9/30/2022

REVIEWED
By Britany Wylie at 10:09 am, Oct 05, 2022

Worklist: 6111

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
C2022-2013	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
C2022-2027	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2033	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2040	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2044	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2048	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2077	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
C2022-2080	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
C2022-2081	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
C2022-2089	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2105	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2122	1	BCK	AM 27 Blood THC Quant by LC-QQQ
C2022-2153	1	BLOOD	AM 27 Blood THC Quant by LC-QQQ

AM# 27: Quantitation of THC and Metabolites in Blood and Urine by LC-MS/MS

Extraction Date 9/28/22 Analyst: Anne Nord
Plate lot#: 220802 Plate re-test: 2/2/23

Mobile phase A: 0.1% Formic Acid in LCMS Water **Mobile phase B:** 0.1% Formic acid in Acetonitrile
MTBE LCMS Methanol Hexane
Blank Blood Lot: 22B52016-1 **Urine Blank:** 7722 **Column**: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 69679

Pre-Analytic:

☒ 1. Check levels of mobile phases and needle wash refill as needed. Ensure waste is not full.
☒ 2. Ensure correct column is installed and begin mobile phase flow allow to equilibrate ~ 30 minutes.

Analytic:

☒ 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
☒ 2. Urine hydrolysis: add 1.5 ml urine to blank plate, add 250 ul 1N KOH mix and incubate at 40 degrees for 15 minutes.
Pipette **1000µL blood (calibrated pipette) Pipette ID: I41142J** in wells of analytical (standards) plate.
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 4. Pipette **500µL 0.1% formic acid in water blood sample**, **500 ul saturated phosphate buffer in urine** in wells of analytical plate.
☒ 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 6. Transfer **800µL of blood+acid or urine acid** mixture to corresponding wells of SLE+ plate.
☒ 7. Apply positive pressure for approx. 10-15 seconds (or until no liquid remains on top of sorbent).
(Load at 85-100 PSI- Selector to the right) Manifold ID: 66792
☒ 8. Wait 5 minutes.
☒ 9. Add **2.25mL MTBE. (*Add in 3 increments of 750uL)***
☒ 10. Wait 5 minutes.
☒ 11. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 12. Add **2.25mL Hexane. (*Add in 3 increments of 750uL)***
☒ 13. Wait 5 minutes.
☒ 14. Apply positive pressure for approx. 15 seconds. ***(10-15 PSI- Selector to the left).***
☒ 15. Remove plate containing eluate. Place on SPE Dry and evaporate to dryness at approx. 35°C.
SPE Dry ID: 66819
☒ 16. Reconstitute in **100µL 100% MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

☒ 1. Create batch and process data.
☒ 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r^2 values ≥0.98 for each analyte
☒ 3. RT +/- 3% or 0.100 min, whichever is greater, +/- 20% Accuracy for greater than (+/- 30% for 10ng/ml or less). Ion ratios must be within +/- 20% of the averaged calibrators
☒ 4. Case sample response for THC 1ng/ml, OH-THC 3ng/mL (quantitative blood), Carboxy-THC: 5 ng/mL (qualitative only) will be reported. Samples with a THC or OH-THC response over 50 ng/mL will be reported out as greater than 50 ng/mL.
☒ 5. Did all QCs pass for each analyte? (if not is it describe in comments section)
☒ 6. Enter QCs into control charting.
☒ 7. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *THC-OH range 3-100 (cal 1 dropped due to ratio)*

Idaho State Police
Forensic Services

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): **TOX-22-02**

Date of Request:
03/02/2022

Requestor/Discipline:
Celena Shrum/Toxicology

Analytical Method/Quality Standard, Revision #:
Toxicology AM #25, AM #26, and AM #27, Revision 13

Temporary or Permanent Deviation:
Permanent

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc):

Deviation will remain in place until the change is made in the next method revision.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual):

Toxicology AM #25 3.3.1.1 Internal standards are prepared by the ToxBox plate manufacturer and contained on the 96 well plate. If the run contains urine samples, a positive external urine
control must also be run.

