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Determination of cannabinoids in hemp flour, hemp oil cake, hemp tea, and hemp seed pesto Report on the 2025 Proficiency Test of the German Reference Laboratory for Mycotoxins and Plant Toxins

The German National Reference Laboratory (NRL) for Mycotoxins and Plant Toxins organised a proficiency test for the determination of cannabinoids in hemp flour, hemp oil cake, hemp tea, hemp seed pesto and a standard solution. A total of 19 laboratories participated and they could choose from the listed materials to analyse up to four materials plus a standard solution. The determination of CBD, Δ^9-THC and their acid forms was mandatory for the laboratories while the quantification of 15 minor cannabinoids was voluntary.

For the regulated cannabinoids Δ^9-THC and THCA, the reproducibility standard deviation RSD_R varied from 15 to 28 % and from 24 to 73 %, respectively, and for the remaining analytes from 13 to 127 %. Across all samples, analytes, and laboratories, on average, 91 % of *z*-scores fell within the satisfactory range ($|z| \leq 2$).

In an experiment separate from the proficiency test, the mutual interference caused by the close chromatographic appearance of Δ^9- and Δ^8-THC was investigated.

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1 Introduction

Since 1 January 2023, levels of Δ^9-THC in food containing hemp are regulated by Regulation (EU) 2023/915. The maximum levels are calculated as Δ^9-THC equivalents (Δ^9-THC + 0.877*THCA) and apply to hemp seeds, ground hemp seeds, (partially) defatted hemp seeds and other processed hemp seed products (3.0 mg kg^{-1}) as well as hemp seed oil (7.5 mg kg^{-1}) [1]. In addition, as part of their routine work, some official control laboratories examine cannabinoids in hemp-containing products and foodstuffs that are not covered by these maximum levels (e.g. hemp tea, gummies).

Biosynthetically, cannabinoids are generated as carboxylic acids (e.g. THCA). Spontaneous or thermal decarboxylation leads to the neutral, bioactive forms of cannabinoids (e.g. Δ^9-THC). Their analytical differentiation is essential for a precise risk assessment. In addition, there are so-called semi-synthetic cannabinoids, which are primarily relevant as artificial additives but may also occur naturally in trace amounts (e.g. Δ^8-THC usually less than 0.25 mg kg^{-1} when detectable) [2]. Gas chromatography (GC) used to be dominant in cannabinoid analysis, and is still widely used today, which necessitates derivatisation prior to analysis for acid-neutral differentiation due to the full thermal decarboxylation in high-temperature GC environments. Modern liquid chromatography methods are able to differentiate without derivatisation.

The analytical scope of this proficiency test covers 8 acidic and 11 neutral cannabinoids as shown in Table 1. In addition to the analytes, the sum parameter Δ^9-THC equivalents (see above) is evaluated.

Table 1: Analytical Scope of this proficiency test.

Acidic cannabinoids	Neutral cannabinoids
Cannabichromenic acid (CBCA)	Cannabichromene (CBC)
Cannabidiolic acid (CBDA)	Cannabidiol (CBD)
Cannabidivarinic acid (CBDVA)	Cannabidivarin (CBDV)
Cannabigerolic acid (CBGA)	Cannabigerol (CBG)
Cannabicyclolic acid (CBLA)	Cannabicyclol (CBL)
Cannabinolic acid (CBNA)	Cannabinol (CBN)
Tetrahydrocannabinolic acid (THCA)	Δ^9-Tetrahydrocannabinol (Δ^9-THC)
Tetrahydrocannabivarinic acid (THCVA)	Tetrahydrocannabidivarin (THCV)
	Δ^8-Tetrahydrocannabinol (Δ^8-THC)*
	9(*R*)-Hexahydrocannabinol (9(*R*)-HHC)*
	9(*S*)-Hexahydrocannabinol (9(*S*)-HHC)*

* When Δ^8-THC and 9(*R*)- and 9(*S*)-HHC are detected in food or edible samples, they typically occur at high concentrations, presumably reflecting their marketing toward consumers seeking a psychoactive effect. However, these compounds have also been identified in low concentrations in natural hemp plant material, complicating a strict classification into natural versus non-natural cannabinoids. The distinction thus lies beyond the scope of the present report.

2 Proficiency test results

The statistical evaluation of the results was performed as described in Section 4. Laboratory results and statistical characteristics for each test material are given in Table 2 to Table 6. The z-scores for each analyte and each laboratory per test material are visualised in Figure 1 to Figure 5.

2.1 Overview of test materials

2.1.1 Hemp flour (sample 1)

Sample 1 is a hemp flour that naturally contains cannabinoids to which no further cannabinoids have been added. According to the manufacturer, the flour was produced from oilseed cake obtained during the oil-pressing process and subsequently ground. For Δ^9-THC and THCA, x_{av} were 0.8 and 0.7 mg kg^{-1}. The success rate (participants with $|z| \leq 2$) ranged from 71 to 100 %, with 95 % for Δ^9-THC and 89 % for THCA.

Table 2: Analytical results and statistical characteristics for all analytes in hemp flour (sample 1). Mandatory analytes are highlighted. Laboratory and assigned values (x_{av}) are given in mg kg^{-1}. Values are rounded to one decimal place; concentrations <0.05 mg kg^{-1} are displayed as 0.0.

Lab	CBC	CBCA	CBD	CBDA*	CBDV	CBDVA	CBG	CBGA	CBL	CBLA	CBN*	CBNA	Δ^9-THC	THCA*	Δ^9-THC eq.	Δ^8-THC	THCV	TCHVA	9(*R*)-HHC	9(*S*)-HHC
L01	0.3	0.3	6.0	15.4	0.4	1.3	0.2	0.8	-	1.2	0.1	0.4	0.9	0.7	1.5	-	0.1	0.1	-	-
L03	-	-	8.0	21.4	-	-	-	-	-	-	-	-	0.9	1.3	2.0	0.0	-	-	-	-
L04	-	-	6.4	14.0	-	-	-	-	-	-	0.6	-	0.9	0.9	1.7	-	-	-	-	-
L05	-	-	6.3	11.4	0.7	-	-	-	-	-	0.1	-	0.7	0.5	1.1	-	-	-	-	-
L06	-	-	5.5	13.0	-	-	-	-	-	-	0.2	-	0.8	0.6	1.3	-	-	-	-	-
L07	-	-	3.7	12.4	-	-	-	-	-	-	0.1	-	0.5	11.8	10.9	0.1	-	-	-	-
L08	-	-	6.4	16.0	-	-	-	-	-	-	0.2	-	0.7	0.7	1.3	-	-	-	-	-
L09	0.5	0.6	5.6	15.6	0.4	-	-	-	-	-	0.5	-	0.8	0.9	1.6	-	-	-	-	-
L10	-	-	5.4	7.3	-	-	-	-	-	-	-	-	0.5	-	0.5	-	-	-	-	-
L11	-	-	4.5	12.7	0.4	-	-	-	-	-	0.2	-	1.0	0.7	1.6	-	-	-	-	-
L12	-	-	6.9	18.1	-	-	-	-	-	-	0.2	-	0.8	0.7	1.4	-	-	-	-	-
L13	0.4	0.7	7.2	18.8	-	-	0.3	-	-	-	-	0.2	0.6	0.5	1.1	-	-	-	-	-
L14	-	-	6.0	16.8	-	-	-	-	-	-	-	-	0.7	0.7	1.3	-	-	-	-	-
L15	0.2	-	5.1	12.3	0.5	1.5	0.2	0.8	0.1	0.4	0.1	0.3	0.7	0.8	1.3	-	0.1	0.1	-	-
L16	-	-	6.1	17.7	-	-	-	-	-	-	-	-	0.7	0.5	1.2	-	-	-	-	-
L17	-	-	5.7	10.4	0.4	-	0.2	-	-	-	0.1	-	0.8	0.4	1.2	-	0.1	-	-	-
L19	-	-	7.2	21.8	-	-	-	-	-	-	0.1	-	0.7	0.8	1.5	-	-	-	-	-
L21	-	-	7.5	19.6	0.5	-	-	-	-	-	0.4	-	1.2	0.7	1.8	-	-	-	-	-
L22	0.3	0.5	5.1	13.2	0.4	1.0	0.2	0.6	0.1	0.2	0.1	0.2	0.8	0.6	1.3	-	0.1	0.1	-	-
n	5	4	19	19	8	3	5	3	2	3	14	4	19	18	19	2	4	3	0	0
x_{av}	0.3	0.5	6.0	15.2	0.4	1.3	0.2	0.8	0.1	0.6	0.2	0.3	0.8	0.7	1.4	0.1	0.1	0.1	-	-
RSD_R	-	-	17.6	26.9	13.5	-	-	-	-	-	52.3	-	19.5	28.1	22.9	-	-	-	-	-
%	-	-	100	100	88	-	-	-	-	-	71	-	95	89	90	-	-	-	-	-

z'-scores were calculated for CBDA, CBN, and THCA due to a non-negligible uncertainty of the assigned value u_{av} ($0.3 \cdot \sigma_{pt} \leq u_{av} \leq 0.7 \cdot \sigma_{pt}$). They are marked with *.

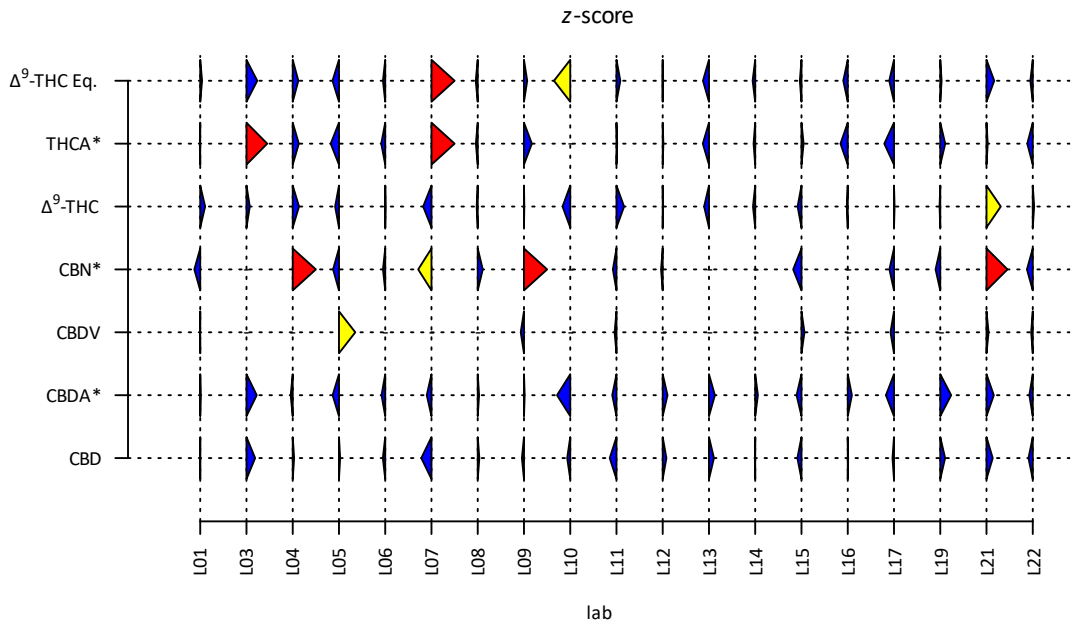

Figure 1: Overview of z-scores for hemp flour (sample 1). The target relative standard deviation is 25 % and corresponds to $|z| = 1$. Size of triangles shows the magnitude of z, their colour the interpretation. Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$). Analytes with z'-scores are marked with *.

2.1.2 Hemp oil cake (sample 2)

Sample 2 is a hemp oil cake that naturally contains cannabinoids to which no further cannabinoids have been added. For Δ^9-THC and THCA, x_{av} were 0.2 and 0.3 mg kg^{-1}. The success rate (participants with $|z| \leq 2$) ranged from 75 to 94 %, with 87 % for Δ^9-THC.

Table 3: Analytical results and statistical characteristics for hemp oil cake (sample 2). Mandatory analytes are highlighted. Laboratory and assigned values (x_{av}) are given in mg kg^{-1}. Values are rounded to one decimal place; concentrations <0.05 mg kg^{-1} are displayed as 0.0.

Lab	CBC	CBCA	CBD	CBDA*	CBDV*	CBDVA	CBG	CBGA	CBL	CBLA	CBN*	CBNA	Δ^9-THC*	THCA	Δ^9-THC eq.	Δ^8-THC	THCV	TCHVA	9(*R*)-HHC	9(*S*)-HHC
L01	0.2	0.1	2.7	4.4	0.2	0.3	0.0	0.1	-	0.4	0.2	-	0.3	0.1	0.3	-	0.0	-	-	-
L03	-	-	4.0	5.7	-	-	-	-	-	-	-	-	0.3	0.2	0.5	0.0	-	-	-	-
L04	-	-	3.4	5.1	-	-	-	-	-	-	0.8	-	0.3	0.3	0.5	-	-	-	-	-
L05	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L06	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L07	-	-	1.8	3.7	-	-	-	-	-	-	0.1	-	0.1	3.3	3.0	0.1	-	-	-	-
L08	-	-	3.2	5.1	-	-	-	-	-	-	0.4	-	0.2	0.1	0.3	-	-	-	-	-
L09	0.4	-	2.7	6.3	0.2	-	-	-	-	-	0.5	-	0.3	0.5	0.7	-	-	-	-	-
L10	-	-	3.0	3.4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L11	-	-	3.4	3.1	0.5	-	-	-	-	-	0.2	-	0.3	0.1	0.3	-	-	-	-	-
L12	-	-	3.4	5.2	-	-	-	-	-	-	0.3	-	0.2	-	0.2	-	-	-	-	-
L13	0.3	0.2	3.5	4.4	-	-	-	-	-	-	-	0.1	0.2	-	0.2	-	-	-	-	-

Lab	CBC	CBCA	CBD	CBDA*	CBDV*	CBDVA	CBG	CBGA	CBL	CBLA	CBN*	CBNA	Δ⁹-THC*	THCA	Δ⁹-THC eq.	Δ⁸-THC	THCV	TCHVA	9(*R*)-HHC	9(*S*)-HHC
L14	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L15	0.1	-	2.5	3.4	0.3	0.5	0.0	0.1	0.1	0.4	0.2	0.2	0.2	0.4	0.5	-	0.0	0.0	-	-
L16	-	-	15.8	48.2	-	-	-	-	-	-	-	-	0.6	0.4	1.0	-	-	-	-	-
L17	-	-	3.0	3.6	0.2	-	0.0	-	-	-	0.2	-	0.2	0.1	0.3	-	0.0	-	-	-
L19	-	-	3.9	5.6	-	-	-	-	-	-	0.2	-	0.2	0.1	0.3	-	-	-	-	-
L21	-	-	4.0	5.7	0.4	-	-	-	-	-	0.4	-	0.4	0.5	0.8	-	-	-	-	-
L22	0.2	0.2	2.7	3.8	0.2	0.3	0.0	0.1	0.1	0.1	0.2	0.1	0.2	-	0.2	-	0.0	0.0	-	-
n	5	3	16	16	7	3	4	3	2	3	12	3	15	12	15	2	4	2	0	0
x_{av}	0.3	0.2	3.2	4.7	0.3	0.4	0.0	0.1	0.1	0.3	0.3	0.1	0.2	0.3	0.5	0.0	0.0	0.0	-	-
RSD_R [%]	-	-	20.8	26.9	32.0	-	-	-	-	-	47.5	-	27.1	73.4	61.5	-	-	-	-	-
% $\|z\| \leq 2$	-	-	94	94	86	-	-	-	-	-	75	-	87	-	-	-	-	-	-	-

z'-scores were calculated for CBDA, CBDV, CBN, and Δ⁹-THC due to a non-negligible uncertainty of the assigned value u_{av} ($0.3 \cdot \sigma_{pt} \leq u_{av} \leq 0.7 \cdot \sigma_{pt}$). They are marked with *. THCA and Δ⁹-THC eq. were excluded from z-score calculation due to uncertainty of the assigned value ($x_{av} > 0.7 \cdot \sigma_{pt}$).

