Analytical Techniques for Phytocannabinoid Profiling of Cannabis and Cannabis-Based Products–A Comprehensive Review

Supplementary Material

Table S1. GC-based analytical methods for cannabinoid profiling, COTP - column oven temperature program, IV- injection volume, LOD - limit of detection, LOQ – limit of quantification.

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
grinded herbal cannabis / 0.1 g	-	GC + FID	COTP: 260°C, isothermal injector: 300°C FID: 300°C IV: 1 μL	THC (Δ^9-THC + Δ^9-THCA)	-	-	2017,-	[1]
	-	Shimadzu GC-2010 + FID Rxi-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 260°C (10 min), 20°Cmin^{-1} to 300°C (2 min) injector: 300°C split mode (1:40) FID: 300°C IV: 1 μL	THC (Δ^9-THC + Δ^9-THCA)	14.00	-	2021, Italy	[2]
		Thermo Focus GC/DSQ II Rxi-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 260°C (10 min), 20°Cmin^{-1} to 300°C (2 min) injector: 300°C split mode (1:40) ion source: 270°C interface: 250°C IV: 1 μL	EI, 70 eV full scan mode (*m/z* 40-450) THC (Δ^9-THC + Δ^9-THCA)				

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* ($ngmL^{-1}$ or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
traditional cannabis / sinsemilla / cannabis resin / 0.25 g	-	Agilent 7890 + FID Zebron ZB-5HT Inferno (30 m x 0.32 mm x 0.25 μm)	COTP: 60°C (3 min), 35°Cmin^{-1} to 200°C, 5 °Cmin^{-1} to 250°C (5 min), 100°Cmin^{-1} to 320°C (3.3 min)	Δ^9-THC, CBD, CBN	25.00	-	2015, England	[3]
herbal cannabis / cannabis resin / marijuana joint / 0.05-0.10 g	-	HP 5890 + FID CPsil8CB (25 m x 0.32 mm x 0.25 μm)	COTP: 250°C, isothermal injector: 280°C split mode (20 m$Lmin^{-1}$) FID: 300°C	Δ^9-THC, CBD, CBN	8.00	Δ^9-THC 0.10 CBD 0.10 CBN 0.10	2000-2004 2013 2014 2005-2015	[4]* [5]* [6]* [7]*
dried / herbal cannabis / resin / other (mainly homemade cigarettes with cannabis material) / 0.05-0.1 g	-	Agilent 7890 + FID HP-5MS (12 m x 0.20 mm x 0.33 μm)	COTP: 220°C (1 min), 6°Cmin^{-1} to 250°C (2 min) injector: 250°C split mode (1:100) FID: 300°C	Δ^9-THC, CBD, CBN	8.00	-	2010-2012, northern Italy 2013, northern Italy	[8] [9]
inflorescences of pistillated plants / 0.05 g	-	HP 5710A GLC + FID DB-5MS (30 m x 0.53 mm x 1.5 μm)	COTP: 200°C (8 min), 4°Cmin^{-1} to 300°C (4 min) injector: 250°C FID: 250°C	Δ^9-THC, CBG, CBGM, CBDV, THCV, CBD, CBC	37.00	-	2004, USA	[10]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* ($ngmL^{-1}$ or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
cannabis leaves/ cannabis oil / cannabis resins / 0.005 – 0.03 g	-	GC + FID DB-1 (10 m x 0.18 mm x 0.4 μm)	COTP: 200°C (4 min), 15°Cmin^{-1} to 280°C (2 min) injector: 250°C split mode (22.2 $mLmin^{-1}$) FID: 280°C	Δ^9-THC, CBD	11.33	-	1976-1995, New Zealand	[11]
whole cannabis plants / 1.0 g	dry extracts in 500 μL pyridine + 500 μL BSTFA + 1% TMCS 60°C, 5 min	Beckman GC-4 and GC-45 + FID 2% OV-17 100-200 mesh GasChrom Q	FID: 210°C	Δ^9-THC, Δ^9-THCA	52.00	-	1971, Mexico	[12]
whole mature cannabis plants / cannabis resin / 1.0 g	-	Pye 104 + FID 3% OV-17 100-200 mesh GasChrom Q (1.5 m x 4 mm)	COTP: 250°C, isothermal IV: 5 μL FID: 300°C	Δ^9-THC, Δ^9-THCA	-	-	1975-1978, United Kingdom	[13]
whole mature cannabis plants / 2.0 g	-	PU4500 + FID 3% OV-17 100-120 mesh Chromosorb WHP (1.5 m x 4 mm)	COTP: 245°C, isothermal	total THC (Δ^9-THC + THCA)	-	-	1984-1989, United Kingdom	[14]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* ($ngmL^{-1}$ or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
dried cannabis leaves and flowering/fruiting tops / 5.0 g	-	HP 5590 + FID SPB-1 (15 m x 0.53 μm x 1.5μm)	COTP: 60°C (1.5 min), 30°Cmin^{-1} to 300°C (8 min) injector: 275°C splitless mode FID: 350°C	Δ^8-THC, Δ^9-THC, CBD, CBG, CBN	17.50	-	1988, Denmark	[15]
dried and pulverised cannabis plant material / cannabis flowers / cannabis resin / 0.05 -0.10 g	-	Carlo Erba GC8000 Top + FID DB-5MS (15 m x 0.25 mm x 0.25 μm)	COTP: 120°C (2 min), 20C min^{-1} to 300°C (3 min)	Δ^9-THC	14.00	-	2004, Switzerland	[16]
upper main stem of flowering cannabis plants, powedered / 0.02 g	-	HP GCD + MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 100°C, 15°Cmin^{-1} to 300°C (8 min) injector: 280°C splitless mode interface: 300°C	EI, 70 eV SIM mode CBD (**231**, 174, 314) CBN (**295**, 238, 310) Δ^9-THC (**299**, 314, 231)	21.33	-	1996, Greece 1999, Greece	[17] [18]
dried cannabis upper flowering plant parts, powdered / 0.1 g [19] **cannabis leaves / reproductive parts / calli / 0.1 g [20]**	-	GC 8000 Top + FID fused silica capillary column (30 m x 0.32 mm x 0.5 μm)	COTP: 220°C-300°C injector: 300°C splitless mode FID: 300°C	Δ^9-THC, CBD, CBG		-	2006, Italy 2008, Italy	[19] [20]

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dried cannabis from illegal farm / dried seized herbal cannabis / dried cannabis from authorized trial fields / 1.0 g **cannabis leaves / 0.5 g**	-	Chrompack 9002 GC + FID DB-1 (30 m x 0.32 mm x 0.25 μm)	COTP: 230°C (7 min), 10°Cmin^{-1} to 260°C (2 min) IV: 1 μL injector: 260°C split mode (1:20) FID: 270°C	Δ^9-THC, CBD, CBN	12.00	-	2008, Northern Thailand 2009, Northern Thailand	[21] [22]
herbal cannabis from coffeeshops **herbal cannabis from coffee shops/ flower tops of pharmaceutical-grade varieties/ hemp dried flowers / 1.0 g**	-	Agilent 6890 + FID DB-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 60°C, 3°Cmin^{1} to 240°C (5 min) IV: 4 μL injector: 230°C split mode (1:20) FID: 250°C	28 [23]/44 [24] major cannabinoids and terpenes, including: THCV, CBD, CBC, CBDV, CBGM, THC, CBG, CBN	65.00	THCV 6 000 000 CBD 6 000 000 CBC 6 000 000 CBDV 6 000 000 CBGM 6 000 000 Δ^9-THC 6 000 000 CBG 6 000 000 CBN 6 000 000 [25]	2011, the Netherlands 2010, the Netherlands 2016, the Netherlands	[23] [25] [24]
homogenized inflorescences/ 0.02 – 0.03 g	-	Agilent 6890 + FID HP-1 (25 m x 0.32 mm x 0.5 μm)	COPT: 250°C, isothermal (9 min) IV:1 μL injector split mode (1:25) FID: 300°C	Δ^9-THC, CBD, CBN	9.00	-	2010, Belgium	[26]
herbal cannabis / 0.2 g **herbal cannabis (stems, leaves and inflorescence) / 0.1 g** **seized cannabis resin/ 0.05 g** **herbal cannabis**	-	Agilent 6890N + FID HP-5MS (30 m x 0.2 mm x 0.2 μm)	COTP: 60°C (2 min), 15°Cmin^{-1} to 280°C (5 min) IV:1 μL injector: 270°C splitless mode FID: 280°C	Δ^9-THC	22.00	-	2005-2014, Morocco	[27]

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herbal cannabis (apical segments) / 1.0 g	-	Becker 3810 + FID 2% OV-17 (2 m x 4 mm)	COTP: 210°C, isothermal IV: 2 μL injector: 260°C detector: 260°C	Δ^9-THC, CBDV, THCV, CBL, CBC, CBV, CBD, CME, Δ^8-THC, CBG, CBN	-	-	1979, South Africa	[28]
		Finnigan 3200/6100 2% OV-1 (20 m x 0.3 mm)	COTP: 100°C (2 min), 6°Cmin^{-1} to 230°C injector: 230°C split mode (1:10) interface: 230°C transfer line: 230°C					
cannabis fruiting tops / cannabis resin / 0.5 g hash oil / 1.0 g	-	HP 5730 + FID 2% OV-17 100-120 mesh Chromosorb WHP	COTP: 250°C, isothermal	Δ^9-THC, CBN, CBC, CBG, THCA	-	-	1979, Colombia	[29]
herbal cannabis, powdered / cannabis resin, powdered / 0.01 g	-	Agilent 6890 + FID HP-5 (30 m x 0.32 mm x 0.25 μm)	COTP: 150°C (1 min), 15°Cmin^{-1} to 250°C (13 min) IV: 1 μL injector: 270°C split mode (1:20) FID: 280°C	Δ^9-THC, CBN, CBD	20.67	Δ^9-THC 0.03/*0.05* CBN 0.03/*0.05* CBD 0.03/*0.05*	2006-2007, Brazil	[30]*
cannabis resin / 0.05 g	-	Agilent 6890 + FID HP-5 (15 m x 0.32 mm x 0.25 μm)	COTP: 180°C, 40°Cmin^{-1} to 220°C, 5°Cmin^{-1} to 240°C, 25°Cmin^{-1} to 280°C injector: 280°C split mode (1:30) FID: 300°C	Δ^9-THC, CBD, CBN	8.00	Δ^9-THC 0.015/*0.030* CBD 0.011/*0.041* CBN 0.007/*0.026*	2002, Italy	[31]
***C. sativa* cultured by micropropagation (buds, leaves, roots and stems) / 0. 1 g**	dried extract + 100 μL BSTFA + 10 μL 2 % DMAP 70ºC, 30min	Agilent 6890N + FID HP-5 (15 m x 0.25 mm x 0.25 μm)	COTP:190°C (1 min), 30°Cmin^{-1} to 230°C (2 min), 5°Cmin^{-1} to 250°C (1 min), 20°Cmin^{-1} to 300°C (2.75 min)	Δ^8-THC, Δ^9-THC, THCV, CBD, CBC, CBG. CBN, CBGA, THCA, CBDA	17.50	THCV 120/*380* CBD 120/*35* CBC 150/*460* Δ^8-THC 140/*430* Δ^9-THC 150/*450* CBG 150/*47*	2017, USA	[32]

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			injector: 275°C split mode (1:20) FID: 300°C			CBN 130/*410* CBDA 140/*430* THCA 190/*560* CBGA 110/*340* CBDV 100/*500*		
dried cannabis flower buds/ 0.1 g	-	Agilent 6890 GC + FID ZB-5 (15 m x 0.25 mm x 0.25 µm)	COTP: 200°C (2 min), 10 $^{9}Cmin^{-1}$ to 240°C (2 min) IV: 1.5 µL injector: 280°C split mode (1:20) FID: 300°C	Δ^9-THC, CBD	8.00	-	2018, Czech Republic	[33]*
dried cannabis flowers (with brachts), upper leaves, lower (large) leaves, stems	-	Varian 204 or 2100 GC + FID 5% SE-30 100/120 mesh Gas Chrom P, 5% XE-60 80/100 mesh Chromosorb W 3% JXR100/120 mesh Gas Chrom Q (all 1.8m x 2-3 mm) LKB 9000 GC-MS	injector: 250-260°C FID: 250-260°C	Δ^9-THC, CBD	-	-	1971, UNODC	[34]
dried cannabis plant material, cannabis resin, reefer / 0.05 g	-	Pye 104 FID 2% OV17 80-100 mesh Chromosorb W (1.524 m x 4 mm)	injector: 235-240°C	CBD, CBN, Δ^9-THC	-	-	1973, United Kingdom	[35]
cannabis plant material/ 0.2 g cannabis resin / 0.1 g	1.5 mL $CHCl_3$ + 100 µL MSTFA 70°C, 30 min	GC-FID 5% diphenyl 95% dimethylpolysilox	COTP: 200°C (2 min), 10°$Cmin^{-1}$ to 240°C (2 min) injector: 280°C splitless mode/split mode (1:20)	CBD, Δ^9-THC, CBN	8.00	-	2009, UNODC	[36]

