Sonication (RT, 10 min)	NR	S11	[109]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Oils and resins						
SE108	Commercial CBD oil / 0.1	H ₂ O 30% (w/w) KBr / 1 and ES (100 µL)	DSLME. Vortexing (30 s) and sonication (40 KHz, 25 °C, 10 min). Vortexing (30 s) and centrifugation (2,200g x 5 min). ES-rich phase was collected and diluted with EtOH	NR	S04,S05	[69]
SE109	1) Oil / 80 µL 2) Resins / 0.001	1) DCM;ACN:MeOH (2:2.2:5.8, v/v) / 10 2) MeOH / 10	Sonication (RT, 15 min)	CBD: 99; CBDA: 97; CBN: 99; CBC: 100; CBG: 80; Δ ⁹ -THC: 96; THCA: 103 (for resins)	40	[45]
SE110	Resins / 0.05	MeOH / 5	Sonication (15 min)	NR	1	[110]
SE111	Resins / 0.5	EtOH / 10	SLE by agitation and grinding in a tube dry system of sample and extractant mixture (3,500 rpm x 4 min). Mixture left to stand at RT for 9 min and centrifugation (3,900 rpm x 3 min)	NR	18	[100]
SE112	Resins / 0.25	MeOH:CHCl ₃ (9:1, v/v) / 10	Vortexing (1 min), US-SLE (15 min) and centrifugation (3,000 rpm x 5 min)	NR	99	[111]
SE113	Resins / 0.05	MeOH:CHCl ₃ (8:2, v/v) / 10	Mixture with extraction solvent, vortexing (1 min). US-SLE (15 min), mechanical agitation (30 min) and centrifugation (1,370g x 1 min)	NR	64	[112]
SE114	Resins / 600	scCO ₂ / NR	SFE with extraction vessel (5 L, 18 MPa, 40 °C) and collection of extract after 1 h	NR	101	[113]
SE115	Resins (hemp concentrate) / 0.1	MeOH / NR ^f	US-SLE (20 min) and centrifugation (13,000 rpm, 10 min)	ACBD: 94.8-120.5	70	[114]
SE116	1) Concentrates (resins, rosins, distillates) / NR 2) Infused products / NR	EtOAc and 10% FA in dH ₂ O / NR	1) DM by homogenization in a Geno/Grinder (1350 cycles/s, 15 min) and centrifugation 2) DM by vortexing (2,000 rpm x 15 min) and centrifugation	NR	90	[27]
Food products						
SE117	Infused ice cream/ 3	CHCl ₃ /H ₂ O (1:3, v/v) / 4	Vortexing (25 s), UAE (20 min), and centrifugation (6,000 rpm x 5 min) (repeated 3x)	CBD: 86.8-92.2; 84.0-94.0 Δ ⁹ -THC:	S20	[16]
SE118	CBD edible products	MeOH / NR or another suitable organic solvents /NR	NR	NR	33	[115]
SE119	Proteic flour / 15	<i>n</i> -Hex / 400	4) Soxhlet extraction. NR	NR	S06	[70]
SE120	Flour, flour by- product (coarse residue of the sieving process) / 1	<i>n</i> -Hex / 1 and MeOH:H ₂ O (8:2, v/v) / 1	Sample was homogenized with <i>n</i> -Hex and second solvent added. Agitation (5 min), US-SLE (5 min) and centrifugation (3,000 rpm x 10 min). The MeOH/H ₂ O phase was collected and the residue reextracted 2x more. The methanolic aqueous phase were combined and washed with <i>n</i> -Hex (1 mL)	NR	30	[106]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
SE121	1) Water-based cannabis products / 0.5 2) Oil-based products / 0.125	1) MeOH / 25 2) <i>n</i> -Hex / 10	1) Centrifugation (15 min) of sample in MeOH and US-LLE (15 min) 2) Centrifugation (15 min) of sample in <i>n</i> -Hex and US-LLE (15 min). Solvent removal by pressurized air (vent hood) and residue reconstitution in MeOH (10 mL), sonication (15 min) and centrifugation (15 min)	CBD: 95-101; Δ ⁹ -THC: 93	88	[116]
SE122	Tea products / 0.2	1) ACN / 50 and 100 2) H ₂ O / NR	1) Teabags content US-SLE (10 min) 2) Teabags added to boiling deionized H ₂ O and left to infuse (5 min)	NR	17	[117]
SE123	CBD infused pomegranate juice/NR	MeOH / NR	10-fold dilution of juice (CBD concentration of 3.5 and 7 mg/L) in solvent, vortexing and centrifugation (4 °C, 1,700g x 15 min)	NR	34	[118]
SE124	1) Solid products / 0.08-0.09 2) Aqueous products / NR	1) EtOAc / 1 2) EtOAc / NR	1) US-SLE (15 min) in ice bath, centrifugation (12,000 rpm x 3 min) 2) Extraction with an equal volume of solvent	NR	53	[119]
SE125	THC in extra virgin olive oil / 0.3 mL	MeOH / 0.6	Extractant with IS (2 drops, ibuprofen 30 µg/mL in MeOH) added to sample, vortexing (30 s), US-LLE (20 min) and centrifugation (6,000 rpm x 15 min).	NR	S29	[120]
SE126	1) Liquid samples (oily samples, e-liquids, gels, honey) / NR 2) Gummy bears, candies / NR 3) Cookies, chewing gum, candy bars / 0.05 4) Tea beverages / NR	1) EtOAc, ACN, ACN:H ₂ O (1:1, v/v), MeOH:H ₂ O (1:1, v/v) / NR 2) MeOH:H ₂ O (1:1, v/v) / NR 3) ACN:FA (98:2, v/v) / 1.5 4) H ₂ O / 200	1) Dissolution and homogenization 2) US-SLE 3) US-SLE (15 min) and centrifugation (12100g x 7 min) (repeated 4x) 4) Infusing of one bag of tea (2x) in boiling for H ₂ O (5 min)	NR	32	[88]
SE127	Δ ⁸ -THC gummies / 1	MTBE:H ₂ O (1:1, v/v) / 200	US-LLE in an ice bath (40 min). Phase separation (10 min) and analysis of MTBE phase after filtration	NR	83	[121]
SE128	1) Chocolate, honey and gummies / NR 2) Drinks (e.g. CBD shots) / 10 mL 3) CBD-sachets coffee, capsules / 0.5	1) H ₂ O / 50 and ACN / 50 2) and 3) ACN / 100	1) US-SLE. Dissolution in H ₂ O and sonication (15 min), addition of ACN and sonication (15 min) 2) US-LLE (15 min) 3) US-SLE (45 min)	CBD: 4.22-87.15	17	[104]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^s	Ref.
Food products						
SE129	1) Aqueous tinctures, oils, e- liquids / NR 2) Drinks / NR	1) IPA:MeOH (1:1, m/m) or IPA / NR 2) MeOH / NR	1) dilution with extractant 2) dilution and US-LLE	NR	S23	[122]
SE130	Chocolate, cookie, cookie dough, and gummy solid / 0.1	ACN plus FA (2%) / 1	Crushing (5 min) of sample mixture with cold extractant using steel beads and centrifugation (13,400 x 5 min). Vortexing of supernatant (2 mL) mixed with H ₂ O (0.5 mL) and filtration through Captiva EMR-Lipid cartridge	NR	91	[123]
SE131	1) Liquid edibles (sparkling water, tea, SCD probiotics + CBD, water soluble CBG oil, CBNight water soluble CBN oil, water soluble full spectrum hemp oil, or terpene rich hemp oil tincture / 0.1 2) CBD gummies, CBD hard candy, and CBD coffee / 1 3) CBD brownies, CBD cookies, CBD krispy cereals, marshmallow treats, CBD popcorn, cat food kitty nugs, CBD horse treats, soft CBD dog treats / 1 4) CBD tablets / 0.1	1) and 4) MeOH / NR 2) MeOH:H ₂ O (95:5, v/v) / NR 3) MeOH / 4	1) and 4) Sonication (10 min) for homogenization and addition of a ACBD solution in MeOH (75 µg/mL) to afford a final sample concentration of 25 mg/mL. US-SLE (20 min) and centrifugation (13,000 rpm x 10 min) 2) Preparation of a H ₂ O-dispersion solution by shaking (3,000 rpm x 5 min) of sample (1 g) in H ₂ O (4 mL). Addition of H ₂ O-dispersion solution (200 µL) to a mixture of MeOH (1.8 mL) and ACBD solution in MeOH (2 mL, 75 µg/mL). Sonication (20 min) and centrifugation (13,000 rpm x 10 min) 3) Shaking (3,000 rpm x 5 min) of sample (1 g) in ACBD solution in MeOH (4 mL, 0.25 mg/mL) and sonication (20 min). Repeated 2x and centrifugation (13,000 rpm x 10 min)	ACBD: 90-108	86	[36]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^s	Ref.
Pharmaceutical e other formulated products						
SE132	Commercial veterinary CBD oil / 100 µL	MeOH / 10	US-LLE (30 °C, 5 min) with MeOH (5 mL) and dilution to the final volume	98-102	80	[124]
SE133	Suk-SaiYas pills / 0.05	MeOH / 5	US-SLE (30 min)	NR	S01	[125]
SE134	Commercial Suk- Saiyasna herbal remedy / 30	CH ₂ Cl ₂ / 300	US-SLE (30 min) (repeated 4x)	CBD: and Δ ⁹ -THC: 99.9	S16	[126]
SE135	Herbal and cosmetic creams / 0.5	ACN / 20	Agitation (15 min) of homogenous powdered sample mixture with ACN and IS (1 mg/mL), centrifugation (2,579g x 15 min, 4 °C)	CBD: 69.10-98.82; CDBA: 73.46-92.80; CBN: 61.37-96.76; CBCA: 67.10-86.35; CBG: 75.50-99.47; CBGA: 64.39-95.35; Δ ⁸ -THC: 62.54-89.85; Δ ⁹ -THC: 74.15-90.90; THCA: 76.85-86.48	98	[127]
SE136	CBD topical products	MeOH / NR or another suitable organic solvents /NR	NR	NR	33	[115]
SE137	Water-based CBD lotions / 0.0001	MeOH / 25	Shaking (15 s) of sample dissolved in MeOH, centrifugation (15 min) and US-LLE (RT, 15 min)	NR	87,89	[128]
SE138	Ointment cannabis leaf-based / 0.15	IPA / 5	US-SLE (15 min), kept at -20 °C for 30 min and centrifugation (4 °C, 10 min)	93.7-104	92	[129]
SE139	CBG emulgel (oil-in- gel emulsion) in 1) tapes and 2) membranes /	1) MeOH / 1) 10; 2) 30	US-LLE (RT, 1 h)	NR	S12	[130]
SE140	CBD adhesive bandages	MeOH and EtOAc / 25	US-SLE (15 min) of bandages small pieces	NR	32	[88]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Pharmaceutical and other formulated products						
SE141	Topicals (e.g. face mask, CBD-lip balm) / 0.5	ACN / 100	US-SLE (45 min)	CBD: 1.54-48.15	17	[104]
SE142	CBD inhalable powders / 0.001-0.002	PBS (pH 7.4) plus 0.5% sodium lauryl sulfate / 350	Stirring (37 °C, 100 rpm, withdraw of aliquots at predetermine times, max 90 min) and centrifugation (37 °C, 4255g x 10 min)	CBD: 103.2	S34	[131]
SE143	DPCC-CBD inhalable powder / 0.005	PBS (pH 7.4) / 50	Stirring (37 °C, 100 rpm, 32 h) and centrifugation (37 °C, 4255g x 10 min)	CBD: 100-110	S34	[132]
SE144	Tablets (oil-based products) / 0.005	ACN / 35	Dissolution with ACN of grinded and weighed sample (five tablets minimum). US-SLE (15 min) with intermitted shaking	CBD: 105.2; Δ ⁹ -THC: 100.2	S15	[133]
SE145	CBD sublingual oil formulations / 0.5	MeOH:CHCl ₃ (9:1) / 10	US-LLE (30 min) and allowed to stand frozen for 90 min before filtration	NR	S31	[134]
SE146	CBD nanoemulsion formulations / NR	ACN / NR	After digestion assay: disruption of nanoemulsion with ACN (ratio of 1:10) in separated mixed micelles and sediment phases. Centrifugation (1,613g x 5 min)	NR	S27	[135]
SE147	CBD nanoemulsion formulation / 0.001	MeOH / 1	US-LLE	NR	S19	[136]
SE148	CBD-loaded organosilica in 1) Cotton swabs, tapes / NR 2) Skin samples / NR	EtOH / NR	1) Shaking in a rotary mixer (RT, 12 h) of samples in EtOH 2) US-SLE (1 h)	NR	S24	[137]
SE149	CBD-loaded NP / 300 µL	EtOH / 300 µL	After gastric- and intestine-like release tests, the CBD nanoparticle suspension aliquots obtained at different time points were extracted with EtOH	NR	S25	[138]
SE150	CBD-loaded NP / 0.015	DCM	Mixture of nanoparticles in extractant solvent	NR	S14	[139]
SE151	Cannabis oil nanoemulsion / 0.05	MeOH / 10	US-SLE (15 min) and centrifugation (4,500g x 5 min)	NR	47	[140]
SE152	CBD-loaded nanostructured lipid carriers / NR	MeOH / NR	US-SLE (15 min)	NR	S30	[141]
SE153	CBD/HP-β-CD inclusion complex / NR	ACN:NH ₄ HCO ₂ (75:25, v/v, 20 mM, pH 3.6) / NR	Dissolution in mobile phase and US-LLE (RT, 40 min)	NR	92	[142]