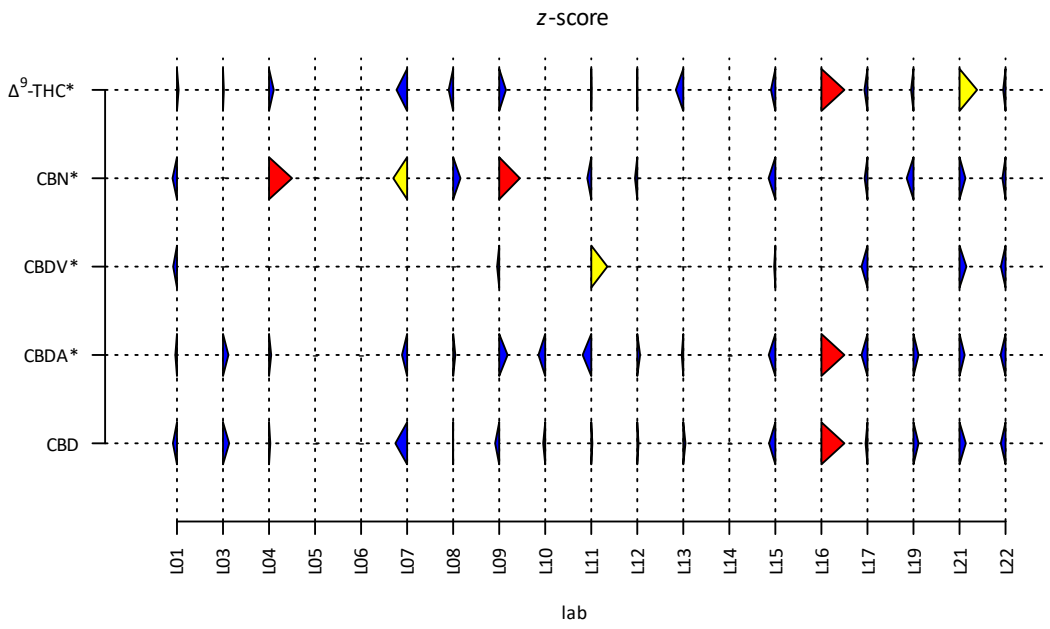

Figure 2: Overview of z-scores for hemp oil cake (sample 2). The target relative standard deviation is 25 % and corresponds to $|z| = 1$. Size of triangles shows the magnitude of z, their colour the interpretation.
Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$). Analytes with z'-scores are marked with *.

2.1.3 Hemp tea (sample 3)

Sample 3 is a hemp tea that naturally contains cannabinoids to which no further cannabinoids have been added. For Δ⁹-THC and THCA, x_{av} were 60.9 and 17.4 mg kg^{-1}. The success rate (participants with $|z| \leq 2$) ranged from 75 to 100 %, with 93 % for Δ⁹-THC and 86 % for THCA.

Table 4: Analytical results and statistical characteristics for hemp tea (sample 3). Mandatory analytes are highlighted. Laboratory and assigned values (x_{av}) are given in mg kg^{-1}. Values are rounded to zero decimal places; concentrations <0.5 mg kg^{-1} are displayed as 0.

Lab	CBC	CBCA	CBD	CBDA	CBDV*	CBDVA	CBG	CBGA	CBL	CBLA	CBN*	CBNA	Δ^9-THC*	THCA*	Δ^9-THC eq.	Δ^8-THC	THCV	TCHVA	9(*R*)-HHC	9(*S*)-HHC
L01	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L03	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L04	-	-	5,259	3,694	-	-	-	-	-	-	78	-	121	64	177	-	-	-	-	-
L05	-	-	4,515	2,043	34	-	-	-	-	-	53	-	56	15	69	-	-	-	-	-
L06	-	-	4,900	2,500	0	-	-	-	-	-	54	-	48	16	62	3	0	-	-	-
L07	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L08	-	-	4,810	2,537	-	-	-	-	-	-	82	-	60	16	74	-	-	-	-	-
L09	312	190	3,670	1,740	27	-	159	-	-	-	78	-	69	20	87	-	-	-	-	-
L10	-	-	4,492	2,210	-	-	-	-	-	-	-	-	34	9	42	-	-	-	-	-
L11	-	-	4,802	2,605	36	-	-	-	-	-	60	-	72	15	85	-	-	-	-	-
L12	-	-	4,396	2,114	-	-	-	-	-	-	55	-	62	16	76	-	-	-	-	-
L13	388	145	3,917	2,006	-	-	163	-	-	-	62	6	59	15	72	-	-	0	-	-
L14	-	-	4,986	2,298	-	-	-	-	-	-	57	-	41	31	68	2	-	-	-	-
L15	211	-	3,862	1,693	35	27	105	79	4	369	43	7	49	11	58	-	1	1	-	-
L16	-	-	4,140	2,360	-	-	-	-	-	-	-	-	81	9	89	-	-	-	-	-
L17	-	-	4,639	2,180	67	-	180	-	-	-	57	-	61	17	76	-	1	-	-	-
L19	-	-	-	-	-	-	-	-	-	-	45	-	-	-	-	-	-	-	-	-
L21	-	-	4,780	2,848	35	-	-	-	-	-	62	-	76	27	100	10	-	-	-	-
L22	301	119	3,750	2,050	34	21	122	92	17	8	49	5	58	-	58	-	-	-	-	-
n	4	3	15	15	8	2	5	2	2	2	14	3	15	14	15	3	3	2	0	0
x_{av}	303	151	4,461	2,271	34	24	146	86	10	188	60	6	61	17	75	4	1	0	-	-
RSD$_R$ [%]	-	-	12.5	17.7	22.6	-	-	-	-	-	22.6	-	25.7	43.4	22.9	-	-	-	-	-
% $\lvert z \rvert \leq$	-	-	100	93	75	-	-	-	-	-	100	-	93	86	93	-	-	-	-	-

z'-scores were calculated for CBDV, CBN, Δ^9-THC, and THCA due to a non-negligible uncertainty of the assigned value u_{av} ($0.3 \cdot \sigma_{pt} \leq u_{av} \leq 0.7 \cdot \sigma_{pt}$). They are marked with *.

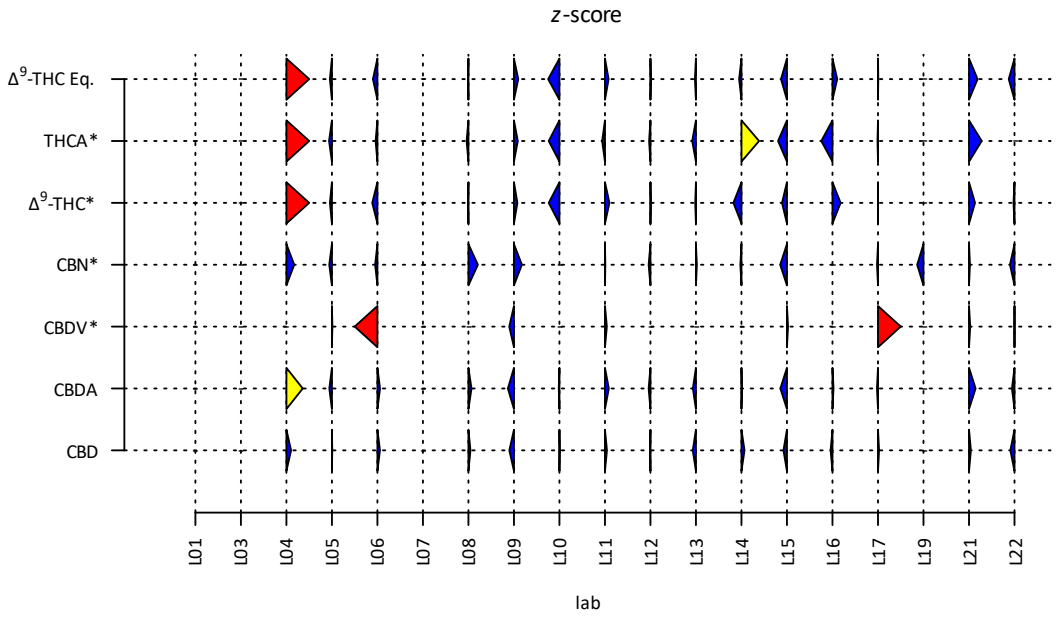

Figure 3: Overview of z-scores for hemp tea (sample 3). The target relative standard deviation is 25 % and corresponds to $|z| = 1$. Size of triangles shows the magnitude of z, their colour the interpretation. Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$). Analytes with z'-scores are marked with *.

2.1.4 Hemp pesto (sample 4)

Sample 4 is a hemp pesto that naturally contains cannabinoids to which no further cannabinoids have been added. For Δ^9-THC and THCA, x_{av} were 0.4 and 0.3 mg kg^{-1}. The success rate (participants with $|z| \leq 2$) ranged from 82 to 93 %, with 83 % for Δ^9-THC and 82 % for THCA.

Table 5: Analytical results and statistical characteristics for hemp pesto (sample 4). Mandatory analytes are highlighted. Laboratory and assigned values (x_{av}) are given in mg kg^{-1}. Values are rounded to one decimal place; concentrations <0.05 mg kg^{-1} are displayed as 0.0.

Lab	CBC	CBCA	CBD	CBDA*	CBDV	CBDVA	CBG	CBGA	CBL	CBLA	CBN	CBNA	Δ9-THC*	THCA*	Δ9-THC eq.*	Δ8-THC	THCV	TCHVA	9(R)-HHC	9(S)-HHC
L01	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L03	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L04	-	-	8.4	23.5	-	-	-	-	-	-	0.6	-	0.6	0.8	1.2	-	-	-	-	-
L05	-	-	6.9	12.1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L06	-	-	7.3	16.0	-	-	-	-	-	-	-	-	0.4	0.4	0.7	-	-	-	-	-
L07	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L08	-	-	7.5	15.0	-	-	-	-	-	-	-	-	0.4	0.3	0.7	-	-	-	-	-
L09	1.5	0.8	8.0	16.2	0.3	-	0.1	-	-	-	0.1	-	0.4	0.3	0.6	-	-	-	-	-
L10	-	-	7.6	14.5	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L11	-	-	5.7	10.3	-	-	-	-	-	-	-	-	0.5	0.3	0.7	-	-	-	-	-
L12	-	-	9.1	14.2	-	-	-	-	-	-	0.1	-	0.4	-	0.4	-	-	-	-	-

Lab	CBC	CBCA	CBD	CBDA*	CBDV	CBDVA	CBG	CBGA	CBL	CBLA	CBN	CBNA	Δ⁹-THC*	THCA*	Δ⁹-THC eq.*	Δ⁸-THC	THCV	TCHVA	9(*R*)-HHC	9(*S*)-HHC
L13	0.5	0.7	8.0	14.4	-	-	0.1	-	-	-	-	-	0.3	0.2	0.4	-	-	-	-	-
L14	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L15	0.1	-	0.7	0.9	0.0	0.1	-	0.0	-	-	-	-	0.1	0.1	0.1	-	-	-	-	-
L16	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L17	-	-	8.4	14.6	0.3	-	0.2	-	-	-	0.1	-	0.4	0.4	0.7	-	-	-	-	-
L19	-	-	8.7	9.2	-	-	-	-	-	-	0.1	-	0.4	0.2	0.6	-	-	-	-	-
L21	-	-	8.9	13.1	0.4	-	-	-	-	-	0.6	-	0.8	0.4	1.1	-	-	-	-	-
L22	0.5	0.6	5.7	9.4	0.2	0.4	0.1	0.3	0.1	0.2	0.1	0.1	0.4	0.3	0.6	-	0.0	-	-	-
n	4	3	14	14	5	2	4	2	1	1	7	1	12	11	12	0	1	0	0	0
x_{av}	0.6	0.7	7.5	13.3	0.2	0.2	0.1	0.2	0.1	0.2	0.2	0.1	0.4	0.3	0.7	-	0.0	-	-	-
RSD$_R$ [%]	-	-	18.0	25.9	-	-	-	-	-	-	127	-	28.4	34.8	40.7	-	-	-	-	-
% $\|z\| \leq 2$	-	-	93	86	-	-	-	-	-	-		-	83	82	73	-	-	-	-	-

z'-scores were calculated for CBDA, Δ⁹-THC, THCA, and Δ⁹-THC eq. due to a non-negligible uncertainty of the assigned value u_{av} ($0.3 \cdot \sigma_{pt} \leq u_{av} \leq 0.7 \cdot \sigma_{pt}$). They are marked with *.

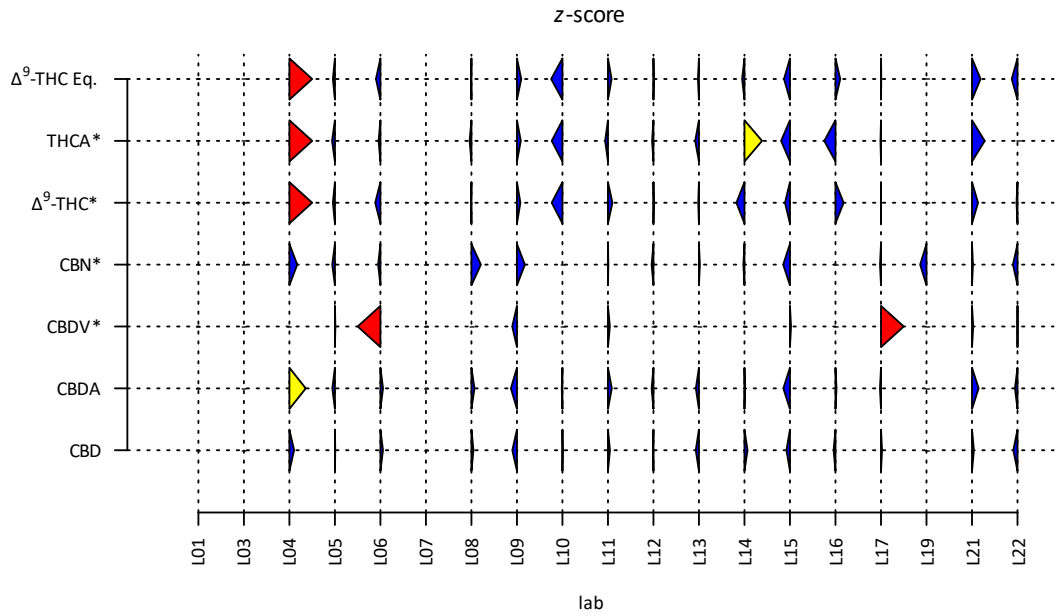

Figure 4: Overview of z-scores for hemp pesto (sample 4). The target relative standard deviation is 25 % and corresponds to $|z| = 1$. Size of triangles shows the magnitude of z, their colour the interpretation. Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$). Analytes with z'-scores are marked with *.

2.1.5 Standard solution (sample 5)

Sample 5 is a cannabinoid standard solution that was prepared at the NRL. For Δ⁹-THC and THCA, x_{av} were 6.1 and 12.1 ng mL^{-1}. The success rate (participants with $|z| \leq 2$) ranged from 87 to 100 %, with 93 % for Δ⁹-THC and 88 % for THCA.

Comparing the target value x_{target}, expected from the preparation scheme with the x_{av} shows that they correspond closely (Table 6). The exception is Δ⁸-THC, which has a recovery

rate of 145 %. Since the theoretical target value of 4.8 ng mL^{-1} is outside the 95 % confidence intervals of the assigned value of 7.0 ng mL^{-1}[†] this observation indicates a statistically significant systematic overestimation of Δ^8-THC across the participating laboratories. As the standard solution not only contained Δ^8-THC but also its isomer Δ^9-THC, the overestimation may be due to insufficient chromatographic separation of both isomers. Further discussion can be found in Section 2.3.

Table 6: Analytical results and statistical characteristics for the cannabinoid standard solution (sample 5). Mandatory analytes are highlighted. Laboratory and assigned values (x_{av}) are given in mg kg^{-1}. Values are rounded to one decimal place; concentrations <0.05 mg kg^{-1} are displayed as 0.0.

Lab	CBC	CBCA	CBD	CBDA	CBDV	CBDVA	CBG	CBGA	CBL	CBLA	CBN	CBNA	Δ^9-THC	THCA	Δ^9-THC eq.	Δ^8-THC	THCV	TCHVA	9(*R*)-HHC	9(S)-HHC
L01	9.8	15.7	30.9	40.6	9.4	11.5	12.9	17.9	14.0	21.2	12.0	22.6	7.0	12.0	17.5	5.9	4.5	8.7	14.7	14.6
L03	-	-	37.6	38.9	-	-	-	-	-	-	-	-	6.2	17.2	21.3	9.5	-	-	-	-
L04	-	-	33.0	45.2	-	-	-	-	-	-	12.4	-	7.9	21.3	26.6	-	-	-	-	-
L05	-	-	24.1	34.5	10.2	-	-	-	-	-	12.5	-	4.9	8.5	12.4	4.9	4.6	-	11.0	-
L06	-	-	32.2	46.3	5.2	-	-	-	-	-	14.1	-	5.8	11.5	15.9	5.6	5.3	-	-	-
L07	-	-	23.6	29.1	-	-	-	-	-	-	7.1	-	6.0	29.3	31.7	6.9	-	-	-	-
L08	-	-	32.0	45.0	-	-	-	-	-	-	13.0	-	5.5	12.0	16.0	-	-	-	-	-
L09	12.2	-	29.4	65.7	13.8	-	16.8	-	-	-	17.5	-	-	-	-	8.5	-	-	8.1	12.2
L10	3.7	-	31.2	39.9	8.1	12.2	9.8	14.8	-	-	9.4	-	3.9	10.9	13.5	-	3.4	-	-	-
L11	-	-	28.0	30.6	8.3	-	11.1	-	-	-	10.0	-	5.2	12.8	16.4	4.0	3.5	-	-	-
L12	-	-	36.8	39.5	-	-	-	-	-	-	14.0	-	6.2	12.0	16.7	-	-	-	-	-
L13	10.2	15.4	36.7	45.2	-	-	15.7	17.7	-	-	14.0	19.8	6.2	12.2	16.9	8.5	4.9	9.1	12.2	11.1
L14	-	-	32.0	42.0	-	-	-	-	-	-	17.0	-	-	12.0	10.5	-	-	-	-	-
L15	7.8	-	29.4	29.6	10.6	13.5	12.2	13.2	10.5	29.5	12.8	16.3	6.2	7.6	12.8	-	5.4	7.2	-	-
L16	-	-	32.9	40.9	-	-	-	-	-	-	-	-	9.8	9.4	18.0	-	-	-	-	-
L17	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L19	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L21	-	-	33.0	44.5	11.4	-	-	-	-	-	13.8	-	6.1	11.9	16.5	13.3	-	-	-	-
L22	9.0	16.2	27.8	39.1	9.8	11.9	13.6	15.6	14.6	17.2	11.6	19.9	6.1	11.6	16.2	5.1	4.1	7.7	12.1	12.5
n	6	3	17	17	9	4	7	5	3	3	15	4	15	16	16	10	8	4	5	4
x_{av}	8.9	15.8	31.4	40.1	9.7	12.3	13.1	15.9	13.0	22.6	12.8	19.7	6.1	12.1	16.6	7.0	4.5	8.2	11.6	12.6
RSD$_R$ [%]	-	-	13.3	16.9	22.7	-	21.3	-	-	-	20.1	-	15.0	24.3	22.5	36.6	19.3	-	-	-
% $\lvert z \rvert \leq 2$	-	-	100	94	100	-	100	-	-	-	100	-	93	88	88	90	100	-	-	-
x_{target}	8	16	35	40	10	12	12	16	14	16	12	20	6.1	12	15.3	4.8	4	8	12	12
x_{av}/x_{target}	1.11	0.99	0.90	1.00	0.97	1.02	1.10	0.99	0.93	1.41	1.07	0.98	1.00	1.01	1.11	1.45	1.12	1.02	0.97	1.05

z'-scores were calculated for CBDV, CBG, Δ^8-THC, THCA, and THCV due to a non-negligible uncertainty of the assigned value u_{av} ($0.3 \cdot \sigma_{pt} \leq u_{av} \leq 0.7 \cdot \sigma_{pt}$). They are marked with *.