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cannabis oil / 0.05 g		ane (15 m x 0.25 mm x 0.25 μm)	FID: 300°C					
cannabis inflorescenses / 0.05 g [37] **cannabis leaves** [38] **herbal cannabis / cannabis resin / sinsemilla / cannabis powder / 0.05 g**	-	HP 6890 + FID **quantitative analysis:** HP-5 (30 m x 0.32 mm x 0.25 μm) **separation of CBC and CBD:** HP-1 (40 m x 0.1 mm x 0.25 μm)	-	-	-	-	2003, Italy	[37]
						Δ^9-THC 1.06/*2.44* CBN 1.06/*1.76* CBD 1.02/*1.68*	2012, Colombia	[38]
						-	2005, England	[39]
cannabis plant parts (bracts, floral leaves)	-	HP 5710A GC + hydrogen FID 3% OV-1 80/100 mesh Supelcoport (2.43 m x 2 mm)	COTP: 180°C, 4°Cmin^{-1} to 240°C (8 min) IV: 0.5 μL inlet: 250°C FID: 300°C	CBD, CBC, Δ^9-THC, CBN	24.00	-	1977, USA	[40]
herbal cannabis / cannabis resin / hashish oil / 0.1 g [41] **herbal cannabis / sinsemilla / kilobrick / cannabis resin /**	-	Varian CP-3380 + dual FID DB-5 MS (15 m x 0.25 mm x 0.25 μm)	COTP: 170°C (1 min), 10°Cmin^{-1} to 250°C (3 min) IV: 1 μL injector 240°C split mode (100 mL/min, 1:50) FID: 260°C	Δ^8-THC, Δ^9-THC, CBD, CBN, CBC, CBG, THCV	12.00	-	1993-2008, USA 1995-2014, USA 2008 - 2018, USA	[41] [42] [43]

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Thai sticks / hashish oil / ditchweed / 0.1 g [42] **herbal cannabis (marijuana, sinsemilla, ditchweed) / cannabis resin / hashish oil / 0.01 g [43]** **herbal cannabis (indoor, outdoor) / 0.1 g [44]**		Varian CP-3380 + dual FID DB-1 (15 m x 0.25 mm x 0.25 μm)					2009, USA	[44]
dried cannabis plant material/ 0.05 g	**Step1:** dry extract + 0.1 mL pyridine + 0.1 mL benzene + 1.0 mg alkylboronic acid room ºC, 30min **Step2:** 0.1mL pyridine + 0.1 mL benzene + 0.5 mg methylboronic acid room ºC, 30 min **Step 3:** 0.1 mL ACN + 0.1 mL BSTFA + 0.05 mL TMCS room ºC, 30 min	Varian 2400 + dual FID glass column 3% SE-30 100-200 mesh Gas Chrom Q	COTP: 100°C, 4°Cmin^{-1} to 330°C IV: 1 μL	-	57.50	-	1977, United Kingdom	[45]

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	dried extract + 0.1 mL ACN + 0.1 mL BSTFA + 0.05 mL TMCS room ºC, 30 min	Varian 2400 + VG Micromass 12B	COTP: 170-280°C, 2°Cmin^{-1} injector: 280°C inlet: 230°C separator: 230°C ion source: 260°C	EI, 25 eV full scan mode (*m*/*z* 40-680) Δ^9-THCA, CBNA, CBDA	55.00			
herbal cannabis / cannabis resin / 0.0025 g	-	Trace Ultra™ (GC x GC) + FID 100% polysiloxane (30 m x 0.25 mm x 0.25 μm) + Carbowax (0.5 m x 0.1 mm x 0.1 μm)	COPT: 40°C (1 min), 10°Cmin^{-1} to 200°C, 2°Cmin^{-1} to 260°C (10 min) IV: 0.5 μL injector: 250°C splitless mode FID: 280°C	Δ^9-THC, CBD, CBN	57.00	-	2008, Germany	[46]
hemp fruits / "seeded" flowers / hash oil / 1.0 g	TMSH	HP 5890 + FID or HP 5870 MSD OV-1 column	-	EI, 70 eV Δ^8-THC, Δ^9-THC, CBN, CBD	-	-	1997, Germany	[47]
dried cannabis leaves / 0.01 g	-	Varian CP-3800 + FID OV-1 (30 m x 0.53 mm x 0.50 μm)	COTP: 240°C isothermal injector:260°C IV: 1 μL injector split mode (1:5) ion source: 250°C quadrupole: 150°C interface: 280°C	Δ^9-THC, CBN, CBD	-	-	2009, Colombia	[48]
		Agilent HP 6890 + HP 5973 MSD DB-1 (30 m x 0.25 mm x 1.00 μm)	COTP: 240°C, isothermal IV: 1 μL injector: 260°C split mode (1:5) ion source: 250°C quadrupole: 150°C interface: 280°C	EI, 70 eV full scan mode (*m*/*z* 40-550) Δ^9-THC, CBN, CBD		Δ^9-THC 1.06/*2.44* CBN 1.06/*1.76* CBD 1.02/*1.68*		

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seized cannabis female inflorescences, grounded / cannabis oil in olive oil / 0.05 g	50 µL BSTFA + 1% TMCS + 50 µL toluene 70ºC, 30 min	Trace 2000 + FID DB-5MS IU (30 m x 0.25 mm x 0.25 µm)	COTP: 200°C, 10°Cmin^{-1} to 300°C (2 min) injector: 280°C split mode (39 mL/min, 1:30) FID: 300°C	Δ^9-THC, CBD, CBN, THCA, CBDA	12.00	-	2017, Italy	[49]
		HP 5973 + MSD Rxi-5MS (30 m x 0.25 mm x 0.25 µm)	COTP: 70°C, 40°Cmin^{-1} to 180°C, 10°Cmin^{-1} to 300°C (6.25 min) injector: 280°C splitless mode transfer line: 300°C ion source: 230°C	EI, 70 eV SIM mode (*m*/*z* 50-600) CBD-2TMS (390, 337, 301) Δ^9-THC-TMS (386, 371, 315) CBN-TMS (382, 368, 367) CBDA-3TMS (559, 491, 453) Δ^9-THCA-2TMS (487, 502)	21.00			
dried, homogenized and grinded herbal cannabis / 0.2 g	-	Agilent 7890N + FID HP-5MS (30 m x 0.32 mm x 0.25 µm)	COPT: 150°C (1 min, at 10°Cmin^{-1} to 280°C, (5 min) injector: 250°C split mode (1:20) interface: 300°C	EI, 70 eV full scan mode (*m*/*z* 40-450) Δ^8-THC, Δ^9-THC, CBD, CBN, CBC, CBG, THCV	19.00	-	2012, Romania	[50]
		Agilent 6890N + 5973N MSD HP-5MS (30 m x 0.25 mm x 0.25 µm)	COPT: 150°C (1 min), 10°Cmin^{-1} to 280°C (5 min) injector: 290°C splitless mode interface: 300°C	Δ^8-THC, Δ^9-THC, CBD, CBN, CBC, CBG, THCV	19.00			
cannabis mature floral clusters / 0.05 g	-	HP 5890 + FID ZB-624 (30 m x 0.32 mm x 0.25 µm)	COTP: 40°C (5 min), 10°Cmin^{-1} to 250°C (40 min) injector split mode (1:10)	Δ^9-THC, CBD, CBC, CBG, CBGM, CBDV, CBCV, THCV	47.00	-	2009, Italy	[51]

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		HP 6890 + VG Trio MSD ZB-5 (30 m x 0.32 mm x 0.25 μm)	COTP: 70°C, 5°Cmin^{-1} to 305°C injector split mode (1:5)	EI, 70 eV Δ^9-THC, CBD, CBC, CBG, CBGM, CBDV, CBCV, THCV	66.00			
sieved powder from whole cannabis plant / herbal cannabis / cannabis resin / 0.2 g	-	Agilent 7890 or Shimadzu GC2010 + FID (for discrimination of CBD and CBC: GCMS-QP2010 Plus) HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 200°C (2 min), 10C°min^{-1} to 240°C (15 min) IV: 1 μL inlet: 250°C split mode (1:20) FID: 300°C	Δ^9-THC, CBN	21.00	-	2010, Japan	[52]
			for CBC and CBD: COTP: 200°C (2 min), 10°Cmin^{-1} to 240°C (15 min) IV: 1 μL injector split mode (1:20)	EI, 70 eV full scan mode (*m*/*z* 40-400) CBC, CBD				
cannabis buds, milled / 0.05 g	-	Agilent 7890A + FID + 5975C MSD D1: HP-5MS (30 m x 0.25 mm x 0.25 μm) D2: DB-17MS (5 m x 0.25 mm x 0.25 μm) + fused silica restrictors	COTP: 60°C, 4°Cmin^{-1} to 102°C, 12°Cmin^{-1} to 165°C, 6°Cmin^{-1} to 300°C (5 min) IV: 2 μL injector splitless mode FID: 300°C quadrupole: 150°C ion source: 230°C	EI, 70 eV full scan mode (*m*/*z* 50-350)	43.25	-	2014, Spain	[53]
	-	Clarus 680 GC + FID + Clarus Q 8T SLB-5MS (30 m x 0.25 m x 0.25 μm)	COTP: 50°C to 350°C at 3°Cmin^{-1} injector: 280°C splitless mode	EI, 70 eV full scan mode (*m*/*z* 40-550) CBDV, CBT, CBL, CBD, CBC, Δ^8-THC, Δ^9-THC	100.00	-	2021, Italy	[54]

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			ion source: 220°C interface: 250°C FID: 300°C					
dried herbal cannabis/ 2.0g or 5.0 g	-	Agilent 7820 + FID HP-5 (30 m x 0.32 mm x 0.25 μm)	COTP: 60°C, 3°Cmin^{-1} to 240°C (10 min) inlet: 250°C split mode (1:20) IV: 5 μL	CBD, CBN, Δ^8-THC, Δ^9-THC, THCA, CBDA, CBD-d$_3$, CBN-d$_3$, Δ^9-THC-d$_3$	70.00	CBDA 7.5/*25.1* CBD 6.1/*20.4* CBN 9.2/*30.7* Δ^9-THC 9.4/*31.3* Δ^8-THC 6.8/*22.6* THCA 12.4/*41.2*	2021, Czech Republic	[55]
	dry extract + 50 μL dry EtAc + 50 μL BSTFA + 1% TMCS 70°C, 30 min		COTP: 180°C (2 min), 20°Cmin^{-1} to 310°C (1.5 min)		10.00			
		Agilent 7890A + HP 5975C MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 180°C (2 min), 20°Cmin^{-1} to 310°C (1.5 min) injector: 260°C split mode (1:9) IV: 5 μL ion source: 230°C	EI, 70 eV SIM mode CBDA-3TMS (73, **491,** 559) CBD-2TMS (73, 337, **390**) CBN-2TMS (73, **367**, 382) Δ^9-THC-TMS (73, **371**, 386) Δ^8-THC-TMS (73, **303**, 386) THCA-2TMS (73, **487**) CBD-d$_3$-2TMS (73, 340, **393**) CBN-d$_3$-TMS (73, **370**, 385) Δ^9-THC-d$_3$-TMS (73, **374**, 389)	10.00	CBDA 3.6/*12.0* CBD 4.6/*15.4* CBN 6.1/*20.4* Δ9-THC 2.6/*8.7* Δ8-THC 4.1/*13.5* THCA 5.9/*19.6*		
herbal cannabis ("loose marijuana", buds, kilobricks and "domestic marijuana", all stemless and seedless) / cannabis resin / 0.1 g	-	Varian CP3880 + FID DB-5 (30 m x 0.25 mm x 0.25 μm)	COTP: 150°C (1 min), 10°Cmin^{-1} to 290°C (2 min) injector: 200°C split mode (50 mL/min, 1:30) FID: 300°C	Δ^9-THC	17.00	-	1997-2004, Modena, Italy	[56]
		Varian 3400 + Saturn 2000 IT DB-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 120°C (1 min), 10°Cmin^{-1} to 290°C (10 min) injector: 250°C split mode (50 mL/min, 1:30)	EI, 70 eV full scan mode (*m/z* 43-500) Δ^9-THC	28.00			