Entry	Sample type / quantity (g) ^a	Extraction solvent / volume (mL)	Extraction method and protocol	Extraction recovery (%)	HPLC Entry ^g	Ref.
Pharmaceutical and other formulated products						
SE154	e-Cigarettes semiliquid / 0.025	MeOH / 10	US-LLE (20 min, 30 °C)	91.6-111.7	81	[60]
SE155	CBD e-cigarettes liquid / NR	ACN:H ₂ O (1:1, v/v) / NR	Dilute-and-shoot approach (1:50)	NR	75	[143]
SE156	Cigarette material / 0.1	MeOH / NR ^c	US-SLE (5 min) and vortexing (repeated 4x). Centrifugation (13,000 rpm, 10 min) of supernatant (2 mL)	ACBD: 93.6-106.1	84	[73]
SE157	Smoking product/ 0.1	MeOH / 10	SLE by shaking horizontally (200 oscillations/min, 90 min) light protected and centrifugation (3000g x 5 min)	NR	76	[74]
Biological materials						
SE158	Human and mouse plasma, mouse lung and liver / 100 µL mouse brain tissue / 200 µL	EtOAc / 500 µL	Addition of IS to sample, Simvastatin (50 µg/mL in MeOH) for human plasma and Perampanel (60 µg/mL in MeOH) for mouse matrices. Protein precipitation with IPA (150 µL) and vortex (60 s). LLE with vortexing-mixing (60 s), centrifugation (12,100g x 3 min) (repeated 2x)	CBD: 80.12-91.71; CBN: 86.81-88.63; CBC: 86.60-94.99; CBG: 77.31-101.89	3	[144]
SE159	Human plasma / 1 mL	NR	An aliquot of each sample (1 mL) from the binding assay was added to an micropartition system (MW cutoff 30,000). Centrifugation (2,000 rpm x 30 min) and unbonded CBD determination in ultrafiltrate assayed	NR	S33	[145]
SE160	1) CBD nanosuspension in rat skin / NR 2) Rat plasma / 100 µL	1) MeOH / 3 2) MeOH:ACN (1:1, v/v) / 0.2	1) Agitation in a shaker (24 h), sonication (30 min) (repeated 2x) 2) Sample mixture with IS (10 µL of Δ ⁹ -THC-D3, 1 µg/mL), vortex (1 min), addition of extractant. Vortexing (1 min) and centrifugation (10,000 rpm x 10 min)	NR	S17	[146]
SE161	1) CBD-polymer coated NP in agarose patch / NR 2) CBD-polymer coated NP in rat brain tissue / NR	1) EtOH / 2 2) Hex / 3	1) Sonication (30 min) of sample and EtOH (1 mL). Aspiration of solvent (700 µL) and repeat procedure after addition of EtOH (1 mL) 2) Protein precipitation followed by LLE. Vortex (5 min) and centrifugation (4,000g x 10 min)	NR	S18	[147]
SE162	Zebrafish larvae tissue / 12 specimens	MeOH + 0.1% FA / 200 µL	SLE by bead beating (4-8 °C, 3 min, 1 mm glass beads), centrifugation of homogenate (18,000g x 30 min)	NR	78	[148]

a) Quantities reported in grams (g) or otherwise clearly stated; b) Cannabinoid extract in MeOH prepared by SFE was used as secondary reference standards; c) No specification to which cannabinoids the values are referred too; d) Linear Relative Response Factors (LRRFM) and Relative Response Factors (RRF) models used to quantify low concentrations of Δ⁹-THC and THCA in cannabis flowers using CBD as reference; e) mixture of solvent that resulted in the highest content of phenolic compounds; f) The sample is mixed with a 75 µg/mL solution of ACBD (abnormal CBD) in MeOH to a final concentration of 25 mg/mL; g) The entry assigned in this column corresponds to the analytical method entry presented in Table 1, Table 2, Table S2, and Table S3. *Abbreviations:* ACN, acetonitrile; BUT, butane; DCM, dichloromethane; DSLME, dispersive solid-liquid microextraction; DM, dynamic maceration; DME, dimethyl ether; EtOAc, ethyl acetate; EtOH, ethanol; ES, eutectic solvent; FA, formic acid; *n*-Hex, *n*-hexane;

HP- β -CD, 2-hydroxypropyl- β -cyclodextrin; IPA, isopropanol; IS, internal standard; LLE, liquid-liquid extraction; MeOH, methanol; MTBE, Methyl *tert*-butyl ether; NP, nanoparticles; NR, not reported; NTP, normal temperature pressure; PBS, phosphate buffer saline; RT, room temperature; scCO₂, supercritical carbon dioxide; SFE, supercritical fluid extraction; SLE, Solid-liquid extraction; SPE, solid phase extraction; SRS, secondary reference standards; UAE, ultrasound-assisted extraction; US-SLE, ultrasound-assisted solid-liquid extraction; US-LLE, ultrasound-assisted liquid-liquid extraction.

Cannabinoids Abbreviations: ACBD, abnormal cannabidiol; CBD, cannabidiol; CBDA, cannabidiolic acid; CBDA-ME, cannabidiolic acid methyl ester; CBDP, cannabidiphlorol; CBE, cannabielsoin; CBEA, cannabielsoic acid; CBDV, cannabidivarin; CBC, cannabichromene; CBCA, cannabichromenic acid; CBCO, cannabichromeorcin; CBCV, cannabichromevarin; CBCVA, cannabichromevarinic acid; CBG, cannabigerol; CBGA, cannabigerolic acid; CBGAQ, cannabigerol quinone acid; CBGV, cannabigerovarin; CBGVA, cannabigerovarinic acid; CBL, cannabicyclol; CBLA, cannabicyclolic acid; CBN, cannabinol; CBNR, cannabinerol; CBT, cannabicitran; DCBD, dihydrocannabinol; 7-OH-CBD, 7-OH-cannabidiol; Δ^8 -THC, Δ^8 -tetrahydrocannabinol; Δ^9 -THC, Δ^9 -tetrahydrocannabinol; THCA, Δ^9 -tetrahydrocannabinolic acid; THCB, Δ^9 -tetrahydrocannabutol; THCH, Δ^9 -tetrahydrocannabihexol; THCP, Δ^9 -tetrahydrocannabiphlorol; THCV, Δ^9 -tetrahydrocannabivarin; THCVA, Δ^9 -tetrahydrocannabivarinic acid.

Table S2. Summary of LC parameters used in gradient chromatographic runs for detection of 1 or 2 cannabinoids

Entry	Method ^a	Mobile Phase ^b			Column	Flow Rate (mL/min)	Run Time (min) ^c	Injection Vol. (μL) ^d	Temp. (°C)	UV Detection (nm) ^e	Cannabinoids Analyzed ^f	Ref.
		Aqueous Phase (A)	Organic Phase (B)	Gradient % B								
SH01	HPLC – DAD		ACN	73.5 – 90	Zorbax Eclipse Plus C18 (100 × 4.6 mm, 3.5 μm)	0.5	32	10	30	220	2: CBD, Δ ⁹ -THC	[125]
SH02	UHPLC – DAD	0.05% FA	ACN	NR	Acquity UPLC HSS T3 (100 × 2.1 mm, 1.8 μm)	0.45	NR	10	40	NR	2: CBD, Δ ⁹ -THC	[149]
SH03	HPLC – DAD	0.1% FA	ACN	5 – 50	Phenomenex Luna C18 (2) (150 × 4.6 mm, 5 μm)	1	41 ^d	20	30	254	1-CBD	[81]
SH04	UHPLC – DAD	0.1% FA	ACN - 0.1%FA	15 – 86	Ascentis Express C18 (150 × 2.1 mm, 2.7 μm)	0.25	67	5	30	270	2: CBD, CBDA	[69]
SH05	UHPLC – DAD	0.1% FA	ACN - 0.1%FA	30 – 86	Ascentis Express C18 (150 × 2.1 mm, 2.7 μm)	0.25	56	3 or 5 ^d	30	270	2: CBD, CBDA	[69]
SH06	HPLC – DAD	0.1% FA	ACN - 0.1%FA	60 – 90	Ascentis C18 (150 × 2 mm) with a guard column	0.1	45	NR	32	210, 220, 235, 275	2: CBD, CBDA	[70]
SH07	HPLC – DAD	0.4% FA	ACN-0.4% FA	50 – 95	Zorbax RP C18 (150 × 4.6 mm, 5 μm)	1	10	20	25	210	2: CBD, CBG	[150]
SH08	HPLC – DAD	0.1% AcOH	ACN – 0.1% AcOH	5 – 95	Waters Sunfire C18 (250 × 4.6 mm, 5 μm)	0.8	60 ^d	20	25	220	2: CBDA, CBDVA	[78]
SH09	HPLC – DAD	2% AcOH	ACN	3 – 95	Zorbax Eclipse Plus C18 (250 × 4.6, 5 μm)	1	90	10	NR	280, 320, 360	1: Δ ⁹ -THC	[103,151]
SH10	HPLC – DAD	0.1%TFA – 2%ACN	ACN - 0.1%TFA – 2%H ₂ O	43 – 56	Phenomenex Kinetex C18 (100 × 4.6 mm, 1.7 μm)	2.0	50 ^d	NR	40	220, 280	2: CBD, CBDA	[18]
SH11	UHPLC – DAD	50 mM AF 10% MeOH	MeOH – 10% H ₂ O – 50mM AF	75 – 100	Kinetex Polar C18 (100 × 3 mm, 2.6 μm)	0.5	24	3	NR	210	1: CBD	[109]
SH12	HPLC – UV	20 mM Na ₃ (PO ₄) ₂ (pH 3.0)	MeOH	70 – 90	Syncronis aQ C18 (150 × 4.6 mm, 5 μm)	1	18.5	20	NR	220	2: CBD, CBG	[130]

AcOH: acetic acid; ACN: acetonitrile; AF: ammonium formate; DAD: Diode Array Detector; FA: formic acid, HPLC: high-performance liquid chromatography, MeOH: methanol; NR: not reported; TFA: trifluoroacetic acid; UHPLC: ultra high-performance liquid chromatography; UV: ultraviolet.

