Data on 'The analysis of delta-9-tetrahydrocannabinol, cannabidiol and cannabinol in oral fluid and selected commercial products by gas chromatography-mass spectrometry' J Jooste, JB Laurens, M Wooding.

1 Method development

1-1. Optimisation of derivatisation agents for Δ^9-THC analyses. Values are response values of analyte (derivisatised with one of three derivatisation agents) to α-5-cholestane.

Derivatisation Agent	AUC of analyte to α-5-cholestane		
None	0.53	0.54	0.44
MSTFA	0.50	0.60	0.60
MTBSTFA	0.95	0.96	0.96

THC Concentration (ng/ml)	Derivatisation agent	
	MSTFA	MTBSTFA
0.5	0.03	0.05
1	0.04	0.18
2	0.10	0.35
4	0.21	0.77
6	0.64	1.27
8	0.71	1.73
10	0.93	2.16

1-2a. Tables. Optimisation of microwave parameters. Values are concentrations calculated from responses of analyte to cholestane internal standard.

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 25 ng/ml				
0.5	25.22	25.75	24.47	21.79	25.12
1	22.54	21.73	26.13	24.71	25.79
2	27.12	18.34	23.44	24.91	26.11
3	32.97	26.60	28.36	24.89	22.92
4	25.14	30.31	19.38	31.55	22.43

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 50 ng/ml				
0.5	44.38	50.42	45.40	50.13	43.12
1	45.96	37.15	39.22	46.45	41.97
2	53.43	40.10	47.55	43.48	53.63
3	46.88	36.85	43.21	49.35	45.11
4	40.38	48.08	47.10	53.64	59.42

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 75 ng/ml				
0.5	62.66	61.40	61.74	57.03	64.48
1	62.70	66.99	64.54	66.23	74.17
2	70.78	96.45	68.45	93.74	74.66
3	72.61	74.21	85.48	86.54	74.45
4	66.51	80.35	75.05	70.17	60.26

Applied Energy (kJ)	THC concentration (ng/ml)								
	Expected concentration of 25 ng/ml			Expected concentration of 50 ng/ml			Expected concentration of 75 ng/ml		
54	22.99	23.02	21.22	47.34	47.01	48.47	75.86	76.76	83.36
90	25.01	26.81	24.61	48.96	48.51	49.84	81.93	70.57	81.82
126	26.91	27.74	26.51	47.15	49.96	47.69	78.94	67.25	68.61
180	26.79	26.99	28.57	53.05	58.96	45.39	70.85	71.52	72.66

1-2b. t-test calculations.

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
25	$\bar{x}_{0.5min}$ = 25 ng/mL	-	-	2.78	-0.76	Accept H_0
	$\bar{x}_{1min}$ = 25 ng/mL			2.78	-0.93	Accept H_0
	$\bar{x}_{2min}$ = 25 ng/mL			2.78	-0.66	Accept H_0
	$\bar{x}_{3min}$ = 25 ng/mL	-	-	2.78	1.25	Accept H_0
	$\bar{x}_{4min}$ = 25 ng/mL			2.78	0.33	Accept H_0
50	$\bar{x}_{0.5min}$ = 50 ng/mL	-	-	2.78	-2.20	Accept H_0
	$\bar{x}_{1min}$ = 50 ng/mL			2.78	-4.30	Reject H_0
	$\bar{x}_{2min}$ = 50 ng/mL			2.78	-0.88	Accept H_0
	$\bar{x}_{3min}$ = 50 ng/mL			2.78	-2.70	Accept H_0
	$\bar{x}_{4min}$ = 50 ng/mL			2.78	-0.09	Accept H_0
75	$\bar{x}_{0.5min}$ = 75 ng/mL			2.78	-11.00	Reject H_0
	$\bar{x}_{1min}$ = 75 ng/mL			2.78	-4.13	Reject H_0
	$\bar{x}_{2min}$ = 75 ng/mL			2.78	0.98	Accept H_0
	$\bar{x}_{3min}$ = 75 ng/mL	-	-	2.78	1.21	Accept H_0
75	$\bar{x}_{4min}$ = 75 ng/mL			2.78	-1.31	Accept H_0
25	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	1.24	2.31	1.37	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	1.82	2.31	0.64	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	1.82	2.31	0.48	Accept H_0
50	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	1.60	2.31	0.98	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	1.44	2.31	0.50	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	2.30	2.31	1.41	Accept H_0

75	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	3.85	2.31	0.32	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	2.94	2.31	1.51	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	1.31	2.31	1.79	Accept H_0

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
25	$\bar{x}_{54kJ}$ = 25 ng/mL	-	-	4.30	-4.36	Reject H_0
	$\bar{x}_{90kJ}$ = 25 ng/mL			4.30	0.71	Accept H_0
	$\bar{x}_{126kJ}$ = 25 ng/mL			4.30	5.75	Reject H_0
	$\bar{x}_{180kJ}$ = 25 ng/mL	-	-	4.30	3.96	Accept H_0
50	$\bar{x}_{54kJ}$ = 50 ng/mL	-	-	4.30	-5.40	Reject H_0
	$\bar{x}_{90kJ}$ = 50 ng/mL			4.30	-2.29	Accept H_0
	$\bar{x}_{126kJ}$ = 50 ng/mL			4.30	-2.01	Accept H_0
	$\bar{x}_{180kJ}$ = 50 ng/mL			4.30	0.63	Accept H_0
75	$\bar{x}_{54kJ}$ = 75 ng/mL			4.30	1.55	Accept H_0
	$\bar{x}_{90kJ}$ = 75 ng/mL			4.30	0.82	Accept H_0
	$\bar{x}_{126kJ}$ = 75 ng/mL			4.30	-0.92	Accept H_0
	$\bar{x}_{180kJ}$ = 75 ng/mL	-	-	4.30	-6.32	Reject H_0
25	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	1.28	2.78	3.40	Reject H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	2.75	2.78	6.66	Reject H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	1.12	2.78	-6.14	Reject H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	3.52	2.78	-2.07	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	1.43	2.78	-2.24	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	2.45	2.78	-0.59	Accept H_0
50	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	1.29	2.78	-2.54	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	3.79	2.78	-0.68	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	78.78	4.30	-1.23	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	0.20	2.78	0.89	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	101.54	4.30	-0.85	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	20.79	4.30	-1.04	Accept H_0

75	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	5.08	2.78	1.18	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	4.55	2.78	1.16	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	20.19	4.30	2.88	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	1.12	2.78	1.81	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	51.29	4.30	1.69	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	49.19	4.30	-0.02	Accept H_0

1-3. Optimisation of microwave parameters. Values are concentrations calculated from responses of analyte d_3-THC internal standard:

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 25 ng/ml				
0.5	29.05	29.09	27.80	29.64	29.57
1	27.76	27.68	28.53	27.68	31.21
2	23.86	24.36	25.04	25.39	27.08
3	25.23	25.42	26.01	25.65	28.18
4	25.90	25.36	28.94	25.59	26.76

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 50 ng/ml				
0.5	51.28	54.76	51.34	50.64	50.86
1	50.11	49.66	48.82	48.37	50.95
2	50.84	44.61	49.11	49.87	47.43
3	49.96	48.48	50.11	51.24	50.26
4	46.40	46.96	45.29	52.91	48.70

Time (min)	THC Concentration (ng/ml)				
	Expected concentration of 75 ng/ml				
0.5	70.82	71.01	69.13	69.20	70.28
1	68.12	73.42	69.93	70.23	74.90
2	73.58	78.67	78.74	74.49	73.23
3	70.11	73.40	75.32	76.63	76.16
4	67.33	74.41	70.22	74.56	74.97