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
			transfer line: 280°C					
beer, liquor, cannabis oil, pastilles, seeds, scented grass / 1.0 g (solid samples) / 1.0 mL (liquid samples)	dry extract + 100 µL MSTFA + 2% TMCS 70°C, 30 min	Agilent 6890 + 5973 N MSD HP-5MS (30 m x 0.25 mm x 0.25 µm)	COTP: 120°C (2 min), 20°Cmin^{-1} to 290°C (10 min) injector: 260°C split mode (1:15)	EI, 70 eV SIM mode Δ^9-THC-2TMS (386, **371**, 303) CBD-2TMS (458, 390, 337) CBN-2TMS (382, 367, **310**)	21.00	Δ^9-THC 0.30/*1.00* CBD 0.30/*1.00* CBN 0.60/*2.00*	2003, Italy	[57]
cannabis tea / 0.05g / chocolate/snack bar / 0.4 g flour, seeds, fruit bar, nibbles / 0.1 g pastilles / 1.0 g oil / 0.1 mL lemonade, beer / 0.5 mL **tea infusion, shampoo / 1.0 mL**	HS-SPME: 25 µL MSTFA 90°C, 8 min (on-coating derivatization) LLE: dry extract + 20 µL pyridine + 50 µL MSTFA + 130 µL isooctane 90ºC, 15 min	Agilent 6890N + 5973 MSD HP-5MS (30 m x 0.25 mm x 0.25 µm)	COTP: 160°C (1 min), 15°Cmin^{-1} to 190°C (1 min), 5°Cmin^{-1} to 250°C (1 min), 20° min^{-1} to 300°C (2 min) injector: 250°C splitless mode ion source: 230°C quadrupole: 150°C interface: 280°C	EI, 70 eV SIM mode Δ^9-THC-2TMS (303**, 371**, 386) CBD-2TMS (301, 337, **390)** CBN-2TMS (**367**, 368, 392)	21.50	**tea leaves** Δ^9-THC 0.01/0.08 CBD 0.12/0.32 CBN 0.01/0.09 **chocolates** Δ^9-THC 0.03/0.06 CBD 0.17/0.35 CBN: 0.03/0.07 **oils** Δ^9-THC 0.05/0.03 CBD 0.09/ 0.34 CBN 0.15/0.43	2002, Germany	[58]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
dried herbal cannabis (stems, leaves and inflorescence) / 0.1 g	-	Agilent 6890N + 5973 MSD HP-5MS (30m x 0.25mm x 0.25 μm)	COTP: 100°C (1 min), 10°Cmin^{-1} to 260°C injector: 280°C split mode (1:20) transfer line: 250°C ion source: 150°C quadrupole: 150°C	EI, 70 eV full scan mode (*m*/*z* 50-500) Δ^9-THC, CBD, CBN, CBC, CBG	17.00	-	2016, Brazil	[59]
cannabis plants (recreational, medical, hemp) / consumer products (oral supplements, foods, candies, beverages, vapes, liquids, topicals) / medical products / illicit products (kief, hash oil)/ 0.03 – 3.0 g	dry extract + 200 μL pyridine + 200 μL BSTFA 80ºC, 30 min	Agilent 6890N + 5973 MSD Rxi-35Sil MS (30 m x 0.25 mm x 0.25 μm)	COTP: 60°C (0.5 min), 25°Cmin^{-1} to 220°C (10 min), 10°Cmin^{-1} to 300°C (15 min) IV: 1 μL injector: 250°C splitless mode transfer line: 280°C	EI, 70 eV full scan mode (*m*/*z* 40-600) CBD, CBDA, Δ^9-THCA, Δ^9-THC	39.90	CBD 1000 CBDA 1000 Δ^9-THC 1000 THCA 1000 CBN 1000	2017, USA	[60]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
cannabis inflorescence (AK-47, amnesia, somango, critical), cryogenically miled / 0.05g for FUSE, 0.1g for SFE	-	Agilent 6890 N + 5973 N MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 60°C, 8°Cmin^{-1} to 90°C, 70°Cmin^{-1} to 192°C, 3°Cmin^{-1} to 195°C, 70°Cmin^{-1} to 285°C, 10°Cmin^{-1} to 300°C (2.5 min) IV: 2 μL injector: 300°C splitless mode transfer line: 310°C ion source: 230°C quadrupole: 150°C	EI, 70 eV full scan mode (*m/z* 50-350) Δ^9-THC, CBD, CBN	11.50	-	2013, Spain	[61]
dry cannabis female flowering head material / 2.0 g	-	Agilent 6890 + MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 80°C (1 min), 50°Cmin^{-1} to 300°C (9.6 min) inlet: 280°C detector: 325°C	EI, 70 eV full scan mode total THC (Δ^9-THC + Δ^9-THCA)	15.00	-	2010, New Zealand	[62]
no real samples	-	Agilent 5975 + 5973 MSD JW Ultra 1 (12 m x 0.2 mm x 0.33 μm) **DB-35MS (30 m x 0.25 mm x 0.25 μm)** DB-1701 (30 m x 0.25 mm x 0.25 μm)	COTP: 90°C (0.5 min), 5°Cmin^{-1} to 300°C (15 min) IV: 1 μL injector split mode (50 mLmin^{-1}) detector: 280°C	EI, 70 eV full scan mode (*m/z* 30-550) CBC, CBD, CBG, CBN, CBDV, THCV, Δ^8-THC, Δ^9-THC	57.50	CBDV 1000/2850 THCV 710/710 CBD 2850/2850 CBC 2850/2850 Δ^8-THC 710/710 Δ^9-THC 710/710 CBG 10000/12500 CBN 1420/2850	2017, USA	[63]
cannabis leaves / 0.1 g	-	Agilent 7890A + 5975C MSD	COTP: 100°C, 10°Cmin^{-1} to 260°C (10 min) IV: 2 μL injector: 280°C	EI, 70 eV SIM mode (*m/z* 30-450) THCV (**271**, 286, 243, 203)	26.00	-	2010, Switzerland	[64]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
		HP-5MS (30 m x 0.25 mm x 0.25 μm)	split mode (1:10) transfer line: 250°C ion source: 230°C quadrupole: 150°C	CBL (**231**, 232, 314, 174) CBD (**231**, 174, 314, 299) Δ^9-THC (**299**, 314, 231, 271) CBG (**193**, 231, 123, 316) CBN (**295,** 238, 310, 223)				
fresh cannabis flowers/ 0.6 g	-	Agilent 7890B GC + 5977A MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 50°C (2 min), 6°Cmin^{-1} to 300°C, 300°C (4 min) IV: 1 μL split mode (1:10) interface: 280°C	EI: 70 eV (*m*/*z* 40–500) CBG, CBD, CBN, Δ^9-THC, CBC	51.00	-	2018, Israel	[65]
cold pressed hemp seed oil/ 3.0 g **hemp seeds/ 3.0 g** **hemp proteins/ 3.0 g** **hemp teas/ 0.25 g** **confectioneries/alcoholic beverages / 5.0 g**	-	Agilent 7890B GC + 5977A MSD -	COTP: 50°C (2 min), 40°Cmin^{-1} to 270°C (7 min), 30°Cmin^{-1} to 280°C (3 min) injector: 260°C split mode (1:10) transfer line: 280°C ion source: 230°C quadrupole: 150°C	EI: 70 eV full scan mode (*m*/*z* 150-330) SIM mode CBD (**231**, 246, 209) CBNN (**299**, 231, 314) Δ^9-THC (**295**, 238, 310)	17.83	<u>CBD</u> oil 0.0005/*0.001* tea 0.01/*0.02* protein 0.001/*0.002* seed 0.001/*0.002* chocolate0.001/*0.002* <u>CBN</u> oil 0.0001/*0.0005* tea 0.002/*0.01* protein 0.0005/*0.001* seed 0.0005/*0.001* chocolate0.0005/*0.001* <u>THC</u> oil 0.0005/*0.001* tea 0.003/*0.010* protein0.0002/*0.0005* seed 0.0005/*0.001* chocolate 0.0002/*0.0005*	2018-2019, Europe	[66]
cannabis resin / 0.05 g	-	Agilent 6890 + 5975C MSD	COTP: 100°C, 10°Cmin^{-1} to 260°C (10 min) IV: 2 μL	EI, 70 eV SIM mode THCV (**271**, 286, 243, 203)	26.00	-	2013, Switzerland	[67]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
		HP-5MS (30 m x 0.25 mm x 0.25 μm)	injector: 280°C split mode (1:10) transfer line: 250°C ion source: 230°C quadrupole: 150°C	CBL (**231**, 232, 314, 174) CBD (**231**, 174, 314, 299) Δ^9-THC (**299**, 314, 231, 271) CBG (**193**, 231, 123, 316) CBN (**295,** 238, 310, 223)				
Hempseeds / 1.0 g hempseed oil/ 0 2 mL	-	Agilent 6890 N GC + 5975 MSD HP-5MS (30 m x 0.25 mm x 2.5 μm)	COTP: 80°C (1 min), 20°Cmin^{-1} to 240°C, 5°Cmin^{-1} to 260°C, 20°Cmin^{-1} to 300°C (10 min) IV: 1 μL	EI, 70 eV full scan mode THC **299**, 314 CBD **231**, 246 CBN **295**, 296, 310 THC-d$_3$ **302**, 317 CBD-d$_3$ **234**, 249 CBN-d$_3$ **298**, 299, 313	25.00	THC sunflower seeds 10.0/*50.0* olive oil 20.0/*50.0* CBD sunflower seeds 5.0/*10.0* olive oil 5.0/*10.0* CBN sunflower seeds 5.0/*10.0* olive oil 5.0/*10.0*	2020, South Korea	[68]
seized herbal cannabis (flower and leaf) / cannabis resin / 0.01 g	-	Agilent 7890 + MSD DB-5 (30 m x 0.25 mm x 025 μm)	COTP: 80°C (2 min), 10°C min^{-1} to 290°C (5 min) injector: 280°C split mode (10:1)	EI, 70 eV full scan mode (*m/z* 50-400) Δ^9-THC, CBD, CBC, CBG, CBN	28.00	-	2019, Brazil	[69]
		GCMS QP 2010 Shimadzu Ultra (GC x GC) D1: DB-5 (30 m x 0.25 mm x 0.25 μm) D2: D-17 (1.8 m x 0.1 mm x 0.1 μm)	COTP: 80°C (5 min), at 7°Cmin^{-1} to 300°C (10 min) IV: 1 μL injector: 280°C splitless mode interface: 300°C ion source: 300°C	EI, 70 eV full scan mode (*m/z* 50-550) Δ^9-THC, CBD, CBC, CBG. CBN, THCA, CBDA	46.45			
cannabis seeds / roots/ leaves / stems / flowers / 0.01 g	-	Shimadzu GC-2010 + Shimadzu QP 2010 S	COTP: 100°C (1 min), 20°Cmin^{-1} to 290°C (10 min)	EI, 70 eV SIM mode (*m/z* 40-600)	20.50	Δ^9-THC 0.005/*0.01* CBD 0.005/*0.01* CBN 0.005/*0.01*	2008-2011, Albania	[70]*