Toxicology AM #26 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and external positive urine control must also be included.

Toxicology AM #27 3.3.2 A negative control will be run with each extraction. If the run contains urine samples, a negative urine control and positive external urine control will also be included in the run.

The deviation is to include the option of using an internal urine control in lieu of an external urine control.

Technical Justification for Analytical Method Deviations:

Internal controls serve the same purpose as external controls but also helps to avoid the possible issues that can occur with using external controls (incorrect spiking, incorrect preparation, evaporation of compounds, etc.). If these errors occur, runs need to be repeated and this wastes time, sample, and supplies.

Technical Review

☒ Departure approved
Comments:

☐ Departure Not Approved
Comments:

Approver: Rachel Cutler Date: 3/2/22
Title: Lab Manager

Quality Review

Quality Approver: Jason Crowe
Title: Quality Manager
Date: 3/2/2022

	1	2	3	4	5	6
a	cal 1	Internal urine	2122-1			
b	cal 2	negative blood	2153-1			
c	cal 3	2027-1	2033-1			
d	cal 4	2040-1	negative urine			
e	Cal 5	2044-1	2013-2			
f	cal 6	2048-1	2077-1			
g	cal 7	2089-1	2080-2			
h	Internal control (blood)	2105-1	2081-1			

Plate position 3

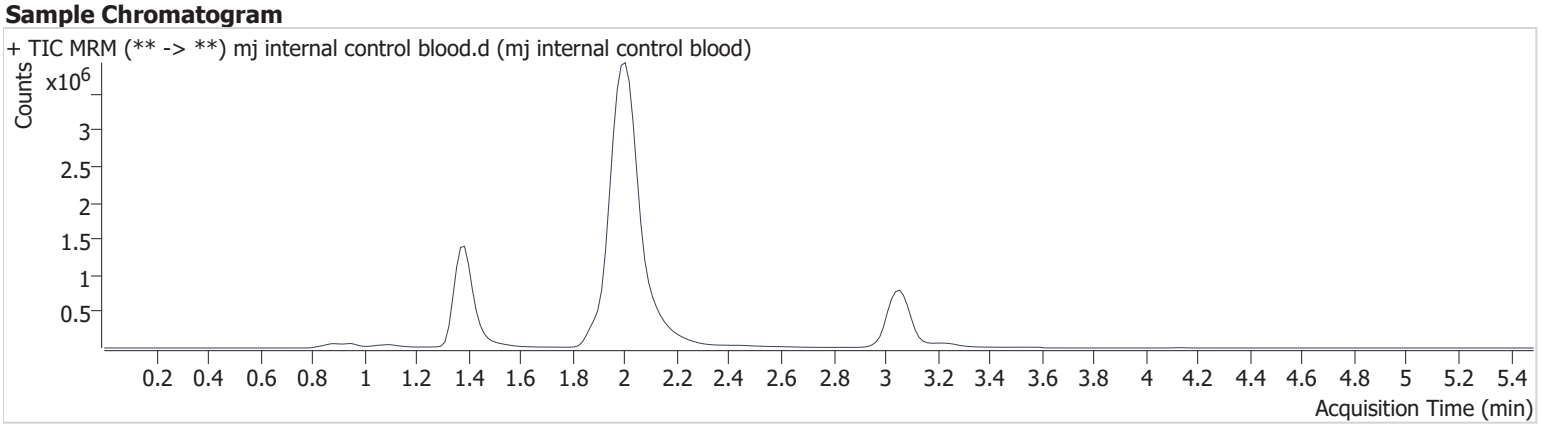
c2022-____-_

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj internal control blood.d
Type	QC	**Sample**	mj internal control blood
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:41:43 PM		
Sample Info.			