Applied Energy (kJ)	THC concentration (ng/ml)								
	Expected concentration of 25 ng/ml			Expected concentration of 50 ng/ml			Expected concentration of 75 ng/ml		
54	25.72	26.63	23.63	48.35	48.03	49.48	73.79	75.84	75.76
90	25.81	26.59	25.99	50.04	48.72	49.01	71.63	71.71	76.21
126	24.53	25.31	25.92	50.48	50.32	51.78	79.30	74.36	77.21
180	25.85	25.48	28.02	52.90	54.14	49.86	69.98	73.27	75.20

1-3b. t-test calculations

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
25	$\bar{x}_{0.5min}$ = 25 ng/mL	-	-	2.78	12.17	Reject H_0
	$\bar{x}_{1min}$ = 25 ng/mL			2.78	5.27	Reject H_0
	$\bar{x}_{2min}$ = 25 ng/mL			2.78	0.27	Accept H_0
	$\bar{x}_{3min}$ = 25 ng/mL	-	-	2.78	2.05	Accept H_0
	$\bar{x}_{4min}$ = 25 ng/mL			2.78	2.32	Accept H_0
50	$\bar{x}_{0.5min}$ = 50 ng/mL	-	-	2.78	2.34	Accept H_0
	$\bar{x}_{1min}$ = 50 ng/mL			2.78	-0.91	Accept H_0
	$\bar{x}_{2min}$ = 50 ng/mL			2.78	-1.49	Accept H_0
	$\bar{x}_{3min}$ = 50 ng/mL			2.78	0.02	Accept H_0
	$\bar{x}_{4min}$ = 50 ng/mL			2.78	-1.46	Accept H_0

75	$\bar{x}_{0.5min}$ = 75 ng/mL			2.78	-12.43	Reject H_0
	$\bar{x}_{1min}$ = 75 ng/mL			2.78	-2.97	Reject H_0
	$\bar{x}_{2min}$ = 75 ng/mL			2.78	0.61	Accept H_0
	$\bar{x}_{3min}$ = 75 ng/mL	-	-	2.78	-0.57	Accept H_0
	$\bar{x}_{4min}$ = 75 ng/mL			2.78	-1.79	Accept H_0
25	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	2.72	2.31	-1.49	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	1.40	2.31	-1.60	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	3.81	2.31	-0.55	Accept H_0
50	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	6.12	2.31	-1.39	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	1.48	2.31	0.18	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	9.07	2.57	1.40	Accept H_0
75	$\bar{x}_{2min} = \bar{x}_{3min}$	6.39	1.22	2.31	1.61	Accept H_0
	$\bar{x}_{2min} = \bar{x}_{4min}$	6.39	1.52	2.31	1.77	Accept H_0
	$\bar{x}_{3min} = \bar{x}_{4min}$	6.39	1.85	2.31	3.26	Reject H_0

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
25	$\bar{x}_{54kJ}$ = 25 ng/mL	-	-	4.30	0.36	Accept H_0
	$\bar{x}_{90kJ}$ = 25 ng/mL			4.30	4.77	Reject H_0
	$\bar{x}_{126kJ}$ = 25 ng/mL			4.30	0.62	Accept H_0
	$\bar{x}_{180kJ}$ = 25 ng/mL	-	-	4.30	2.04	Accept H_0
50	$\bar{x}_{54kJ}$ = 50 ng/mL	-	-	4.30	-3.15	Accept H_0
	$\bar{x}_{90kJ}$ = 50 ng/mL			4.30	-1.86	Accept H_0
	$\bar{x}_{126kJ}$ = 50 ng/mL			4.30	1.86	Accept H_0
	$\bar{x}_{180kJ}$ = 50 ng/mL			4.30	1.92	Accept H_0
75	$\bar{x}_{54kJ}$ = 75 ng/mL			4.30	0.19	Accept H_0
	$\bar{x}_{90kJ}$ = 75 ng/mL			4.30	-1.20	Accept H_0
	$\bar{x}_{126kJ}$ = 75 ng/mL			4.30	1.37	Accept H_0
	$\bar{x}_{180kJ}$ = 75 ng/mL	-	-	4.30	-1.35	Accept H_0

25	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	13.85	2.78	-0.88	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	4.86	2.78	0.08	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	1.25	2.78	-0.95	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	2.85	2.78	1.88	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	11.06	2.78	-0.38	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	3.88	2.78	-1.35	Accept H_0
50	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	1.23	2.78	-1.07	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	1.09	2.78	-3.51	Reject H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	8.33	2.78	-2.73	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	1.34	2.78	-2.63	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	10.22	2.78	-2.28	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	7.63	2.78	-1.06	Accept H_0
75	$\bar{x}_{54\ kJ} = \bar{x}_{90\ kJ}$	19.00	5.08	2.78	1.18	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{126\ kJ}$	19.00	4.55	2.78	-1.16	Accept H_0
	$\bar{x}_{54\ kJ} = \bar{x}_{180\ kJ}$	19.00	5.15	2.78	1.39	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{126\ kJ}$	19.00	1.12	2.78	-1.81	Accept H_0
	$\bar{x}_{90\ kJ} = \bar{x}_{180\ kJ}$	19.00	1.01	2.78	0.17	Accept H_0
	$\bar{x}_{126\ kJ} = \bar{x}_{180\ kJ}$	19.00	1.13	2.78	1.98	Accept H_0

1-4. Comparison of reconstitutions inside two different vial types. Values are ratios of responses of analyte to cholestane internal standard

THC concentration (ng/ml)	Normal vial insert	Micro vial insert
0.5	0.025	0.027
1.25	0.201	0.179
2.5	0.416	0.376
5	0.877	0.814
6.25	1.457	1.332
7.5	2.031	1.891
10	2.586	2.500

1-5. Optimisation of extraction procedures for THC and CBD. Values are percentage recoveries calculated using expected concentrations and responses of analyte to d_3-THC external standard

THC

pH	Recovery (%)			Average Recovery (%)	Standard deviation (%)
2	64.9	67.32	57.02	63.77	6.26
5	69.82	86.84	76.42	73.83	3.52
8	78.78	90.12	83.9	80.16	3.28
11	100.55	105.04	91.5	96.57	4.62

CBD

pH	Recovery (%)			Average Recovery (%)	Standard deviation (%)
2	66.12	58.01	77.82	67.32	9.96
5	80.89	91.04	88.59	86.84	5.3
8	89.43	99.06	81.86	90.12	8.62
11	107.94	102.13	--	105.04	4.11

1-5b. t-test calculations

THC:

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
10	$\bar{x}_{pH\,2}$ = 10 ng/mL			4.303	-10.03	Reject H_0
	$\bar{x}_{pH\,5}$ = 10 ng/mL			4.303	-12.88	Reject H_0
	$\bar{x}_{pH\,8}$ = 10 ng/mL			4.303	-10.48	Reject H_0
	$\bar{x}_{pH\,11}$ = 10 ng/mL			4.303	-1.77	Accept H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,5}$	19.00	3.16	2.78	-2.43	Accept H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,8}$	19.00	3.64	2.78	-4.02	Reject H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,11}$	19.00	2.07	3.18	-5.93	Reject H_0
	$\bar{x}_{pH\,5} = \bar{x}_{pH\,8}$	19.00	1.15	2.78	-2.28	Accept H_0
	$\bar{x}_{pH\,5} = \bar{x}_{pH\,11}$	19.00	1.53	3.18	-5.96	Reject H_0
	$\bar{x}_{pH\,8} = \bar{x}_{pH\,11}$	19.00	1.76	3.18	-4.30	Reject H_0

CBD:

Concentration (ng/mL)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
10	$\bar{x}_{pH\,2}$ = 10 ng/mL			4.30	-5.68	Reject H_0
	$\bar{x}_{pH\,5}$ = 10 ng/mL			4.30	-1.98	Accept H_0
	$\bar{x}_{pH\,8}$ = 10 ng/mL			4.30	-4.30	Accept H_0
	$\bar{x}_{pH\,11}$ = 10 ng/mL			4.30	2.12	Accept H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,5}$	19	3.54	2.78	-3.00	Reject H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,8}$	19	1.33	2.78	-3.00	Reject H_0
	$\bar{x}_{pH\,2} = \bar{x}_{pH\,11}$	19	5.88	3.18	-4.88	Reject H_0
	$\bar{x}_{pH\,5} = \bar{x}_{pH\,8}$	19	2.65	2.78	-0.56	Accept H_0
	$\bar{x}_{pH\,5} = \bar{x}_{pH\,11}$	19	1.66	3.18	-4.04	Reject H_0
	$\bar{x}_{pH\,8} = \bar{x}_{pH\,11}$	19	4.40	3.18	-2.20	Accept H_0

1-6. Optimisation of the number of consecutive extractions. Values are concentrations calculated from responses of analyte to cholestane internal standard

Consecutive extractions	THC concentration (ng/ml)				
1	28.88	33.50	28.20	27.40	29.34
2	42.43	33.42	33.83	31.04	29.82
3	33.30	30.46	33.00	35.26	31.97

1-7. Investigation into the stability of THC stock solutions when prepared within a methanol or acetonitrile solvent. *Values are concentrations calculated from responses of analyte to cholestane internal standard*

Methanol stock solutions

Container Material	Sample	t (days)				
		0	1	3	5	8
Untreated glass	1	6.10	4.52	4.58	2.46	1.95
	2	6.65	5.76	5.33	3.12	2.12
	3	6.93	6.38	5.35	3.15	2.17
	4	6.41	5.91	5.83	3.07	2.60
	5	7.06	6.09	5.82	3.11	2.62

Silanised glass	1	4.90	5.31	5.25	2.67	1.97
	2	5.88	5.88	5.57	3.03	2.68
	3	5.87	6.26	5.83	3.04	2.61
	4	5.99	6.64	6.08	3.17	2.54
	5	6.32	6.32	5.34	3.26	2.57

Acetonitrile stock solutions

Container Material	Sample	t (days)				
		0	1	3	5	8
Untreated glass	1	3.83	4.36	3.40	4.52	3.88
	2	5.80	6.22	4.88	5.50	5.30
	3	5.94	5.82	5.61	5.64	5.31
	4	5.72	6.61	5.23	6.22	5.12
	5	6.11	6.33	5.37	5.72	5.20
Silanised glass	1	4.90	4.72	3.77	4.31	3.38
	2	5.84	6.25	5.37	5.97	4.96
	3	5.73	6.85	5.38	5.65	5.25
	4	5.64	6.33	5.20	5.76	5.27
	5	5.72	6.49	5.09	5.99	5.10

1-7b. t-tests calculations

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Untreated glass	$\bar{x}_{D0} = \bar{x}_{D1}$	6.39	3.40	2.31	2.47	Reject H_0
	$\bar{x}_{D0} = \bar{x}_{D3}$	6.39	1.71	2.31	4.37	Reject H_0
	$\bar{x}_{D0} = \bar{x}_{D5}$	6.39	1.74	2.31	16.76	Reject H_0
	$\bar{x}_{D0} = \bar{x}_{D8}$	6.39	1.65	2.31	19.73	Reject H_0
Silanised glass	$\bar{x}_{D0} = \bar{x}_{D1}$	6.39	1.09	2.31	-0.89	Accept H_0
	$\bar{x}_{D0} = \bar{x}_{D3}$	6.39	2.37	2.31	0.62	Accept H_0
	$\bar{x}_{D0} = \bar{x}_{D5}$	6.39	5.65	2.31	10.68	Reject H_0
	$\bar{x}_{D0} = \bar{x}_{D8}$	6.39	3.42	2.31	12.28	Reject H_0
	$\bar{x}_{N0} = \bar{x}_{S0}$	6.39	6.07	2.31	-0.19	Accept H_0
	$\bar{x}_{N3} = \bar{x}_{S3}$	6.39	1.67	2.31	-0.13	Accept H_0
	$\bar{x}_{N8} = \bar{x}_{S8}$	6.39	1.71	2.31	0.38	Accept H_0
	$\bar{x}_{N11} = \bar{x}_{S11}$	6.39	1.06	2.31	0.09	Accept H_0

2a. Method validation in aqueous matrix. Values are concentrations calculated from responses of analyte to deuterated internal standard

THC Concentration	Relative response of analyte to d_3-THC					
2.5	0.63	0.62	0.59	0.67	0.55	0.55
5	1.22	1.09	1.04	1.22	1.12	1.17
6.25	1.59	1.27	1.31	1.39	1.37	1.37
7.5	1.83	1.61	1.57	1.77	1.63	1.64
10	2.34	1.97	1.91	2.32	2.15	2.06

CBD Concentration	Relative response of analyte to d_3-CBD			
125	0.22	0.20	0.21	0.30
250	0.40	0.43	0.42	0.57
500	0.79	0.80	0.76	1.06
625	0.97	0.98	1.07	1.10
750	1.11	1.15	1.17	1.28
1000	1.43	1.57	1.56	1.77

Sample	THC Concentration (ng/ml)					
	2.5	2.5*	5.0	5.0*	7.5	7.5*
1	2.10	2.31	4.75	4.64	8.51	8.19
2	2.18	2.21	5.20	5.06	7.85	7.57
3	2.70	2.70	5.91	5.73	8.24	7.93
4	2.45	2.47	5.34	5.20	7.57	7.30
5	2.45	2.47	5.11	4.98	7.50	7.24
6	2.45	2.46	5.06	4.92	7.76	7.48
7	2.55	2.56	5.68	5.51	8.04	7.74
8	2.76	2.76	5.14	5.00	8.91	8.57
9	2.31	2.33	5.18	5.04	7.84	7.56
10	2.61	2.61	5.30	5.15	7.68	7.41
11	2.75	2.75	4.59	4.49	7.93	7.64
12	2.21	2.23	4.85	4.73	6.95	6.71
13	2.63	2.63	5.15	5.01	7.40	7.14
14	2.47	2.49	4.96	4.84	7.16	6.92
15	2.70	2.70	5.41	5.26	8.24	7.93
16	2.45	2.47	5.11	4.98	7.50	7.24
17	2.36	2.38	5.36	5.21	7.67	7.39
18	2.69	2.69	5.03	4.91	7.80	7.52
19	2.85	2.85	5.21	5.07	7.24	6.99
20	1.94	1.98	4.95	4.83	7.37	7.11
21	2.26	2.28	4.83	4.71	8.26	7.95
22	2.70	2.70	5.15	5.01	7.59	7.32

*With bias correction

Replicates	THC Concentration (ng/ml)					
	2.5	2.5*	5.0	5.0*	7.5	7.5*
1	2.20	2.23	4.96	4.83	8.09	7.80
2	2.13	2.16	4.67	4.56	8.11	7.82
3	2.19	2.21	4.75	4.63	8.97	8.62
4	2.41	2.43	5.56	5.40	7.86	7.58
5	2.39	2.41	4.73	4.62	7.88	7.60
average	2.26	2.29	4.93	4.81	8.18	7.88
std	0.13	0.122	0.36	0.345	0.45	0.429
%RSD	5.72	5.35	7.40	7.17	5.55	5.45

*With bias correction

Sample	CBD Concentration (ng/ml)		
	250	500	750
1	234.74	482.47	739.27
2	269.46	478.19	732.75
3	219.37	504.86	736.02
4	236.02	486.13	760.48
5	207.29	472.94	749.61
6	239.44	513.33	743.89
7	269.22	514.97	748.55
8	262.05	485.89	759.54
9	285.88	578.28	--

Replicates	CBD Concentration (ng/ml)		
	250	500	750
1	235	482	739
2	269	478	733
3	219	505	736
4	236	486	760
5	207	473	--
average	233	485	742
std	23	12	13
%RSD	10.02	2.51	1.69