Cannabinoids Abbreviations: CBD: cannabidiol; CBDA: cannabidiolic acid; CBDVA: cannabidivarinic acid; CBG: cannabigerol; Δ⁹-THC: Δ⁹-tetrahydrocannabinol.

^a Type of liquid chromatography apparatus and UV detector used; when not specified, the UV term is used. Entries S04 and S05: Qualitative analysis by UHPLC-PDA-ESI-MS/MS. ^b Entry S01: Only water was used as aqueous phase. ^c When reported, run time includes column equilibration; Entries S3, S08, and S10: No column equilibration was reported in the description of the gradient. ^d Entry S05: CBD oil: 5 μL; Inflorescence: 3 μL. ^e Wavelengths used for cannabinoid detection and/ or quantification. ^f Entry S12: Both CBG and CBD have the same retention time.

Table S3: Summary of LC parameters used in isocratic chromatographic runs for detection of 1 or 2 cannabinoids

Entry	Method ^a	Mobile Phase ^b			Column	Flow Rate (mL/min)	Run Time (min)	Injection Vol. (μL) ^c	Temp. (°C)	UV Detection (nm) ^d	Cannabinoids Analyzed	Ref.
		Aqueous Phase (A)	Organic Phase (B)	Gradient % B								
SH13	HPLC – DAD		ACN	20:80	RP-8 (250 × 4mm, 5 μm) with a guard column	1	8	10	30	220, 240	2: CBD, Δ ⁹ -THC	[152,153]
SH14	HPLC – DAD		ACN	25:75	Restek, C18 (150 × 4.6 mm, 5 μm)	1	10	NR	NR	205	1: CBD	[139]
SH15	HPLC – UV/Vis		ACN	25:75	SOLAS C18 (150 × 4.6 mm, 5 μm)	1.5	20	10	25	214	2: CBD, Δ ⁹ -THC	[154]
SH16	HPLC – DAD		ACN	25:75	C18 (250 × 4.6 mm, 5 μm)	1	30	10	NR	208	2: CBD, Δ ⁹ -THC	[126]
SH17	HPLC – UV		ACN	30:70	YMC C18 (250 × 4.6 mm, 5 μm)	1	NR	NR	30	220	1: CBD	[146]
SH18	HPLC – DAD		ACN	38:62	ACE C18-PFP (150 × 4.6 mm, 3 μm) with a guard column	1	30	40	55	220	1: CBD	[147]
SH19	HPLC – DAD		ACN	55:45	Princeton Sphere Ultima C-18 (250 × 4.6 mm, 5 μm)	1	15	20	30	210	1: CBD	[136]
SH20	HPLC – DAD	0.1% FA	ACN – 0.1% FA	20:80	Purospher STAR RP-18 (120 × 4.6 mm, 5 μm)	1	10	NR	25	208: CBD 280: Δ ⁹ -THC	2: CBD, Δ ⁹ -THC	[16]
SH21	HPLC – DAD	0.1% FA	ACN – 0.1% FA	20:80	Zorbax SB-C18 (250 × 4.6 mm, 5 μm)	1	20	10	35	220	1- CBD	[155]
SH22	HPLC – DAD	0.1% FA	ACN – 0.1% FA	40:60	Cortecs shield RP18 (150 × 4.6, 2.7 μm) with a guard column	1.6	25	10 or 50 ^e	30	223	1: THCA	[59]
SH23	HPLC – UV	AF 5 mM – 0.1% FA	ACN – 0.1% FA	25:75	Raptor ARC-18 (150 × 4.6 mm, 2.7 μm)	1.5	10	5	30	228	2: CBD, CBDA	[122]
SH24	HPLC – UV	0.1% TFA	ACN	25:75	Grace Davison C18 (250 × 4.6 mm; 5 μm)	0.8	20	20	NR	210	1: CBD	[137]
SH25	HPLC – DAD	0.1% AcOH	ACN – 0.1% AcOH	15:85	Kinetex C18 (150 × 2.1 mm × 2.9 μm) with a guard column	0.25	5	1-4	30	NR	1: CBD	[138]
SH26	HPLC – DAD	0.1% AcOH	ACN – 0.1% AcOH	40:60	Waters Sunfire C18 (250 × 4.6 mm, 5 μm)	0.8	40	20	25	220	2: CBDA, CBDVA	[78]
SH27	HPLC – DAD	0.2% AcOH	ACN	15:85	Infinity Lab Poroshell 120 EC-C18 (250 × 4.6 mm, 2.7 μm) with a guard column	0.6	NR	5	25	280	1: CBD	[135]
SH28	HPLC – UV	0.5% AcOH	ACN	30:70	ACE C18-AR	2	20	10	25	220	1: Δ ⁹ -THC	[156]
SH29	HPLC – UV/Vis	Phosphate buffer (pH 2.5)	ACN	30:70	ACE RP C18 (250 × 4.6 mm; 5 μm)	2	13.5	20	40	220	1: Δ ⁹ -THC	[120]

Entry	Method ^a	Mobile Phase ^b			Column	Flow Rate (mL/min)	Run Time (min)	Injection Vol. (μL) ^c	Temp. (°C)	UV Detection (nm) ^d	Cannabinoids Analyzed	Ref.
		Aqueous Phase (A)	Organic Phase (B)	Gradient % B								
SH30	HPLC – UV		MeOH	15:85	Dikma Platisil ODS (250 × 4.6 mm, 5 μm)	1.2	10	20	NR	220	1-CBD	[141]
SH31	HPLC – DAD		MeOH	17.5:82.5	Shimadzu shim-pack GIST C-18 (150 × 4.6 mm, 5 μm)	1	15	10	30	210, 228	1: CBD	[134]
SH32	HPLC – DAD		MeOH	20:80	RP-C18 with a guard column	1	55	20	30	220	2: CBD, Δ ⁹ -THC	[44,47]
SH33	HPLC – UV	Phosphate buffer (0.1% PA, pH 2.16)	MeOH	10:90	Phenomenex C18 (250 × 4.6 mm, 5 μm) with a guard column	0.9	NR	40	30	220	1: CBD	[145]
SH34	HPLC – UV/Vis		MeOH:THF (93.4:6.6)	24:76	Zobrax SB-C18 (150 × 4.60 mm, 5 μm)	1	NR	NR	NR	228	1: CBD	[131,132]

AcOH: acetic acid; ACN: acetonitrile; AF: ammonium formate; DAD: Diode Array Detector, FA: formic acid, HPLC: high-performance liquid chromatography, MeOH: methanol; NR: not reported; TFA: trifluoroacetic acid; THF: Tetrahydrofuran; UHPLC: ultra high-performance liquid chromatography; UV: ultraviolet; Vis: visible.

Cannabinoids Abbreviations: CBD: cannabidiol; CBDA, cannabidiolic acid; CBDVA: cannabidivarinic acid; CBG: cannabigerol; Δ⁹-THC: Δ⁹-tetrahydrocannabinol; THCA: Δ⁹-tetrahydrocannabinolic acid.

^a Type of liquid chromatography apparatus and UV detector used; when not specified, the UV term is used. ^b Entry S13–19, S30–32, and S34: Only water was used as aqueous phase. ^c Entry S22: standards: 10 μL; Samples: 50 μL. ^d Wavelengths used for cannabinoid detection and/ or quantification.

Table S4. Summary of validation parameters: Linearity, Precision, Accuracy, LOD and LOQ.

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c					Guidelines	HPLC entry ^d	Ref
SV01				Spiking placebo solutions				Repeatability (6 samples, n = 2)	Intermediate (IP) (6 samples, n = 2)		Inter- Analyst (IA)				
	CBD Δ ⁹ -THC	0.9999 0.9999	5 – 15 0.5 – 1.5	100 – 105 98 – 100		NR NR	NR NR	0.23 0.43	0.14 0.64		0.26 0.94		ICH Q2 (R1)	S15	[133]
SV02				Spiking surrogate matrices (SM) (1-3 conc)		Based on SD of a linear response and a slope		Instr. P	Method P		Method IP (IA)		ICH Q2 (R1)	65	[67]
				For Inflorescences (chamomile)	For Oil (olive oil)	Inflorescence (µg/g)		Solv	Infl	Oil	Infl SM	Oil SM			
	CBC	0.99999	3.1 – 250	100.8	NR	20	60	0.32	1.41	NR	1.23	NR			
	CBD	0.99998	3.0 – 240	95.3 – 102.4	98.2 – 103.1	35	107	1.49	1.15	1.27	0.67	1.60			
	CBDA	0.99998	2.8 – 230	102.4	101.9	41	123	0.10	1.24	NR	3.01	NR			
	CBDV	0.99998	3.1 – 240	103.1	NR	39	118	0.44	NR	NR	NR	NR			
	CBG	0.99996	2.9 – 230	95.4	NR	57	172	2.00	1.61	NR	1.76	NR			
	CBGA	0.99998	3.0 – 240	97.9 – 109.3	NR	31	94	0.68	1.76	NR	1.70	NR			
	CBN	0.99999	2.8 – 220	102.9	NR	55	167	0.38	3.58	NR	4.58	NR			
	Δ ⁸ -THC	0.99998	3.1 – 250	90.1	NR	48	144	1.14	NR	NR	NR	NR			
	Δ ⁹ -THC	0.99993	3.0 – 240	100 – 105.1	95.4 – 98.2	78	238	1.21	1.23	1.32	2.34	1.28			
THCA	1.00000	3.0 – 240	96.9	100.7	32	96	0.68	2.09	NR	3.97	NR				
SV03				Spiking placebo matrix – cannabis extraction waste (CEW) (3 conc)		Based on S/N		Method P Samples (n = 3)		Spiking CEW (3 conc.)					
				Intra-day Day1	Inter-day	S/N ≥ 3	S/N ≥ 10			Intra-day (n = 6)	Inter-day (3 days)				
	CBC	0.9997	5 – 75	90 – 94	90.57 – 94.22	1.1	3.3	0.58 – 10.63		0.53 – 7.86		2.12			
	CBCA	0.9996	5 – 75	81 – 93	83.15 – 92.33	1.6	4.8	5.00 – 13.63		2.21 – 9.84		5.26			
	CBD	0.9998	5 – 75	95 – 102	95.94 – 101.04	0.3	0.9	1.48 – 8.34		0.58 – 1.74		2.72			
	CBDA	0.9997	5 – 75	87 – 100	91.42 – 100.67	0.3	0.9	1.13 – 6.70		0.73 – 2.06		4.81			
	CBDV	0.9997	5 – 75	95 – 101	95.60 – 99.17	1.3	3.9	4.61 – 5.46		0.35 – 1.41		1.96			
	CBDVA	0.9999	5 – 75	86 – 97	90.10 – 97.67	1.3	3.9	4.13 – 8.45		0.44 – 1.36		4.05			
	CBG	0.9995	5 – 75	95 – 100	95.53 – 98.03	0.4	1.2	2.63 – 14.58		1.37 – 2.21		1.32			
	CBGA	0.9996	5 – 75	89 – 101	90.84 – 99.22	1.6	4.8	1.13 – 11.92		1.53 – 2.84		4.44			
	CBL	0.9998	5 – 75	93 – 98	94.28 – 96.28	0.7	2.1	NR		0.71 – 2.55		1.11			
	CBN	0.9998	5 – 75	93 – 97	93.24 – 97.37	0.5	1.5	1.43 – 14.46		0.43 – 1.25		2.31			
	Δ ⁸ -THC	0.9994	5 – 75	93 – 96	91.11 – 95.02	1.1	3.3	0.82 – 13.78		1.52 – 4.15		2.27			
	Δ ⁹ -THC	0.9997	5 – 75	94 – 99	94.81 – 96.89	0.8	2.4	1.34 – 8.61		0.92 – 2.32		1.16			
	THCA	0.9995	5 – 75	85 – 93	88.83 – 91.96	1.5	4.5	0.75 – 9.17		0.84 – 7.71		1.80			
	THCV	0.9998	5 – 75	95 – 101	95.68 – 98.56	0.2	0.6	1.86 – 8.97		0.65 – 1.60		1.63			
	THCVA	0.9909	5 – 75	84 – 95	88.66 – 94.41	1.5	4.5	NR		0.53 – 7.86		3.18			