Sample Chromatogram



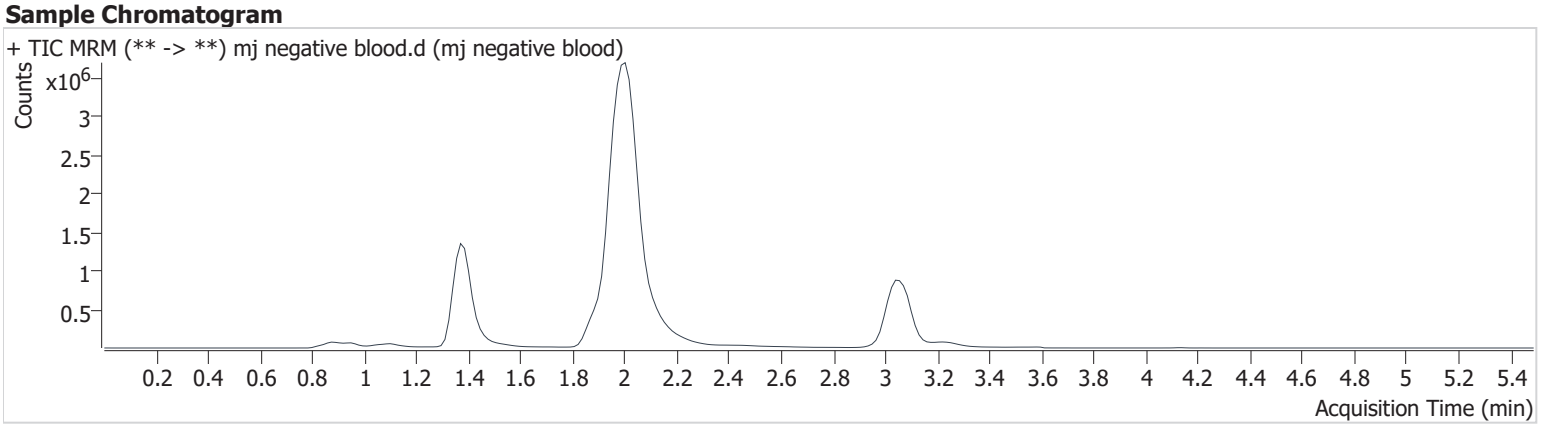
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	67839	∞	932.0	∞	4257219	4.984 ng/ml
THC-COOH	1.403	99569	886.2	273.5	365.5	1239074	14.881 ng/ml
THC	3.077	526426	10144.1	24.0	∞	4158048	4.767 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update	9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj negative blood.d
Type	Sample	**Sample**	mj negative blood
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:48:27 PM		
Sample Info.			

Sample Chromatogram

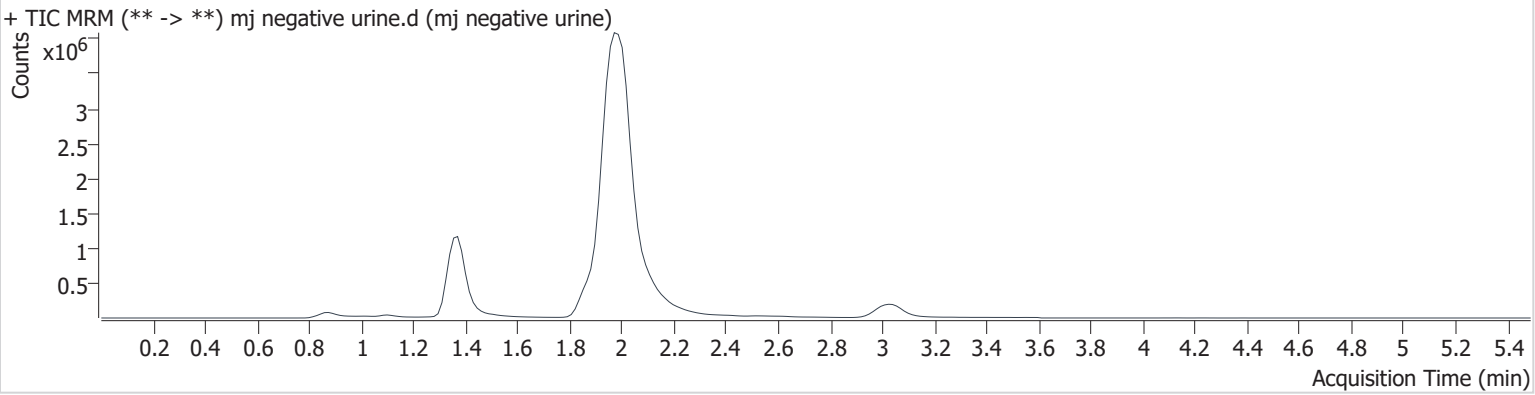


AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj negative urine.d
Type	Sample	**Sample**	mj negative urine
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-D3	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 10:02:16 PM		
Sample Info.			