2b. Method validation in oral fluid matrix

THC Concentration	Relative response of analyte to d_3-THC				
2.5	0.64	0.51	0.48	0.54	0.53
5	1.26	1.12	0.95	1.07	1.06
6.25	1.30	1.37	1.20	1.20	1.35
7.5	1.53	1.82	1.49	1.62	1.68
10	2.00	2.14	2.12	1.98	1.97

CBD Concentration	Relative response of analyte to d_3-CBD				
12.5	0.16	0.10	0.11	0.10	0.13
25	0.31	0.19	0.20	0.19	0.21
50	0.54	0.36	0.39	0.40	0.40
62.5	0.58	0.49	0.52	0.49	0.48
75	0.71	0.63	0.61	0.59	0.56
100	0.82	0.81	0.86	0.81	0.76

Sample	THC Concentration (ng/ml)		
	2.5	5.0	7.5
1	2.84	5.63	8.56
2	2.99	5.69	8.59
3	2.54	4.15	7.02
4	2.77	4.33	7.15
5	2.62	4.60	7.95
6	2.75	5.04	8.15
7	2.74	4.69	8.21
8	2.14	4.84	9.07
9	2.32	5.79	7.32
10	2.31	5.03	8.16
11	2.57	4.78	7.34
12	2.55	5.19	7.97
13	2.36	5.01	8.07
14	2.43	4.72	7.91
15	2.61	5.15	7.26
16	2.65	5.33	7.09
17	2.50	5.10	7.20
18	2.58	4.96	7.66
19	2.60	4.92	7.41

Replicates	THC Concentration (ng/ml)		
	2.5	5.0	7.5
1	2.54	4.15	7.02
2	2.77	4.33	7.15
3	2.62	4.60	7.95
4	2.75	5.04	8.15
5	2.74	4.69	8.21
average	2.680	4.563	7.697
std	0.099	0.344	0.568
%RSD	3.70	7.54	7.38

Sample	CBD Concentration (ng/ml)		
	25	50	75
1	31.82	44.15	69.12
2	25.52	46.07	77.83
3	25.10	47.48	73.27
4	25.04	50.19	73.70
5	24.30	50.77	72.89
6	25.86	56.31	78.61
7	23.63	44.95	77.90
8	24.14	47.06	73.47
9	24.29	49.76	73.56
10	24.44	50.91	73.03

Replicates	CBD Concentration (ng/ml)		
	25	50	75
1	24.29	44.15	69.12
2	25.52	46.07	77.83
3	25.10	47.48	73.27
4	25.04	50.19	73.70
5	24.30	50.77	72.89
average	24.9	47.7	73.4
std	0.5	2.8	3.1
%RSD	2.17	5.83	4.22

2c. ANOVA tests

Matrix: Cannabinoid	Null Hypothesis (H_0)	Total degrees of freedom	F-critical	F-statistic	Outcome
Aqueous: THC	Between group variation = Within group variation	53	2.30	412.51	Reject H_0
Aqueous: CBD		23	2.77	100.02	Reject H_0
Oral Fluid: THC		24	2.87	174.77	Reject H_0
Oral fluid: CBD		34	2.45	282.46	Reject H_0

Matrix: Cannabinoid	Null Hypothesis (H_0)	Total degrees of freedom	F-critical	F-statistic	Outcome
Aqueous: THC	Between group variation = Within group variation	14	3.89	370.28	Reject H_0
Aqueous: CBD		25	3.42	835.06	Reject H_0
Oral Fluid: THC		56	3.17	688.86	Reject H_0
Oral fluid: CBD		29	3.35	665.45	Reject H_0

Internal standard: Vial type	Null Hypothesis (H_0)	Total degrees of freedom	F-critical	F-statistic	Outcome
d_3-THC: Untreated	Between group variation = Within group variation	53	2.15	0.29	Accept H_0
d_3-THC: Silanised		53	2.15	0.56	Accept H_0
Cholestane: Untreated		53	2.15	1.02	Accept H_0
Cholestane: Silanised		53	2.15	1.17	Accept H_0

3. Optimisation of pre-analytical parameters in aqueous matrix. Values are concentrations calculated from responses of analyte to deuterated internal standard

ACN: water	Time (days)																	
	0			1			2			3			4			7		
0:01	3.23	2.55	2.90	3.15	3.05	2.60	4.78	5.00	5.28	0.85	1.12	1.47	5.08	3.48	1.55			
1:19	4.64	5.77	9.05	3.41	4.47	3.59	3.02	2.88	2.57	3.18	3.56	3.27	2.16	7.68	3.38	1.55	3.12	3.32
1:09	9.56	8.79	8.78	7.45	7.53	6.51	8.01	7.79	6.67	2.35	5.67	5.42	9.92	10.24	9.07	5.22	6.97	6.32
1:04	10.55	10.78	11.06	10.91	10.30	10.80	11.41	10.94	9.66	9.64	9.67	10.28	10.58	10.42	9.52	9.45	10.20	9.82

3-2a. Container material: Analyses without vortexing containers

Container material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	4.13	4.42	3.39	2.81	3.67
	1	1.01	2.53	1.22	0.78	1.43
	2	0.37	1.32	0.73	0.42	0.90
Silanised glass	0	5.14	4.25	4.87	5.31	5.47
	1	4.00	3.85	4.27	3.69	3.82
	2	3.69	2.99	3.28	3.16	3.62
	3	1.90	2.39	2.26	2.46	2.58

3-2b. Analyses with vortexing containers

Container material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	4.98	5.65	6.22	6.33	6.15
	1	2.18	1.54	1.68	1.70	1.39
	2	2.09	2.00	2.89	2.56	1.82
	3	1.39	1.42	2.02	2.07	1.57
	4	1.32	<LOQ	1.60	1.53	1.32
Silanised glass	0	3.72	5.67	3.82	5.57	6.13
	1	1.03	1.99	1.75	1.85	1.96
	2	1.33	2.31	2.02	2.23	1.96
	3	<LOQ	1.66	1.83	1.88	1.65
	4	<LOQ	1.35	<LOQ	<LOQ	<LOQ

3-3. Analyses at 2^{o}C.

Container material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	4.17	2.90	3.46	<LOQ	4.74
	1	3.77	1.63	1.64	2.25	1.77
	2	1.35	1.28	1.57	1.47	<LOQ
	3	1.68	1.89	1.91	2.83	2.44
	4	3.73	2.57	2.99	2.31	4.56
Silanised glass	0	3.21	<LOQ	2.39	3.36	3.38
	1	2.27	1.93	1.28	2.08	1.93
	2	1.38	1.54	<LOQ	1.31	1.32
	3	2.47	2.16	1.46	2.29	2.46
	4	2.13	1.94	1.61	1.55	2.26

3-4. Analyses at 40°C.