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref
SV04				(1 conc; MeOH solutions)		Based on S/N S/N ≥ 3 S/N ≥ 10		Intra-day (1 Std solution, n= 3)	Inter-day (1 Std solution, 3 days)			
	CBC	0.998	2.5 – 50	91.9		1.6	5.2	0.1 – 5.8	6.0	ICH Q2 (R2)	29	[157]
	CBD	0.999	51.5 – 206	104.0 (synthetic) 106.2 (natural)		0.4	1.3	4.8 – 6.5	6.1			
	CBDDB	0.998	2.5–50	94.0		0.6	2.1	5.2 – 6.3	6.0			
	CBDV	0.998	2.5–50	93.9		0.3	1.0	4.5 – 9.3	7.4			
	Δ ⁹ -THC	0.999	2.5–50	82.9		0.3	0.8	5 – 5.4	5.6			
	Δ ⁸ -THC	0.996	2.5–50	96.6		1.0	3.4	1.5 – 7	6.2			
SV05				Standard addition method (3 conc)		Based on SD of a linear response and a slope		Intra-day (3 conc, n= 3)	Inter-day (3 conc, 3 days)			
	CBD	0.9951	10 – 60	97.84 – 99.34		0.483622	1.465521	0.24 – 0.54	0.19 – 0.47	ICH	S19	[136]
SV06				Standard addition method Inflorescences Resins		Based on standard deviation of a linear response and a slope		Intra-day (3 Std solutions; n = 3)	Inter-day (3 Std solutions; 3 days)			
	CBC	0.9997	5 – 100	98	100	1.04	3.15	2.44	1.71	ICH Q2 (R2)	40	[45]
	CBD	0.9999	5 – 150	98	99	1.76	5.33	3.14	2.62			
	CBD A	0.9999	5 – 100	115	97	1.96	5.96	2.34	1.16			
	CBG	0.9998	5 – 100	97	80	1.00	3.03	2.68	1.61			
	CBN	0.9994	5 – 150	98	99	3.66	11.09	3.32	2.53			
	Δ ⁹ -THC	0.9998	5 – 100	94	96	0.91	2.78	4.82	3.15			
	THCA	0.9998	5 – 100	99	103	1.82	5.59	4.27	1.84			
SV07				Standard addition method (before extraction; 3 conc) Hemp pool Seized pool		Based on SD of a linear response and a slope		Intra-day (3 Std solutions; n = 6)	Inter-day (3 Std solutions; 3 days)			
	CBC	0.9999	0.5 – 50	83 – 87.7	86.9 – 89.5	0.03	0.09	0.05 – 0.25	0.36 – 1.16	ICH Q2 (R1)	15	[39]
	CBCT	0.9996	0.5 – 50	NR	NR	0.05	0.17	0.26 – 0.85	0.96 – 1.74			
	CBD	0.991	0.5 – 100	81.3 – 84.8	95.0 – 103.9	0.03	0.09	0.06 – 0.16	1.34 – 1.84			
	CBD A	0.9992	0.5 – 100	105.0 – 114.8	104.0 – 114.1	0.04	0.13	0.10 – 0.17	0.18 – 0.56			
	CBDV	0.9995	0.5 – 50	96.5 – 98.8	105.9 – 118.4	0.04	0.12	0.19 – 0.39	1.06 – 1.85			
	CBDVA	0.9997	0.5 – 50	102.2 – 104.3	93.4 – 94.6	0.04	0.14	0.07 – 0.99	0.21 – 2.04			
	CBE	0.9996	0.5 – 50	104.9 – 106.8	96.5 – 101.9	0.05	0.16	0.24 – 1.31	1.17 – 1.62			
	CBN	0.9998	0.5 – 50	112.5 – 116.3	107.8 – 110.2	0.02	0.06	0.11 – 0.31	0.57 – 1.30			
	CBGA	0.9999	0.5 – 50	100.8 – 108.2	96.6 – 96.8	0.03	0.10	0.09 – 0.18	0.20 – 1.39			
	Δ ⁹ -THC	0.996	0.5 – 100	105.9 – 116.2	108.3 – 117.3	0.04	0.14	0.09 – 0.42	1.12 – 1.86			
	Δ ⁹ -cis- THC	0.9995	0.5 – 50	97.4 – 103.0	96.9 – 106.0	0.06	0.17	0.16 – 0.64	1.07 – 1.74			
	THCA	0.9997	0.5 – 100	100.7 – 103.1	110.2 – 111.3	0.04	0.11	0.07 – 0.21	0.22 – 0.29			
SV08	% in oil			Spiking blank matrix (MCT oil) (before dilution, 3 conc), % Bias		% in oil		Intra-day (Std solutions; n = 3)	Inter-day (Std solutions; 3 days)			
	Δ ⁹ -THC	0.9957	0.03125 – 0.5	-5.0016 – 1.8606		0.01	0.03125	8.35 – 13.68	3.80 – 12.50	ICH Q2 (R1)	S28	[156]

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c				Guidelines	HPLC entry ^d	Ref	
SV09				<i>Intra-day</i> (4 QC in human plasma; n = 5, % Bias)	<i>Inter-day</i> (4 QC in human plasma; % Bias)			<i>Intraday</i> (4 QC in human plasma; n = 5)	<i>Interday</i> (4 QC in human plasma)						
	CBC	0.9958	0.1 – 4	-13.72 – -0.77	-2.28 – 0.29	NR	0.1	5.66 – 12.81	5.72 – 11.13	ICH M10	3	[144]			
	CBD	0.9963	0.05 – 4	-18.30 – -2.61	-11.61 – 1.03	NR	0.05	2.16 – 10.12	6.51 – 15.93						
	CBG	0.9873	0.15 – 4	-14.11 – 7.62	-4.17 – 7.55	NR	0.15	2.52 – 8.21	5.58 – 13.25						
	CBN	0.9951	0.05 – 4	-11.57 – -3.49	-2.10 – 4.69	NR	0.05	1.69 – 11.09	8.84 – 11.75						
SV10				<i>Spiking experiment</i> (enrichment oil samples, 3 conc)		<i>Based on SD of a linear response and a slope S/N was also checked</i>		<i>Intra-day</i> (Std solutions; n = 18)	<i>Inter-day (Std solutions; 3 days)</i>	ICH Q2 (R1)	80	[124]			
	CBC	0.99940	0.1 – 85	99.51		0.10	0.50	5.12	4.64						
	CBD	0.99951	0.1 – 85	99.43		0.05	0.50	5.41	4.96						
	CBDA	0.99937	0.1 – 85	99.75		0.05	0.50	3.76	3.31						
	CBDV	0.99947	0.1 – 85	98.32		0.05	0.50	4.67	4.13						
	CBG	0.99945	0.1 – 85	98.67		0.05	0.60	4.89	4.61						
	CBGA	0.99939	0.1 – 85	98.15		0.05	0.50	3.81	2.97						
	CBL	0.99959	0.1 – 85	97.24		0.10	0.61	5.30	4.74						
	CBN	0.99954	0.1 – 85	99.37		0.05	0.50	2.59	2.65						
	Δ ⁸ -THC	0.99951	0.1 – 85	100.85		0.10	0.60	3.81	3.58						
	Δ ⁹ -THC	0.99951	0.1 – 85	98.56		0.10	0.50	3.05	3.17						
	THCA	0.99940	0.1 – 85	101.47		0.13	0.59	5.42	5.04						
	THCV	0.99973	0.1 – 85	101.24		0.05	0.50	4.43	3.78						
SV11				<i>Spiking experiment</i> (Extra virgin olive oil - EVOO, 3 conc)		<i>Based on visual evaluation</i>		<i>Intra-day (Std in EVOO; 6 conc; n = 3)</i>	<i>Inter-day (Std in EVOO; 6 conc; 3 days)</i>						
	Δ ⁹ -THC	0.9998	0.039 – 5.0	92.308 – 100		0.019	0.039	0.580 – 4.487	0.949 – 6.897	ICH Q2	S29	[120]			
SV12				<i>Spiking experiment</i> <i>Intra-day (Std sol; 3 conc; n = 6)</i> <i>Inter-day (Std sol; 3 conc; 3 days)</i>		<i>Based on SD of a linear response and a slope S/N was also checked</i>		<i>Intra-day (Std sol; 3 conc; n = 6)</i>	<i>Inter-day (Std sol; 3 conc; 3 days)</i>						
	CBD	0.9988	0.10 – 100	100.7 – 101.4	100.3 – 102.2	0.19	0.57	0.987 – 1.003	0.995 – 1.008	ICH Q2	S07	[150]			
	CBG	0.9993	0.10 – 100	100.2 – 101.3	100.2 – 102.7	0.17	0.51	0.989 – 1.004	0.992 – 1.015						
SV13				<i>Intra-day/ Inter-day SM (1 conc)</i> <i>Olive oil Sunflower</i>		<i>Standard addition</i> <i>Samples</i>	<i>Based on S/N</i> <i>S/N ≥ 3 S/N ≥ 10</i>		<i>Intra-day</i> (1 conc; n = 3) <i>Olive oil Sunflower</i>		<i>Inter-day</i> (1 conc, 2 days) <i>Olive oil Sunflower</i>				
	CBD	0.9973 – 0.9995	0.3 – 40	102.2/ 97.0	110.9/ 107.8	121	0.03	0.05	0.3	1.4	8.1	4.2	ICH Q2 (R2)	82	[158]
	CBN	0.9973 – 0.9995	0.3 – 40	100/ 94.6	108.7/ 105.7	105	0.03	0.05	0.3	0.4	7.4	4.8			
	Δ ⁹ -THC	0.9976 – 0.9995	0.3 – 40	100/ 94.6	106.1/ 103.3	100	0.03	0.05	1.7	1.2	7.9	4.6			