[†] The confidence interval for Δ^8-THC is [6.08; 7.84] ng mL^{-1}. The assigned value and relative standard deviation (RSD$_R$) were determined according to Section 4.2. The 95 % confidence interval was calculated using the standard error $\left(\mathrm{SE} = s/\sqrt{n}\right)$ multiplied by the critical t-value $\left(t_{n-1;\alpha=0.05}\right)$ based on the number of participating laboratories (n).

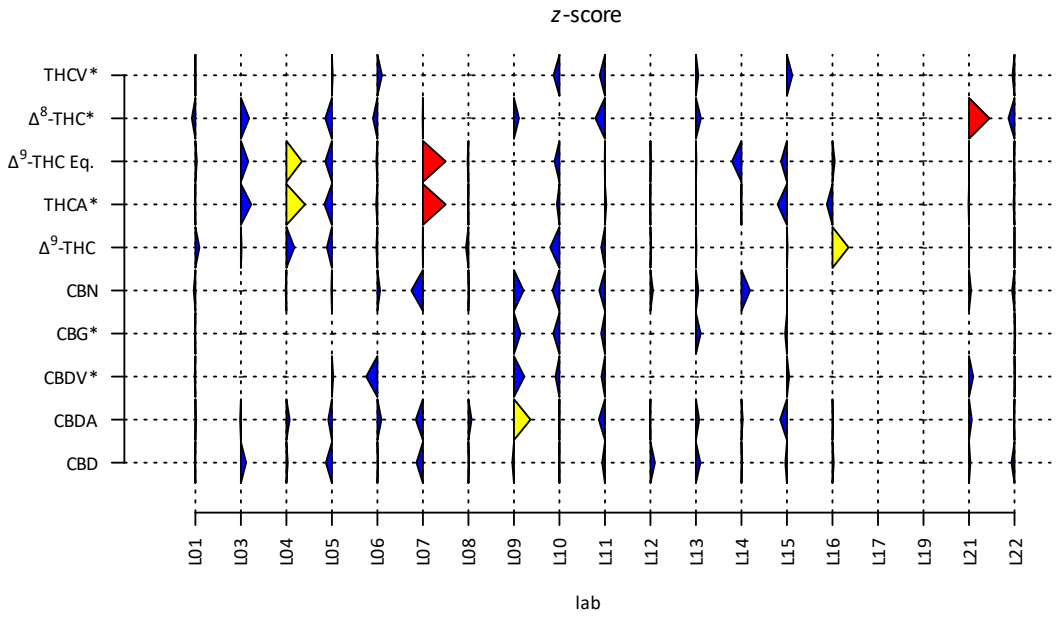

Figure 5: Overview of z-scores for the standard solution (sample 5). The target relative standard deviation is 25 % and corresponds to $|z| = 1$. Size of triangles shows the magnitude of z, their colour the interpretation. Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$). Analytes with z'-scores are marked with *.

2.2 Evaluation of the laboratory performance in terms of bias, precision and absolute mean z-score

One of the main objectives of proficiency testing is to evaluate the performance of laboratories and the methods they use, often referred to as fit-for-purpose testing. Proficiency testing results provide an estimate of the laboratory or method bias (also known as systematic error), as the assigned value for an analyte in a test material serves as a reliable estimate of the true value. This is similar to estimating the bias using a reference material.

The laboratory's bias was calculated as the mean deviation of all tested analyte-matrix combination from the respective assigned values (see Section 4.3.2). The mean bias of the laboratories across the samples is shown in Figure 6. The figures in Appendix B – Visual summary per participant) allow a laboratory to evaluate the influence of individual analytes on its overall bias, revealing specific contributions to under- or overestimations. Consistent deviations across analytes indicate a systematic error. In addition, Figure 6 illustrates the dispersion of bias results for all analyte-matrix combinations around their mean bias, which can serve as a measure of precision within this proficiency test.

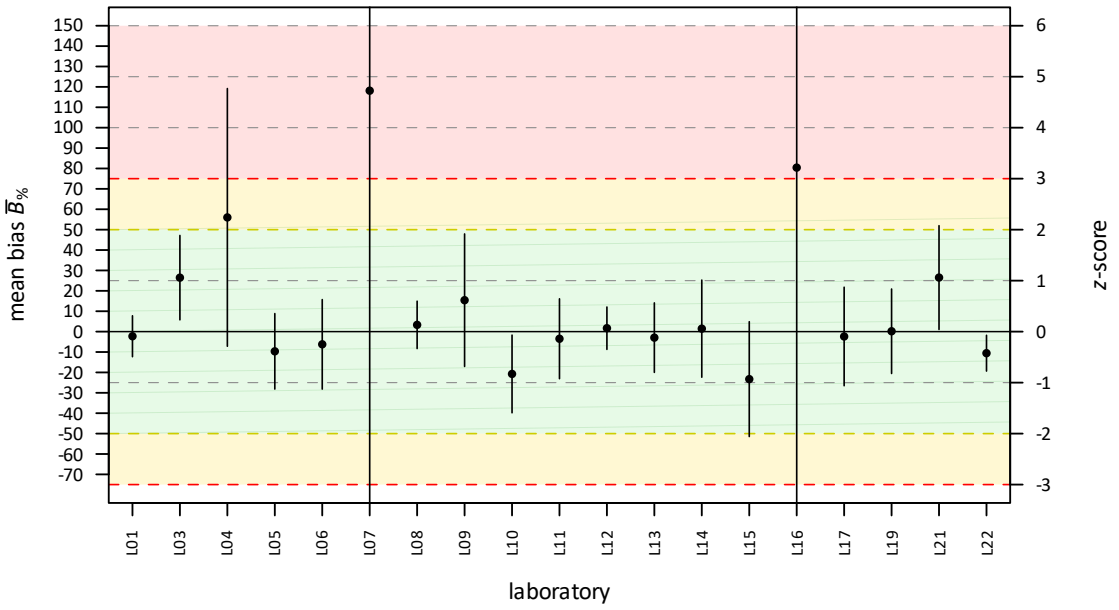

Figure 6: Laboratory performance across all matrix samples (i.e. excluding the standard solution) in terms of mean bias (black dots) and precision. The mean bias $\bar{\boldsymbol{B}}_{\%}$ is calculated as the mean deviation from the assigned values $\boldsymbol{x}_{\text{av}}$ (see Equation 8) across all analyte-matrix combinations. The distance from zero shows the magnitude and direction of systematic deviation. The mean bias (left $\boldsymbol{y}$-axis) is directly proportional to the laboratory's mean $\boldsymbol{z}$-score (right $\boldsymbol{y}$-axis). For example, since the target standard deviation of 25 % corresponds to $\boldsymbol{z} = \mathbf{1.0}$, a deviation of 12.5 % corresponds to $\boldsymbol{z} = \mathbf{0.5}$. The vertical lines represent the standard deviation around the mean bias and can serve as a measure of precision within this proficiency test (see Appendix B – Visual summary per participant). Standard deviations for L07 and L16 are truncated.

According to Regulation (EU) 2023/915, a laboratory can report a default expanded measurement uncertainty of 50 % if it meets the specific requirements for intra-laboratory precision and successfully participates in proficiency tests. A mean absolute z-score ≤ 2 has to be achieved, demonstrating that the required precision under reproducibility conditions (RSD_R) is met. Table 7 shows the mean absolute z-scores obtained by the laboratories across samples 1–5.

Table 7: Absolute mean $\boldsymbol{z}$-scores for all participants.

Lab code	$\lvert\bar{z}\rvert$ for all analytes	$\lvert\bar{z}\rvert$ for mandatory analytes*	$\lvert\bar{z}\rvert$ for regulated analytes#
L01	0.29	0.30	0.44
L03	1.08	0.97	1.03
L04	2.27	1.76	2.91
L05	0.63	0.58	0.73
L06	0.57	0.41	0.39
L07	6.58	6.36	12.62
L08	0.34	0.29	0.32
L09	0.98	0.71	0.62
L10	0.87	0.73	1.47

Lab code	$\lvert\bar{z}\rvert$ for all analytes	$\lvert\bar{z}\rvert$ for mandatory analytes*	$\lvert\bar{z}\rvert$ for regulated analytes#
L11	0.62	0.65	0.58
L12	0.28	0.28	0.09
L13	0.56	0.62	0.77
L14	0.59	0.58	1.14
L15	1.06	1.29	1.23
L16	3.75	4.63	2.12
L17	0.59	0.44	0.41
L19	0.69	0.72	0.44
L21	1.07	1.17	1.76
L22	0.45	0.51	0.37

* includes CBD, CBDA, Δ^9-THC, and THCA
# includes Δ^9-THC and THCA

2.3 Issues regarding chromatographic separation of Δ^9- and Δ^8-THC

When evaluating the standard solution (sample 5), the assigned values agreed with the known target concentrations (recovery 90–111 %) for most analytes, with the exception of Δ^8-THC (145 %). The Δ^8-THC assigned value of 7.0 ng mL^{-1} exceeds the target concentration of 4.8 ng mL^{-1} by 45 %, and this discrepancy is statistically significant: the target value falls outside the 95 % confidence interval of the assigned value.[‡] Because Δ^9- and Δ^8-THC are structural isomers that are generally acquired on the same MRM transition (*m/z* 315.2 > 193.1) and typically elute in close succession, it was hypothesised that insufficient chromatographic resolution leads to systematic signal interference between the two peaks, inflating the apparent Δ^8-THC area (or to be precise: the area of the later eluting peak). Experiments were conducted to investigate whether these potential effects could account for the inflated assigned value and, more broadly, the impact of close elution of Δ^9- and Δ^8-THC on their quantification in hemp matrix samples.

The chromatographic resolution achieved for Δ^9- and Δ^8-THC in the method used for the present investigation was R_{FWHM} = 1.62 (Figure 7), which nominally meets the conventional criterion for baseline separation ($R \geq 1.5$) and is not untypical for LC-MS/MS methods applied to this isomer pair. However, R_{FWHM} is based on peak widths at half height and is consequently insensitive to the tailing region below approximately 5 % of the peak maximum. The classical resolution R_s, defined using peak widths at baseline, is lower by a factor of ~0.59 for Gaussian peaks, yielding R_s = 0.95 from the actual peak shapes analysed here. At R_s < 1.0, baseline separation in the strict sense is not achieved and peak overlap (tailing) is visible in the chromatogram (Figure 7a). Simulated chromatograms of the pair using the same peak shapes at different concentration ratios and resolutions are shown in Figure 16 and Figure 17 (Appendix D – Chromatography of Δ^9- and Δ^8-THC).

[‡] Confidence intervals: Δ^9-THC: [5.60; 6.52] ng mL^{-1}; Δ^8-THC: [6.08; 7.84] ng mL^{-1}. The assigned values were determined after outlier elimination according to Section 4.2. The 95 % confidence interval was calculated using the standard error $\left(\mathrm{SE} = s/\sqrt{n}\right)$ multiplied by the critical t-value $\left(t_{n-1;\alpha=0.05}\right)$ based on the number of participating laboratories (n).

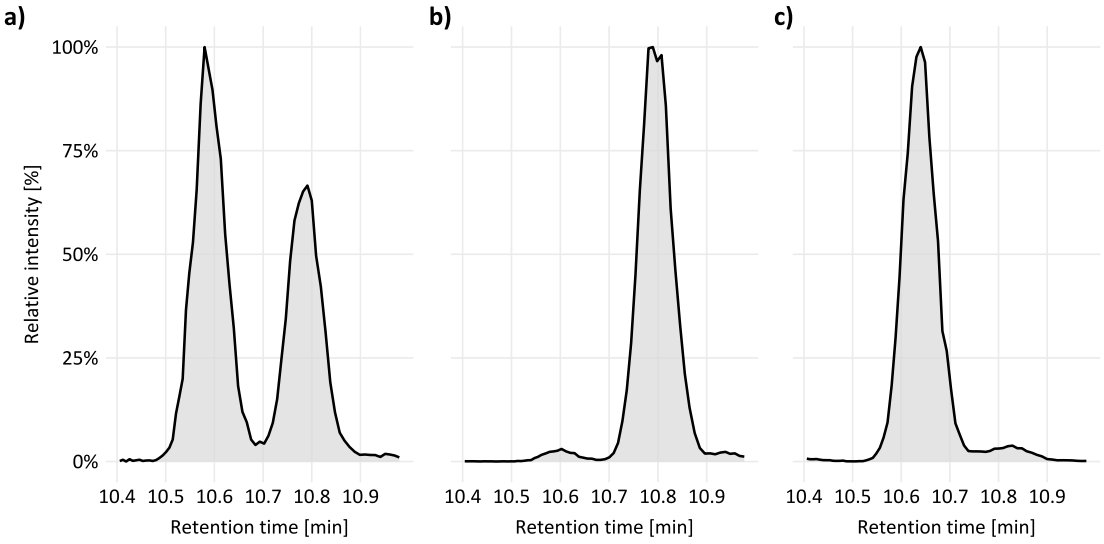

Figure 7: Excerpts of unsmoothed extracted ion chromatograms (LC-MS/MS, m/z 315.2 > 193.1).
Peak 1 (t_R = 10.58 min) corresponds to Δ^9-THC, Peak 2 (t_R = 10.79 min) corresponds to Δ^8-THC.
a) is a sample with concentrations of c = 10 ng mL^{-1} for Δ^9-THC and Δ^8-THC.
b) is a sample with concentrations of c = 1 ng mL^{-1} for Δ^9-THC and of c = 50 ng mL^{-1} for Δ^8-THC.
c) is a sample with concentrations of c = 50 ng mL^{-1} for Δ^9-THC and of c = 1 ng mL^{-1} for Δ^8-THC.

2.3.1 Recoveries for early and late eluting peaks

The following experiment and simulation are intended to illustrate what quantitative consequences different resolutions could have, not to characterise any specific laboratory's performance. To investigate whether and to what extent an overlap causes quantitative errors, a spiking series was prepared in which each isomer in turn was held constant at 1 ng mL^{-1} while the other was varied from 0 to 50 ng mL^{-1}. All solutions were measured in triplicate by LC-MS/MS and quantified using the internal standard method.

The recovery of Δ^8-THC as a function of Δ^9-THC concentration is shown in Figure 8. No relevant interference is observed at Δ^9-/Δ^8-THC ratios up to 10:1. At ratios of 25:1 and 50:1, the recovery increases to 187 % and 282 %, respectively. The mechanism is caused by peak tailing: because Δ^9-THC elutes first and tails to the right, the tail extends into the integration window of Δ^8-THC (Figure 7c). Under perpendicular-drop integration, where the valley minimum between the two peaks defines the boundary, all signal to the right of the valley is attributed to Δ^8-THC, regardless of its origin. The larger the Δ^9-THC concentration, the more tail area crosses into the Δ^8-THC window. Integration bias therefore acts predominantly on the later-eluting peak (Δ^8-THC).

The recovery of Δ^9-THC as a function of Δ^8-THC concentration is shown in Figure 9. In contrast to the pattern above, Δ^9-THC recoveries decrease steadily with increasing Δ^8-THC concentration, falling below 80 % at ratios of 25:1 and 50:1 (73 % and 70 %, respectively). This suppression is directionally opposite to what the perpendicular-drop integration model predicts for the early-eluting peak (which should be largely unaffected by the later one) and

is therefore not attributable to the same mechanism. It possibly arises from ion suppression effects, though further investigation into this or other reasons will not be discussed here.