Container material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	4.17	2.90	3.46	<LOQ	4.74
	1	3.77	1.63	1.64	2.25	1.77
	2	1.35	1.28	1.57	1.47	<LOQ
	3	1.68	1.89	1.91	2.83	2.44
	4	3.73	2.57	2.99	2.31	4.56
Silanised glass	0	3.21	<LOQ	2.39	3.36	3.38
	1	2.27	1.93	1.28	2.08	1.93
	2	1.38	1.54	<LOQ	1.31	1.32
	3	2.47	2.16	1.46	2.29	2.46
	4	2.13	1.94	1.61	1.55	2.26

3-5. Analyses at different pH levels

pH level	Time Stored (Days)	THC concentration (ng/ml)				
5	0	6.67	7.35	8.69	8.12	9.49
	1	3.99	3.33	5.52	5.29	3.96
	2	5.43	3.21	3.86	3.06	4.16
	3	3.14	2.58	1.60	2.87	3.11
	4	2.28	3.57	1.88	2.33	3.09
8	0	8.37	7.26	8.65	8.77	7.97
	1	9.45	7.63	5.00	8.44	9.09
	2	7.43	8.35	6.66	7.12	6.98
	3	6.39	4.95	2.46	4.59	5.02
	4	3.75	3.21	2.58	3.35	2.38
11	0	9.12	9.56	10.03	8.42	8.76
	1	7.64	8.67	6.44	7.14	9.70
	2	7.47	7.73	6.83	6.86	6.73
	3	5.38	4.81	5.51	4.90	5.16
	4	4.31	2.80	2.45	3.63	2.58

3b. t-tests calculations

Acetonitrile: water (*v:v*)	Null hypothesis (H_0)	t-critical	t-statistic	Outcome
0:1	D0 = 10 ng/mL	3.182	-47.57	Reject H_0
	D1= 10 ng/mL	2.776	-60.81	Reject H_0
	D2 = 10 ng/mL	2.776	-8.27	Reject H_0
	D4 = 10 ng/mL	3.182	-8.94	Reject H_0
1:19	D0 = 10 ng/mL	4.303	-2.66	Accept H_0
	D1= 10 ng/mL	4.303	-18.79	Reject H_0
	D2 = 10 ng/mL	4.303	-53.46	Reject H_0
	D3 = 10 ng/mL	4.303	-57.11	Reject H_0
	D4 = 10 ng/mL	4.303	-3.34	Accept H_0
	D7 = 10 ng/mL	4.303	-13.11	Reject H_0
1:9	D0 = 10 ng/mL	4.303	-3.72	Accept H_0
	D1= 10 ng/mL	4.303	-8.70	Reject H_0
	D2 = 10 ng/mL	4.303	-6.05	Reject H_0
	D3 = 10 ng/mL	4.303	-5.18	Reject H_0
	D7 = 10 ng/mL	4.303	-7.51	Reject H_0
1:4	D0 = 10 ng/mL	4.303	5.36	Reject H_0
	D1= 10 ng/mL	4.303	3.55	Accept H_0
	D2 = 10 ng/mL	4.303	1.28	Accept H_0
	D4 = 10 ng/mL	4.303	0.54	Accept H_0
	D7 = 10 ng/mL	4.303	-0.82	Accept H_0

Null hypothesis (H_0)	t-critical	t-statistic	Outcome
$\bar{x}_{UG\ 2h} = 5$ ng/mL	2.776	-4.69	Reject H_0
$\bar{x}_{SG\ 2h} = 5$ ng/mL	2.776	0.047	Accept H_0
$\bar{x}_{SG\ \mathrm{D3}} = 0$ ng/mL	2.776	19.95	Reject H_0
$\bar{x}_{SG\ \mathrm{D3}} = 5$ ng/mL	2.776	-23.05	Reject H_0

Time stored (days)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
0	$\bar{x}_{UG} = \bar{x}_{SG}$	6.39	4.03	2.31	1.57	Accept H_0
1	$\bar{x}_{UG} = \bar{x}_{SG}$	6.39	1.80	2.31	-0.08	Accept H_0
2	$\bar{x}_{UG} = \bar{x}_{SG}$	6.39	1.32	2.31	1.15	Accept H_0
3	$\bar{x}_{UG} = \bar{x}_{SG}$	6.39	1.03	2.31	0.38	Accept H_0
4	$\bar{x}_{UG} = \bar{x}_{SG}$	6.39	1.50	2.31	2.15	Accept H_0

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Silanised glass	$\bar{x}_{1a\ D1} = \bar{x}_{1c\ D1}$	6.39	3.24	2.31	10.85	Reject H_0
	$\bar{x}_{1a\ D2} = \bar{x}_{1c\ D2}$	6.39	1.66	2.31	6.31	Reject H_0
	$\bar{x}_{1a\ D3} = \bar{x}_{1c\ D3}$	6.39	1.62	2.31	3.74	Reject H_0
Untreated glass	$\bar{x}_{1a\ D1} = \bar{x}_{1c\ D1}$	6.39	5.24	2.31	-0.93	Accept H_0

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Untreated glass	$\bar{x}_{4b\ D0}$= 5 ng/mL			2.78	-9.68	Reject H_0
	$\bar{x}_{4b\ D1}$= 5 ng/mL			2.78	-37.78	Reject H_0
	$\bar{x}_{4b\ D2}$= 5 ng/mL			2.78	-65.31	Reject H_0
	$\bar{x}_{4b\ D3}$= 5 ng/mL			2.78	-31.44	Reject H_0
	$\bar{x}_{4b\ D4}$= 5 ng/mL			2.78	-93.08	Reject H_0
Silanised glass	$\bar{x}_{4b\ D0}$= 5 ng/mL			2.78	-10.37	Reject H_0
	$\bar{x}_{4b\ D1}$= 5 ng/mL			2.78	-16.27	Reject H_0
	$\bar{x}_{4b\ D2}$= 5 ng/mL			2.78	-30.16	Reject H_0
	$\bar{x}_{4b\ D3}$= 5 ng/mL			2.78	-13.20	Reject H_0
	$\bar{x}_{4b\ D4}$= 5 ng/mL			2.78	-99.04	Reject H_0

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Untreated glass	$\bar{x}_{3b\ D0}$= 5 ng/mL			2.78	-4.31	Reject H_0
	$\bar{x}_{3b\ D1}$= 5 ng/mL			2.78	-2.93	Reject H_0
Silanised glass	$\bar{x}_{3b\ D0}$= 5 ng/mL			2.78	-22.25	Reject H_0
	$\bar{x}_{3b\ D1}$= 5 ng/mL			2.78	-8.09	Reject H_0
	$\bar{x}_{3b\ D2}$= 5 ng/mL			2.78	-18.66	Reject H_0
	$\bar{x}_{3b\ D3}$= 5 ng/mL			2.78	-28.39	Reject H_0
	$\bar{x}_{3b\ D4}$= 5 ng/mL			2.78	-15.24	Reject H_0

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Silanised glass	$\bar{x}_{3b\ D1} = \bar{x}_{1a\ D1}$	6.39	12.65	2.57	1.21	Accept H_0
	$\bar{x}_{3b\ D2} = \bar{x}_{1a\ D2}$	6.39	8.18	2.57	1.59	Accept H_0
	$\bar{x}_{3b\ D3} = \bar{x}_{1a\ D3}$	6.39	2.20	2.31	4.82	Reject H_0
Untreated glass	$\bar{x}_{3b\ D1} = \bar{x}_{1a\ D1}$	6.39	1.73	2.31	-6.36	Reject H_0

Vial Type	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Silanised glass	$\bar{x}_{3a\ D0} = \bar{x}_{1c\ D0}$	6.39	5.68	2.31	-3.12	Reject H_0
	$\bar{x}_{3a\ D1} = \bar{x}_{1c\ D1}$	6.39	1.14	2.31	0.73	Accept H_0
	$\bar{x}_{3a\ D2} = \bar{x}_{1c\ D2}$	6.39	1.71	2.31	-3.27	Reject H_0
	$\bar{x}_{3a\ D3} = \bar{x}_{1c\ D3}$	6.39	1.57	2.31	2.35	Reject H_0
	$\bar{x}_{3a\ D4} = \bar{x}_{1c\ D4}$	6.39	2.43	2.31	4.52	Reject H_0
Untreated glass	$\bar{x}_{3a\ D0} = \bar{x}_{1c\ D0}$	6.39	2.06	2.31	-4.51	Reject H_0
	$\bar{x}_{3a\ D1} = \bar{x}_{1c\ D1}$	6.39	9.28	2.57	1.21	Accept H_0
	$\bar{x}_{3a\ D2} = \bar{x}_{1c\ D2}$	6.39	6.88	2.57	-4.30	Reject H_0
	$\bar{x}_{3a\ D3} = \bar{x}_{1c\ D3}$	6.39	2.13	2.31	1.77	Accept H_0
	$\bar{x}_{3a\ D4} = \bar{x}_{1c\ D4}$	6.39	31.33	2.57	4.41	Reject H_0