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a	LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref
SV14				Standard addition method	Based on SD of a linear response and a slope		Intra-day (sample; n = 6)	Inter-day (sample; 6 days)			
	CBC	0.9996	5 – 100	94.11	1.1	3.4	NR	NR	ICH Q2 (R1)	19	[97,15 9]
	CBD	0.9990	5 – 100	90.96	1.6	4.9	0.6	2			
	CBDA	0.9988	5 – 100	90.0	6.1	18.4	0.7	6.6			
	CBG	0.9955	5 – 100	84.2	2.5	7.7	NR	NR			
	CBGA	0.9997	5 – 100	84.7	1.3	3.9	0.9	2			
	CBN	0.9948	5 – 100	93.16	0.3	1.0	NR	NR			
	Δ ⁸ -THC	0.999	5 – 100	67.7	1.4	4.2	NR	NR			
	Δ ⁹ -THC	0.9977	5 – 100	89.67	2.3	6.9	0.7	3.2			
	THCA	0.9998	5 – 100	91.49	1.1	3.4	1.2	1.7			
THCV	0.9999	5 – 100	91.75	0.7	2.1	NR	NR				
SV15				(Std sol; 4 conc)	Based on S/N S/N ≥ 3 S/N ≥ 10		Intra-day (Std sol; 4 conc; n = 5)	Inter-day (Std sol; 4 conc; 3 days)			
	CBC	0.996	1 – 100	7	0.85	2.54	0.4	0.2	AOAC Appendix K	93	[1,6– 8,160]
	CBCA	0.996	5 – 100	5	3.15	9.50	0.8	0.2			
	CBD	0.999	1 – 500	10	0.80	2.68	0.6	0.08			
	CBDA	0.999	1 – 1000	3	0.36	1.21	0.7	0.1			
	CBDVA	0.999	1 – 50	3	0.21	0.70	1.7	0.8			
	CBG	0.999	1 – 100	2	0.77	2.57	0.5	0.2			
	CBGA	0.999	1 – 100	2	0.46	1.55	0.6	0.2			
	CBL	0.996	1 – 100	10	0.81	2.60	0.7	0.1			
	CBN	0.997	1 – 100	5	0.43	1.43	0.6	0.04			
	Δ ⁸ -THC	0.999	1 – 100	10	1.05	3.12	0.8	0.2			
	Δ ⁹ -THC	0.996	1 – 500	8	1.11	3.69	1.4	1.9			
	THCA	0.999	1 – 1000	4	0.76	2.52	0.5	0.4			
	THCV	0.999	1 – 100	11	0.63	2.51	0.7	0.1			
	THCVA	0.999	1 – 50	2	0.34	1.15	2.2	0.9			
SV16				Spiking enriched cannabinoid mixtures to ointment base	Based on S/N S/N ≥ 3 S/N ≥ 10		Intra-day (Std sol; 3 conc; n = 3)	Inter-day (Std sol; 3 conc; 3 days)			
	CBC	0.9998	2.5 – 50	104	0.10	1.0	0.5 – 3.4	3.0 – 4.5	AOAC Appendix K	92	[129]
	CBD	0.9999	2.5 – 50	101.1	0.25	2.5	0.6 – 3.9	1.4 – 3.7			
	CBDA	0.9998	2.5 – 50	100.7	0.10	1.0	0.5 – 2.6	1.1 – 3.7			
	CBG	0.9999	2.5 – 50	100.3	0.25	2.5	0.2 – 3.5	1.5 – 3.8			
	CBGA	0.9999	2.5 – 50	93.7	0.10	1.0	0.6 – 2.9	1.2 – 3.9			
	CBN	0.9999	2.5 – 50	101.3	0.10	1.0	0.4 – 4.1	1.1 – 3.5			
	Δ ⁸ -THC	0.9999	2.5 – 50	NR	0.25	2.5	1.1 – 2.9	1.7 – 3.3			
	Δ ⁹ -THC	0.9999	2.5 – 50	103.5	0.25	2.5	0.6 – 4.2	0.8 – 3.7			
	THCA	0.9998	2.5 – 50	94.4	0.25	2.5	0.2 – 2.1	1.6 – 4.6			
THCV	0.9999	2.5 – 50	103.1	0.25	2.5	0.5 – 2.9	0.9 – 4.0				

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref
SV17				Spiking experiment (Blank matrix: Fibre-type herbal cannabis material, 3 conc, 3 – 4 days) Relative Bias % β-expectation tolerance interval %		Based on S/N S/N ≥ 3 (% w / w) S/N ≥ 3 (% w / w)		Repeatability (blank matrix; 3 conc; n = 3)	IP (blank matrix; 3 conc; 3 – 4 days)			
	CBD	0.99819	6.2 – 143.9	-1.02 – 3.28	-11.70 – 12.48	0.02	0.05	0.76 – 2.45	2.72 – 4.61	ISO-17025	76	[28]
	Δ ⁹ -THC	0.99671	5.0 – 100.9	-0.57 – 2.21	-9.04 – 7.91	0.03	0.06	1.07 – 2.68	2.01 – 3.63			
	THCA	0.99761	4.96 – 99.2	0.12 – 2.93	-3.94 – 5.45	0.01	0.03	1.23 – 2.03	1.23 – 2.03	ISO-17025	76	[74]
	CBDA	0.9979	10 – 120	-0.24 – 0.63	-13.13 – 14.39	0.02	0.2	1.15 – 3.81	2.86 – 4.08			
SV18				total overall bias %	β-expectation tolerance interval %	µg/g		Repeatability	IP			
	CBD	1	2.5 – 50	-0.27 – 2.33	-6.54 – 5.99	1	5	0.387 – 0.62	0.387 – 1.29	ISO-17025	75	[143]
	CBDA	0.9998	2.5 – 50	-2.27 – -1.09	-9.95 – 7.77	1	5	0.45 – 2.95	0.651 – 3.391			
	Δ ⁹ -THC	1	2.5 – 50	-2.80 – -0.18	-9.83 – 4.95	1	5	0.5 – 2.903	1.115 – 2.903			
	THCA	0.9998	2.5 – 50	-5.25 – 1.02	-10.35 – 10.19	1	5	0.403 – 1.366	0.965 – 2.533			
SV19				Intra-day – Day 1 (QC in solv; 3 conc)	Inter-day (QC in solv; 3 days)			Intra-day (QC in solv; 3 conc; n = 3)	Inter-day (QC in solv; 3 days)			
	CBC	≥ 0.994	0.02 – 25	97.4 – 108.3	100 – 105.5	NR	0.02	0.1 – 2.5	1.7 – 2.7	ISO-17025	84	[73]
	CBCA		0.02 – 25	90.2 – 102.3	95.2 – 105.7	NR	0.02	0.1 – 5.4	2.8 – 5.4			
	CBD		0.02 – 25	97.5 – 100.2	100.5 – 103.7	NR	0.02	0.3 – 2.7	3.3 – 5.6			
	CBDA		0.02 – 25	98.5 – 103.3	99.9 – 102.7	NR	0.02	0.1 – 3.1	0.5 – 3.6			
	CBDV		0.02 – 25	97.6 – 104.9	98.5 – 105.2	NR	0.02	0.0 – 4.9	0.8 – 2.7			
	CBDVA		0.02 – 25	100.3 – 106.2	99.4 – 103.6	NR	0.02	0.1 – 8.6	2.2 – 5.4			
	CBG		0.02 – 25	96.2 – 102.7	99.6 – 100.6	NR	0.02	0.1 – 4.6	4.7 – 8.1			
	CBGA		0.02 – 25	98.1 – 105.4	100.1 – 103.8	NR	0.02	0.1 – 3.1	1.3 – 3.6			
	CBL		0.02 – 25	89.6 – 105.6	92.2 – 105	NR	0.02	0.2 – 4.2	1.1 – 6.0			
	CBLA		0.02 – 25	90.0 – 107.0	96.7 – 104.9	NR	0.02	0.1 – 5.3	1.7 – 8.7			
	CBN		0.02 – 25	97.7 – 104.5	99.8 – 104.5	NR	0.02	0.3 – 3.3	0.5 – 4.1			
	CBNA		0.02 – 25	97.1 – 109.4	99.6 – 106	NR	0.02	0.3 – 3.8	1.0 – 5.8			
	CBT		0.02 – 25	90.9 – 104.3	99.5 – 103.5	NR	0.02	0.7 – 8.0	1.2 – 8.2			
	Δ ⁸ -THC		0.02 – 25	95.9 – 99.0	100.5 – 103.4	NR	0.02	0.1 – 3.8	2.4 – 6.9			
	Δ ⁹ -THC		0.02 – 25	96.5 – 103.4	98.1 – 102.5	NR	0.02	0.2 – 5.3	0.8 – 4.7			
	THCA		0.02 – 25	91.1 – 112.3	99 – 107	NR	0.02	0.1 – 4.9	3.0 – 9.4			
	THCV		0.02 – 25	97.7 – 104.1	100.2 – 103.8	NR	0.02	0.4 – 3.4	0.4 – 4.1			
	THCVA		0.02 – 25	98.6 – 105.0	97.1 – 104.8	NR	0.02	0.1 – 7.0	0.2 – 3.7			
SV20				Intra-day – Day 1 (QC in solv; 3 conc)	Inter-day (QC in solv; 3 days)			Intra-day (QC in solv; 3 conc; n = 3)	Inter-day (QC in solv; 3 days)			
	Δ ⁹ -THC	0.9990- 0.9992	0.04 – 50	101.3 – 104.0	100.8 – 106.8	NR	0.04	0.1 – 5.7	0.1 – 3.7	ISO-17025 + ANSI/ASB Standard 036	102	[34]
	THCA	0.9978- 0.9987	0.04 – 50	100.8 – 105.0	99.3 – 104.5	NR	0.04	0.1 – 6.3	0.3 – 2.3			

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref
SV21				<i>Intra-day – Day 1 (QC in solv; 3 conc)</i>	<i>Inter-day (QC in solv; 3 days)</i>			<i>Intra-day (QC in solv; 3 conc, n = 3)</i>	<i>Inter-day (QC in solv; 3 days)</i>	ISO-17025	86	[36]
	CBC	≥ 0.9938	0.02 – 25	95.5 – 100.4	94.8 – 100.7	NR	0.02	0.2 – 3.2	0.8 – 2.6			
	CBCA		0.02 – 25	96.6 – 102.8	96.1 – 107.4	NR	0.02	0.3 – 2.7	1.1 – 3.8			
	CBD		0.02 – 25	92.8 – 112.4	90.9 – 105.2	NR	0.02	0.1 – 5.6	0.6 – 5.9			
	CBD A		0.02 – 25	94.2 – 103.1	95.9 – 101.9	NR	0.02	0.1 – 4.5	1 – 4.7			
	CBDV		0.02 – 25	98.3 – 105.1	96.1 – 102.6	NR	0.02	0.2 – 3.7	1.3 – 3.3			
	CBDVA		0.02 – 25	94.6 – 99.7	94.7 – 100.5	NR	0.02	0.2 – 7.3	0.3 – 3.3			
	CBG		0.02 – 25	97.8 – 103.3	96.3 – 101.5	NR	0.02	0.1 – 5.1	1.8 – 6.2			
	CBGA		0.02 – 25	91.6 – 98.4	94.6 – 99.2	NR	0.02	0.2 – 7.9	0.6 – 7.3			
	CBN		0.02 – 25	65.2 – 104.4	94.9 – 102.4	NR	0.02	0.0 – 7.2	0.7 – 3.5			
	CBT		0.02 – 25	94.6 – 108.6	94.7 – 103.4	NR	0.02	0.2 – 4.2	0.4 – 9.7			
	Δ ⁸ -THC		0.02 – 25	95.3 – 109.9	94.6 – 108.2	NR	0.02	0.3 – 5	0.9 – 4.7			
	Δ ⁹ -THC		0.02 – 25	95.3 – 110.7	94.7 – 105.8	NR	0.02	0.1 – 6.3	0.8 – 5.4			
	THCA		0.02 – 25	95.3 – 98.7	95.3 – 98.8	NR	0.02	0.1 – 5.6	0.3 – 3.6			
THCV	0.02 – 25		95.1 – 103.5	94.8 – 101	NR	0.02	0.1 – 4.8	0.4 – 3.1				
THCVA	0.02 – 25	95.1 – 102.6	96.8 – 106	NR	0.02	0.2 – 4.3	2.8 – 5.4					
SV22				<i>Intra-day – Day 1 (QC in solv; 3 conc)</i>	<i>Inter-day (QC in solv; 3 days)</i>	<i>Based on S/N</i>		<i>Intra-day (QC in solv; 3 conc, n = 3)</i>	<i>Inter-day (QC in solv; 3 days)</i>	ISO-17025	61	[35]
	CBC	≥ 0.9899	0.04 – 50	98.2 – 102.6	98.3 – 101.9	0.009	0.029	0.1 – 7.2	0.9 – 2.3			
	CBCA		0.04 – 50	94.2 – 111.6	95.7 – 110.9	0.007	0.022	0.1 – 4.5	0.5 – 4.5			
	CBDV		0.04 – 50	98.2 – 103.1	97.3 – 103.6	0.005	0.016	0.4 – 3.2	0.3 – 1.1			
	CBDVA		0.04 – 50	98.4 – 102.8	96.7 – 103.4	0.004	0.012	0.2 – 5.3	0.1 – 3.8			
	CBD		0.04 – 50	97.5 – 101.5	97.1 – 100.7	0.007	0.023	0.0 – 9.6	0.7 – 1.4			
	CBD A		0.04 – 50	99.1 – 105.5	98.7 – 106.3	0.005	0.018	0.2 – 7.5	0.3 – 1.5			
	CBG		0.04 – 50	98.5 – 105.4	98.5 – 104.6	0.006	0.022	0.1 – 6.1	0.2 – 0.7			
	CBGA		0.04 – 50	97.3 – 104.2	98.2 – 105.1	0.007	0.022	0.3 – 4.9	1 – 3.3			
	CBL		0.04 – 50	99.3 – 104.0	99.8 – 107.4	0.010	0.034	0.7 – 3.7	0.4 – 3.1			
	CBL A		0.04 – 50	96.2 – 104.7	96.2 – 105	0.012	0.039	0.1 – 8.2	0.7 – 2			
	CBN		0.04 – 50	98.3 – 105.7	98.1 – 102.8	0.004	0.015	0.0 – 4.5	0.2 – 3.2			
	CBNA		0.04 – 50	98.4 – 104.0	98.6 – 108.6	0.003	0.011	0.1 – 7.2	0.1 – 1.2			
	Δ ⁸ -THC		0.04 – 50	100.7 – 105.0	101.6 – 104.7	0.012	0.040	0.5 – 5.1	0.7 – 2.7			
	Δ ⁹ -THC		0.04 – 50	93.9 – 101.8	95.1 – 102.3	0.011	0.038	0.3 – 9.5	0.4 – 1.5			
	THCV		0.04 – 50	84.8 – 103.3	89.5 – 103.7	0.007	0.024	0.1 – 5.8	0.7 – 5.3			
	THCA		0.04 – 50	99.2 – 105.7	98.1 – 106.2	0.009	0.029	0.0 – 5.9	0.9 – 2.7			
	THCVA		0.04 – 50	97.9 – 105.5	98.2 – 105.4	0.007	0.025	0.7 – 4.2	0.7 – 2.5			
	CBT		0.04 – 50	95.8 – 105.0	97 – 103.6	0.010	0.033	0.1 – 2.8	0.5 – 1.6			