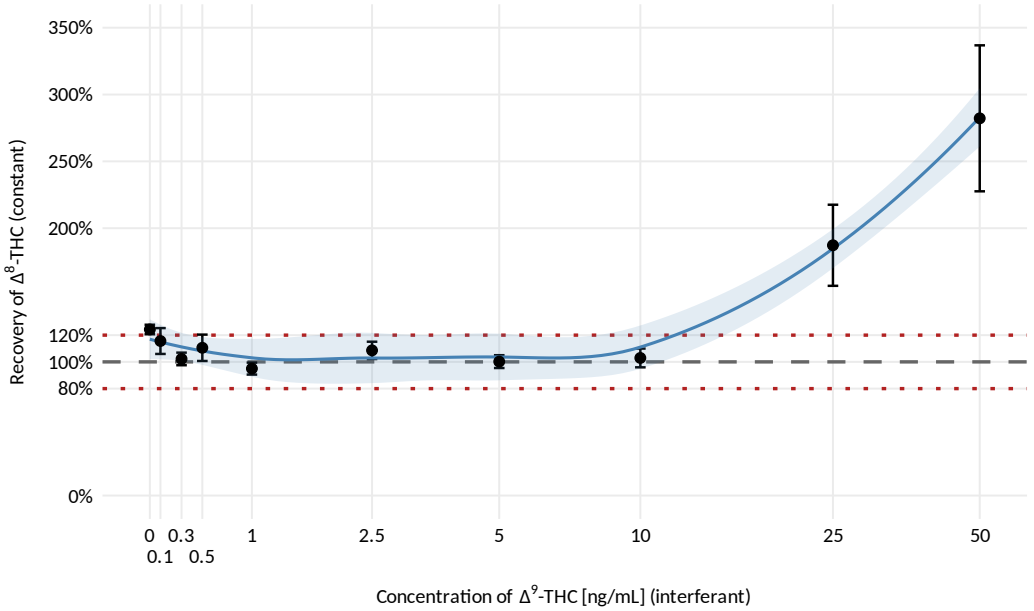

Figure 8: Recovery (mean ± standard deviation) of Δ^8-THC as a function of Δ^9-THC concentration. The solid blue line shows a LOESS trend with 95 % confidence interval (shaded). The x-axis is pseudo-logarithmically scaled to resolve the low-concentration range visually. Axis tick labels are vertically staggered for readability.

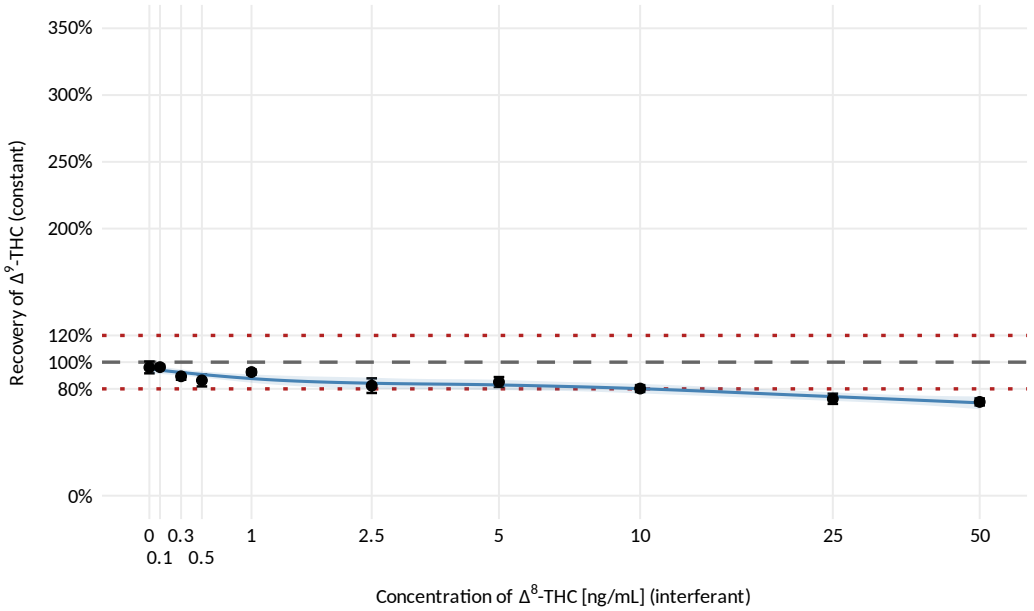

Figure 9: Recovery (mean ± standard deviation) of Δ^9-THC as a function of Δ^8-THC concentration. The solid blue line shows a LOESS trend with 95 % confidence interval (shaded). The x-axis is pseudo-logarithmically scaled to resolve the low-concentration range visually. Axis tick labels are vertically staggered for readability.

2.3.2 Influence of resolution and concentration ratio on recovery

The experiment was conducted at a single operating resolution (R_{FWHM} = 1.6) and does not by itself answer the question most relevant for method assessment: how large would the integration error be at a different resolution, i.e. using different methods in different laboratories? Still, to address this rudimentarily on the basis of one method from one laboratory, the peak shapes determined from single-standard measurements were used to generate artificial co-eluting chromatograms across a resolution range of R_{FWHM} = 0.5–3.0 and the full range of concentration ratios, with perpendicular-drop integration applied at each point. The peak shapes measured here serve as a representative starting point; the simulation is designed to illustrate the general effect of resolution and concentration ratio on recovery of the later peak, not to make specific, absolute predictions about any particular laboratory or method.

The underlying principle is straightforward: given known geometric peak shapes and a defined resolution, one can calculate how much area from the Δ^9-THC tail falls into the Δ^8-THC integration window, and therefore what the expected (over-)recovery would be. The simulated recoveries are shown in Figure 10; selected values are given in Table 8.

First, at the resolution used in the present experiment (R_{FWHM} ≈ 1.4–1.6), the model predicts Δ^8-THC recoveries of approximately 107, 117, 134, and 181 % at Δ^9-/Δ^8-THC ratios of 5:1, 13:1, 25:1, and 63:1, respectively. The experimentally measured values (187 % and 282 % at 25:1 and 50:1) exceed the predictions, indicating that the simulation provides a conservative estimate. The actual magnitude of the effect will depend on the specific tailing behaviour of the method under evaluation. Second, within the practically common resolution range of R_{FWHM} = 1–2, the integration error is affected more strongly by the concentration ratio than by the resolution. Moving from R_{FWHM} = 1.4 to 2.0 at a 25:1 concentration ratio reduces the over-recovery from 134 % to 119 %; at a 63:1 ratio, from 181 % to 145 %. In neither case does the improvement in resolution bring the recovery into a satisfactory range.

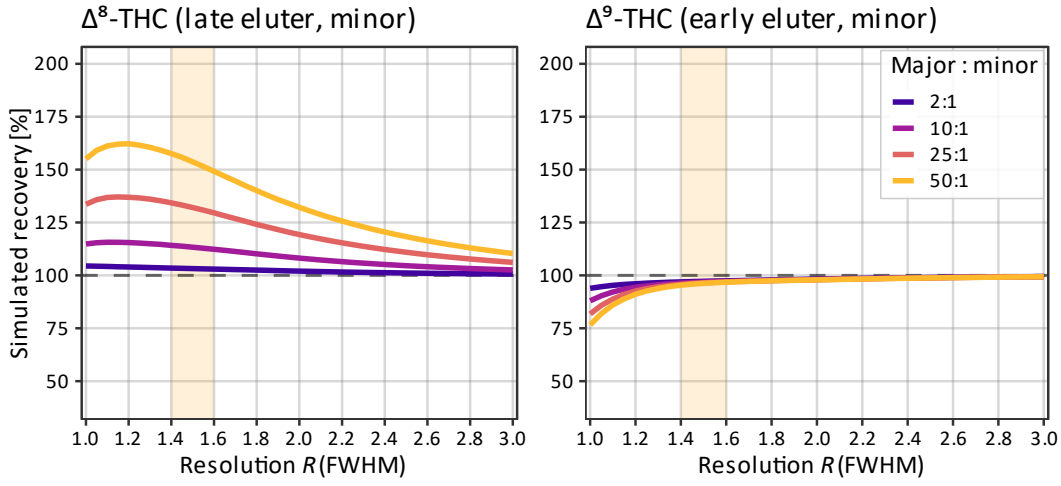

Figure 10: Simulated recovery of Δ^8-THC (left) and Δ^9-THC (right) as minor-component peaks under perpendicular-drop integration, as a function of chromatographic resolution R_{FWHM} and concentration ratio (major:minor). For example, given the peak shapes from Figure 7a, at a resolution of 1.6 and a concentration ratio of Δ^9- to Δ^8-THC of 50:1, the expected recovery for Δ^8-THC is 150 %. The orange band marks the operating resolution of the method applied for this experiment.

Table 8: Simulated recovery of Δ^8-THC (perpendicular-drop integration) as a function of chromatographic resolution and concentration ratio Δ^9-/Δ^8-THC.

Simulated Resolution R_{FWHM}	5:1	13:1	25:1	63:1
1.0	108 %	117 %	129 %	164 %
1.4	107 %	117 %	134 %	181 %
2.0	104 %	110 %	119 %	145 %
2.5	102 %	105 %	111 %	126 %
3.0	101 %	103 %	106 %	115%

The experiment and simulation show that the over-recovery of Δ^8-THC (or more precisely: the later eluting peak) is relatively small and easily overlooked when both isomers are present at similar concentrations, but can become large when Δ^9-THC is present at a 25-fold or greater excess, which is a realistic scenario in hemp-derived food and feed matrices. More fundamentally, the results indicate that even a seemingly adequate $R_{FWHM} > 1.5$ does not preclude substantial integration errors when peak tailing is present and concentration ratios are asymmetric. These results could serve as an explanation as to why, in this proficiency test, the assigned value for Δ^8-THC in the standard solution exceeds the expected value significantly.

If Δ^9- and Δ^8-THC are analysed as a sum, the problems are expected to be not relevant. If they are analysed separately, insufficient baseline separation could amplify the over-recovery of Δ^8-THC, especially at realistic, highly asymmetric concentration ratios. If a laboratory is interested in preventing such errors, resolution should be increased.

3 Scope and study design

3.1 Participants

A total of 19 laboratories participated in the proficiency test by submitting results. The participating laboratories are active in food control, either as official control laboratories of the federal states or as private contract laboratories. The participants are listed in Table 9.

Table 9: Participating laboratories in alphabetical order. All laboratories are based in Germany.

Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Erlangen, Bayern
Bundesinstitut für Risikobewertung, Berlin
Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL) – AöR, Münster, Nordrhein-Westfalen
Chemisches und Veterinäruntersuchungsamt Rhein-Ruhr-Wupper, Krefeld, Nordrhein-Westfalen
Eurofins WEJ Contaminants GmbH, Hamburg
GALAB Laboratories GmbH, Hamburg
GBA Gesellschaft für Bioanalytik mbH, Hamburg
Institut für Hygiene und Umwelt, Hamburg
Institut Kirchhoff Berlin GmbH, Berlin
Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern, Rostock, Mecklenburg-Vorpommern
Landesbetrieb Hessisches Landeslabor, Gießen, Hessen
Landeslabor Berlin-Brandenburg, Berlin, Brandenburg
Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen, Dresden, Sachsen
Landwirtschaftliche Untersuchungs- und Forschungsanstalt Speyer, Speyer, Rheinland-Pfalz
Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit – Futtermittelinstitut Stade, Stade, Niedersachsen
PLANTON GmbH, Kiel, Schleswig-Holstein
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3.2 Test material characterisation

Four hemp-containing materials and one standard solution were prepared for this proficiency test.

Test material 1: Hemp flour

Three 1 kg packages of hemp flour from the same lot were mixed in a rotary tumbler for 24 h. Homogeneity on the basis of a sample weight of 5 g was tested and confirmed on 23 July 2024. The homogenised sample was stored at room temperature until shipment.

Test material 2: Hemp oil cake

3.5 kg of one 8 kg package of hemp oil cake were homogenised using a centrifugal mill equipped with a 500 µm distance sieve at 12,000 rpm. The milled sample was then mixed in a rotary tumbler for 24 h. Homogeneity on the basis of a sample weight of 5 g was tested and confirmed on 23 July 2024. The homogenised sample was stored at room temperature until shipment.

Test material 3: Hemp tea

A total of 2.5 kg hemp tea from several packages were homogenised using a centrifugal mill equipped with a 500 µm distance sieve at 12,000 rpm. The milled sample was then mixed in a rotary tumbler for 24 h. Homogeneity on the basis of a sample weight of 3 g was tested and confirmed on 18 July 2024. According to the product declaration, the tea was made purely from hemp leaves. The homogenised sample was stored at room temperature until shipment.

Test material 4: Hemp pesto

Ten 130 g jars of hemp pesto were mixed using an Ultra-Turrax® to form a homogeneous mass with no visible seeds or seed fragments. Homogeneity on the basis of a sample weight of 2 g was tested and confirmed on 17 June 2025. According to the product declaration, the pesto contained 45 % cold pressed hemp seed oil, 40 % hulled hemp seeds, 12 % salt, and potato flakes. The homogenised sample was stored at -20 °C until shipment.

Test material 5: Cannabinoid standard solution

A mixture containing different concentrations of all the cannabinoids listed in Table 1 was prepared in acetonitrile. This mixture was then diluted to generate a working solution. Aliquots of this working solution, ready to be measured, were sent to participants with expected concentrations ranging from approximately 2 to 40 ng mL^{-1}.

3.3 Test material homogeneity

The homogeneity of the samples was determined in accordance with Annex B, Point B.1 of ISO 13528 [3]. For this purpose, ten units of each test material were randomly selected and analysed in duplicate under repeatability conditions. The homogeneity of the samples was confirmed on the basis of a sample weights of 5 g (hemp flour and hemp oil cake), 3 g (hemp tea), and 2 g (hemp pesto) for the cannabinoids CBD, CBDA, THCA, Δ^9-THC, and CBN, as these are reliably present in most hemp-containing food and feed.

The analyte distribution c was considered as sufficiently homogeneous if at least the extended condition for homogeneity according to Point B.2.3 in ISO 13528 is fulfilled where:

$$s_s = \sqrt{\max\left(0, s_{\bar{x}}^2 - \frac{s_w^2}{2}\right)}$$ Equation 1

$$s_w = \sqrt{\sum_{t=1}^{g} \frac{w_t^2}{2g}}$$ Equation 2

$$c = F_1 \cdot \left(0.3\sigma_{\mathrm{pt}}\right)^2 + F_2 \cdot s_w^2$$ Equation 3

s_s	between-samples standard deviation
σ_{pt}	target standard deviation for the proficiency test (pt) (here: 25 %)
s_w	within-sample standard deviation (here: duplicate analysis)
w_t	between-sample ranges
F_1, F_2	test values from Table B.1 in Annex B, ISO 13528

3.4 Shipment and instructions

Samples and accompanying information (digital only) were sent on 18 November 2025, and the deadline for submitting the results was 30 January 2026. A reporting sheet in .xlsx format was provided.

4 Statistical methodology and evaluation

4.1 Blunder removal and treatment of data below the reported Limit of Quantification

A visual pre-evaluation of the data was carried out to identify systematically deviating laboratories (obvious blunders) [4]. A systematic deviation of a laboratory is present if a consistent trend can be determined for the majority of the analytes in the test materials. Laboratories showing such deviations were contacted and asked to review their submitted data. Where laboratories provided corrected values, these replacement values were used in the statistical evaluation. Where no corrected values were submitted, the originally reported data were retained and included in the calculation. The influence of systematically deviating results on the assigned value and reproducibility standard deviation is minimised by the robust statistical procedure applied.

4.2 Assigned value and reproducibility standard deviation

The statistical evaluation was carried out in accordance with the "Statistical methods for use in proficiency testing by interlaboratory comparison" published in ISO 13528 [3]. Further specific procedures were adopted from the background document "Performance assessment in proficiency tests organised by the EURL mycotoxins & plant toxins in food and feed" [5].

The assigned value x_{av} was derived from participant results using robust statistics (Algorithm A, Huber estimator, Annex C.3 [3]). This approach yields robust estimates for the mean (assigned value) and the reproducibility standard deviation s_{R} by downweighing extreme laboratory results. Unlike conventional outlier elimination methods, robust statistics do not require the data to be normally distributed [5]. To determine an assigned value, a minimum of seven results per analyte and test material is required [5].

The assigned value is subject to uncertainty, depending on the number and dispersion of the results. This uncertainty is passed on directly to the z-scores. Consequently, high uncertainty caused by all participants collectively could falsely indicate unsatisfactory performance, with an individual laboratory being penalised for statistical variance rather than actual measurement error. To prevent inappropriate conclusions being drawn about laboratory competence, the uncertainty of the assigned value is formally factored into the evaluation (see Section 4.3).

4.2.1 Uncertainty of the assigned value

The uncertainty of the assigned value u_{av} was calculated from its estimated standard deviation and the number of results used for its determination:

$$u_{\mathrm{av}} = 1.25 \cdot \frac{s_{\mathrm{R}}}{\sqrt{n}} \qquad \text{Equation 4}$$

u_{av} uncertainty of the assigned value
n number of values used to calculate the assigned value
s_{R} robust reproducibility standard deviation of the laboratory results

Note: According to ISO 13528 Chapter 7.7.7 the factor 1.25 is based on the standard deviation of the median, or the efficiency of the median as an estimate of the mean, in a large set of results drawn from a normal distribution.