Sample Chromatogram

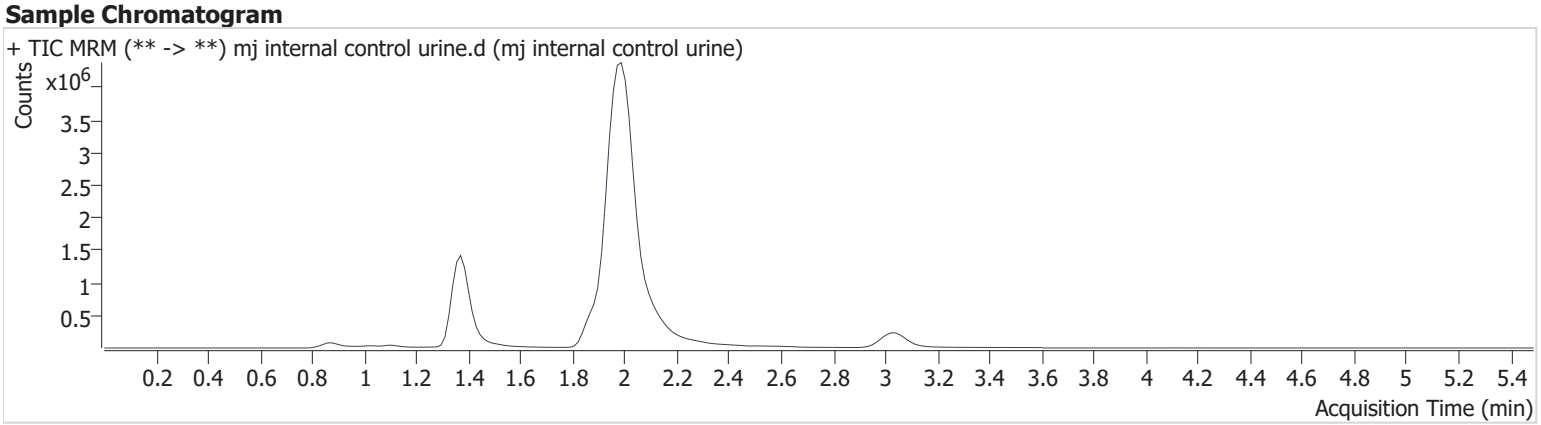


AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj internal control urine.d
Type	Sample	**Sample**	mj internal control urine
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 11:02:30 PM		
Sample Info.			