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
crushed and riddled plant		HP-5MS (30 m x 0.25 mm x 0.25 μm)	IV: 1 μL injector split mode (1:10)	Δ^9-THC (**299**, 314, 231) CBD (**231**, 174, 314) CBN (**295**, 238, 310)				
***C.indica* resin, grounded/ 10.0 g**	-	Shimadzu GC-QP 2010 Plus DB-5MS (3 0m x 0.25 mm x 0.25 μm)	COTP: 100°C (2 min), 10°Cmin^{-1} to 300°C (10 min) IV: 1 μL injector: 300°C split mode ion source: 280°C	EI, 70 eV full scan mode (*m*/*z* 85-380) Δ^9-THC, CBD, CBN, CBC, CBD, CBG, THCV, CBP, CBV, HHCBN	42.00	-	2012, Pakistan	[71]
dried *C. indica* leaves and stems, powdered / 10.0 g	-	Shimadzu GC- QP 2010 Plus DB-5MS (30 m x 0.25 mm x 0.25 μm)	IV: 1 μL injector: 300°C split mode ion source: 280°C interface: 280°C	EI, 70 eV full scan mode THCV, CBV, CBD, CBC, CBG, Δ^9-THC, CBN	-	-	2015, Pakistan	[72]
dried hemp inflorescence (*C.sativa L.* futura, 75 variety), powdered / 0.025 g	50 μL pyridine + 150 μL MSTFA + 1% TMCS	Shimadzu QP 2010 Plus Restek RTX-5 (10 m x 0.1 mm x 0.1 μm)	COTP: 180°C (0.5 min), 10°Cmin^{-1} to 250°C, 60°Cmin^{-1} to 350°C (5 min) IV: 1 μL injector: 300°C split mode (1:30) interface: 330°C ion source: 200°C	EI, 70 eV SIM mode THCV-TMS (**343**, 358, 315, 278) CBD-2TMS (**390**, 458, 301, 337) CBC-TMS (**303**, 371, 386, 246) Δ^8-THC-TMS (**386**, 303, 265, 330) Δ^9-THC-TMS (**371**, 386, 315, 303) CBG-2TMS (**337**, 321, 460, 391) CBN-TMS (**367**, 310, 382, 295) CBDA-2TMS (**491**, 453, 559, 492) THCA-2TMS (**487**, 488, 550, 413) CBGA-3TMS (**561**, 5662, 417, 453)	8.33	THCV 3.97/*12.00* CBD 4.29/*6.63* CBC 4.62/*8.91* Δ^8-THC 2.91/*8.82* Δ^9-THC 9.08/*18.40* CBG 4.07/*12.30* CBN 4.12/*12.50* CBDA 7.66/*23.20* THCA 7.75/23.50 CBGA 9.40/25.50	2018, Italy	[73]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* ($ngmL^{-1}$ or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
fresh cannabis seeds, whole / 0.5 g	-	HP 5890A + 5970A MSD DB-1 (15 m x 0.25 mm x 0.25 μm)	COTP: 170°C (1 min), 10°Cmin^{-1} to 250°C (10 min) splitless mode	EI, 70 eV SIM mode Δ^9-THC (314, 299, 231)	19.00	-	2000, USA	[74]
cannabis resin / 0.1 g	-	HP 5890 + 5971 MSD SE-52 (30 m x 0.25 mm x 0.25 μm)	COPT: 80°C (3 min), 20°Cmin^{-1} to 260°C IV: 1 μL injector: 280°C split mode (1:20) detector: 285°C ion source: 180°C	EI, 70 eV full scan mode (*m/z* 50-500) Δ^9-THC, CBD, CBN	12.00	-	2006, Italy	[75]
dried herbal cannabis, grounded / 0.06 g	-	HP 5890 + HP 5972 MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 150°C (2 min), 30°Cmin^{-1} to 210°C, 5°Cmin^{-1} to 250°C, 10°Cmin^{-1} to 280°C injector: 280°C splitless mode transfer line: 280°C	EI, 70 eV full scan mode (*m/z* 30-400) Δ^9-THC, CBD, CBN, THCV, CBG, CBV, CBC, CBCL	15.00	-	2003, Switzerland	[76]
dried herbal cannabis, grinded / 0.1 g	-	HP 5980 II^+ + 5989B MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 200-280°C IV: 2 μL injector: 280°C split mode (1:50) transfer line: 260°C ion source: 250°C quadrupole: 120°C	EI, 70 eV SIM mode THC (299, 314) CBD (231, 314)	-	-	1999, Austria	[77]
cannabis resin bars / 0.025 g	-	HP 5890 II + HP 5972 MSD BP-5 (30 m x 0.25 mm x 0.25 μm)	COTP: 100°C (2 min), 10°Cmin^{-1} to 300°C (15 min) IV: 1 μL ion source: 280°C	EI, 70 eV full scan mode (*m/z* 100-600) Δ^9-THC, CBD, CBN	37.00	-	2005, United Kingdom	[78]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
herbal cannabis, powdered / 0.06 g	-	HP 5890 + HP 5972 MSD HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 150°C (2 min), 30°Cmin^{-1} to 210°C, 5°Cmin^{-1} to 250°C, 10°Cmin^{-1} to 280°C (3 min) injector: 280°C splitless mode transfer line: 280°C	EI, 70 eV full scan mode (*m/z* 30-400) Δ^{9}-THC, CBD, CBN	18.00	-	2005, Switzerland	[79]
commercial-grade hempseed oil / 1.0 mL	-	Trace GC + DSQ II MSD ZB-5MS (30 m x 0.25 mm, 0.25 μm)	COTP: 60°C (2 min), 15°Cmin^{-1} to 260°C (5 min) IV: 1 μL injector: 260°C splitless mode transfer line: 270°C ion source: 200°C	EI, 70 eV SIM mode Δ^{9}-THC (231, 299, 314) CBD (231, 246, 314) CBN (295, 296, 310)	21.00	-	2014, Croatia	[80]
cannabis inflorescences, cannabis-based drugs, cannabis extracts / 0.5-1.0 mg	-	Agilent 7890 GC + 5977 MSD (+ Aviv Analytical SMB interface and its dual cage flight-through ion source) DB-1HT (15 m x 0.32 mm x 0.1 μm)	COTP: 50°C (0 min), 20°Cmin^{-1} to 330°C (3 min) split mode (1:10)	cold EI, 70 eV full scan mode	17.00	-	2021, Israel	[81]
cannabis oil / 1.0 g	-	Perkin Elmer GC AutoSystem XL + MS Turbo Mass MDN-5S (30 m x 0.25 mm x 0.25 μm)	COTP: 50°C (2 min), 10°Cmin^{-1} to 300°C, 300°C (3 min) IV: 1 μL injector: 250°C splitless mode ion source: 280°C interface: 280°C	EI, 70 eV SIM mode (*m/z* 40-400) Δ^{9}-THC (231), CBD (231), CBC (231), CBN (295)	30.00	Δ^{9}-THC 5 000 000 CBD 5 000 000 CBC 5 000 000 CBN 4 000 000	2005, Japan	[82]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
cannabis inflorescences (fiber-type), powdered / 0.4 g	-	Varian 3400 GC + Finnigan SSQ 710 single-stage quadrupole MSD	COTP: 120°C (1 min), 30°Cmin^{-1} to 295°C, 295°C (13 min) injector: 295°C splitless mode	THC (299, 314)	20.00	THC 0.003	2005, Australia	[83]*
no real samples	-	Varian 380 GC + Saturn 2000 IT DB-1MS (30 m x 0.25 mm x 0.1 μm) HP-50+ (30 m x 0.25 mm x 0.15 μm)	COTP: 100°C, 10°C min^{-1} to 280°C (12 min) injector: 280°C split mode (1:50) detector: 290°C	EI, 70 eV full scan mode Δ^9-THC, Δ^8-THC, THCV, CBL, CBD, CBC, CBG, CBN	30.00	-	2005, Switzerland	[84]
fresh herbal cannabis (inflorescences, lower-/upper-part leaves (1:1))/ dried herbal cannabis / 0.1 g / powdered cannabis / 0.01 g	-	Varian CP-3800 + Saturn 2000 IT HP-5 (25 m x 0.2 mm x 0.11 μm)	COTP: 60°C (2 min), 15°Cmin^{-1} to 280°C (5 min) IV: 1 μL injector: 270°C splitless mode	Δ^9-THC, CBD, CBN, Δ^9-THCA, CBDA, CBNA	22.00	-	2004, Morocco	[85]
dried *C. ruderalis* female inflorescences, pulverized / 0.0005-0.002 g	125 μL pyridine + 225 μL HMDS + 25 μL TFA 100°C, 90 min	Varian 240 GC-IT HP-5MS (30 m x 0.25 mm x 0.25 μm)	COTP: 100°C, 20°Cmin^{-1} to 300°C (10 min for TBDMS derivatives) IV: 1 μL injector: 300°C (4 min) transfer line: 300°C IT: 210°C manifold: 80°C	EI, 70 eV full scan mode Δ^9-THC, CBC, CBD, CBG, CBN	13.00 20.00 (TBDMS derivatives)	20-80 ng/mL injected sample THC *-/0.03* CBN *-/0.02*	2018, Hungary	[86]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
no real samples	dried standard solutions + 50 µL EtAc + 50 µL BSTFA + 1% TMCS 70°C, 30 min (for Δ^9-THCA)	Shimadzu GC-MS 8030 + QQQ Rxi-5MS (20 m x 0.18 mm x 0.18 µm)	COTP: 40°C (1 min), 20°Cmin^{-1} to 200°C, 3°Cmin^{-1} to 300°C (3 min) IV: 1 µL injector splitless mode (THCV, CBD, CBC, Δ^8-THC, CBG, CBN) split mode (1:80 Δ^9-THC, Δ^9-THCA, in some cases CBD) ion source: 230°C interface: 250°C	EI, 70 eV SIM mode THCV (41, 43, 271, 203) CBD (231, 232, 174, 246) CBC (231, 232, 174, 41) Δ^8-THC (231, 314, 258, 271) CBG (193, 123, 231, 41) CBN (295, 296, 238, 310) Δ^9-THC (299, 314, 231, 271) Δ^9-THCA-2TMS (487, 73, 365, 147)	46.00	THCV 20.00 CBD 10.00 CBC 10.00 Δ^8-THC 20.00 CBG 9.00 CBN 10.00 CBD 1300.00 Δ^9-THC 1300.00 Δ^9-THC-TMS (BSTFA + 1% TMCS) 850.00 Δ^9-THC-TMS (MSTFA) 1210.00 Δ^9-THCA-2TMS (BSTFA + 1% TMCS) 3800.00 Δ^9-THCA-2TMS (MSTFA) 6000.00	2016, USA	[87]
dried cannabis leaves and inflorescences (17 outdoor + 5 indoor cultivars) / 0.1 g	dried extracts + 270 µL BSTFA + 2% TMCS + 30 µL pyridine 37°C, 60 min	Agilent 7890A GC + Agilent 7200 UHD Accurate-Mass QTOF DB-5MS-UI (30 m × 0.25 mm × 0.25 µm)	COTP: 50°C, 2°Cmin^{-1} to 104°C (27 min), 20°Cmin^{-1} to 120°C (0.8 min), 4°Cmin^{-1} to 160°C (10 min), 25°Cmin^{-1} to 232°C (2.9 min), 1.5°Cmin^{-1} to 242°C (6.7 min), 2°Cmin^{-1} to 250°C (4 min), 25°Cmin^{-1} to 300°C (12 min) IV: 1 µL injector: 250°C splitless mode (derivatized samples, split mode (1:10)) transfer line: 305°C ion source: 305°C quadrupole: 200°C	EI, 70 eV (*m/z* 50-750) - (untargeted analysis)	89.40	-	2019, Spain	[88]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* ($ngmL^{-1}$ or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
no real samples	dried solution + 500 µL EtAc + 500 µL BSTFA + 1% TMCS 70ºC, 30 min	GC 2010 + VGA 100 (VUV) RTX-5 (30 m x 0.25 mm x 0.25 µm)	COTP: 40°C (1 min), 30°$Cmin^{-1}$ to 220°C, 10°$Cmin^{-1}$ to 260°C (5 min) IV: l µL injector: 250°C splitless mode transfer line: 250°C	THCV, CBD, CBC, Δ^8-THC, Δ^9-THC, CBG, CBN, Δ^9-THCA	16.00	THCV 3*/5**/- CBD 3*/5**/ - CBC 3*5**/- Δ^8-THC 3* / 5**/- Δ^9-THC 5*/10**/- CBG 3*/5**/- CBN 3*/5**/- Δ^9-THCA 3*/5**/- 11-nor-9-carboxy- Δ^9-THC 3*/5**/- 11-OH-Δ^9-THC 3*/5**/- *derivatized **underivatized	2018, USA	[89]
fresh cannabis female flower tops / 0.3 g (PLE), 2.0 g (LLE)	dried extract + 100 µL BSTFA + 1% TMCS room °C, 15 min	Shimadzu GC-17A + FID ZB-5 (30 m x 0.32 mm x 0.50 µm)	COTP: 170°C, 15°$Cmin^{-1}$ to 260°C (10 min), 5°$Cmin^{-1}$ to 280°C IV: 1 µL	Δ^9-THC, CBN, Δ^9-THCA	18.00	-	2015, Poland	[90]
cannabis herbal material, crude / 25.0 g	-	GC-MSD DB-5 (15 m x 0.25 mm x 0.25 µm)	COTP: 200°C (2 min), 10°$Cmin^{-1}$ to 240°C (2 min) IV: 1.5 µL injector: 280°C split mode (1:20) detector: 300°C	Δ^9-THC, CBD, CBN	8.00	-	2014, Pakistan	[91]
dried female flower tops / 0.05 g, 0.1 g	-	Chromapack CP9000 GC-FID DB-1 (30 m x 0.25 mm x 0.1 µm) Varian 3800 GC + Saturn 2000 IT	COTP: 100°C, 10°$Cmin^{-1}$ to 280°C injector: 280°C split mode (1:50) FID: 290°C	Δ^9-THC, CBD, CBG, CBN, Δ^9-THCA, CBDA EI, 70 eV Δ^9-THC, CBD, CBG, CBN, Δ^9-THCA, CBDA,	30.00	-	2004, the Netherlands	[92]

Matrix/amount	Derivatization conditions (derivatization agent amount, temperature, time)	instrument type /column	GC and detector conditions (COTP and other t)	compound identification	runtime (min)	LOD/*LOQ* (ngmL^{-1} or ng/g), * -LOD/*LOQ* expressed in % *(w/w)*	Year, country	Ref.
		VA-5MS (30 m x 0.25 mm x 0.25 μm)						

Table S2. LC-based analytical methods for cannabinoid profiling. APCI - atmospheric pressure-chemical ionization, CT - column temperature, CV - capillary voltage, ESI - electrospray ionization, FR - flow rate, IV - injection volume, MP - mobile phase, MPGP – mobile phases gradient program, MRM - Multiple Reaction Monitoring, SC-CO_2 – supercritical carbon dioxide, WL – wavelength.