In accordance with ISO 13528 Chapter 9.2.1, the uncertainty of the assigned value (u_{av}) may be considered to be negligible if:

$$u_{\mathrm{av}} \leq 0.3 \cdot \sigma_{\mathrm{pt}} = 0.3 \cdot 0.25 \cdot x_{\mathrm{av}} \qquad \text{Equation 5}$$

4.3 Performance characteristics of the laboratories with regard to the accuracy

4.3.1 Calculation of *z*-scores and *z*'-scores

A primary objective of proficiency testing is to evaluate the performance and fitness for purpose of participating laboratories and their applied methods. For this evaluation, the difference between a laboratory's measured value and the assigned value x_{av} is expressed as a z-score. Commission Regulation (EU) 2023/2783 specifies a target standard deviation σ_{pt} of 25 %. Thus, a 25 % deviation from the assigned value corresponds to a z-score of 1 [6].

$$z = \frac{(x_{\mathrm{lab}} - x_{\mathrm{av}})}{\sigma_{\mathrm{pt}}} \qquad \text{Equation 6}$$

As described in Section 4.2, the uncertainty of the assigned value u_{av} can be high and must then be considered in laboratory evaluations. If the uncertainty is non-negligible (see Section 4.2.1), z'-scores have to be calculated instead of z-scores [6].

$$z' = \frac{x_{\mathrm{lab}} - x_{\mathrm{av}}}{\sqrt{\sigma_{\mathrm{pt}}^2 + u_{\mathrm{av}}^2}} \qquad \text{Equation 7}$$

For example, with a non-negligible $u_{\mathrm{av}} = 15\ \%$, $x_{\mathrm{lab}} = 151$, $x_{\mathrm{av}} = 100$, and $u_{\mathrm{av}} = 8\ \%$, applying the z'-score is required: it adjusts an otherwise questionable evaluation ($z = 2.06$) to a satisfactory result ($z' = 1.96$), ensuring laboratories are not penalized for the inherent variance of the reference value.

The resulting *z*- and *z*'-scores are interpreted as follows [3]:

$\lvert z \rvert \leq 2$	satisfactory
$2 < \lvert z \rvert < 3$	questionable (or warning signal)
$\lvert z \rvert \geq 3$	unsatisfactory (or action signal)

4.3.2 Calculation of the laboratory bias and precision

Participation in proficiency tests is a good opportunity to estimate the laboratory/method bias (also called systematic error). The assigned value derived for an analyte in a test material is a sufficiently good estimate of the true value. Therefore, the laboratory's deviation from the assigned value (calculated as a z-score) can be used in the same way as the laboratory's deviation when analysing a certified reference material.

According to ISO 11352, Chapter 8.3.3 states that a laboratory should analyse at least six samples in one or two proficiency test rounds in order to reliably estimate bias [7]. For many laboratories and methods, this approach would mean that bias estimation would only be possible after a long period of time. This method of estimating bias is appropriate if bias is an intrinsic factor, e.g. analyte loss during sample processing. However, it would be incorrect if the bias were not an intrinsic factor and the laboratory recognised an error based on the deviation in the proficiency test, correcting it immediately. This would be the case, for example, if the concentration of the standard solution was incorrect.

Since up to 19 cannabinoids are tested (Table 1), the confidence in the bias estimate appears to be sufficiently high even with fewer than the recommended six samples. This approach is already used in the field of pesticides for bias estimation from proficiency testing data, see SANTE/11312/2021, Annex C, Approach 2 [4].

The bias can be calculated in different ways:

(1) Calculating the Root Mean Squares of the sum of the squared bias divided by the number of PT results [4].
    a. This approach has the disadvantage that information about the bias is lost, e.g. whether the laboratory under- or overestimated the assigned value (recovery below or above 100 %). In other words, this procedure calculates a precision parameter rather than a bias.

(2) A direct method for assessing a laboratory's bias is to use the deviation of an analyte-matrix combination from the assigned values (approximating the true value), similar to analysing a reference material. The mean deviation of all tested analyte-matrix combinations reflects the mean bias.
    a. The disadvantage of this approach is that inaccurate methods that both underestimate and overestimate the assigned value (recovery below or above 100 %) can lead to a bias of zero. Therefore, this type of bias estimation must be supported by an additional precision estimation that enables the laboratory to distinguish between bias and (im)precision.

To obtain information about the direction of the bias information, option 2 is preferred and the mean percent bias $\bar{B}_{\%}$ is calculated as follows:

$$\bar{B}_{\%} = \frac{\sum_1^n \left(\frac{x_{\mathrm{m}} - x_{\mathrm{av,m}}}{x_{\mathrm{av,m}} \cdot 100} \right)}{n}$$ Equation 8

x_{m} result of the laboratory for analyte m
$x_{\mathrm{av,m}}$ assigned value of analyte m
n number of analytes

The mean z-score $\bar{z}$ of all analyte-matrix-combinations tested can be calculated as follows (mean deviation of a laboratory from the assigned value for all analyte-matrix-combination tested):

$$\bar{z} = \frac{\sum_1^n z}{n}$$ Equation 9

z for calculation refer to equation 6.
n number of z-scores (evaluated analyte-matrix combinations)

To distinguish between bias and (im)precision, the dispersion of each laboratory's individual analyte-matrix bias results around its mean bias (mean deviation from the assigned values) was used as a measure of precision. This is an indicator of how the laboratory reproduces its measurements within this PT. The standard deviation of the individual analyte-matrix bias results was calculated according to the following equation and reported as the precision of the laboratory:

$$z_{\text{precision}} = \sqrt{\frac{1}{n-1}\sum_{\text{m}}^{n}(z_{\text{m}} - \bar{z})}$$ Equation 10

z_{m} z-score of the laboratory for analyte m
$\bar{z}$ mean z-score of the laboratory for all analytes, see Equation 9

Note: Calculating bias in terms of mean percentage and precision in terms of standard deviation percentage, or z-score mean and z-score precision, is equivalent. This is because the percentage deviation of a laboratory result from its assigned value is equivalent to the laboratory's z-score multiplied by 25.

4.3.3 Calculation of the laboratory absolute mean z-score

According to Commission Regulation (EU) 2023/915, a laboratory may declare an expanded standard uncertainty of 50 %, provided it meets the specified intra-laboratory precision requirements and successfully participates in proficiency testing programmes [1]. To this end, $|\bar{z}| \leq 2$ must be achieved to demonstrate that the required accuracy under reproducibility conditions has been met.

$$|\bar{z}| = \frac{\sum_1^n |z|}{n}$$ Equation 11

5 Summary

The 2025 proficiency test for cannabinoids in hemp-based food matrices was successfully conducted with 19 participating laboratories, covering 19 analytes across four naturally contaminated test materials (hemp flour, hemp oil cake, hemp tea, hemp seed pesto) and one cannabinoid standard solution. Across all samples, analytes, and laboratories, 91 % of z-scores fell within the satisfactory range ($|z| \leq 2$), indicating a generally satisfactory level of analytical competence among the participating laboratories.

For Δ^9-THC, which is the primary regulated analyte under Regulation (EU) 2023/915, per-sample success rates ranged from 83 % (hemp pesto) to 95 % (hemp flour), with RSD_R values between 19.5 and 28 %. These figures demonstrate that the majority of laboratories are capable of determining Δ^9-THC reliably across the tested concentration range of 0.2 to 60.9 mg kg^{-1}. Performance was comparably robust for the standard solution (15.0 % RSD_R and 93 % satisfactory z-scores), confirming that the observed variability in food matrices reflects genuine method challenges rather than fundamental calibration deficiencies. For THCA, which together with Δ^9-THC determines the regulatory Δ^9-THC equivalents, inter-laboratory reproducibility was markedly lower, with RSD_R ranging from 28.1 to 73 % across the sample materials and 24.3 % within the standard solution. As the variation in the measurement results was already considerably higher in the standard solution, the wider range is likely to be due to several factors.

Almost all laboratories meet the precision requirements under reproducibility conditions and could report a default expanded measurement uncertainty of 50 % (EU-Regulation 2023/2783). If there are indications of a systematic bias (in cases of elevated mean deviations), the individual bias patterns and their analyte-specific contributions may be helpful for method review (Appendix B – Visual summary per participant).

A review of the reported method details (instrumentation, extraction approach, clean-up procedure, calibration strategy) revealed no systematic association between any of these parameters and overall laboratory performance. Elevated mean absolute z-scores are more likely attributable to individual, laboratory-specific factors than to a common methodological characteristic.

Due to the overestimation of the assigned value for the Δ^8-THC concentration in the standard solution, an investigation was conducted on the chromatographic separation of Δ^9- and Δ^8-THC and its impact on quantification. Results revealed that, even seemingly adequate resolution ($R_{FWHM} > 1.5$) can lead to a substantial overestimation of the later-eluting peak (generally assumed to be Δ^8-THC). If Δ^9- and Δ^8-THC are analysed as a sum (total content), the problems are likely to be negligible, since the total content would not be overestimated. When determining individual concentrations, insufficient baseline separation could lead to an overestimation of Δ^8-THC, particularly, when the concentration ratios are as asymmetric as they typically are in hemp food matrices. To avoid such errors, the focus on resolution needs to be increased.

6 References

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[2] EFSA Panel on Contaminants in the Food Chain (CONTAM) *et al.,* "Derivation of a health‐based guidance value for Δ8‐tetrahydrocannabinol (Δ8‐THC) and its occurrence in food," *EFSA Journal*, vol. 23, no. 11, e9735, 2025. doi: 10.2903/j.efsa.2025.9735. [Online]. Available: https://efsa.onlinelibrary.wiley.com/doi/full/10.2903/j.efsa.2025.9735

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[4] European Commission, "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed: SANTE 11312/2021 v2026," European Commission, Directorate-General for Health and Food Safety (DG SANTE), Brussels, 2021.

[5] EURL mycotoxins & plant toxins, "Performance assessment in proficiency tests organised by the EURL mycotoxins & plant toxins in food and feed v1.1," Wageningen Food Safety Research, part of Wageningen University & Research, Rep. EURL-MP-background doc_001 v1.1, 2023.

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[7] *Wasserbeschaffenheit – Abschätzung der Messunsicherheit beruhend auf Validierungs- und Kontrolldaten (ISO 11352:2012)*, 241, DIN ISO, 2013.

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9 Appendix A – Mandel's h plots

This appendix shows plots for Mandel's h statistics for each sample.

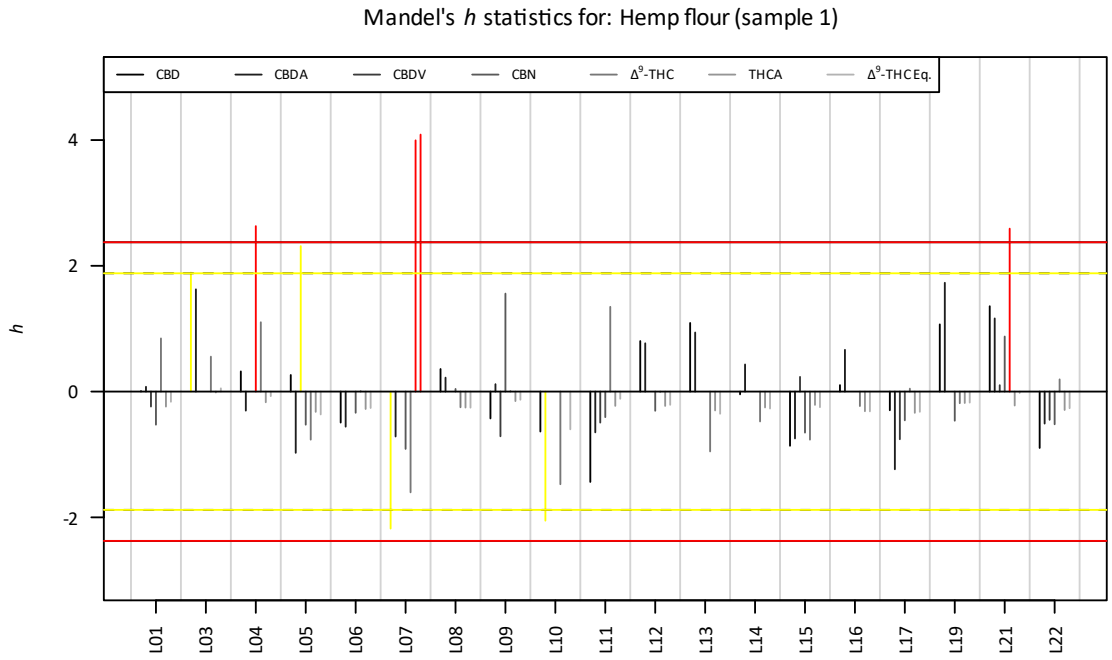


Figure 11: Mandel's h statistics for hemp flour (sample 1) by laboratory. Values outside ±2 (dashed lines) indicate laboratories with significant mean bias relative to the consensus.

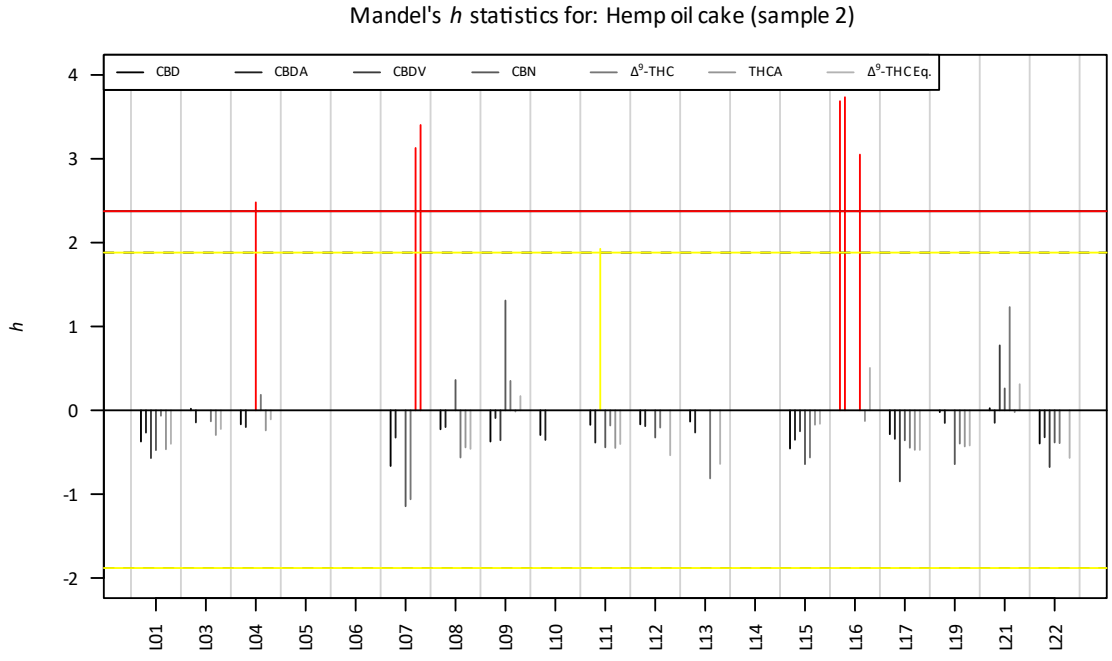


Figure 12: Mandel's ***h*** statistics for hemp oil cake (sample 2) by laboratory. Values outside ±2 (dashed lines) indicate laboratories with significant mean bias relative to the consensus.

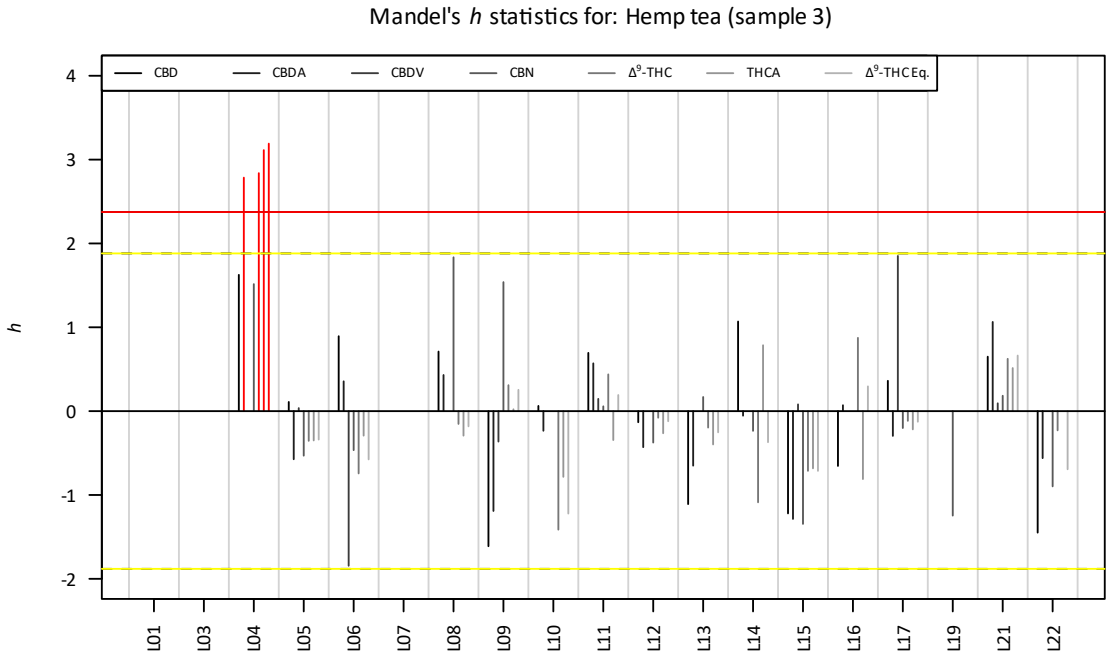


Figure 13: Mandel's ***h*** statistics for hemp tea (sample 3) by laboratory. Values outside ±2 (dashed lines) indicate laboratories with significant mean bias relative to the consensus.