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref
SV23				<i>Intra-day – Day 1 (QC in solv; 3 conc)</i>	<i>Inter-day (QC in solv; 3 days)</i>			<i>Intra-day (QC in solv; 3 conc, n = 3)</i>	<i>Inter-day (QC in solv; 3 days)</i>	ISO-17025	70	[114]
	CBC	≥ 0.9880	0.02 – 25	94.7 – 107.5	98.2 – 107.4	NR	0.02	0.2 – 4.2	0.8 – 3.6			
	CBCA		0.02 – 25	97.5 – 114.7	94.8 – 114.1	NR	0.02	0.6 – 7.8	1.7 – 3.7			
	CBD		0.02 – 25	98.6 – 101.5	98.1 – 106.1	NR	0.02	0.5 – 4.4	1.0 – 3.8			
	CBD A		0.02 – 25	93.9 – 107.6	92.4 – 108.2	NR	0.02	0.2 – 2.4	0.7 – 1.8			
	CBDV		0.02 – 25	84.5 – 107.6	93.8 – 106.5	NR	0.02	0.4 – 4.2	1.0 – 8.8			
	CBDVA		0.02 – 25	93.9 – 110.4	91.7 – 108.8	NR	0.02	0.4 – 4.8	0.8 – 4.7			
	CBG		0.02 – 25	98.6 – 101.5	99.0 – 104.0	NR	0.02	0.1 – 5.4	0.9 – 2.1			
	CBGA		0.02 – 25	96.2 – 106.3	96.1 – 108.2	NR	0.02	0.7 – 10.3	0.2 – 1.7			
	CBN		0.02 – 25	93.9 – 105.4	93.9 – 106.2	NR	0.02	0.2 – 1.7	0.9 – 1.8			
	Δ ⁸ -THC		0.02 – 25	90.5 – 108.6	92.1 – 104.5	NR	0.02	0.4 – 8.9	3.4 – 5.8			
	Δ ⁹ -THC		0.02 – 25	97.0 – 104.7	98.2 – 105.2	NR	0.02	0.6 – 4.1	0.7 – 2.6			
	THCA		0.02 – 25	96.4 – 109.8	95.1 – 108.9	NR	0.02	0.2 – 9.6	0.9 – 5.6			
	THCV		0.02 – 25	98.6 – 114.5	98.5 – 105.4	NR	0.02	0.3 – 9.8	1.3 – 14			
	THCVA		0.02 – 25	96.4 – 102.6	97.1 – 105.3	NR	0.02	0.2 – 7.9	0.7 – 2.9			
SV24				<i>(1 conc, n = 10)</i>				<i>(1 conc, n = 10)</i>		ISO–17025	33	[115]
	CBC	NR	NR	96		0.118	0.393	2.631				
	CBCA	NR	NR	103		0.091	0.304	3.802				
	CBD	NR	NR	98		0.156	0.519	6.809				
	CBD A	NR	NR	110		0.109	0.364	2.118				
	CBDV	NR	NR	100		0.113	0.375	4.830				
	CBDVA	NR	NR	105		0.076	0.253	3.074				
	CBG	NR	NR	88		0.099	0.331	4.798				
	CBGA	NR	NR	110		0.211	0.705	8.179				
	CBL	NR	NR	109		0.237	0.790	2.311				
	CBLA	NR	NR	97		0.197	0.658	2.168				
	CBN	NR	NR	97		0.068	0.226	2.997				
	CBNA	NR	NR	104		0.084	0.280	3.454				
	Δ ⁹ -THC	NR	NR	106		0.163	0.545	6.598				
	THCA	NR	NR	93		0.186	0.619	8.512				
	THCV	NR	NR	85		0.241	0.803	12.154				
	THCVA	NR	NR	101		0.110	0.366	4.632				
SV25				<i>Hemp-free blank products</i>		<i>Based on SD of a linear response and a slope and</i>		<i>Precision (RSD %)</i>	<i>Product</i>			
				<i>% Bias</i>	<i>% Recovery</i>	<i>S/N ≥ 3 (mg/g)</i>	<i>S/N ≥ 10 (mg/g)</i>					
	CBD	0.997	14 – 75	3	101	0.025	0.400	NR	Shikai Lotion	ANSI/ASB standard	88	[116]
	CBD	0.997	1 – 20	-4	101	0.062	0.207	NR	Hempz Lotion			
	CBD	0.997	20 – 100	2	95	0.121	0.403	NR	Zen Renu Cream and			
	Δ ⁹ -THC	0.999	1 – 20	6	93	0.005	0.016	NR	Neu Hemp Cream			
CBD	0.996	1 – 50	0	97	0.340	1.134	NR	Pure Ratios Salve				

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c				Guidelines	HPLC entry ^d	Ref
SV26				Spiking experiment (coconut oil, 3 conc)		Based on S/N S/N ≥ 2 or 3 S/N ≥ 10		3 conc, n = 6						
	CBD	0.999	0.5 – 5	87.92 – 102.21		0.10	0.5	0.67 – 5.97				SANTE	S02	[149]
	Δ ⁹ -THC	0.998	0.5 – 5	95.85 – 106.44		0.12	0.5	1.68 – 6.54						
SV27				3 conc Standard Addition Method				Intra-day (3 conc)		Inter-day (3 conc)				
	CBD	1	0.78 – 50	100.04 – 101.7	91.2 – 102.8	0.28	0.84	0.05 – 0.39		0.78 – 1.42		FDA (ORA-LAB.5.4.5)	17	[104,17]
	CBDA	0.9996	0.78 – 50	98.66 – 101.23	ND	1.26	3.82	0.08 – 0.45		0.32 – 0.96				
	Δ ⁹ -THC	0.9996	0.78 – 50	100.47 – 101.60	ND	1.28	3.89	0.05 – 0.27		0.71 – 1.25				
	THCA	0.9987	0.78 – 50	99.03 – 100.17	ND	0.23	0.71	0.10 – 0.38		0.26 – 0.80				
SV28				Spiking experiment (3 conc) Sunflower: coconut oil (60:40) - SM SM oil in ice cream		Based on SD of a linear response and a slope LOQ recovery checked		Intra-day (1 conc, n = 6) Oil Ice Cream		Inter-day (1 conc, 3 days) Oil Ice Cream				
	CBD	0.9977	1 – 5	98.8 – 101.1	86.8 – 92.2	0.11	0.33	1.5	1.7	3	3.5	ICH Q2 (R2) + AOAC Appendix F	S20	[16]
	Δ ⁹ -THC	0.9918	20 – 100	98.8 – 102.5	84 – 94	0.76	2.30	1.7	1.6	3	3.2			
SV29				Spiking experiment – blank matrix (3 conc)		Based on SD of a linear response and a slope		Intra-day (QC in solv; 3 conc, n = 3)		Inter-day (QC in solv; 3 days)				
	CBD	0.9997	31.25 – 250	210 nm: 98.42 – 100.96 228 nm: 98.25 – 101.48		5.93 5.77	17.96 17.49	0.8 – 1.3 0.7 – 1.4		0.6 – 1.9 0.6 – 1.9		ICH Q2 (R1) + AOAC Appendix F	S31	[134]
SV30				Standard addition method (5 conc)		Based on S/N S/N ≥ 3 S/N ≥ 10		Repeatability (1 conc, n = 6)		IP (1 conc; 3 days)		ICH Q2 (R1) + AOAC SMPR 2019.003 and Appendix K	S01	[125]
	CBD	0.9998	0.25 – 10.0	92 – 107		0.03	0.10	0.25 – 0.43		1.83				
	Δ ⁹ -THC	0.9999	0.25 – 10.0	97 – 109		0.03	0.10	0.67 – 0.99		1.22				
SV31				Standard addition (3 conc) %Bias	% Extraction recovery (3 conc)	Based on SD of a linear response and a slope (confirmed by S/N)		Repeatability (samples, n = 6)		Intermediate (samples, n= 6, 2 days)		ICH Q2 (R1) + ANVISA RDC 166/2017 + USP + FDA	46	[30]
	CBD	0.9998	10 – 100	1.2 – 4.3	91.3 – 97.4	3.6	10.8	NR		NR				
	CBDA	0.9995	10 – 100	3.7 – 7.1	NR	3.3	10.1	NR		NR				
	CBN	0.9993	10 – 100	0.0 – 4.8	NR	4.1	12.5	NR		NR				
	Δ ⁹ -THC	0.9995	10 – 100	0.9 – 2.2	NR	4.2	12.6	4 – 4.8		5.1				
	THCA	0.9995	10 – 100	0.4 – 1.7	NR	3.4	10.2	4.3 – 4.8		4.5				
SV32				Spiking experiment – cannabis extraction waste (CEW)		LOD based on visual evaluation (QC from CRS)		Intra-day (QC from SRM in solv; 3 conc)		IP (QC from SRM in solv; 3 conc)				
	CBD	1.0000	2 – 110	94.77 – 99.45		0.80	2	0.40 – 3.20		1.16 – 2.66		ICH Q2 (R1) + ANVISA RDC 166/2017	56	[48]
	CBDA	0.9997	2 – 110	88.32 – 94.71		0.60	2	0.38 – 1.69		2.16 – 3.33				
	CBN	1.0000	2 – 110	113.21 – 116.85		0.50	2	0.31 – 1.93		1.79 – 3.82				
	Δ ⁹ -THC	1.0000	2 – 110	109.76 – 119.28		0.70	2	1.44 – 3.33		2.02 – 3.86				
	THCA	0.9999	2 – 110	76.92 – 82.07		0.40	2	0.32 – 3.18		1.47 – 4.27				