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
dried cannabis inflorescence / 0.2 g	Agilent 1100 HPLC + Waters 2996 DAD XTerra® MS C_{18} (250 mm x 2.1 mm x 5 μm) + XTerra® MS C_{18} (10 mm x 2.1mm x 5μm)	MP A: H_2O + 50 mM NH_4COOH MP B: MeOH + 50 mM NH_4COOH 0 min (32/68), 25 min (9.5/90.5), 26 min (5/95), 29 min (5/95), 30 min (32/68), 36 min (32/68) FR: 0.3 $mLmin^{-1}$ IV: 30 μL CT: 30°C	200-400 nm	36.00	Δ^9-THCA0.05/*0.025* Δ^9-THC 0.05/*0.025* CBDA 0.05/*0.05* CBD 0.075/*0.075* CBGA 0.05/*0.05* CBG 0.15/*0.10* CBN 0.05/*0.025*	2009, Belgium	[93]
	Shimadzu HPLC + DAD Waters X-Bridge RP-C_{18} (150 mm x 4.6 mm, 3.5 μm) + Opti-Guard C_{18} (2.1 mm x 1 mm)	MP A: 50 mM NH_4COOH + 10% ACN MP B: 90% ACN 0 min (30/70), 15 min (10/90), 30 min (10/ 90), 31 min (30/70), 40 min (30/70) FR: 1 $mLmin^{-1}$ IV: 5 uL CT: 25°C	272 nm	40.00	Δ^9-THC, CBD, CBG, CBC), CBN, THCV, Δ^9-THCA, CBDA, CBGA	2013, Australia	[94]
dried and homogenized herbal cannabis (only flowers and leaves) / 0.5 g	HPLC + DAD LiChrospher® 60 RP-select B (250 mm x 4.0 mm, 5μm) + LiChrospher® 60 RP-select B (4.0 mm x 4.0 mm x 5.0 μm)	MP A: HPLC-grade H_2O MP B: ACN isocratic, 20:80, *v/v* FR: 1 $mLmin^{-1}$ IV: 10 μL CT: 30°C	220 nm, 240 nm	8.00	CBD, CBN, Δ^9-THC, Δ^9-THCA	2009, UNODC	[36]
fresh cannabis inflorescences / 0.5 g	HPLC + DAD Poroshell 120 EC-C_{18} (150 mm x 3.0 mm x 2.7 μm) or equivalent + C_{18} (5 mm x 3 mm x 2.7 μm)	MP A: 85% o-H_3PO_4 in H_2O MP B: ACN 0 min (36/64), 16 min (18/82), 17 min (36/64), 20 min (36/64) FR: 1 $mLmin^{-1}$ IV: 10 μL CT: 40°C	225 nm, 306 nm	20.00	Δ^9-THC, CBD, CBN, CBDA, Δ^9-THCA	2018, DAB 2018, Ph.Helv.	[95,96]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
cannabis SC-CO_2 extracts	Prominmence LC-2030c 3D UHPLC + DAD Shim-pack XR-ODSII RP C_{18} (2.2 μm)	MP A: H_2O + 0.07% H_3PO_4 MP B: MeOH + 0.07% H_3PO_4 0-1 min (35/65) to (28/72) in 25 min, to (5/95) in 5 min FR: 1 $mLmin^{-1}$ IV: 10 μL CT: 50˚C	-	32.00	Δ^9-THC 370/*1260* Δ^8-THC 510/*1710* CBD 340/*1130* CBN 330/*1102* CBDA 320/*1080* Δ^9-THCA 270/*920* CBC 290/*990* CBG 310/*1030* CBGA 320/*1060* THCV 330/*1110* CBDV 420/*1410*	2021, Australia	[97]
dried cannabis herbal material / 20.0 g	Waters HPLC 900 + 996 PDA Ace® 5 Phenyl (250 mm x 4.6 mm x 5 μm) + Nova-Pak® C_8 (20 mm x 3.9 mm)	MP A: H_2O + 0.1% TFA MP B: H_2O/ACN (65:45, *v*/*v*) + 0.1% TFA MP C: ACN 0 min (70/30/0), 10 min (60/40/0), 38 min (40/60/0), 40 min (5/95/0), 55 min (0/100/ 0), 74 min (70/30/0) + post-phase (63-71 min, 0/0/100) FR 0.9 mL/min IV: 10 μL/30 μL CT: 25°C	214 nm	80.00	- (fingerprinting method)	2017, United Kingdom	[98]
	Waters HPLC 900 + 996 PDA Zorbax RX-C_{18} (250 mm x 4.6 mm x 5 μm)	MP A: H_2O/ACN (65:45, *v*/*v*) + 0.1% TFA MP B: ACN 0 min (70/30), 30 min (35/65), 48 min (70/30), 40 min (5/95)	/	55.00			
no real samples	HPLC Agilent 1260 + QTRAP Agilent Eclipse Plus 95A C_{18} (100 mm x 4.6 mm x 3.5 μm) with guard column	MF A: H_2O + 0.1% HCOOH MF B: ACN + 0.1% HCOOH isocratic, 10:90, *v*/*v* FR: 0.5 $mLmin^{-1}$ IV: 20 μL CT: 40°C	ESI (-) CBDA 357.0 → 339.0 357.0 → 179.0 Δ^9-THCA 357.0 → 313.0 357.0 → 245.0 ESI (+) CBD, Δ^9-THC	11.00	CBD 0.048 Δ^9-THC 0.048 CBDA 0.024 Δ^9-THCA 0.024	2018, Canada	[99]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
			315.0 → 193.0 315.0 → 259.0				
dried cannabis female flowers / 0.1 g	Thermo Scientific UHPLC + Q Exactive™ Orbitrap Kinetex C_{18} (150 mm × 2.1 mm x 2.6 μm) + guard column (0.5 μm depth filter × 0.1 mm)	MP A: Milli Q + 0.1% CH_3COOH MP B: CAN + 0.1% CH_3COOH MP C: MeOH 0 min (45/50/5), 2 min (28/67/5), 6 min (28/67/5), 10 min (5/90/5), 14 min (5/90/5), 15 min (45/50/5), 20 min (45/50/5) FR: 0.3 $mLmin^{-1}$ IV: 1 μL CT: 30°C	ESI (-) full scan mode (150-500 *m/z*) MS/MS mode	20.00	Δ⁹-THCA *5.0* CBDA *1.0* Δ⁹-THC *2.0* CBDVA *0.25* CBD *1.0* CBGA *2.5* CBG *2.0* CBDV *1.25* CBC *0.25* THCV *0.25* CBN *1.25* Δ⁸-THC *1.25* CBL *1.25*	2018, Israel	[100]
cannabis extract microdepots	Thermo Scientific UHPLC + Q-Exactive™ Orbitrap Halo C_{18} (150 mm × 2.1 mm x 2.7 μm) + guard (5 mm x 2.1 mm)	MP A: Milli Q + 0.1% CH_3COOH MP B: ACN + 0.1% CH_3COOH MP C: MeOH 0 min (45/50/5), 2 min (28/67/5), 6 min (28/67/5), 10 min (5/90/5), 14 min (5/90/5), 15 min (45/50/5), 20 min (45/50/5) FR: 0.25 $mLmin^{-1}$ IV: 5 μL CT: 30°C	ESI (-) full scan mode (150-500 *m/z)*	20.00	Δ⁹-THC, Δ⁹-THCA, CBDA, CBD, CBG, CBGA, CBC, CBN, CBDV	2020, Israel	[101]
	Thermo Scientific UHPLC + Q Kinetex C_{18} (150 mm × 2.1 mm x 2.6 μm) + SecurityGuard Ultra (2 mm × 2.1 mm)	MP A: Milli Q + 0.1% CH_3COOH MP B: ACN + 0.1% CH_3COOH MP C: MeOH 0 min, at 0.8 $mLmin^{-1}$ (45/50/5), 4 min, at 0.8 $mLmin^{-1}$ (45/50/5), 6 min, 0.3 mL/min (28/67/5), 10 min, at 0.3 $mLmin^{-1}$ (28/67/5), 14 min at 0.3 $mLmin^{-1}$ (5/90/5), 18 min at 0.3 $mLmin^{-1}$ (5/90/5), 18 min at 0.8	ESI (-) SIM mode	24.00			