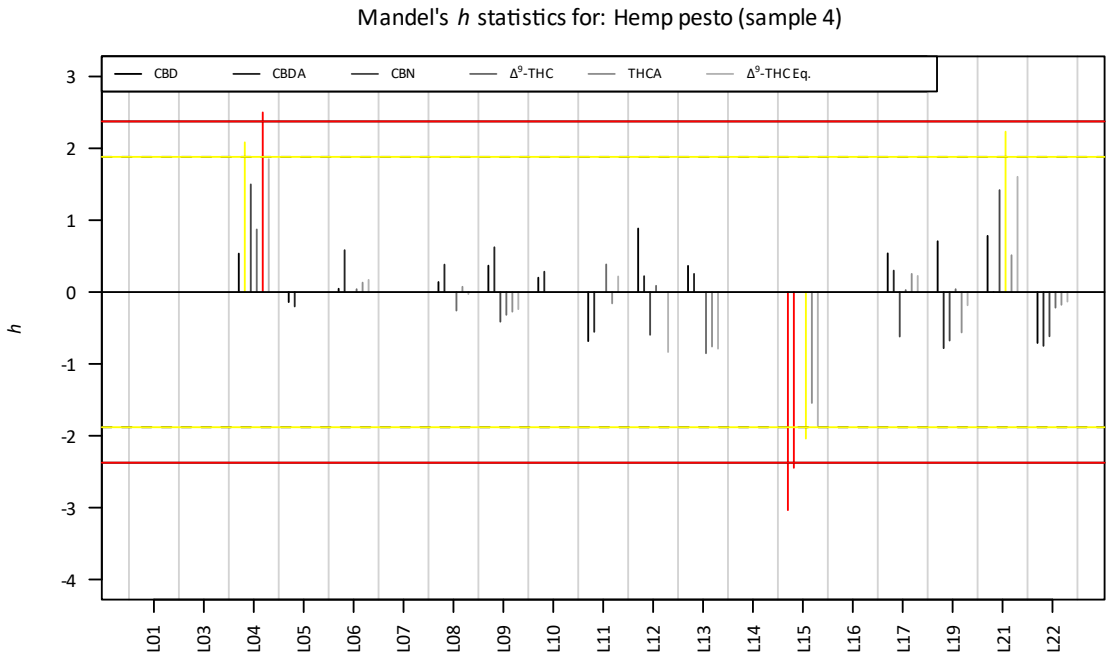


Figure 14: Mandel's ***h*** statistics for hemp pesto (sample 4) by laboratory. Values outside ±2 (dashed lines) indicate laboratories with significant mean bias relative to the consensus.

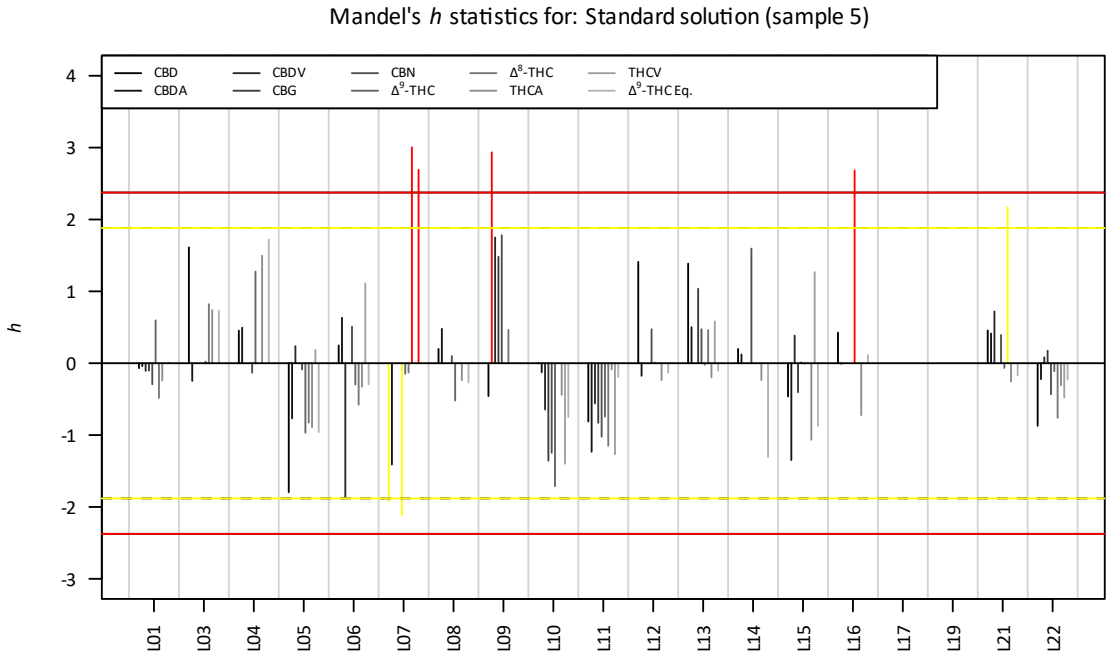
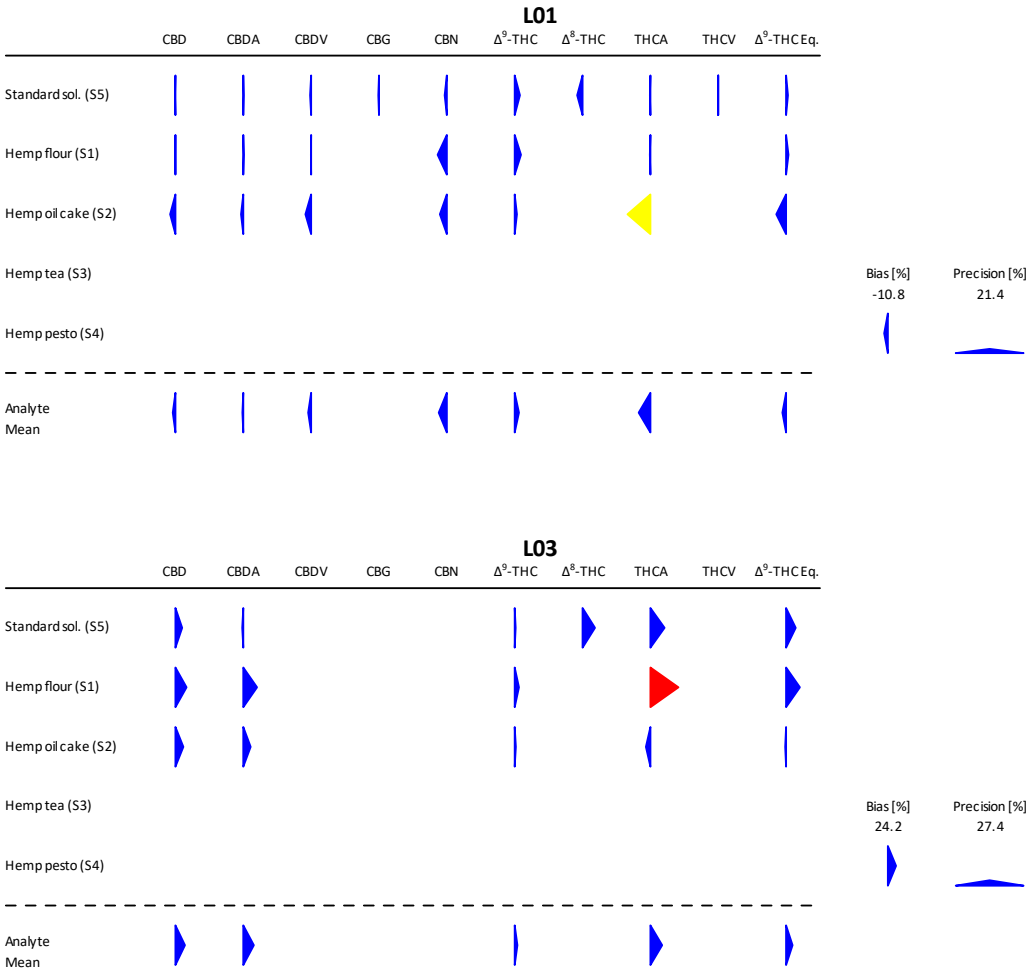
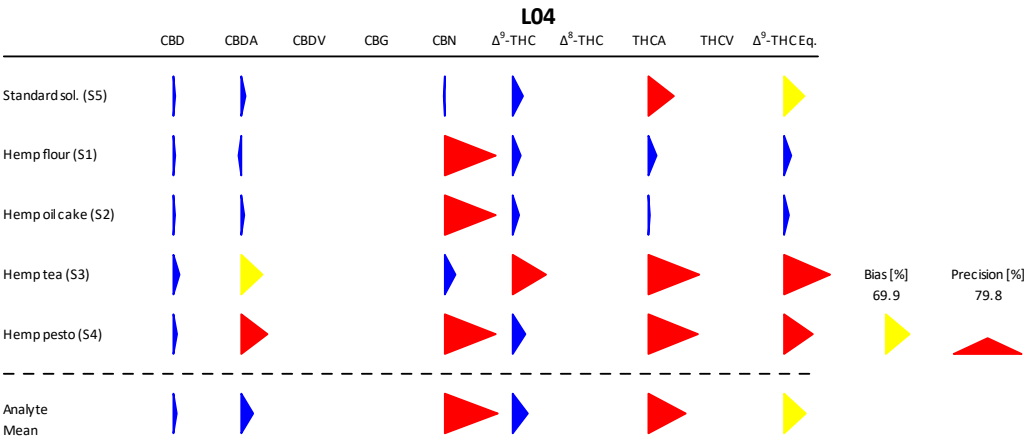


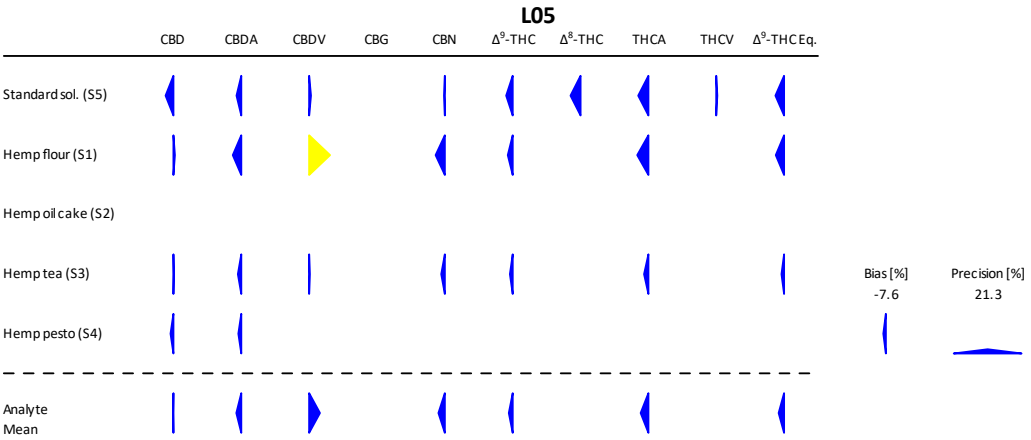
Figure 15: Mandel's ***h*** statistics for the standard solution (sample 5) by laboratory. Values outside ±2 (dashed lines) indicate laboratories with significant mean bias relative to the consensus.

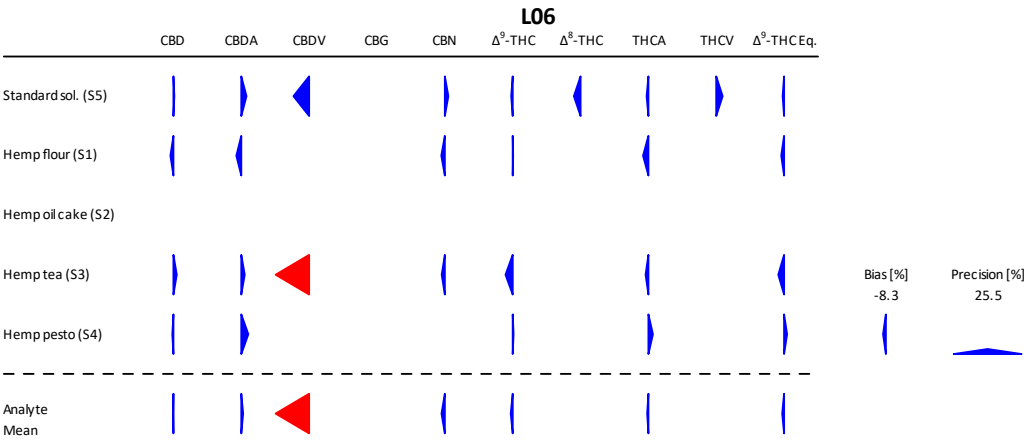
10 Appendix B – Visual summary per participant

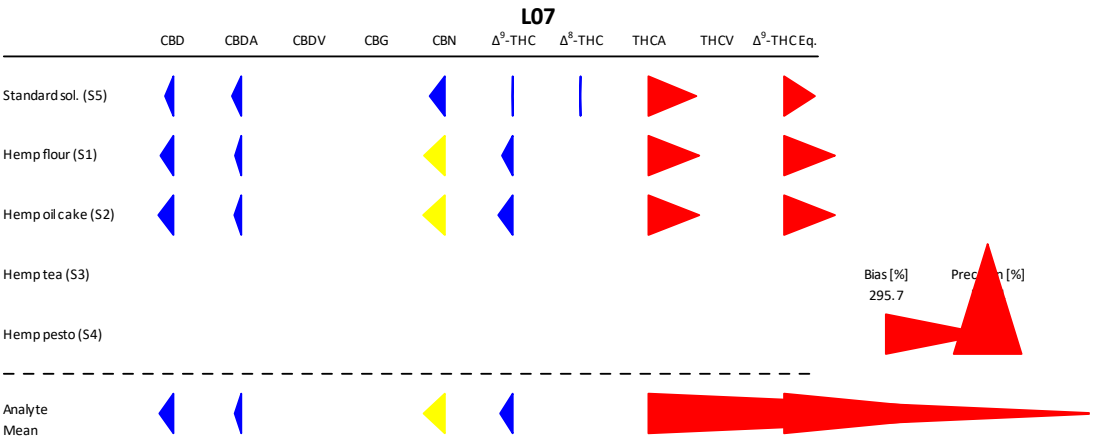
Appendix B shows the Wimpel-plots for each participant across the tested analyte-matrix-combinations as well as the mean for all materials for each analyte, and the mean bias and precision. The target relative standard deviation is 25 % and corresponds to $|z| = 1$. The size of the triangles shows the magnitude of z and their colour shows the interpretation. Blue = satisfactory ($|z| \leq 2$), yellow = questionable ($2 < |z| < 3$), red = unsatisfactory ($|z| \geq 3$).