Time stored (days)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
0	$\bar{x}_{pH\ 5} = \bar{x}_{pH8}$	6.39	3.25	2.31	0.25	Accept H_0
1	$\bar{x}_{pH\ 5} = \bar{x}_{pH8}$	6.39	3.54	2.31	3.90	Reject H_0
2	$\bar{x}_{pH\ 5} = \bar{x}_{pH8}$	6.39	2.16	2.31	6.58	Reject H_0
3	$\bar{x}_{pH\ 5} = \bar{x}_{pH8}$	6.39	4.97	2.31	2.91	Reject H_0
4	$\bar{x}_{pH\ 5} = \bar{x}_{pH8}$	6.39	1.46	2.31	1.06	Accept H_0
0	$\bar{x}_{pH\ 5} = \bar{x}_{pH11}$	6.39	1.09	2.31	2.46	Reject H_0
1	$\bar{x}_{pH\ 5} = \bar{x}_{pH11}$	6.39	1.91	2.31	0.00	Accept H_0
2	$\bar{x}_{pH\ 5} = \bar{x}_{pH11}$	6.39	2.08	2.31	-0.53	Accept H_0
3	$\bar{x}_{pH\ 5} = \bar{x}_{pH11}$	6.39	22.52	2.57	0.72	Accept H_0
4	$\bar{x}_{pH\ 5} = \bar{x}_{pH11}$	6.39	1.96	2.31	0.23	Accept H_0
0	$\bar{x}_{pH8} = \bar{x}_{pH11}$	6.39	2.99	2.31	1.96	Accept H_0
1	$\bar{x}_{pH8} = \bar{x}_{pH11}$	6.39	1.85	2.31	4.92	Reject H_0
2	$\bar{x}_{pH8} = \bar{x}_{pH11}$	6.39	4.50	2.31	6.79	Reject H_0
3	$\bar{x}_{pH8} = \bar{x}_{pH11}$	6.39	4.53	2.31	7.92	Reject H_0
4	$\bar{x}_{pH8} = \bar{x}_{pH11}$	6.39	1.34	2.31	1.11	Accept H_0

Temperature (°C)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
2	$\bar{x}_{Study\ 3a\ D1} = \bar{x}_{Study\ 5\ D1}$	6.39	26.35	2.57	1.18	Accept H_0
	$\bar{x}_{Study\ 3a\ D1} = \bar{x}_{Study\ 8\ D1}$	6.39	1.80	2.31	-2.21	Accept H_0
	$\bar{x}_{Study\ 5\ D1} = \bar{x}_{Study\ 8\ D1}$	6.39	14.63	2.57	-5.14	Reject H_0
	$\bar{x}_{Study\ 3a\ D2} = \bar{x}_{Study\ 6\ D2}$	6.39	2.93	2.31	2.30	Accept H_0
	$\bar{x}_{Study\ 3a\ D2} = \bar{x}_{Study\ 8\ D2}$	6.39	15.28	2.57	-1.25	Accept H_0
	$\bar{x}_{Study\ 5\ D2} = \bar{x}_{Study\ 8\ D2}$	6.39	44.79	2.57	-1.95	Accept H_0
	$\bar{x}_{Study\ 3a\ D3} = \bar{x}_{Study\ 5\ D3}$	6.39	8.79	2.57	-3.39	Reject H_0
	$\bar{x}_{Study\ 3a\ D3} = \bar{x}_{Study\ 8\ D3}$	6.39	2.56	2.31	-2.38	Reject H_0
	$\bar{x}_{Study\ 5\ D3} = \bar{x}_{Study\ 8\ D3}$	6.39	22.51	2.57	-0.55	Accept H_0
	$\bar{x}_{Study\ 3a\ D4} = \bar{x}_{Study\ 5\ D4}$	6.39	13.60	2.57	-4.57	Reject H_0
	$\bar{x}_{Study\ 3a\ D4} = \bar{x}_{Study\ 8\ D4}$	6.39	6.94	2.57	-5.71	Reject H_0
	$\bar{x}_{Study\ 5\ D4} = \bar{x}_{Study\ 8\ D4}$	6.39	1.96	2.31	-2.92	Reject H_0

25	$\bar{x}_{\text{Study 1c D1}} = \bar{x}_{\text{Study 6 D1}}$	6.39	1.02	2.31	2.08	Accept H_0
	$\bar{x}_{\text{Study 1c D1}} = \bar{x}_{\text{Study 9D1}}$	6.39	12.20	2.57	1.48	Accept H_0
	$\bar{x}_{\text{Study 6 D1}} = \bar{x}_{\text{Study 9 D1}}$	6.39	12.43	2.57	0.67	Accept H_0
	$\bar{x}_{\text{Study 1c D2}} = \bar{x}_{\text{Study 6 D2}}$	6.39	2.05	2.31	-0.15	Accept H_0
	$\bar{x}_{\text{Study 1c D2}} = \bar{x}_{\text{Study 9 D2}}$	6.39	1.45	2.31	-1.20	Accept H_0
	$\bar{x}_{\text{Study 6 D2}} = \bar{x}_{\text{Study 9 D2}}$	6.39	2.98	2.31	-1.22	Accept H_0
	$\bar{x}_{\text{Study 1c D3}} = \bar{x}_{\text{Study 6 D3}}$	6.39	6.32	2.31	2.52	Reject H_0
	$\bar{x}_{\text{Study 1c D3}} = \bar{x}_{\text{Study 9 D3}}$	6.39	4.46	2.31	-0.58	Accept H_0
	$\bar{x}_{\text{Study 6 D3}} = \bar{x}_{\text{Study 9 D3}}$	6.39	28.17	2.57	-1.89	Accept H_0
	$\bar{x}_{\text{Study 1c D4}} = \bar{x}_{\text{Study 6 D4}}$	6.39	2.69	2.31	-2.72	Reject H_0
	$\bar{x}_{\text{Study 1c D4}} = \bar{x}_{\text{Study 9 D4}}$	6.39	31.51	2.57	-0.29	Accept H_0
	$\bar{x}_{\text{Study 6 D4}} = \bar{x}_{\text{Study 9 D4}}$	6.39	84.62	2.57	0.27	Accept H_0
40	$\bar{x}_{\text{Study 4 D1}} = \bar{x}_{\text{Study 7 D1}}$	6.39	7.43	2.57	0.16	Accept H_0
	$\bar{x}_{\text{Study 7 D1}} = \bar{x}_{\text{Study 10 D1}}$	6.39	2.02	2.31	-3.54	Reject H_0
	$\bar{x}_{\text{Study 4 D2}} = \bar{x}_{\text{Study 7 D2}}$	6.39	12.07	2.57	-4.70	Reject H_0
	$\bar{x}_{\text{Study 4 D2}} = \bar{x}_{\text{Study 10 D2}}$	6.39	18.40	2.57	-3.56	Reject H_0
	$\bar{x}_{\text{Study 7 D2}} = \bar{x}_{\text{Study 10 D2}}$	6.39	1.52	2.31	0.24	Accept H_0
	$\bar{x}_{\text{Study 4 D3}} = \bar{x}_{\text{Study 7 D3}}$	6.39	7.20	2.57	-6.68	Reject H_0
	$\bar{x}_{\text{Study 4 D3}} = \bar{x}_{\text{Study 10 D3}}$	6.39	3.75	2.31	-0.59	Accept H_0
	$\bar{x}_{\text{Study 7 D3}} = \bar{x}_{\text{Study 10 D3}}$	6.39	1.92	2.31	10.16	Reject H_0
	$\bar{x}_{\text{Study 7 D4}} = \bar{x}_{\text{Study 10 D4}}$	6.39	1.01	2.31	4.17	Reject H_0

4. Optimisation of pre-analytical parameters in oral fluid matrix. Values are concentrations calculated from responses of analyte to deuterated internal standard

4-1. Analyses at constant and varying surface area to volume ratios.