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
		$mLmin^{-1}$ (45/50/5), 24 min at 0.8 $mLmin^{-1}$ (45/50/5) FR: 0.25 $mLmin^{-1}$ CT: 30°C IV: 5 µL					
dried cannabis leaves and female inflorescences / 0.1 g	Agilent 1200 LC+ 6540 UHD QTOF RP-C_{18} (250 mm x 4.6 mm x 3 µm)	MP A: H_2O + 5% ACN + 0.1% HCOOH MP B: ACN + 5% H_2O + 0.1 % HCCOH 0 min (96/4), 1 min (96/4), 5 min (80/20), 10 min (30/70), 20 min (10/90), 32 min (0/100), 42 min (0/100) + post-time re-equilibration, 10 min (96/4) FR: 0.7 mL/min IV: 5 µL CT: 34°C	ESI (+/-) full scan mode (60-1100 *m/z)* MS/MS mode CBDVA 329.1758 CBDV 287.1992 CBDA 357.2072 CBG 317.2480 CBGA 359.2227 CBD 315.2331 THCV 287.1992 CBN 309.1863 Δ^9-THC 315.2300 Δ^8-THC 315.2316 CBC 315.2303 CBL 313.2173 Δ^9-THCA 357.2071	52.00	-	2019, Spain	[88]
dried cannabis leaves and flowers (mixed) / 0.1 g	Waters 1515® HPLC + DAD Nucleodur® C_{18} Gravity (250 mm x 4.6 mm x 5 µm)	MPA: 50mM o-H_3PO_4 in H_2O MP B: ACN isocratic, 15:85 v/v FR: 1-3 $mLmin^{-1}$ CT: 35°C	Δ^9-THC 211 nm Δ^9-THCA 220 nm	5.00	Δ^9-THC 4540/*15130*	2019, Switzerland	[102]
dried cannabis inflorescences / 0.025g	HPLC Prominence-i LC2030C + UV RP-C_{18} Nex-Leaf CBX Potency (150 mm x 4.6 mm x 2.7 µm) + NexLeaf	MP A: H_2O + 0.085% o-H_3PO_4 MP B: ACN + 0.085% o- H_3PO_4 0 min (30/70), 3 min (30/70), 7 min (15/85), 7.01 min (15/85), 8 min (5/95), 10 min (30/70) FR: 1.6 $mLmin^{-1}$	220 nm	8.00	CBDA 340/*1050* CBGA 320/*980* CBG 620/*1870* CBD 630/*1910* THCV 950/*2870* CBN 280/*840* Δ^9-THC 1250/*3790*	2019, Italy	[103]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	CBX (5 mm x 4.6 mm x 2.7 μm)	IV: 5 μL CT: 35°C			Δ^8-THC 1020/*3100* CBC 290/*880* Δ^9-THCA 430/1290		
dried cannabis inflorescences / 2.5 g	Agilent 1100 HPLC + MSD trap (SL) Poroshell 120 SB-C_{18} (75 mm x 3.0 mm x 2.7 μm)	MP A: 25 mM NH4CH2COOH MP B: MeOH 0 min (32/68), 9 min (15/85), 10 min (32/68) FR: 0.7 mLmin^{-1} IV: 10 μL CT: 30°C	235 nm	10.00	CBDA 62.5/*250* CBGA 62.5/*250* CBG 62.5/*250* CBD 62.5/*250* THCV 62.5/*250* CBN 62.5/*250* Δ^9-THC 62.5/*250* Δ^8-THC 62.5/*250* CBC 62.5/*250* Δ^9-THCA 62.5/*250*	2017, USA	[104]
fresh cannabis female inflorescences / 0.25 g	Agilent 1100 HPLC + UV/DAD Ascentis Express C_{18} (150 mm x 3.0 mm x 2.7 μm)	MP A: 0.1% HCOOH in H_20 MP B: 0.1% HCOOH in ACN 0 min (40/60), 13 min (40/60), 17 min (20/80), 22 min (10/90) FR: 0.4 mLmin^{-1} IV: 3 μL CT: 30°C	210 nm, 220 nm	37.00	CBDA, CBGA, CBD, CBG	2018, Italy	[105]
	Agilent 1100 HPLC + IT Ascentis Express C_{18} (150 mm x 3.0 mm x 2.7 μm)		ESI (+/-) full scan mode (200-1200 *m*/*z*) MS/MS mode (50-1500 *m*/*z*) CBDA 359, 341/ 357* CBGA 361, 343 / 359* CBG 317/315* CBD 315/313* *(-) mode	37.00	-		
dried cannabis plant material (recreational, medical, hemp) / consumer products (oral supplements, foods, candies, beverages, vapes, liquids, topicals) /	Agilent 1100, 1200, or 1260 HPLC + DAD MacMod Ace® 5 C_{18}-AR (250 mm x 4.6 mm x 5 μm)	MP A: 0.5% CH_3COOH MP B: ACN isocratic, 34:66, *v*/*v* FR: 1 mLmin^{-1} IV: 25 μL	220 nm 240 nm 270 nm 307 nm	50.00	CBD, CBDA, Δ^9-THC, THCA, CBN, Δ^8-THC, CBG, CBGA, CBDV, THCV, CBC concentration-based 200/*500* method-based *10000*	2018, USA	[106]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
medical products / illicit products (kief, hash oil) / 0.03 – 3.0 g							
hexane (+0.1% HCOOH) cannabis inflorescence extracts	Agilent 1100 HPLC + G1315 DAD + 6320 IT Luna Omega PS C_{18} (150 mm x 2.1 mm x 5 μm)	MPA: H_2O + 0.1% HCOOH MPB: ACN 0 min (50/580) + 6 min, 12 min (43/57), 23 min (50/50) + 2 min FR: 0.4 $mLmin^{-1}$ IV: 5 μL CT: 28°C	(190-6020 nm for UV-Vis spectra acquisition) 220 nm ESI (+) and (-) SIM mode ESI (-) CBDA **357**, 339, 245 CBGA **359**, 341 CBNA **353**, 309, 279 THCAA **357**, 313, 245 ESI (+) CBG **317**, 207, 233 CBD **315,** 259, 233 CBN **311**, 223, 43 Δ^9-THC **315**, 245, 193	25.00	-	2021, Italy	[107]
cannabis tinctures/oils / 0.5 mL beverages / 1.0 mL powders, edibles. gummies and candies / 1.0 g	Agilent 1290 HPLC + DAD/FLD ACE Excel 3 C_{18} (150 mm x 2.1 mm x 3.0 μm)	MP A: 0.5% CH_3COOH MP B: ACN 0 min (33/67), 17 min (5/95) + re-equilibration 18 min (33/67) FR: 0.3 mL IV: 2 μL CT: 25°C	DAD: 220 nm 240 nm 270 nm 307 nm FLD: 234/311 nm (0-8.5 min) 261/378 nm (8.5-11.0 min) 234/315 nm (11.0-13.5 min) 272/346 nm (13.5-24.0 min)	24.00	CBD, CBDA, Δ^9-THC, THCA, CBN, Δ^8-THC, CBG, CBGA, CBDV, THCV, CBC	2021, USA	[108]
CBD e-liquids	Shimadzu HPLC + Applied	MP A: DI H_2O MP B: MeOH isocratic, 90:10, *v/v*	CBD 315>193, 315>259	8.00	CBD	2016, USA	[109]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	Bioscience 3200 QTRAP Zorbax Eclipse XDBC_{18} (75 mm x 4.6 mm x 3.5 µm)	FR: 0.5 $mLmin^{-1}$ IV: 10 µL					
fresh cannabis plant material / 0.05 g	Agilent HPLC Infinity + Agilent 6430 QQQ Kinetex C_{18} (150 mm x 3 mm x 2.6 µm) + guard column (0.5 µm depth filter x 0.1 mm)	MP A: H_2O + 0.1% HCOOH MF B: MeOH + 0.1% HCOOH 0 min (50/50), 1 min (20/80), 11 min (20/80), 13 min 5 (5/95), 16 min (5/95), 18 min (50/50), 28 min (50/50) FR: 0.25 $mLmin^{-1}$ IV: 10 µL CT: 30°C	APCI (+) MRM mode CBD 315.1 → 192.8 315.1 → 259.0 THCV 287.1 → 165.0 287.1 → 231.0 CBG 317.2 → 193.2 317.2 → 123.0 CBN 311.0 → 222.9 311.0 → 293.0 Δ^9-THC 315.0 → 193.0 315.0 → 259.0 Δ^9-THCA 315.1 → 193.0 315.1 → 259.1	28.00	CBD 0.2 THCV 0.05 CBG 0.02 CBN 0.05 Δ^9-THC 0.05 Δ^9-THCA 0.02	2014, Spain	[110]
	Waters ACQUITY UPLC + SYNAPT G2 QTOF Kinetex C_{18} (150 mm x 3 mm x 2.6 µm) + guard column (0.5 µm depth filter x 0.1 mm)		APCI (+) untargeted analysis	22.00	CBD, THCV, CBG, CBN, Δ^9-THC, Δ^9-THCA		
	Waters Thar SFC s	MP: SC-CO_2 + MeOH (15%) FR: 1.5 $mLmin^{-1}$ IV: 5 µL CT 40°C	220 nm	7.00			

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	Kromasil NP-DIOL (250 mm x 4.6 mm x 5 μm)						
fresh cannabis extracts / 0.05 g	Waters UHPSFC + PDA + Q ACQUITY UPC² BEH 2-EP (150 mm x 3.0 mm x 1.7 μm)	MP A: SC-CO_2 MP B: isopropanol/ACN (80:20, *v*/*v*) + 1% H_2O 0 min (96/4), 4.5 min (91/9), 7.0 min (70/30), 10 min (70/30) FR: 1.4 $mLmin^{-1}$ IV: 1.0 μL CT: 30°C	scan mode (19-400 nm) 220 nm	16.50	CBD, THCV, CBG, CBN, Δ⁹-THC, Δ⁹-THCA, CBDA, CBGA	2016, USA	[111]
cannabis seed oil / 0.1 mL	Agilent HPLC 1200 + DAD Poroshell 120 EC-C_{18} (100 mm x 3.0 mm x 2.7 μm)	MP A: H_2O + 0.1% HCOOH MP B: ACN + 0.1% HCCOH 0 min (30/70), 10 min (20/80), 10.1 min (5/95), 11.0 min (5/95), 11.1 min (30/70), 15 min (30/70) FR: 0.4 $mLmin^{-1}$ IV: 5 μL CT: 25°C	228 nm	15.00	CBDV, CBDA, CBG, CBD, CBN, Δ⁹-THC, Δ⁹-THCA	2018, Italy	[112]
	Agilent HPLC 1200 + 6540 QTOF Poroshell 120 EC-C_{18} (100 mm x 3.0 mm x 2.7 μm)		ESI (+/-) full scan mode (50-700 *m*/*z*) MS/MS mode, (+) CBDA 359.2217 Δ⁹-THCA 359.2217 CBD 315.2300 Δ⁹-THC 315.2300 CBDV 287.1998 CBG 317.2468 CBN 311.2024 MS/MS mode, (-) CBDA 357.2164 Δ⁹-THCA 357.2164 CBD 313.2012 Δ⁹-THC 313.2012 CBDV 285.1830 CBG 315.2385	15.00			