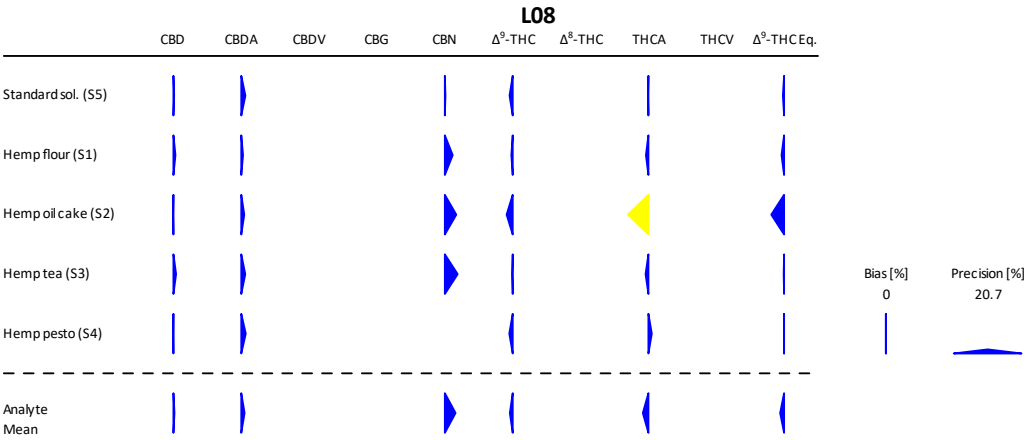


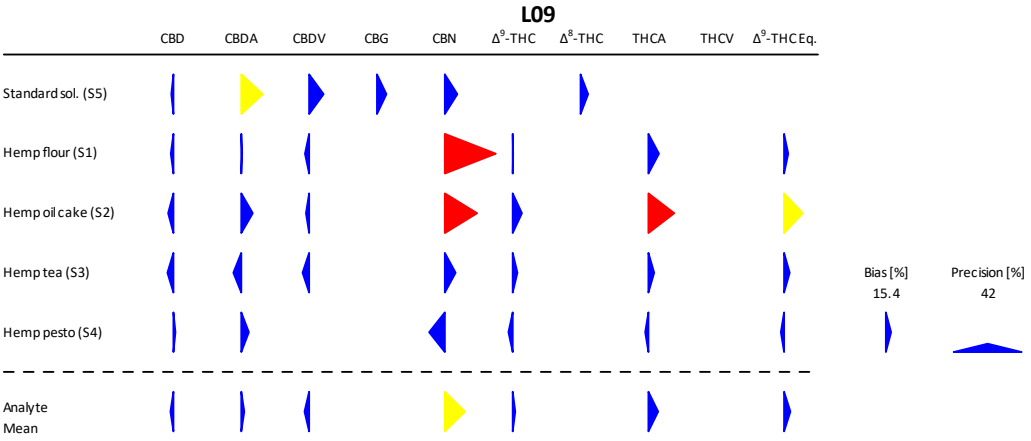

L04
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
69.9
Precision [%]
79.8

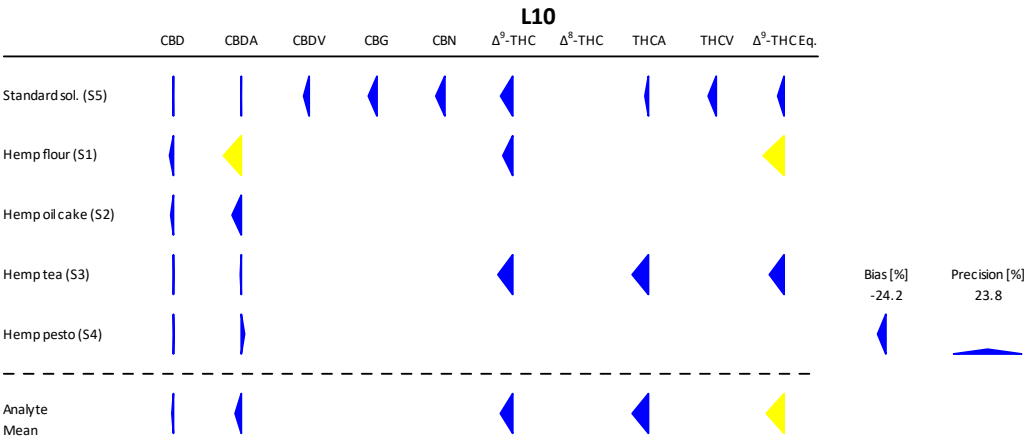

L05
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-7.6
Precision [%]
21.3

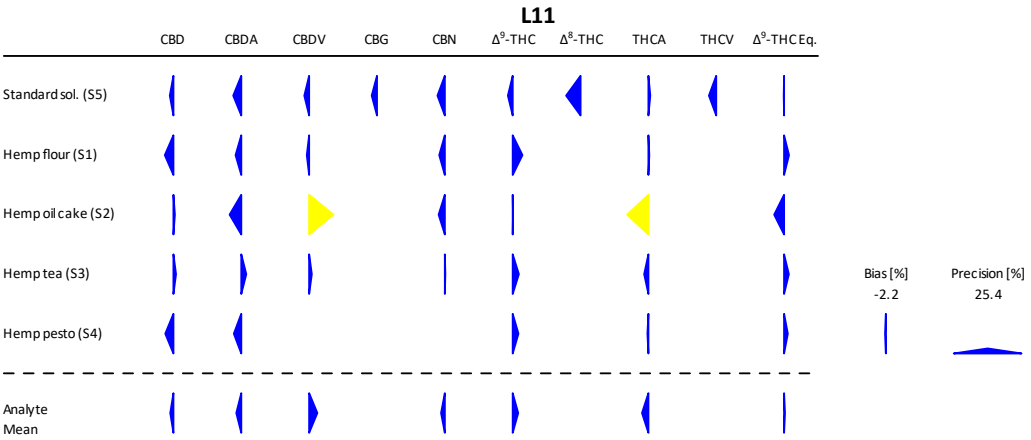

L06
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-8.3
Precision [%]
25.5

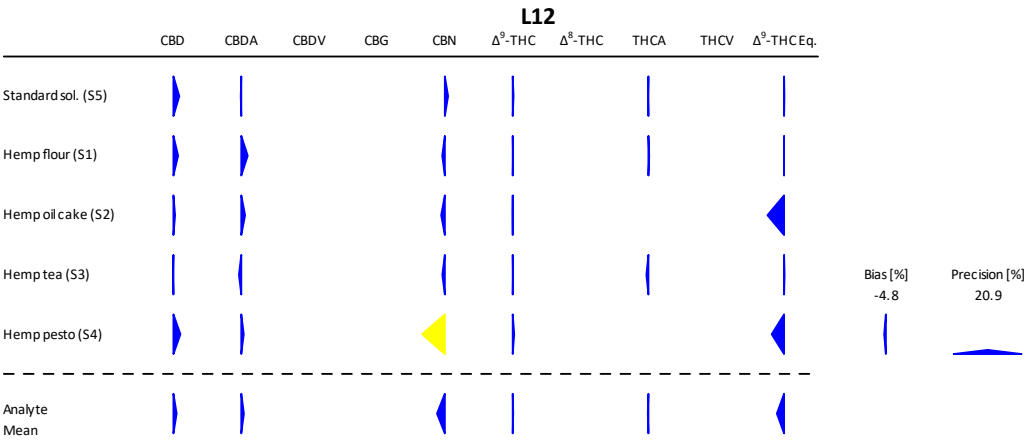

L07
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
295.7
Precision [%]

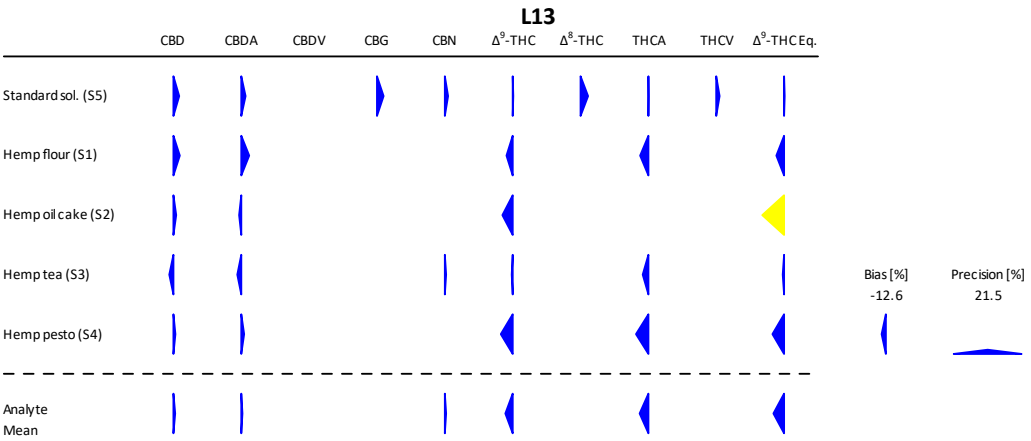

L08
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
0
Precision [%]
20.7

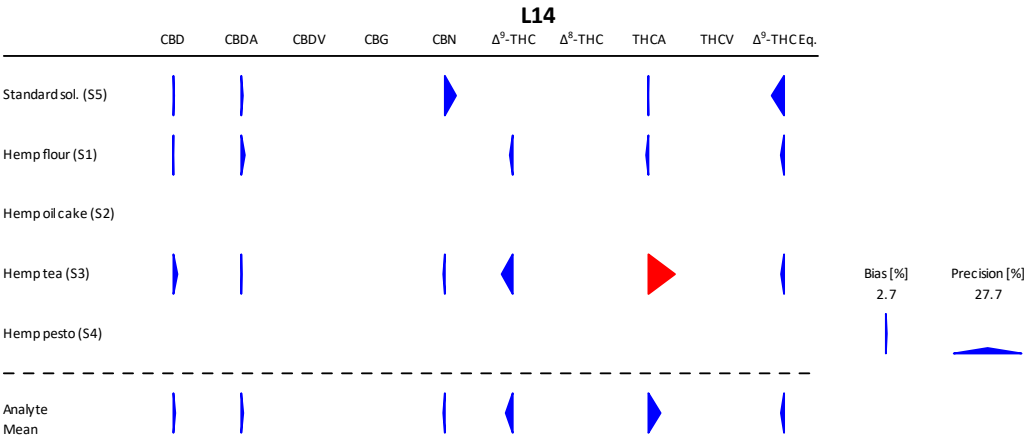

L09
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
15.4
Precision [%]
42

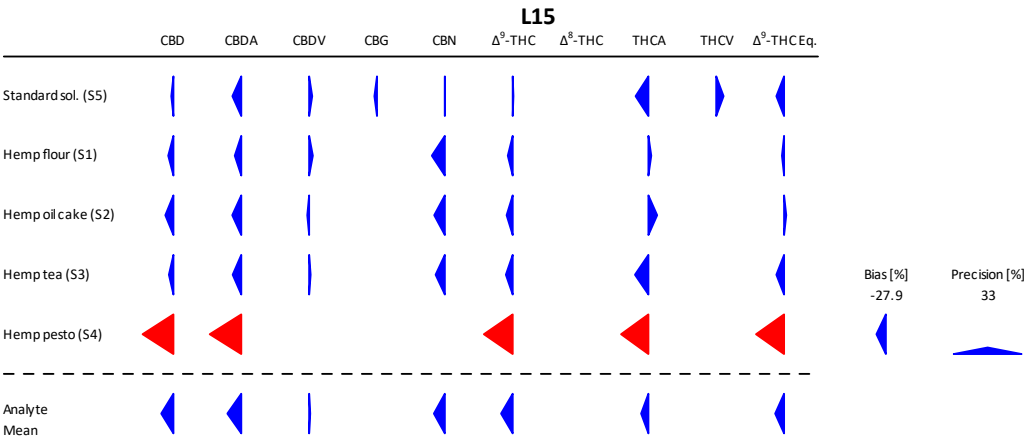

L10
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-24.2
Precision [%]
23.8

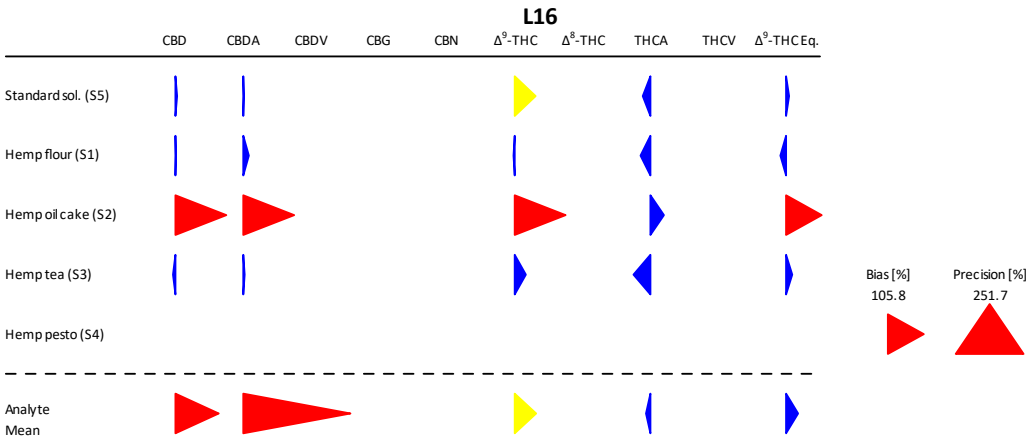

L11
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-2.2
Precision [%]
25.4

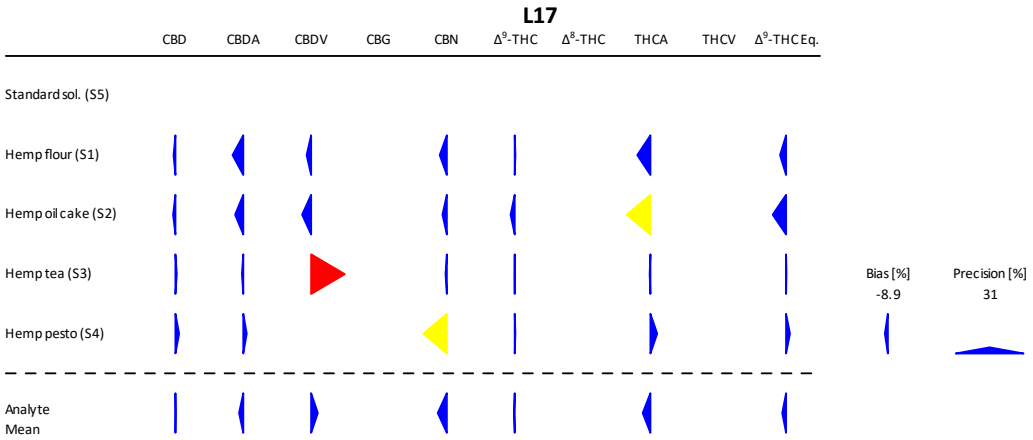

L12
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-4.8
Precision [%]
20.9

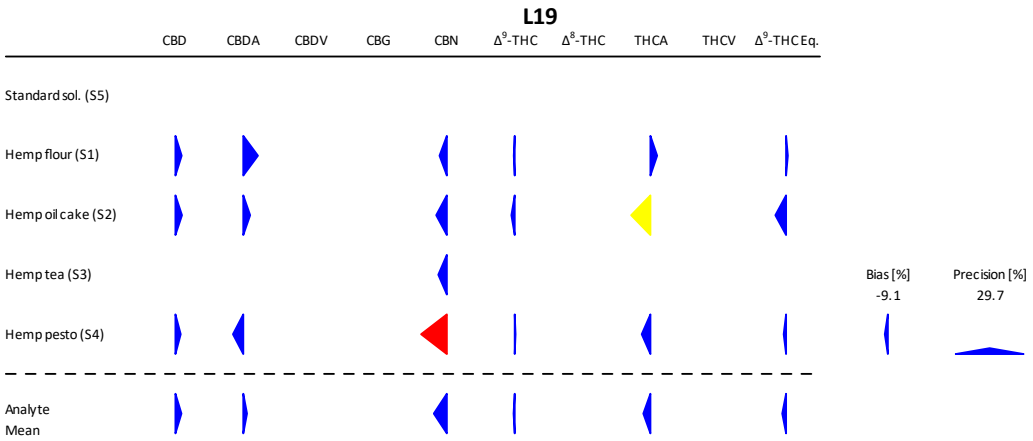

L13
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-12.6
Precision [%]
21.5

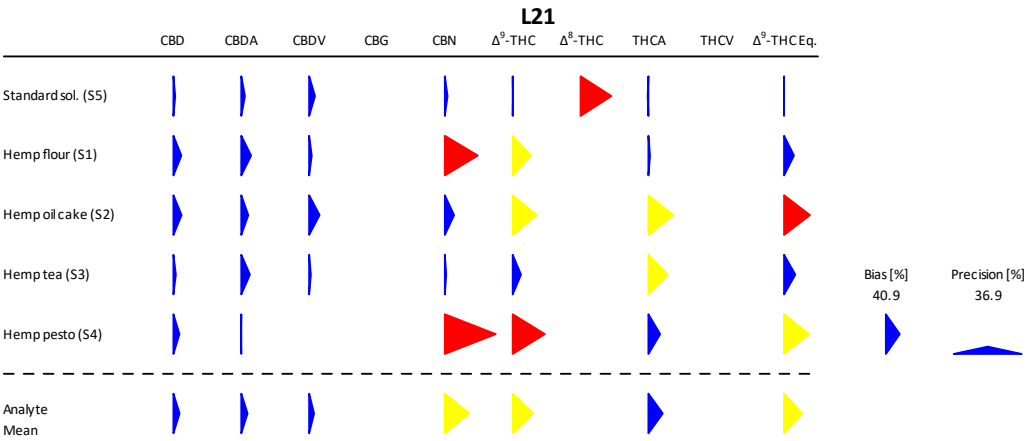

L14
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
2.7
Precision [%]
27.7

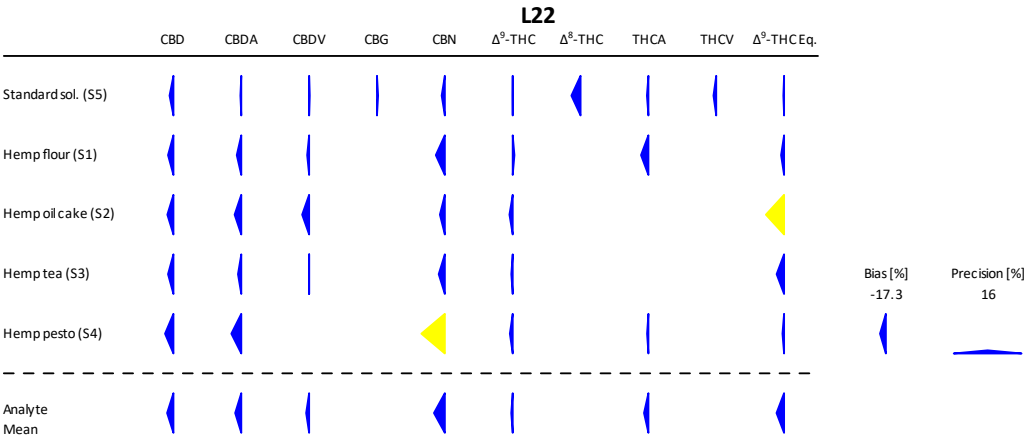

L15
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-27.9
Precision [%]
33


L16
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
105.8
Precision [%]
251.7


L17
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-8.9
Precision [%]
31


L19
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-9.1
Precision [%]
29.7


L21
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
40.9
Precision [%]
36.9


L22
CBD
CBDA
CBDV
CBG
CBN
Δ9-THC
Δ8-THC
THCA
THCV
Δ9-THC Eq.
Standard sol. (S5)
Hemp flour (S1)
Hemp oil cake (S2)
Hemp tea (S3)
Hemp pesto (S4)
Analyte Mean
Bias [%]
-17.3
Precision [%]
16

11 Appendix C – *z*-scores

Table 10: Overview of ***z***-scores achieved by the participants in hemp flour (test material 1). For CBDA, CBN, and THCA ***z***'-scores were calculated and they were marked with *. No ***z***-scores were calculated for analytes not listed.