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a			LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c		Guidelines	HPLC entry ^d	Ref	
SV33				<i>Intra-day</i> (3 conc, n = 7)	<i>Inter-day</i> (3 conc, 3 days)	<i>Based on SD of a linear response and a slope LOQ by Visual evaluation</i>			<i>Repeatability</i> (3 conc, n = 7)	<i>IP</i> (3 conc, 3 days)	ICH + Eurachem + SANTE 11312/ 2021 + ANVISA RDC 166/2017 + FDA	48	[94]	
	CBD	0.9856	10 – 120	101.3 – 113.7	99.3 – 119.3	0.19	5		3.6 – 4.8	2.3 – 4.6				
SV34				% Bias <i>Intra-day (QC in solv; 3 conc, n = 5)</i>			<i>Inter-day (QC in solv; 3 conc, 3 days)</i>	<i>Based on S/N and visual evaluation</i>		<i>Intra-day (QC in solv; 3 conc, n = 5)</i>	<i>Inter-day (QC in solv; 3 conc, 3 days)</i>			Eurachem + UNODC
	CBD	[0.9978 – 0.9988]	0.5 – 25.0	-5.30 – 14.49	-13.00 – 4.00	0.125	0.5		0.32 – 8.02	1.00 – 4.90				
	CBDA		0.5 – 25.0	-9.61 – 0.99	0.20 – 5.80	0.250	0.5		0.44 – 6.96	0.90 – 6.90				
	CBN		0.5 – 25.0	-4.81 – 13.02	-13.30 – 3.10	0.125	0.5		0.02 – 4.91	0.80 – 4.40				
	Δ ⁸ -THC		0.5 – 25.0	-1.95 – 3.39	-3.30 – 1.10	0.125	0.5		0.01 – 12.48	0.80 – 3.60				
	Δ ⁹ -THC		0.5 – 25.0	-1.24 – 7.68	-6.40 – 1.10	0.125	0.5		0.53 – 12.72	1.50 – 13.00				
	THCA		0.5 – 25.0	-6.84 – 14.50	-10.10 – 5.90	0.250	0.5		0.01 – 10.45	0.90 – 8.90				
SV35				<i>Standard addition method - Hemp essential oil (EO) (3 conc)</i>			<i>Based on S/N</i> <i>S/N ≥ 3 S/N ≥ 10</i>		<i>Intra-day (QC in EO, 3 conc, n = 3)</i>	<i>Inter-day (QC in EO, 3 conc, 3 days)</i>	NR	20	[102]	
	CBD	0.9995	0.5 – 100	86 – 90		0.11	0.35		1.84 – 3.30	9.80				
	CBDA	0.9999	0.5 – 100	81 – 98		0.16	0.48		0.99 – 3.60	6.32 – 10.40				
	CBN	0.9997	0.5 – 100	90 – 97		0.13	0.40		0.83 – 3.00	3.75 – 4.80				
	Δ ⁹ -THC	0.9997	0.5 – 100	79 – 99		0.12	0.38		0.83 – 3.00	5.22 – 9.72				
	THCA	0.9993	0.5 – 100	78 – 100		0.15	0.47		0.92 – 2.10	7.75 – 9.55				
SV36				<i>Standard addition method (2 conc)</i>			<i>Based on SD of a linear response and a slope</i>		<i>Repeatability</i> (2 conc; n = 6)	<i>IP (2 conc; 3 days)</i>	Eurachem	7	[41]	
	CBD	0.9999	0.15 – 25	91.1 – 94.4		0.05	0.15		0.08 – 0.23	1.86 – 3.49				
	CBDA	0.9999	0.25 – 25	93.8 – 97.5		0.08	0.25		0.04 – 0.05	0.07 – 0.31				
	CBN	0.9998	0.15 – 25	87.2 – 89.0		0.05	0.15		0.19 – 1.51	1.50 – 1.72				
	Δ ⁹ -THC	0.9998	0.2 – 25	94.0 – 95.0		0.07	0.20		0.36 – 0.84	2.37 – 1.59				
	THCA	0.9999	0.25 – 25	90.0 – 106.6		0.07	0.25		0.10 – 0.14	0.34 – 1.18				
SV37				<i>Spiking experiments – cannabis extraction waste (CEW)</i>			<i>Based on S/N</i>		<i>Repeatability (QC in CEW, 6 conc; n = 6)</i>	<i>IP (QC in CEW, 3 conc; 6 days)</i>	ICH Q2 (R1), AOAC, FDA, USP	71	[93]	
				<i>Intra-day (% Bias)</i>	<i>Inter-day (% Bias)</i>	<i>Extraction Recovery</i>	<i>S/N ≥ 3</i>	<i>S/N ≥ 10</i>						
	CBC	0.999	3.125 – 100	-7.63 – 4.50	-1.26 – 9.77	79 – 98	0.284	0.948	1.11 – 4.91	0.89 – 4.22				
	CBD	0.999	3.125 – 100	-0.51 – 3.66	-30.80 – 9.62	83 – 113	0.450	1.502	0.78 – 6.63	0.43 – 4.91				
	CBDA	0.999	3.125 – 100	0.89 – 2.54	-74.67 – 4.44	77 – 102	0.367	1.226	0.74 – 2.89	0.45 – 2.82				
	CBDV	0.999	3.125 – 100	-2.07 – 7.63	6.86 – 9.85	84 – 114	0.335	1.116	0.63 – 1.29	0.34 – 1.00				
	CBG	0.999	3.125 – 100	-1.72 – 4.92	-6.16 – 8.22	81 – 121	0.367	1.224	0.86 – 4.00	0.87 – 2.82				
	CBGA	0.999	3.125 – 100	1.43 – 3.27	1.99 – 8.40	80 – 109	0.367	1.226	0.42 – 0.79	0.45 – 1.04				
	CBL	0.999	3.125 – 100	0.57 – 6.23	6.13 – 9.82	NR	0.351	1.171	1.05 – 9.39	0.61 – 8.19				
	CBN	0.999	3.125 – 100	0.42 – 4.41	8.21 – 9.57	NR	0.468	1.560	0.64 – 4.41	0.33 – 7.32				
	Δ ⁸ -THC	0.999	3.125 – 100	-1.37 – 6.67	5.54 – 10.49	77 – 111	0.200	0.669	0.38 – 8.73	0.44 – 7.28				
	Δ ⁹ -THC	0.999	3.125 – 100	-4.03 – 2.49	-182.83–4.66	74 – 89	0.229	0.766	1.31 – 5.53	1.02 – 6.81				
	THCA	0.999	3.125 – 100	-13.78 – 3.41	-6.16 – 8.34	76 – 93	0.342	1.140	3.10 – 3.75	1.88 – 4.04				
	THCV	0.999	3.125 – 100	-7.89 – 5.32	8.69 – 9.28	83 – 113	0.340	1.134	0.57 – 5.92	0.58 – 5.21				

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a			LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c			Guidelines	HPLC entry ^d	Ref	
SV38				% Relative error											
				<i>Intra-day</i> (QC in sample; 4-5 conc; n = 6)	<i>Inter-day</i> (QC in sample; 8-11 conc; 5 days, n = 5)	<i>Interm. (QC</i> <i>in sample; 4-</i> <i>5 conc; 5</i> <i>days, n = 15)</i>	<i>Based on visual evaluation</i> µg/mL or µg/mg (semi-solid or solid)		<i>Intra-day</i> (QC in sample; 4-5 conc; n = 6)	<i>Inter-day</i> (QC in sample; 8-11 conc; 5 days, n = 5)	<i>Intermediate</i> (QC in sample; 4-5 conc; 5 days, n = 15)				
	<i>Beverage / oil</i>											ICH M10 + FDA + USP	98	[127]	
	CBCA	0.9987	0.8 – 100	-5.12 – 3.24	-5.50 – 3.66	-7.65 – 6.40		0.8	3.44 – 11.28	2.25 – 11.10	3.23 – 13.30				
	CBD	0.9990	0.4 – 100	-5.51 – 2.36	4.60 – 2.77	-10.23 – -3.85		0.4	3.00 – 9.49	1.06 – 10.74	5.52 – 12.03				
	CBDa	0.9986	0.4 – 100	-4.28 – 7.48	-6.42 – 2.26	-9.60 – 2.76		0.4	3.27 – 9.32	2.32 – 10.36	3.04 – 12.49				
	CBN	0.9984	0.1 – 100	-12.36 – 10.17	-6.18 – 3.34	-8.02 – 7.24		0.1	3.34 – 10.27	1.65 – 12.58	3.62 – 11.49				
	CBG	0.9979	0.4 – 100	-5.96 – 3.76	-6.31 – 5.48	-6.29 – 0.28		0.4	3.48 – 6.73	1.90 – 10.81	5.65 – 11.94				
	CBGA	0.9987	0.4 – 100	-4.03 – 9.88	-6.03 – 3.29	-6.15 – 2.15		0.4	3.19 – 10.49	2.42 – 10.15	3.63 – 12.00				
	Δ ⁸ -THC	0.9989	0.4 – 100	-8.96 – 2.87	-6.54 – 4.48	-10.78 – -3.58		0.4	3.73 – 9.14	2.21 – 9.76	4.88 – 10.74				
	Δ ⁹ -THC	0.9988	0.4 – 100	-8.90 – 3.10	-5.02 – 2.52	-7.78 – -1.28		0.4	3.75 – 9.29	0.90 – 11.19	4.81 – 14.67				
	THCA	0.9967	0.4 – 250	-12.56 – 7.52	-9.27 – 6.00	-9.00 – 3.30		0.4	3.23 – 8.15	3.73 – 17.02	1.91 – 15.51				
	<i>Herbal samples</i>														
	CBCA	0.9953	0.08 – 10	-5.12 – 3.24	-5.50 – 3.66	-7.65 – 6.40		0.08	3.44 – 11.28	2.25 – 11.10	3.23 – 13.30				
	CBD	0.9953	0.04 – 10	-8.75 – -2.75	-6.00 – 4.25	-14.60 – 4.04		0.04	1.65 – 11.63	1.47 – 11.43	2.13 – 11.42				
	CBDa	0.9974	0.04 – 10	-14.5 – -0.15	8.75 – 2.00	13.2 – -0.04		0.04	3.62 – 11.39	1.45 – 10.61	1.81 – 12.42				
	CBN	0.9937	0.01 – 10	-12.36 – 10.17	-6.18 – 3.34	-8.02 – 7.24		0.01	3.34 – 10.27	1.65 – 12.58	3.62 – 11.49				
	CBG	0.9953	0.04 – 10	-5.96 – 3.76	-6.31 – 5.48	-6.29 – 0.28		0.04	3.48 – 6.76	1.90 – 10.81	5.65 – 11.94				
	CBGA	0.9945	0.04 – 10	-4.03 – 9.88	-6.03 – 3.29	-6.15 – 2.15		0.04	3.41 – 10.49	2.42 – 10.15	3.63 – 12.00				
	Δ ⁸ -THC	0.9963	0.04 – 10	-9.96 – 2.87	-6.54 – 4.48	-10.78 – -3.58		0.04	3.73 – 9.14	2.21 – 9.76	4.88 – 10.74				
	Δ ⁹ -THC	0.9975	0.04 – 10	-8.90 – 3.10	-5.02 – 2.52	4.81 – 14.67		0.04	3.75 – 9.29	0.90 – 11.19	4.81 – 14.67				
	THCA	0.9930	0.04 – 25	-12.56 – 7.52	-9.27 – 6.00	-9.00 – 3.03		0.04	3.23 – 8.15	3.73 – 17.02	1.91 – 15.51				
	<i>Cosmetic products</i>														
	CBCA	0.9940	0.08 – 10	-12.00 – 7.80	0.26 – 13.33	-12.92 – 4.90		0.08	2.34 – 5.87	1.68 – 12.12	1.12 – 3.40				
	CBD	0.9943	0.04 – 10	-2.70 – 5.42	-5.55 – 7.08	-4.63 – 6.67		0.04	1.90 – 4.57	1.80 – 8.43	1.50 – 8.87				
	CBDa	0.9959	0.04 – 10	-8.33 – 9.52	-6.25 – 9.18	-5.83 – 8.67		0.04	2.62 – 7.01	1.27 – 13.16	2.15 – 3.28				
	CBN	0.9928	0.01 – 10	-12.00 – 10.13	-11.00 – 6.34	-8.67 – 6.57		0.01	1.14 – 7.92	0.77 – 10.11	2.14 – 7.29				
	CBG	0.9941	0.04 – 10	5.40 – 10.97	-5.83 – 11.67	-5.00 – 5.42		0.04	3.06 – 7.79	1.24 – 10.73	1.49 – 4.16				
	CBGA	0.9935	0.04 – 10	-5.42 – 6.00	-7.10 – 12.92	-6.67 – 4.33		0.04	2.63 – 7.39	1.81 – 6.32	1.59 – 8.56				
	Δ ⁸ -THC	0.9960	0.04 – 10	1.00 – 6.33	-11.04 – 12.08	-8.50 – 9.00		0.04	2.48 – 7.72	3.16 – 10.20	3.27 – 7.61				
	Δ ⁹ -THC	0.9961	0.04 – 10	-7.50 – -0.70	-13.25 – 10.96	-3.88 – 5.33		0.04	0.63 – 4.16	3.16 – 10.20	4.70 – 7.94				
	THCA	0.9939	0.04 – 25	-9.50 – 5.12	-8.33 – 8.29	-1.83 – 10.33		0.04	4.59 – 8.49	1.18 – 14.13	1.47 – 4.76				
SV39									<i>Intra-day (3 conc)</i>	<i>Inter-day (3 conc)</i>					
	CBC	0.9997	1 – 1000	99.69			0.11025	0.36383	1.10 – 1.21	1.14 – 1.19	NR			97	[51]
	CBD	0.9999	1 – 1000	99.49			0.11512	0.37989	1.12 – 1.13	1.15 – 1.75					
	CBG	0.9999	1 – 1000	99.67			0.1112	0.36669	1.11 – 1.15	1.06 – 1.09					
	CBN	0.9998	1 – 1000	99.53			0.12545	0.41401	1.01 – 1.44	1.15 – 1.31					
	Δ ⁹ -THC	0.9999	1 – 100	99.79			0.13212	0.43599	1.12 – 1.39	1.01 – 1.12					