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
			CBN 309.1902				
fresh cannabis female inflorescences / 0.25 g cannabis oil / 50 µL cannabis balm/ 0.25 g cannabis extract / 0.02 g	Agilent HPLC 1100 + UV Ascentis Express C_{18} (150 mm x 3.0 mm x 2.7µm)	MP A: H_2O + 0.1% HCOOH MP B: ACN + 0.1% HCOOH 0 min (40/60), 13 min (40/60), 17 min (20/80), 22 min (10/90), 30 min (10/90) FR: 0.4 mLmin^{-1} IV: 3 µL CT: 30°C	210 nm 220 nm	45.00	CBDA 800/*2500* CBGA 800/*2500* CBG 500/*1800* CBD 700/*1300*	2017, Italy	[113]
	Agilent HPLC 1200 + 6310A IT Ascentis Express C_{18} (150 mm x 3.0 mm x 2.7µm)		ESI (+/-) full scan mode (200-1200 *m/z*) MS/MS mode (50-1500 *m/z*)		-		
multi-floral / dandelion / chestnut honey / 20.0 g fresh cannabis male inflorescences and their pollen / 0.25 g	Agilent 1100 HPLC + UV/DAD Ascentis Express C_{18} (150 mm x 3 mm x 2.7 µm)		210 nm 220 nm				
	Agilent 1200 HPLC + AB SCIEX API 4000 QTRAP Kinetex EVO C_{18} (100 mm x 2.1 mm x 5 µm)	MP A: 2 mM $CH_3COOHNH_4$ in H_2O MF B: 2 mM $CH_3COOHNH_4$ in ACN 0 min (70/30), 10 min (10/90), 15 min (10/90), 18 min (70/30) FR: 0.35 mLmin^{-1} IV: 25 µL CT: 40°C	ESI (-) MRM mode CBDA 357 → **245**, 179, 271 CBGA 359 → **341**, 315, 217 Δ^9-THCA 357 → **191**, 245 CBG 315 → **136**, 191, 177 CBD 313 → **245**, 107 Δ^9-THC 313 → **245**, 191, 203	32.00	CBDA 0.3/*0.5* CBGA 0.3/*0.5* Δ^9-THCA 0.3/*0.5* CBG 0.3/*0.5* CBD 0.3/*0.5* Δ^9-THC 0.3/*0.5*	2019, Italy	[114]
foods, beverages and feeds / 1.0 g	Agilent 1200 HPLC + AB SCIEX API 4000 QTRAP Ascentis Express RP-amide (50 mm	MPA: H_2O + 0.1% HCOOH MPB: ACN + 0.1% HCOOH 0-10.0 min (60/40), 19.0 min (5/95), 22.0 (60/40) + re-equilibration 7 min FR: 0.8 mLmin^{-1}	ESI (+) and (–) MRM mode (+) Δ^9-THC 315.4 → 193.3 315.4 → 259.4 Δ^8-THC 315.4 → 193.3 315.4 → 259.4	22.00	CBD, CBN, CBG, Δ^8-THC, Δ^9-THC, THCV, CBDA, CBGA, Δ^9-THCA honey, coffee and eggs: 6.0/*20.0* beverages: 0.6/*2.0*	2021, Italy	[115]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	x 4.6 mm x 2.7 μm)	IV: 100 μL CT: 25°C	CBD 315.5 → 193.2 315.5 → 259.1 CBN 315.5 → 223.2 315.5 → 241.2 CBG 317.3 → 193.4 317.3 → 123.4 THCV 287.4 → 165.3 287.4 → 135.3 (-) THCA 357.5 → 313.5 357.5 → 245.2 CBDA 357.5 → 245.4 357.5 → 339.5 CBGA 359.4 → 341.5 359.4 → 315.5		feed: 30.0/*100.0*		
cannabis extracts / 2.0 g, 5.0 g, 20.0 g (depending on extraction methodology)	Agilent HPLC 1200 + UV Poroshell 120 SB-C_{18} (100 mm x 2.1 mm x 2.7 μm)	MP A: H_2O + 0.1% HCOOH MP B: ACN + 0.1% HCOOH isocratic FR: 0.5 mL/min IV: 5 μL CT: 25°C	228 nm	10.00	CBDA CBD, CBN, Δ^9-THC, Δ^9-THCA	2016, Italy	[116]
	Agilent HPLC 1200 + 6540 QTOF Poroshell 120 SB-C_{18} (100 mm x 2.1 mm x 2.7 μm)		ESI (+) full scan mode (50-500 *m/z*) MS/MS mode (50-1700 *m/z*) CBDA 359.2224 CBD 315.2314 CBN 311.2000 Δ^9-THC 315.2311 Δ^9-THCA 359.2216	10.00	CBDA, CBD, CBN, Δ^9-THC, Δ^9-THCA		
dried cannabis plant material / 0.3 g marihuana / 0.5 g	HPLC Waters 2695 + LiChrospher 60, RP-Select B LiChroCart (125 mm x 4 mm x 5 μm) +	MP A: 1M triethylammoniumphosphate in Milli-Q MP B: ACN isocratic, 36:64, *v/v* FR: 1 $mLmin^{-1}$	210 nm	~16.00	Δ^9-THC 1000/*6000* Δ^9-THCA 4000/1*6000* CBD 1000/*40000* CBN 1000/*40000*	2014, Switzerland	[117]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	LiChrospher 60, RP- Select B (5 μm)	IV: 10 μL					
capsule, oil tincture, soft chew or powder / 0.02 g, 0.1 g or 1.0 g	Acquity UPLC + DAD	**-**	190-500 nm	-	CBGA, CBG, CBDA, CBD, Δ^9-THCA, Δ^9-THC, CBN, exo-THC, Δ^8-THC, CBC, THCV, CBDV	2020, USA	[118]
fresh cannabis inflorescences, cannabis tea, cannabis oil / 0.5 g	Waters Acquity UPLC + QQQ Acquity UPLC HSS C_{18} (150 mm x 2.1 mm x 1.8 μm)	MP A: H_2O + 0.1% HCOOH MP B: ACN 0 min (40/60), 0.5 min (40/60), 4.5 min (10/90), 6.5 min (10/90), 7.0 min (40/60), 10 min (40/60) FR: 0.4 mLmin^{-1} IV: 30 μL CT 30°C	ESI (+) CBDA 359.4 → **219.3**, 261.3 CBG 317.5 → **193.3**, 123.2 CBD 315.4 → **193.3**, 123.2 CBN 311.4 → **223.3**, 293.4 Δ^9-THC 315.4 → **123.2**, 193.3 CBC 315.4 → **193.3**, 123.2 Δ^9-THCA 359.4 → **219.3**, 261.3	10.00	CBDA, CBG, CBD, CBN, Δ^9-THC, CBC, Δ^9-THCA	2017, Italy	[119]
dried cannabis plant material / 0.1 g	HPLC + DAD Luna C_{18} (150 mm x 4.60 mm x 3 μm) + C_{18} guard column cartridge	MP A: H_2O + 0.1% HCOOH MP B: ACN + 0.1% HCCOH 0 min (30/70), 6 min (30/70), 12 min (23/77), 22 min (23/77), 22.2 min (30/70) FR: 1.2 mLmin^{-1} IV: 10 μL CT: 28°C	220 nm	22.50	CBDA 100/*18400* CBGA 1300/*3900* CBG 2500/*7700* CBD 1600/*4900* THCV 700/*2100* CBN 300/*1000* Δ^9-THC 2300/*6900* Δ^8-THC 1400/*4200* CBL 1100/*3400* CBC 1100/*3400* Δ^9-THCA 1100/*3400*	2015, Egypt	[120]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
cannabis inflorescences / 0.1 g [121] **cannabis plant material, cannabis oil / 0.1 g [122]** **dried cannabis inflorescences / 1.0 g [123]**	Thermo Fisher HPLC + Q-Exactive Orbitrap® Synergi Hydro RP (150 mm x 2 mm x 4.0 μm) + C_{18} (4 mm x 3 mm)	MP A: H_2O + 0.1% HCOOH MP B: ACN + 0.1% HCOOH [121,123] 0 min (95/5), 35 min (5/95) [121,123] MP A: H_2O + 0.1% HCOOH MP B: ACN 0 min (60/40), 10 min (95/5), 14 min (95/5) [122,124] FR: 0.3 $mLmin^{-1}$ IV: 2 μL CT: 30°C	ESI (+/-) full scan mode (100-900 *m/z*) MS/MS mode [121,123] full scan mode (215-500 *m/z*) MS/MS mode [122,124]	35.00 [121,123] 20 min [122,124]	CBD, Δ^9-THC, CBN, CBG, CBC, CBDV, THCV, CBDA, Δ^9-THCA, CBNA, CBGA, CBCA, CBDVA, THCVA [121,123] CBD *0.1* Δ^9-THC *0.1* CBN *0.1* CBG *0.1* CBDA *0.05* THCA *0.05* CBGA *0.05* [122] CBD, ΔTHC, CBN, CBG, CBDA, Δ^9-THCA, CBGA [124]	2019, Italy 2018, Italy 2020, Italy 2018, Italy	[121] [122] [123] [124]
dried cannabis inflorescences, leaves, stem barks and roots / 2.0-4.0 g	Agilent 1260 Infinity II + Q Zorbax RX-C_{18} (150 mm x 4.6 mm x 3.5 μm)	MP A: H_2O + 0.2% HCOOH MP B: MeOH 0 min (25/75), 13 min (10/90), 26 min (10/90) FR: 0.6 $mLmin^{-1}$ IV: 5 μL CT: 30°C	ESI (+) CBDV 287.2 CBDVA 331.2 CBG 317.3 CBD 315.3 CBDA 359.2 THCV 287.2 CBGA 343.3 CBN 311.2 Δ^9 -THC 315.3 Δ^8 -THC 315.3 THCVA 331.2 CBC 315.2 Δ^9-THCA 359.3 CBCA 359.3	30.00	CBDV 1.0/*3.0* CBDVA 2.0/*5.0* CBG 2.0/*5.0* CBDA 1.0/*3.0* THCV 0.4/*1.0* CBGA 3.0/*8.0* CBN 1.0/*2.0* Δ^9-THC 1.0/*3.0* Δ^8-THC 2.0/*5.0* THCVA 3.0/*10.0* CBC 2.0/*7.0* Δ^9-THCA 3.0/*9.0* CBCA 4.0/*12.0*	2020, Canada	[125]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
galenic cannabis oils/ 10 µL or 50 µL	Acquity® UPLC + TQD AcquityHSS-T3 (30 mm x 2.1 mm x 1.8 µm)	MPA: H_2O/ACN (30/70) + 0.05% HCOH MPB: isopropanol/ACN (80/20) + 0.05% HCOH 0 min (100/0), 4.6 min (0/100) + 1.5 min + reconditioning at (0/100) FR: 0.4 mLmin^{-1} IV: 4 µL	ESI (+) SRM mode THC 315.2 → 193.1 CBD 315.2 → 259.2 CBDA 359.15 → 261.1 THCA 341.15 → 219.15 THC-d_3 318.2 → 196.1 CBD-d_3 318.2 → 296.5	7.00	-	2021, Italy	[126]
	Agilent 1200 HPLC + 6430 QQQ Zorbax Eclipse Plus C_{18} (50 mm x 2.1 mm x 1.8 µm)	MPA: H_2O + 0.1% HCOOH in ACN MPB: 0.1% HCOOH in ACN 0 min (50/50), 2.8 min (0/100) + 2 min + reconditioning at (50/50) FR: 0.3 mLmin^{-1} IV: 5 µL	ESI (+) SRM mode THC 315.2 → 123.0 CBD 315.2 → 193.1 CBDA 357.2 → 245.1 THCA 357.2 → 245.1 THC-d_3 318.2 → 196.1 CBD-d_3 262.0 → 196.1	8.50			
dried cannabis plant material / 0.1 g	Agilent Infinity 1290 UHPLC + 6430 QQQ Zorbax Eclipse Plus C_{18} Rapid Resolution	MPA: H_2O + 5 mM ammonium formate MPB: ACN + 0.1% HCOOH 0 min (35/65), 4.5 min (20/80), 7.5 min (19.1/80.90), 9.5 min (0/100) + re-equilibration FR: 0.6 mLmin^{-1} IV: 5 µL CT: 30°C	ESI (+) and (-) MRM mode ESI (+) CBG-d_9 **326.3 → 202.2** 326.3 → 123.0 CBG **317.3 → 193.1** 317.3 → 123.0 CBD-d_3 **318.3 → 196.1** 318.3 → 123.0 CBD **315.2 → 193.1** 315.2 → 123.0 THCV **287.2 → 123.0** 287.2 → 231.1 THC-d_3 **318.3 → 196.1** 318.3 → 123.0 THC **315.2 → 193.1** 315.2 → 123.0 CBL **315.2 → 235.2**	14.00	THC 0.014/*10.0* THCA 0.01/*10.0* CBD 0.2/*0.5* CBDA 0.04/*0.4* CBN 0.25/*0.25* CBNA 0.005/*1.0* CBG 1.0/*1.0* CBGA 0.1/*1.0* CBC 0.5/*0.5* CBCA 0.1/*10.0* CBL 0.2/*0.2* CBLA 0.013/*1.0* THCV 0.25/*0.5* THCVA 0.005/*1.0* CBDV 0.5/*1.25* CBDVA 0.02/*0.1*	2021, Germany	[127]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
			315.2 → 165.1 CBC-d9 **324.3 → 202.2** 324.3 → 268.2 CBC **315.2 → 193.1** 315.2 → 259.2 <u>ESI (-)</u> CBDA **329.2 → 311.2** 329.2 → 217.1 THC-COOH-d9 **352.3 → 308.3** 352.3 → 254.3 CBDV **285.2 → 217.1** 285.2 → 107.0 CBDA **357.2 → 339.2** 357.2 → 245.2 CBGA **359.2 → 341.2** 359.2 → 315.2 THCVA **329.2 → 285.2** 329.2 → 217.1 CBN-d3 **312.2 → 282.1** 312.2 → 222.1 CBN **309.2 → 279.1** 309.2 → 222.1 CBNA **353.2 → 309.2** 353.2 → 279.1 CBC-d9 **324.3 → 200.2** 324.3 → 268.2 THCA **357.2 → 313.2** 357.2 → 245.2 CBCA **357.2 → 313.2** 357.2 → 191.1 CBLA **357.2 → 313.2** 357.2 → 191.1				
dried cannabis plant material	ThermoFisher UltiMate® 3000	MPA: H_2O + 0.1% HCOOH MPB: ACN + 0.1% HCOOH	ESI (+) MRM mode	15.00	CBD 0.20/*0.61* CBN 0.03/*0.09* THC 0.06/*0.17*	2021, South Africa	[128]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	HPLC + Bruker Compact QTOF C_{18} Wate (100 mm x 4.6 mm x 3.5 μm) + guard column	- FR: 0.3 $mLmin^{-1}$ IV: 10 μL CT: 30°C					
lyophilized non-drug-type cannabis plant material	HP HPLC 1050 + G1315B DAD Luna C_{18} (150 mm x 2 mm x 3 μm)	MPA: 5% ACN in H_2O + 0.1% o-H_3PO_4 MPB: 80% CAN in H_2O + 0.1% o-H_3PO_4 isocratic (17/83, *v/v*) FR: 0.25 $mLmin^{-1}$ IV: 5 μL CT: 35°C	220 nm	15.00	CBD, CBDA, CBG 13.0/*44.0*	2021, Czech Republic	[129]
	Thermo Fischer LCQ Accela Fleet + IT Luna C_{18} (150 mm x 2 mm x 3 μm)	MPA: 5% ACN in H_2O + 0.1% HCOOH MPB: 80% ACN in H_2O + 0.1% HCOOH isocratic (17/83, v/v) FR: 0.25 $mLmin^{-1}$ IV: 5 μL CT: 35°C	APCI (-)		-		
cannabis plant material, cannabis resins / 0.5 g	Acquity UPLC + PDA Poroshell 120 EC C_{18} (150 mm x 2.1 mm x 2.7 μm) + Poroshell 120 EC-C_{18} guard (5 mm x 2.1 mm x 2.7 μm)	MPA: H_2O + 0.1% HCOOH MPB: ACN + 0.1% HCOOH 0 min (32/68), 2.8 min (27/73), 7.0 min (0/95) +1.0 min + re-equilibration (4.5 min) FR: 0.5 $mLmin^{-1}$ IV: 1 μL CT: 30°C	214 nm	18.00	CBD, CBN, THC, CBDA, THCA	2020, Belgium 2021, Belgium	[130]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
no real samples	Agilent 1260 Infinity + DAD ACE 3 C_{18}-PFP (150 mm x 3.0 mm x 3.0 μm)	MPA: H_2O MPB: MeOH isocratic, 17/83 *v*/*v* FR: 0.4 $mLmin^{-1}$ IV: 5 μL CT: 25°C	222 nm	20.00	CBD 25.0/*100.0* CBN 25.0/*100.0* Δ^9-THC50.0/*100.0*	2021, Thailand	[131]
lyophilized cannabis flowers	Agilent HPLC 1100 + DAD Kinetex® C_{18} (150 mm x 2.1 mm x 2.6 μm)	MPA: H_2O + 0.1% TFA MPB: MeOH + 0.1% TFA 0 min (32/68), 13 min (15/85) + 7 min FR: 0.25 $mLmin^{-1}$ CT: 60°C	230 nm	20.00	CBG, CBGA, CBD, CBDA, CBN, Δ^9-THC, CBC, CBCA, Δ^9-THCA, THCV, CBDV, CBGVA	2021, Canada	[132]
upper cannabis leaves / 1.5 g	Nexera XR LC-20AD + DAD Atlantis T3 C_{18} (150 mm x 4.6 mm x 3.0 μm)	MPA: ACN MPB: H_2O + 0.85% phosphoric acid 0 min (53/47), 15 min (20/80) + 11 min, 28 min (0/100) + 5 min + re-equilibration 5 min	-	38.00	THC 810.0/*2680.0*	2021, Austria	[133]
cannabis inflorescence / 0.05 g **cannabis oil**	Shimadzu HPLC + SPD-20A UV Raptor ARC-18 (150 mm x 4.6 mm x 2.7 μm)	MPA: 5 Mm ammonium formate + 0.1% HCOOH MPB: ACN + 0.1% HCOOH isocratic, 25/75 *v*/*v*	228 nm	11.00	THCA, CBDA *-/ ≤ 1050* Δ^9-THC, CBD *-/ ≤ 5250* CBDV, CBDVA, CBGA, CBG, THCV, THCVA, CBN, CBNA, Δ^8-THC, CBC, CBL, CBLA, CBCA *-/ ≤ 500*	2021, Australia	[134]
dried cannabis inflorescences and inflorescence leaves / 0.05 g	Jasco 2000 Plus HPLC + PDA	MPA: ACN MPB: H_2O + 0.1% HCOOH isocratic, 75/255 *v*/*v* FR: 1.0 $mLmin^{-1}$	200-650 nm CBC, CBCA, CBCV, CBG, CBGA, CBN, CBNA, CBD, CBDA, CBL, CBDV, CBDVA,	-	-	2021, Israel	[135]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	Luna Omega Polar C_{18} (150 mm x 2.1 mm x 3 μm)		THCVA, THCA, Δ^9-THC, CBT				
hemp pollen from industrial cannabis inflorescences	Nexera UHPLC + Ab SCIEX TripleTOF 4600 hybrid system Luna® Omega C_{18} (50 mm x 2.1 mm x 1.6 μm)	MPA: H_2O + 0.1% HCOOH MPB: ACN + 0.1% HCOOH 0 min (75/25), 1 min (45/55), 8.5 min (5/95) + 1 min + re-equilibration FR: 0.5 mLmin^{-1} IV: 2 μL	ESI (-) CBDA, CBCA, THCA	11.50	-	2021, Italy	[136]
CBD cosmetic products / 1.0 g	Agilent 1100 HPLC + Agilent 6410B QQQ Zorbax SB-C_{18} (50 mm x 2.1 mm x 1.8 μm)	MPA: MeOH + 0.1% HCOOJH MPB: H_2O + 0.1% HCOOH isocratic, 80/20 *v*/*v* FR: 0.2 mLmin^{-1} IV: 10 μL CT: 35°C	ESI (+) MRM mode CBD **315 → 193** 315 → 41 315 → 123 CBD-d_3 **318 → 196** 318 → 41 318 → 123	4.00	CBD 0.22/*0.74*	2021, Spain	[137]
hempseed oil / 0.5 g raw milk/ 10.0 g hemp seeds, hemp proteins, tea, raw milk, skimmed powder milk, coffee and chocolate / 2.0 g	Waters UPLC + Sciex QTRAP 6500 Acquity BEH Shield RP18 (100 mm x 2.1 mm x 1.7 μm)	MPA: H_2O + 0.1% HCOOH MPB: ACN 0 min (50/50), 9.0 min (0/100), 2 min + 2.0 min re-equilibration FR: 0.5 mLmin^{-1} IV: 5 μL CT: 40°C	ESI (+) and (-) MRM mode CBD **315.3 → 193.1** 315.3 → 259.2 315.3 → 135.0 CBDA **357.3 → 245.1** 357.3 → 339.2 357.3 → 226.9 CBN **311.2 → 223.1** 311.2 → 293.2 311.2 → 195.1 Δ^9-THC **315.3 → 193.1** 315.3 → 259.1 315.3 → 195.1 THCA-A **357.2 → 213.1** 357.2 → 245.0 357.2 → 191.0	13.00	hemp seeds hemp protein */150.0* hemp seed oil -/600.0 raw milk skimmed milk -/5.0	2020, Switzerland	[138]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
			CBC **315.2 → 193.0** 315.2 → 259.2 315.2 → 123.1 CBCA **357.2 → 191.0** 357.2 → 313.1 357.2 → 339.2 CBDV **287.2 → 165.1** 287.2 → 123.0 287.2 → 231.0 CBDVA **329.2 → 217.0** 329.2 → 283.1 329.2 → 311.1 CBG **317.2 → 193.0** 317.2 → 123.1 317.2 → 207.0 CBGA **359.2 → 341.1** 359.2 → 315.2 359.2 → 297.0 THCV **287.2 → 165.1** 287.2 → 135.1 287.2 → 123.0 THCVA **329.2 → 285.1** 329.2 → 217.0 329.2 → 163.1 Δ^8-THC **315.3 → 193.1** 315.3 → 259.1 315.3 → 123.0 THC-COOH-d_3 **346.2 → 302.2** 346.2 → 248.1 346.2 → 194.1				
spiked cannabis-infused chocolate / 0.5 g	Nexera Thermo trace 1310 UHPLC + Shimadzu 8060 QQQ	MPA: H_2O + 5 mM ammonium formate + 0.1% HCOOH MPB: ACN + 0.1% HCOOH isocratic, (25/75, *v*/*v*)	ESI (+) and (-) MRM mode CBD 315.3 → 193.0 315.3 → 123.1 CBN 311.3 → 223.3 311.3 → 293.3	10.00	-	2020, USA	[139]