Lab	CBD	CBDA*	CBDV	CBN*	Δ^9-THC	THCA*	Δ^9-THC eq.
L01	-0.01	0.06	-0.03	-0.89	0.74	-0.06	0.28
L03	1.31	1.55			0.5	3.14	1.63
L04	0.21	-0.31		8.29	0.95	0.92	0.85
L05	0.17	-0.96	2.47	-0.89	-0.58	-1.25	-0.98
L06	-0.36	-0.56		-0.34	0.05	-0.6	-0.34
L07	-1.54	-0.71		-2.01	-1.26	60.13	26.93
L08	0.24	0.2		0.76	-0.16	-0.28	-0.3
L09	-0.31	0.1	-0.49	5.17	0.05	1.19	0.49
L10	-0.46	-2			-1.16		-2.46
L11	-1.02	-0.64	-0.28	-0.54	1.15	0.1	0.58
L12	0.55	0.72		-0.25	0.04	0.09	-0.02
L13	0.75	0.89			-0.73	-0.93	-0.91
L14	-0.04	0.4			-0.34	-0.25	-0.39
L15	-0.62	-0.74	0.44	-1.26	-0.58	0.32	-0.25
L16	0.06	0.62			-0.14	-1.1	-0.67
L17	-0.22	-1.21	-0.53	-0.69	0.09	-1.47	-0.72
L19	0.74	1.65		-0.71	-0.1	0.75	0.2
L21	0.94	1.11	0.31	3.18	2.17	0.18	1.17
L22	-0.64	-0.51	-0.23	-0.87	0.21	-0.83	-0.36

Table 11: Overview of ***z***-scores achieved by the participants in hemp oil cake (test material 2). CBDA, CBDV, CBN, and Δ^9-THC ***z***'-scores were calculated and they were marked with *. No ***z***-scores were calculated for analytes not listed.

Lab	CBD	CBDA*	CBDV*	CBN*	Δ^9-THC*
L01	-0.62	-0.25	-0.58	-0.68	0.25
L03	0.94	0.82	-	-	0.13
L04	0.19	0.33	-	5.64	0.72
L05	-	-	-	-	-
L06	-	-	-	-	-
L07	-1.79	-0.78	-	-2.11	-1.61
L08	-0.04	0.33	-	1.11	-0.68
L09	-0.62	1.28	-0.32	3.14	1.03
L10	-0.31	-1.03	-	-	-
L11	0.17	-1.31	2.45	-0.6	0.04
L12	0.19	0.43	-	-0.35	-0.01
L13	0.33	-0.24	-	-	-1.14
L14	-	-	-	-	-
L15	-0.96	-1.01	-0.19	-1.03	-0.68
L16	15.54	35.16	-	-	6.06
L17	-0.28	-0.91	-0.91	-0.43	-0.46
L19	0.77	0.77	-	-1.03	-0.37
L21	0.96	0.78	1.05	0.9	2.67
L22	-0.72	-0.75	-0.71	-0.48	-0.36

Table 12: Overview of z-scores achieved by the participants in hemp tea (test material 3). For CBDV, CBN, Δ^9-THC, and THCA z'-scores were calculated and they were marked with *. No z-scores were calculated for analytes not listed.

Lab	CBD	CBDA	CBDV*	CBN*	Δ^9-THC*	THCA*	Δ^9-THC eq.
L01	-	-	-	-	-	-	-
L03	-	-	-	-	-	-	-
L04	0.72	2.51	-	1.2	3.74	9.18	5.43
L05	0.05	-0.4	0.07	-0.41	-0.31	-0.44	-0.31
L06	0.39	0.4	-3.67	-0.35	-0.8	-0.28	-0.69
L07	-	-	-	-	-	-	-
L08	0.31	0.47	-	1.45	-0.05	-0.28	-0.05
L09	-0.71	-0.93	-0.72	1.22	0.53	0.59	0.66
L10	0.03	-0.11	-	-	-1.65	-1.65	-1.74
L11	0.31	0.59	0.29	0.06	0.69	-0.43	0.56
L12	-0.06	-0.28	-	-0.28	0.04	-0.21	0.05
L13	-0.49	-0.47	-	0.14	-0.11	-0.58	-0.16
L14	0.47	0.05	-	-0.17	-1.24	2.71	-0.36
L15	-0.54	-1.02	0.16	-1.04	-0.76	-1.37	-0.91
L16	-0.29	0.16	-	-	1.25	-1.72	0.73
L17	0.16	-0.16	3.68	-0.15	-0.01	-0.08	0.04
L19	-	-	-	-0.97	-	-	-
L21	0.29	1.02	0.19	0.15	0.93	1.96	1.32
L22	-0.64	-0.39	0	-0.69	-0.15	-	-0.88

Table 13: Overview of z-scores achieved by the participants in hemp pesto (test material 4). For CBDA, Δ^9-THC, THCA, and Δ^9-THC eq. z'-scores were calculated and they were marked with *. No z-scores were calculated for analytes not listed.

Lab	CBD	CBDA*	Δ^9-THC*	THCA*	Δ^9-THC eq.*
L01	-	-	-	-	-
L03	-	-	-	-	-
L04	0.43	2.92	1.39	5.17	2.92
L05	-0.34	-0.33	-	-	-
L06	-0.13	0.79	0.09	0.47	0.31
L07	-	-	-	-	-
L08	-0.02	0.5	-0.37	0.36	0
L09	0.24	0.84	-0.46	-0.33	-0.33
L10	0.05	0.36	-	-	-
L11	-0.96	-0.83	0.63	-0.1	0.38
L12	0.82	0.27	0.17	-	-1.26
L13	0.23	0.32	-1.3	-1.29	-1.19
L14	-	-	-	-	-
L15	-3.64	-3.52	-3.15	-2.85	-2.86
L16	-	-	-	-	-
L17	0.43	0.38	0.07	0.71	0.39
L19	0.62	-1.15	0.09	-0.9	-0.24
L21	0.71	-0.04	3.51	1.23	2.54
L22	-0.99	-1.11	-0.31	-0.14	-0.16

Table 14: Overview of z-scores achieved by the participants in the cannabinoid standard solution (test material 5). For CBDV, CBG, Δ^8-THC, THCA, and THCV z'-scores were calculated and they were marked with *. No z-scores were calculated for analytes not listed.

Lab	CBD	CBDA	CBDV*	CBG*	CBN	Δ^9-THC	THCA*	Δ^9-THC eq.	Δ^8-THC*	THCV*
L01	-0.05	0.05	-0.11	-0.07	-0.26	0.62	-0.55	-0.05	0	0.22
L03	0.8	-0.12	-	-	-	0.11	1.27	1.6	-	1.14
L04	0.21	0.5	-	-	-0.12	1.22	-	2.87	-	2.4
L05	-0.93	-0.56	0.2	-	-0.09	-0.77	-1.02	-1.15	0.12	-1.02
L06	0.11	0.61	-1.73	-	0.41	-0.17	-0.68	-0.2	0.71	-0.17
L07	-0.99	-1.1	-	-	-1.79	-0.04	-0.05	5.41	-	3.64
L08	0.08	0.48	-	-	0.07	-0.37	-	-0.04	-	-0.14
L09	-0.25	2.55	1.6	1.03	1.48	-	0.77	-	-	-
L10	-0.02	-0.02	-0.61	-0.94	-1.06	-1.43	-	-0.39	-0.9	-0.76
L11	-0.43	-0.95	-0.53	-0.58	-0.87	-0.57	-1.47	0.21	-0.81	-0.04
L12	0.69	-0.06	-	-	0.38	0.1	-	-0.04	-	0.03
L13	0.68	0.5	-	0.72	0.38	0.07	0.77	0.02	0.37	0.07
L14	0.08	0.19	-	-	1.32	-	-	-0.04	-	-1.46
L15	-0.25	-1.05	0.34	-0.28	0	0.09	-	-1.44	0.82	-0.91
L16	0.2	0.08	-	-	-	2.47	-	-0.86	-	0.35
L17	-	-	-	-	-	-	-	-	-	-
L19	-	-	-	-	-	-	-	-	-	-
L21	0.21	0.43	0.65	-	0.32	0.03	3.15	-0.08	-	-0.01
L22	-0.46	-0.1	0.06	0.12	-0.37	-0.01	-0.93	-0.16	-0.31	-0.09

12 Appendix D – Chromatography of Δ^9- and Δ^8-THC

Simulated chromatogramms of Δ^9- and Δ^8-THC are shown here.

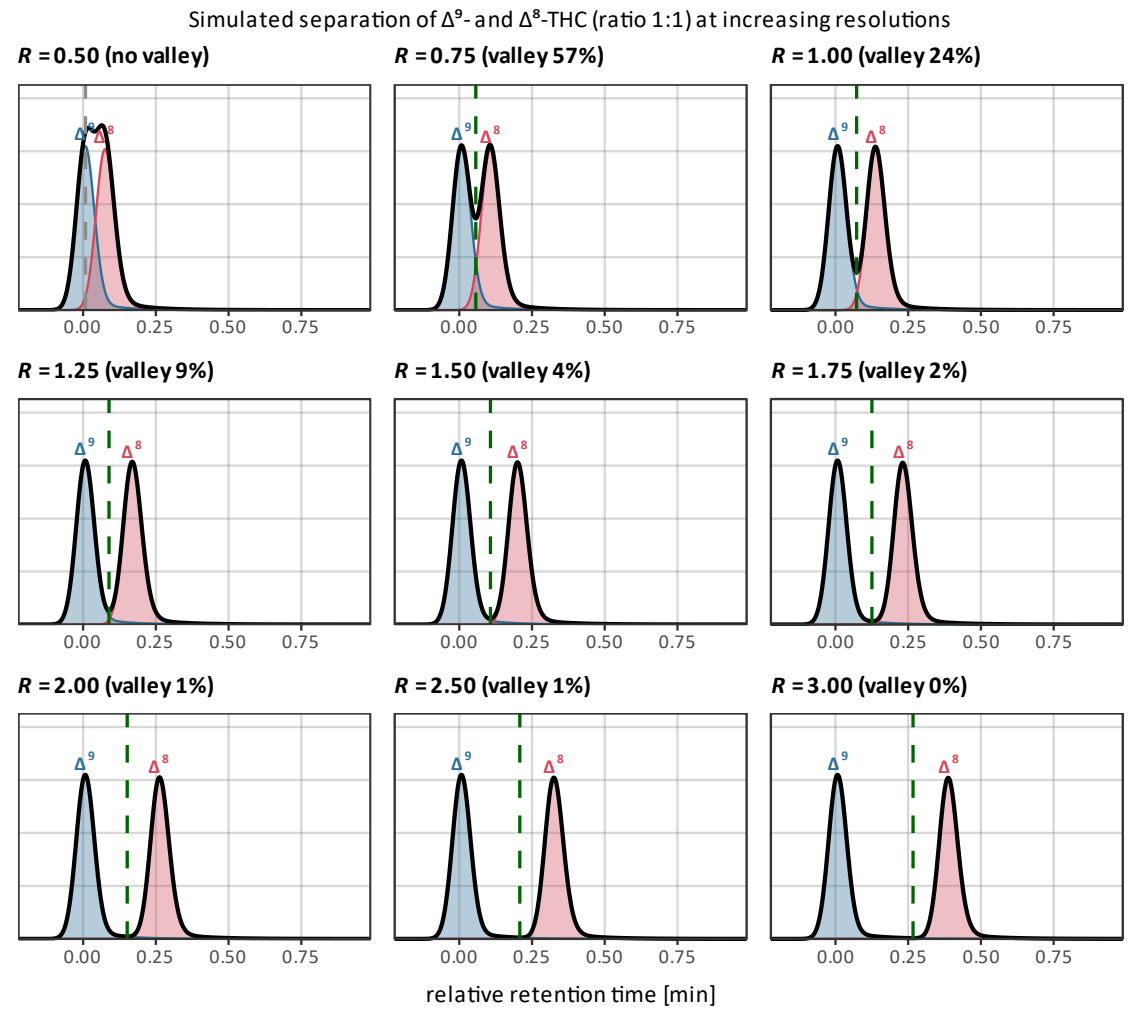


Figure 16: Simulated separation of Δ^9- and Δ^8-THC assuming the peak shapes of the NRL in-house method. Here, the concentration ratio of both compounds is assumed to be 1, putting focus on the idealised, geometrical separation of both peaks.

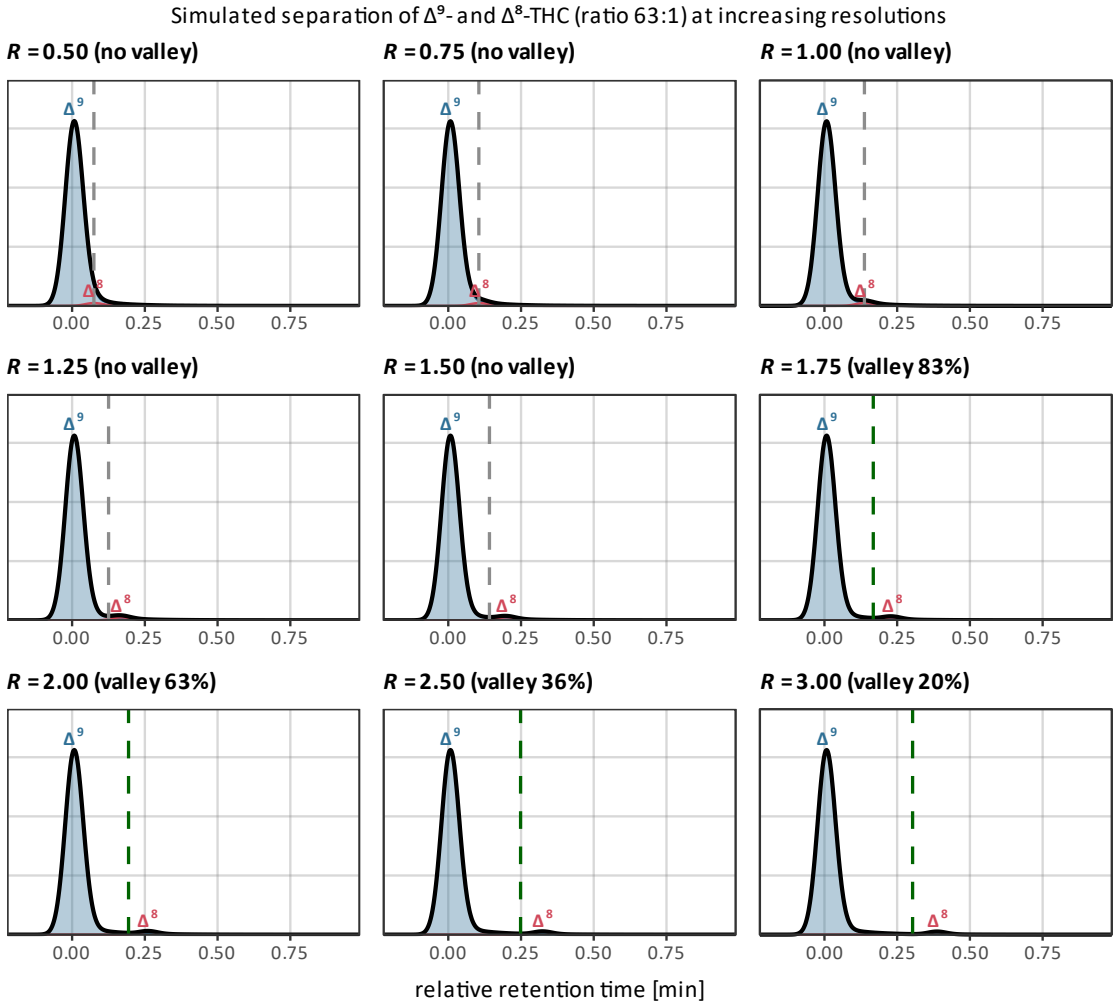


Figure 17: Simulated separation of Δ9- and Δ8-THC assuming the peak shapes of the NRL in-house method. Here, the concentration ratio of both compounds is assumed to be 63, meaning the separation is close to what can be expected from real hemp samples.

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