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a		LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c				Guidelines	HPLC entry ^d	Ref
SV40				Standard addition method (2 conc.) Pre-extraction % Recovery		Based on SD of a linear response and a slope		(QC in solv; 3 conc; n = 7)						
	CBDA THCA	0.997 0.998	1 – 100 1 – 100	71.2 – 91.8 68.6 – 101.0	Post-extraction % Matrix effect 85.9 – 100.8 99.8 – 102.4	0.10 0.12	0.24 0.37	0.58 – 1.37 0.82 – 1.47				Peters et al. [161]	23	[29]
SV41				Spiking experiments – Diluted extracts (3 conc)		Based on S/N and visual evaluation		Intra-day QC in solv; n = 10)		Inter-day QC in samples; 10 days; 10 days)				
	Δ ⁹ -THC	0.9975	5 – 50	94.29 – 103.44		0.5	1	0.16	0.08 – 0.14	0.73	1.19–2.65	NR	67	[32]
SV42								Intra-day		Inter-day (2 days)				
	10 CB	>0.99	0.5 – 100	> 99		≤ 0.8	< 2	< 10				NR	99	[111]
SV43														
	CBD CBDA	NR NR	NR NR	95 – 112 99 – 102		NR NR	NR NR	≤ 5.5 ≤ 4.6				NR	S23	[122]
SV44						Based on SD of a linear response and a slope		Intra-day (n = 6)						
	CBCA	0.99952	0.16 – 10	NR		0.039	0.118	3.42				NR	94	[57]
	CBNA	0.99998	0.16 – 10	NR		0.010	0.030	1.87						
	THCA	0.99994	0.16 – 100	NR		0.043	0.130	3.15						
SV45				Spiking experiments – honey (SM)		Based on S/N S/N ≥ 3 S/N ≥ 10								
	CBD	0.999	1 – 100	≥ 95%		1	5	NR				NR	32	[88]
	CBDA	0.999	1 – 100	≥ 95%		1	5	NR						
	Δ ⁹ -THC	0.999	1 – 50	≥ 95%		1	5	NR						
	THCA	0.999	1 – 50	≥ 95%		1	5	NR						
SV46								Intra-day (3 conc)		Inter-day (3 conc)				
	CBC	0.9988	0.2 – 50	NR		0.1	NR	0.34 – 6.80		1.08 – 1.68		NR	91	[123]
	CBD	0.9977	0.1 – 50	NR		0.02	NR	0.21 – 3.51		0.86 – 1.07				
	CBDA	0.9978	0.1 – 50	NR		0.02	NR	0.81 – 4.97		0.87 – 1.05				
	CBDV	0.9972	0.1 – 50	NR		0.02	NR	0.26 – 1.74		1.71 – 0.82				
	CBG	0.9979	0.1 – 50	NR		0.02	NR	0.22 – 4.20		0.90 – 1.13				
	CBGA	0.9983	0.1 – 50	NR		0.02	NR	0.19 – 4.17		1.02 – 1.25				
	CBL	0.9985	0.2–50	NR		0.1	NR	0.33 – 5.61		1.05 – 1.55				
	CBN	0.9981	0.1 – 50	NR		0.02	NR	0.22 – 2.91		1.03 – 1.40				
	Δ ⁸ -THC	0.9983	0.2 – 50	NR		0.1	NR	1.80 – 3.91		1.03 – 1.71				
	Δ ⁹ -THC	0.9982	0.2 – 50	NR		0.1	NR	1.06 – 3.28		1.03 – 1.50				
	THCA	0.9992	0.2 – 50	NR		0.1	NR	0.24 – 4.72		1.11 – 1.95				
	THCV	0.9974	0.1 – 50	NR		0.02	NR	0.28 – 1.66		0.89 – 1.14				
	THCVA	0.9978	0.1 – 50	NR		0.02	NR	0.63 – 2.83		1.05 – 1.34				

Entry	CB	R ²	L. range (µg/mL)	Accuracy % ^a	LOD ^b (µg/mL)	LOQ ^b (µg/mL)	Precision (RSD%) ^c	Guidelines	HPLC entry ^d	Ref
SV47				(QC in solvent; 2 batches; 3 conc; n = 3)	Based on S/N S/N ≥ 3 S/N ≥ 10		(QC in solvent; 2 batches; 3 conc; n = 3)			
	Δ ⁸ -THC	1	NR	100.58 – 112	0.27	1.98	0.21 – 1.93	NR	41	[162]
	Δ ⁹ -THC	1	NR	100.38 – 112.90	0.25	1.55	0.25 – 1.76			
SV48							Intra-day	Inter-day		
	CBD	> 0.99	1.25 – 40	90 – 110	NR	NR	< 2	< 2	NR	S30 [141]
SV49							Inter-day (n = 10)			
	CBC	0.9999	0.1 – 10	NR	0.03	0.08	1.3	NR	78	[148]
	CBD	0.9997	0.1 – 5	NR	0.02	0.08	7.7			
	CBG	0.9999	0.1 – 10	NR	0.02	0.07	5.6			
	CBN	1	0.1 – 10	NR	0.03	0.05	3.1			
	Δ ⁸ -THC	0.9994	0.1 – 5	NR	0.01	0.10	8.6			
	Δ ⁹ -THC	0.9997	0.1 – 5	NR	0.03	0.08	5.9			
SV50				Spiking experiments – blank samples (normal gummy extract; 3 conc; n = 3) % Bias	Based on SD of a linear response and a slope Confirmed by visual evaluation		(3 conc; n = 3)			
	Ag(I) HPLC-DAD							SWGTOX	104	[121]
	CBD	0.9988	5.00 – 333	-5.0 – 4.2	0.90	NR	0.62 – 5.6			
	Δ ⁽⁴⁾ ⁸ -iso-THC	NR	NR	-12 – 7.6	NR	NR	0.68 – 5.6			
	Δ ⁸ -iso-THC	NR	NR	3.2 – 7.2	NR	NR	0.26 – 6.1			
	Δ ⁸ -THC	0.9983	5.00 – 333	-3.6 – 5.6	0.50	NR	0.86 – 6.1			
	Δ ⁹ -THC	0.9988	5.00 – 333	-3.4 – 8.6	0.40	NR	2.2 – 5.5			

CB: Cannabinoids; CEW: Cannabis extraction waste (obtained after exhaustive extraction of cannabis material); conc: Concentration; CRS: certified reference standards EO: Essential oil; EVOO: Extra virgin olive oil; IA: Inter-analyst; Infl: Inflorescence; IP: Intermediate precision; L. range: Linear range; Instr. P: Instrument precision; Method P: Method precision; NR: not reported, not determined or not present in the corresponding sample; QC: Quality control; RSD: Relative standard deviation; SD: Standard deviation, SM: surrogate matrix; S/N: Signal-to-noise ratio; solv: Solvent; SRM: secondary reference material; Std: Standard.

Cannabinoids Abbreviations: CBD: cannabidiol; CBDA, cannabidiolic acid; CBDV, cannabidivarin; CBC: cannabichromene; CBCA: cannabichromenic acid; CBG: cannabigerol; CBGA: cannabigerolic acid; CBL: cannabicyclol; CBLA: cannabicyclic acid; CBN: cannabinol; CBT: cannabicitran; Δ⁽⁴⁾⁸-iso-THC: Δ⁽⁴⁾⁸-iso-tetrahydrocannabinol; Δ⁸-iso-THC: Δ⁸-iso-tetrahydrocannabinol; Δ⁸-THC: Δ⁸-tetrahydrocannabinol; Δ⁹-THC: Δ⁹-tetrahydrocannabinol; THCA: Δ⁹-tetrahydrocannabinolic acid; THCv: tetrahydrocannabivarin; THCA: tetrahydrocannabivarinic acid.

^a Accuracy: In this column, the main type of accuracy, trueness, bias, recovery described in the analyzed papers are presented. Accuracy intervals correspond to the smallest and the biggest values obtained for any concentration and replicate for the type of accuracy mentioned. When not specified, accuracy is reported as % of recovery. ^b LOD and LOQ values are expressed as mg/mL unless stated otherwise. ^c Precision intervals described in this table correspond to the smallest and the biggest RSD values obtained for any concentration and replicate for the type of precision mentioned. ^d The entry assigned in this column corresponds to the analytical method entry presented in Table 1, Table 2, Table S2, and Table S3.

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