4-1a. Constant surface area to volume ratios

Vial Material	Time Stored (Days)	THC concentration (ng/ml)				
untreated glass	0	2.20	2.75	3.71	4.13	4.37
	1	4.36	3.89	4.45	5.17	4.67
	2	4.42	4.52	4.69	4.58	4.21
	3	4.11	3.64	4.28	5.33	4.76
	4	2.83	3.64	4.23	4.33	4.85
silanised glass	0	4.38	3.35	3.65	4.34	4.13
	1	4.01	4.45	4.31	4.23	4.43
	2	3.69	3.99	4.53	3.33	3.13
	3	4.15	4.83	5.49	3.77	7.23
	4	4.30	4.37	4.08	4.21	4.34

4-1b. Varying surface area to volume ratios

Vial Material	Vial set	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	Day 0	1	4.88	5.34	5.13	4.93	5.15
		2	5.07	5.18	4.99	5.54	4.88
		3	4.57	5.13	5.00	5.84	5.00
		4	5.08	4.64	4.86	4.61	4.12
	Day 1	2	3.22	3.77	3.49	4.08	3.98
		3	4.95	4.24	4.12	4.63	4.14
		4	3.43	2.77	3.37	4.58	4.55
	Day 2	3	4.42	4.19	5.02	4.17	4.15
		4	3.51	4.87	5.31	4.46	4.61
	Day 3	4	4.12	4.56	5.39	4.71	5.16
Silanised glass	Day 0	1	5.24	4.83	4.83	5.66	5.52
		2	5.34	5.09	4.74	4.79	5.34
		3	4.36	4.89	3.74	5.35	4.34
		4	5.09	5.29	5.01	5.10	4.40

	Day 1	2	3.42	2.97	3.49	3.81	3.35
		3	4.09	4.68	4.95	4.55	4.27
		4	3.97	3.22	4.99	4.51	4.45
	Day 2	3	3.52	4.75	4.30	4.35	3.76
		4	4.23	4.43	4.68	5.12	4.30
	Day 3	4	3.71	5.06	4.74	4.65	4.44

4-2. Analyses without vortexing samples.

Vial Material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	5.03	4.94	4.54	4.52	4.98
	1	5.28	5.32	5.20	5.15	5.14
	2	5.02	4.69	4.54	4.96	5.01
	3	5.62	4.98	5.09	4.02	5.17
	4	5.01	4.77	4.31	4.85	4.82
Silanised glass	0	5.29	4.74	5.07	3.77	5.18
	1	3.44	4.66	4.15	4.20	4.58
	2	4.32	4.70	4.93	4.66	7.38
	3	4.39	5.20	5.03	4.44	4.68
	4	6.19	5.37	4.93	4.73	4.60

4-3. Analyses at 2^{o}C

Vial Material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	3.79	4.30	4.19	4.71	3.97
	1	4.94	5.15	5.39	4.71	4.14
	2	3.87	4.75	4.81	4.47	4.69
	3	5.43	5.23	5.29	5.60	5.78
	4	4.21	4.82	6.99	5.72	5.52

4-4. Analyses at 40°C

Vial Material	Time Stored (Days)	THC concentration (ng/ml)				
Untreated glass	0	3.27	4.69	4.90	4.66	4.78
	1	3.97	3.65	4.75	3.84	4.94
	2	3.39	3.33	3.35	2.78	3.61
	3	3.89	3.91	3.98	3.90	3.84
	4	3.62	3.91	3.87	3.82	3.65

4-5. Analyses at different pH levels

pH Level	Time Stored (Days)	THC concentration (ng/ml)				
2	0	11.08	10.23	9.74	11.24	10.38
	1	7.84	8.51	8.27	9.45	10.24
	2	6.87	7.63	6.47	7.23	6.53
	3	6.18	6.01	5.89	6.54	6.20
	4	6.04	4.94	5.81	6.33	5.41
	11	3.08	3.04	3.24	3.62	2.54
5	0	11.02	10.74	11.30	10.25	10.66
	1	9.74	10.77	10.47	10.53	10.91
	2	9.16	9.46	10.44	10.42	8.92
	3	8.69	9.71	9.59	9.10	8.42
	4	9.16	9.08	9.65	9.01	7.80
	11	4.36	6.00	5.48	4.66	5.49
8	0	10.05	10.42	10.58	10.17	10.29
	1	9.48	11.32	11.61	12.56	10.82
	2	10.93	10.55	10.77	9.98	8.74
	3	10.35	11.28	11.47	10.58	10.62
	4	9.78	10.53	10.26	10.56	10.63
	11	5.39	6.31	6.16	6.20	6.27
11	0	10.48	10.71	10.34	10.80	10.03
	1	9.85	10.27	10.45	10.45	10.81
	2	9.67	8.84	10.86	10.25	10.78
	3	7.24	6.80	7.93	7.45	6.49
	4	4.35	3.41	5.10	3.96	5.34
	11	0.97	0.22	0.98	0.86	0.67

4b. t-test calculations.

Vial Type	Null hypothesis (H_0)	t-critical	t-statistic	Outcome
Untreated glass	$\bar{x}_{Constant\ D0} = 5$ ng/mL	2.78	-0.82	Accept H_0
	$\bar{x}_{Constant\ D1} = 5$ ng/mL	2.78	-0.47	Accept H_0
	$\bar{x}_{Constant\ D2} = 5$ ng/mL	2.78	-0.85	Accept H_0
	$\bar{x}_{Constant\ D3} = 5$ ng/mL	2.78	-0.42	Accept H_0
	$\bar{x}_{Constant\ D4} = 5$ ng/mL	2.78	-0.63	Accept H_0
	$\bar{x}_{Variable\ D0-1} = 5$ ng/mL	2.78	-0.82	Accept H_0
	$\bar{x}_{Variable\ D0-2} = 5$ ng/mL	2.78	0.14	Accept H_0
	$\bar{x}_{Variable\ D0-3} = 5$ ng/mL	2.78	0.19	Accept H_0
	$\bar{x}_{Variable\ D0-4} = 5$ ng/mL	2.78	0.10	Accept H_0
	$\bar{x}_{Variable\ D1-2} = 5$ ng/mL	2.78	-1.50	Accept H_0
	$\bar{x}_{Variable\ D1-3} = 5$ ng/mL	2.78	-0.67	Accept H_0
	$\bar{x}_{Variable\ D1-4} = 5$ ng/mL	2.78	-0.76	Accept H_0
	$\bar{x}_{Variable\ D2-3} = 5$ ng/mL	2.78	-0.69	Accept H_0
	$\bar{x}_{Variable\ D2-4} = 5$ ng/mL	2.78	-0.32	Accept H_0
	$\bar{x}_{Variable\ D3-4} = 5$ ng/mL	2.78	-0.63	Accept H_0
Silanised glass	$\bar{x}_{Constant\ D0} = 5$ ng/mL	2.78	-1.01	Accept H_0
	$\bar{x}_{Constant\ D1} = 5$ ng/mL	2.78	-1.18	Accept H_0
	$\bar{x}_{Constant\ D2} = 5$ ng/mL	2.78	-1.04	Accept H_0
	$\bar{x}_{Constant\ D3} = 5$ ng/mL	2.78	0.03	Accept H_0
	$\bar{x}_{Constant\ D4} = 5$ ng/mL	2.78	-1.37	Accept H_0
	$\bar{x}_{Variable\ D0-1} = 5$ ng/mL	2.78	-1.01	Accept H_0
	$\bar{x}_{Variable\ D0-2} = 5$ ng/mL	2.78	0.23	Accept H_0
	$\bar{x}_{Variable\ D0-3} = 5$ ng/mL	2.78	0.08	Accept H_0
	$\bar{x}_{Variable\ D0-4} = 5$ ng/mL	2.78	-0.35	Accept H_0
	$\bar{x}_{Variable\ D1-2} = 5$ ng/mL	2.78	-2.04	Accept H_0
	$\bar{x}_{Variable\ D1-3} = 5$ ng/mL	2.78	-0.59	Accept H_0
	$\bar{x}_{Variable\ D1-4} = 5$ ng/mL	2.78	-0.54	Accept H_0
	$\bar{x}_{Variable\ D2-3} = 5$ ng/mL	2.78	-0.79	Accept H_0
	$\bar{x}_{Variable\ D2-4} = 5$ ng/mL	2.78	-0.51	Accept H_0