Sample type/matrix/ amount (in grams or concentration)	Instrument type and column	LC conditions: mobile phases, MPGP (A%/B%/C%), FR, IV, CT	compounds MS data (quantification ion/MRM), WL	Runtime (min)	LOD/LOQ (ng/mL or ng/g), * - LOD/*LOQ* expressed in % (w/w)	Year, country	Reference
	Raptor ARC-18 (100 mm x 2.1 mm x 2.7 μm) + Raptor ARC-18 EXP guard column (5 mm x 2.1 mm x 2.7 μm)	FR: 0.4 mLmin^{-1} IV: 1 μL CT: 30°C	Δ^{9}-THC 315.3 → 193.0 315.3 → 123.1				
	Waters Acquity HPLC + PDA		CBD, CBN, CBG, CBDA, Δ^{9}-THC, THCA 228 nm		-		
cannabis plant material/ 2.0 g or 5.0 g		MPA: H_2O + 0.1% HCOOH MPB: ACN + 0.1% HCOOH 0 min (40/60), 11 min (20/80), 12.5 min (0/100) + re-equilibration 4.5 min FR: 0.3 mLmin^{-1} IV: 2 μL CT: 40°C	MRM mode ESI (+): CBD 315 → 259 315 → 193 CBN 311 → 293 311 → 233 Δ^{9}-THC 315 → 259 315 → 193 Δ^{8}-THC 315 → 259 315 → 193 CBD-d_3 318 → 262 318 → 196 CBN-d_3 314 → 296 314 → 223 Δ^{9}-THC-d_3 318 → 262 318 → 196 ESI (-): CBDA 357 → 339 357 → 311 THCA 357 → 339 357 → 245	17.00	CBDA 1.9/*5.9* CBD 6.4/*19.3* CBN 6.9/*20.9* Δ9-THC 8.5/*25.8* Δ8-THC 5.6/*17.1* THCA 5.7/*17.3*	2021, Czech Republic	[55]
fresh cannabis inflorescences / 0.05 g	Thermo Fischer UlitMate® 3000 + TSQ Quantum Access Max QQQ Luna Omega Polar C_{18} (100 mm x 2.1 mm x 1.6 μm)				-	2021, Czech Republic	[140]

Table S3. Vibrational spectroscopy-based analytical methods in conjunction with multivariate data analysis for phytocannabinoid profiling and/or classification of cannabis plant material. ATR-MIR – attenuated total reflection mid infrared spectroscopy; HCA - hierarchical cluster analysis; OPLS-DA, orthogonal projections to latent structures- discriminatory analysis; PCA – principal component analysis; PLS – partial least square; SEE – standard error of estimation; SVM-DA - support vector machine-discriminatory analysis.

Sample type	Vibrational spectroscopy technique	Quantified phytocannabinoids	Spectral region (cm^{-1})	Statistical model used	Number of main model components	Statistical accuracy descriptors					Reference analytical technique	Ref.
						Calibration set			Prediction set			
						R^2	SEE (%)	SEEcv (%)	R^2	SEE (%)		
dried cannabis leaves and inflorescences / 30.0 g	NIR (dispersive)	CBDV	2500 - 800	PLS	12	0.95	0.10	0.15	0.92	0.16	GC-FID	[141]
		Δ^9-THCV	2500 - 1100		12	0.92	0.02	0.02	0.87	0.03		
		CBD	2500 - 1100		10	0.99	0.35	0.42	0.98	0.58		
		CBC	2500 - 800		9	0.97	0.03	0.04	0.93	0.05		
		Δ^8-THC	2500 - 1100		9	0.97	0.03	0.03	0.85	0.07		
		Δ^9-THC	2500 - 1100		11	0.99	0.58	0.77	0.90	1.72		
		CBG	2500 - 800		8	0.94	0.25	0.28	0.54	0.79		
		CBN	2500 - 800		7	0.95	0.02	0.03	0.76	0.05		
	FT-NIR	CBDV	9403.7-8447.2; 6102-4242.9		11	0.89	0.13	0.17	0.93	0.21		
		Δ^9-THCV	9403.7-5446.3		10	0.89	0.02	0.03	0.86	0.04		
		CBD	7506-5446.3; 4428-4242.9		12	0.99	0.29	0.38	0.99	0.62		
		CBC	9403.7-4597.7		10	0.96	0.04	0.05	0.96	0.05		

Sample type	Vibrational spectroscopy technique	Quantified phytocannabinoids	Spectral region (cm^{-1})	Statistical model used	Number of main model components	Statistical accuracy descriptors					Reference analytical technique	Ref.
						Calibration set			Prediction set			
						R^2	SEE (%)	SEEcv (%)	R^2	SEE (%)		
		Δ^8-THC	9403.7-7498.3; 6102-4242.9		10	0.98	0.02	0.03	0.91	0.07		
		Δ^9-THC	9403.7-5446.3		12	0.99	0.49	0.62	0.95	1.79		
		CBG	7506-6796.3; 4428-4242.9		12	0.96	0.18	0.22	0.78	0.68		
		CBN	6102-5446.3; 4605.4-4242.9		10	0.96	0.02	0.02	0.83	0.06		
medium-chain triglyceride based formulations, PG-based formulations	FT NIR	CBD	9020-4000	PLS	4	0.99	0.43	-	0.99	0.37	HPLC-DAD	[142]
			9000-4000		6	0.99	0.30	-	0.99	0.32		
dried cannabis leaves, stems and inflorescences	NIR (integrating sphere)	-	4375-4000	PCA HCA PLS-DA SVM-DA	3	-	-	-	-	-	-	[143]
dried cannabis plant material	FT-NIR (dispersive)	-	6000-4000	PCA HCA	3	0.97	-	-	-	-	GC-FID	[144]
				SIMCA	3	-	-	-	-	-		

Sample type	Vibrational spectroscopy technique	Quantified phytocannabinoids	Spectral region (cm^{-1})	Statistical model used	Number of main model components	Statistical accuracy descriptors					Reference analytical technique	Ref.
						Calibration set			Prediction set			
						R^2	SEE (%)	SEEcv (%)	R^2	SEE (%)		
	NIR handheld			PCA HCA	3	0.99	-	-	-	-		
				SIMCA	2	-	-	-	-	-		
veterinary feed spiked with CBD, THC, CBG	NIR handheld	Δ⁹-THC	1700-900	PLS	3	-	0.002	0.006	-	0.009	GC-MS	[145]
		CBD			3	-	0.004	0.004	-	0.005		
		CBG			3	-	0.001	0.002	-	0.005		
seized cannabis inflorescneces entire	NIR handheld NIR-S-G1	Δ⁹-THC	950-1650	PLS and ensemble regression models	-	0.93	-	-	0.73	-	UHPLC-UV	[146]
ground						0.96	-	-	0.74	-		
sieved						0.98	-	-	0.93	-		
cannabis resins						0.72	-	-	0.02	-		
entire	NIR handheld MicroNIR		900-1700		-	0.98	-	-	0.93	-		
ground						0.94	-	-	0.76	-		
sieved						0.98	-	-	0.77	-		
cannabis resins						0.87	-	-	0.67	-		
dried cannabis inflorescences	ATR-MIR	Δ⁹-THCA	4000-400	PLS	-	0.95	-	-	-	0.86	HPLC-DAD	[147]
		Δ⁹-THC				0.95	-	-	-	0.13		
		CBDA				0.93	-	-	-	0.80		
		CBD				0.90	-	-	-	0.08		
		CBGA				0.90	-	-	-	0.12		
		CBG				0.78	-	-	-	0.02		
		THCVA				0.70	-	-	-	0.01		
decarboxylated cannabis flowers	ATR-MIR	Δ⁹-THC	1800-400	PLS	5	0.99	0.43	1.53	-	2.32	HPLC-UV	[148]
					5	0.99	0.21	1.41	-	1.31		
cannabis extracts		CBD	1800-400	PLS	3	0.95	4.67	5.25	-	3.79		
					3	0.99	1.21	2.62	-	1.44		
cannabis extracts	handheld Raman spectrometer	-	1700-701	OPLS-DA	1+2	-	-	-	-	-	CoA from the plant	[149,150]

Sample type	Vibrational spectroscopy technique	Quantified phytocannabinoids	Spectral region (cm^{-1})	Statistical model used	Number of main model components	Statistical accuracy descriptors					Reference analytical technique	Ref.
						Calibration set			Prediction set			
						R^2	SEE (%)	SEEcv (%)	R^2	SEE (%)		
											producer	

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