	$\bar{x}_{Variable\ D3-4} = 5$ ng/mL	2.78	-1.37	Accept H_0

Study	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
1b D0	$\bar{x}_{UG\ D0} = \bar{x}_{SG\ D0}$	6.39	4.24	2.31	-1.17	Accept H_0
	$\bar{x}_{UG\ D1} = \bar{x}_{SG\ D1}$	6.39	4.36	2.31	-0.68	Accept H_0
	$\bar{x}_{UG\ D2} = \bar{x}_{SG\ D2}$	6.39	1.28	2.31	0.42	Accept H_0
	$\bar{x}_{UG\ D3} = \bar{x}_{SG\ D3}$	6.39	1.77	2.31	1.67	Accept H_0
	$\bar{x}_{UG\ D4} = \bar{x}_{SG\ D4}$	6.39	1.13	2.31	-1.43	Accept H_0
2	$\bar{x}_{UG\ D0} = \bar{x}_{SG\ D0}$	6.39	6.10	2.31	-0.03	Accept H_0
	$\bar{x}_{UG\ D1} = \bar{x}_{SG\ D1}$	6.39	37.78	2.57	4.60	Reject H_0
	$\bar{x}_{UG\ D2} = \bar{x}_{SG\ D2}$	6.39	32.04	2.57	-0.63	Accept H_0
	$\bar{x}_{UG\ D3} = \bar{x}_{SG\ D3}$	6.39	2.68	2.31	0.75	Accept H_0
	$\bar{x}_{UG\ D4} = \bar{x}_{SG\ D4}$	6.39	6.04	2.31	-1.32	Accept H_0

Temperature (°C)	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
2	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	1.86	2.31	-2.54	Accept H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	1.54	2.31	-1.27	Accept H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	2.83	2.31	-4.74	Reject H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	20.80	2.57	-0.03	Accept H_0
40	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	1.35	2.31	0.59	Accept H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	3.47	2.31	-3.20	Reject H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	38.18	2.57	-4.37	Reject H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	6.93	2.57	-2.06	Accept H_0

pH level	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
2	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	2.44	2.31	3.25	Reject H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	3.96	2.31	-3.95	Reject H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	3.86	2.31	3.19	Reject H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	4.85	2.31	-1.71	Accept H_0

5	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	1.97	2.31	8.67	Reject H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	1.31	2.31	1.15	Accept H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	2.49	2.31	-2.13	Accept H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	1.64	2.31	1.42	Accept H_0
8	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	1.52	2.31	-0.43	Accept H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	1.05	2.31	8.71	Reject H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	29.78	2.57	-1.67	Accept H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	1.61	2.31	-1.50	Accept H_0
11	$\bar{x}_{D0} = \bar{x}_{D1}$	6.89	3.34	2.31	-1.47	Accept H_0
	$\bar{x}_{D1} = \bar{x}_{D2}$	6.89	1.99	2.31	-1.91	Accept H_0
	$\bar{x}_{D2} = \bar{x}_{D3}$	6.89	1.21	2.31	18.63	Reject H_0
	$\bar{x}_{D3} = \bar{x}_{D4}$	6.89	1.28	2.31	0.52	Accept H_0

Parameter	Null hypothesis (H_0)	F-critical	F-statistic	t-critical	t-statistic	Outcome
Recovery	$\bar{x}_{50\ ng/mL} = \bar{x}_{500\ ng/mL}$	19.00	2.03	2.78	-1.25	Accept H_0

5. Analysis of CBD products

5-1. Oil-based CBD product

Product Number	CBD Content (mg)			CBN Content (mg)			Δ^9-THC Content (μg)		
1	20.95	19.27	18.38	30.33	30.65	22.25	331475	295123	313309
2	27.75	29.85	29.91	44.70	44.57	44.57	62.22	49.32	61.76
3	154.98	163.66	164.20	<LOD	<LOD	<LOD	9.81	10.14	9.47
4	158.92	158.27	158.29	<LOD	<LOD	<LOD	56.18	67.01	63.73
5	369.62	366.56	354.17	<LOD	<LOD	<LOD	138.02	143.36	177.10
6	444.02	416.87	488.33	<LOD	<LOD	<LOD	91.96	74.91	83.41
7	0.21	0.16	0.21	2653	1513	2437	5494	5993	5601
8	132.24	136.99	152.46	<LOD	<LOD	<LOD	167.81	194.53	178.67
9	587.01	577.13	631.09	<LOD	<LOD	<LOD	113.15	66.28	63.35
10	640.05	599.87	669.65	<LOD	<LOD	<LOD	182.32	120.60	115.83

5-1b. Statistical calculations

Null Hypothesis (H_0): $\bar{x}_{product} = [\text{purported content}]$

Product Number	Purported CBD content (mg)	CBD content (mg)	Standard deviation (mg)	t-critical	t-statistic	Outcome
3	100	161	5	4.30	21.13	Reject H_0
4	600	158.5	0.4	4.30	-1912	Reject H_0
5	200	636	8	4.30	94.40	Reject H_0
6	250	450	36	4.30	9.62	Reject H_0
7	1000	0.19	0.03	4.30	-57724	Reject H_0
8	50	141	11	4.30	14.33	Reject H_0
9	300	598	29	4.30	17.80	Reject H_0
10	300	637	35	4.30	16.68	Reject H_0

5-2. Water-based CBD products

Product Number	Product Type	CBD Content (mg)		Δ^9-THC Content (ng)	
11	Unprepared Tea	0.47	0.53	9.83×10^3	8.60×10^3
	Prepared Tea	0.0008	0.011	172.56	157.16
12	Unprepared Tea	2.14	1.94	1.30×10^3	0.27×10^3
	Prepared Tea	0.042	0.035	9.20	63.95
13	Unprepared Tea	2.19	2.97	25.90×10^3	24.50×10^3
	Prepared Tea	0.13	0.29	53.32	17.54
14	Energy Drink	0.0009	0.0007	44.93	69.31
15	Sparkling water	3.93	4.04	7.94×10^3	2.26×10^3
16	Still water	2.85	1.98	4.40×10^3	7.25×10^3

5-2b. Statistical calculations

Product Number	Null Hypothesis	t-critical	t-statistic	Outcome
11	$\bar{x}_{Unprepared\ Tea\ 1} = 0$ ng	3.182	20.45	Reject H_0
	$\bar{x}_{Prepared\ Tea\ 1} = 0$ ng	3.182	0.99	Accept H_0
12	$\bar{x}_{Unprepared\ Tea\ 2} = 0$ ng	3.182	1.48	Accept H_0
	$\bar{x}_{Prepared\ Tea\ 2} = 0$ ng	3.182	1.95	Accept H_0
13	$\bar{x}_{Unprepared\ Tea\ 3} = 0$ ng	3.182	52.12	Reject H_0
	$\bar{x}_{Prepared\ Tea\ 3} = 0$ ng	3.182	2.37	Accept H_0
14	$\bar{x}_{Energy\ Drink} = 0$ ng	4.303	0.88	Accept H_0
15	$\bar{x}_{Sparkling\ Water} = 0$ ng	4.303	2.81	Accept H_0
16	$\bar{x}_{Still\ Water} = 0$ ng	4.303	1.37	Accept H_0