Sample Chromatogram



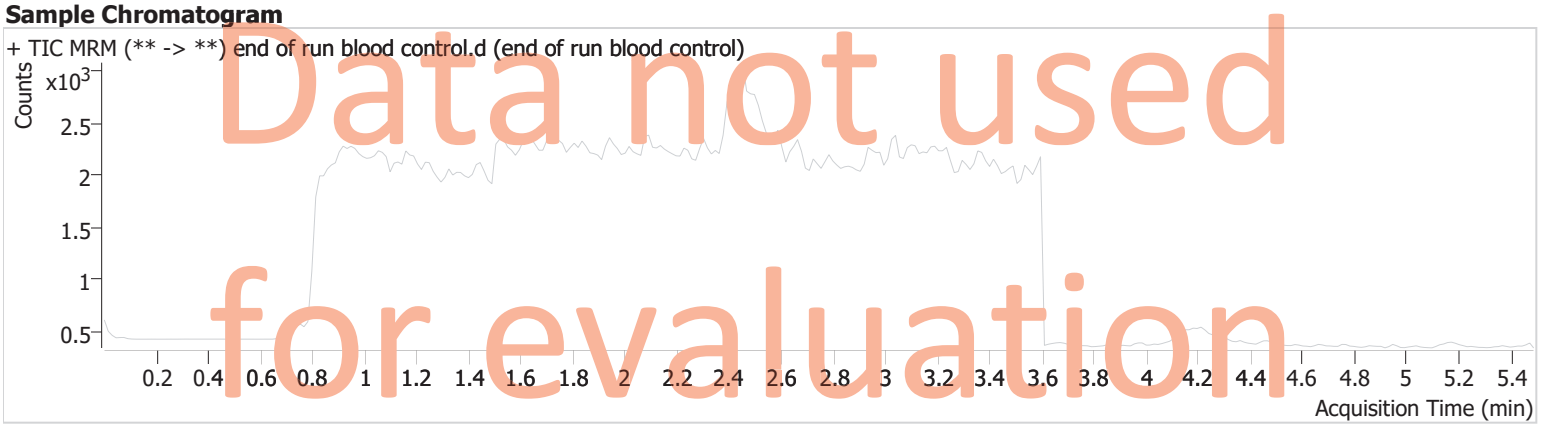
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	72468	∞	869.6	∞	4551774	4.980 ng/ml
THC-COOH	1.403	74691	406.1	286.9	∞	1007326	13.806 ng/ml
THC	3.046	171165	1477.4	23.6	154.8	1424626	4.542 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update	9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	end of run blood control.d
Type	Sample	**Sample**	end of run blood control
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 11:09:14 PM		
Sample Info.			

Sample Chromatogram



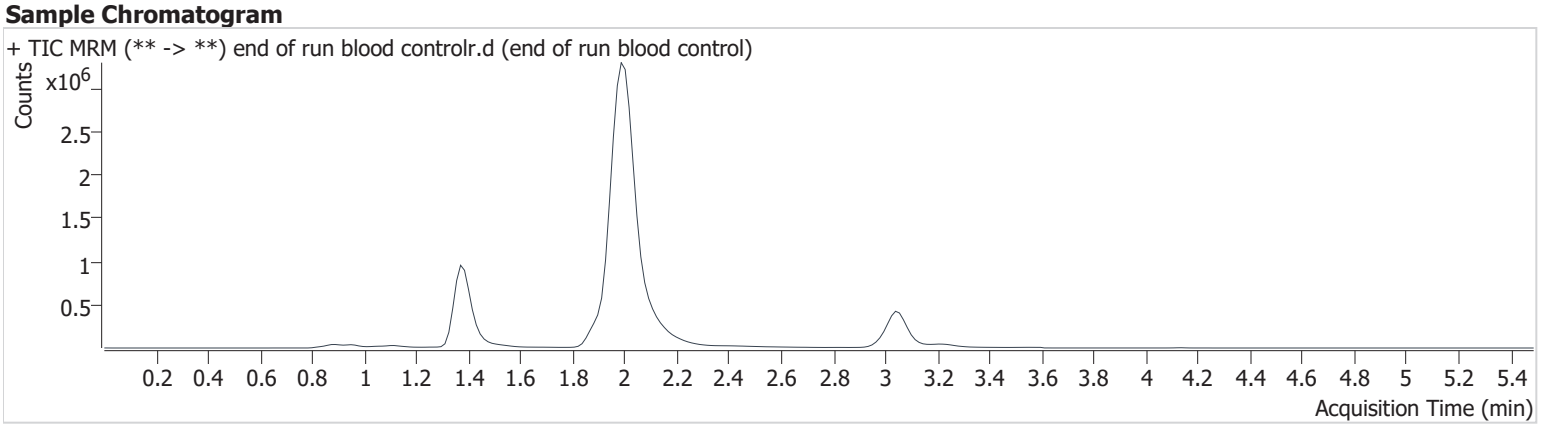
sample evaporated and did not inject, sample was reconstituted and injected.

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	end of run blood controlr.d
Type	Sample	**Sample**	end of run blood control
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/29/2022 8:52:04 AM		
Sample Info.			

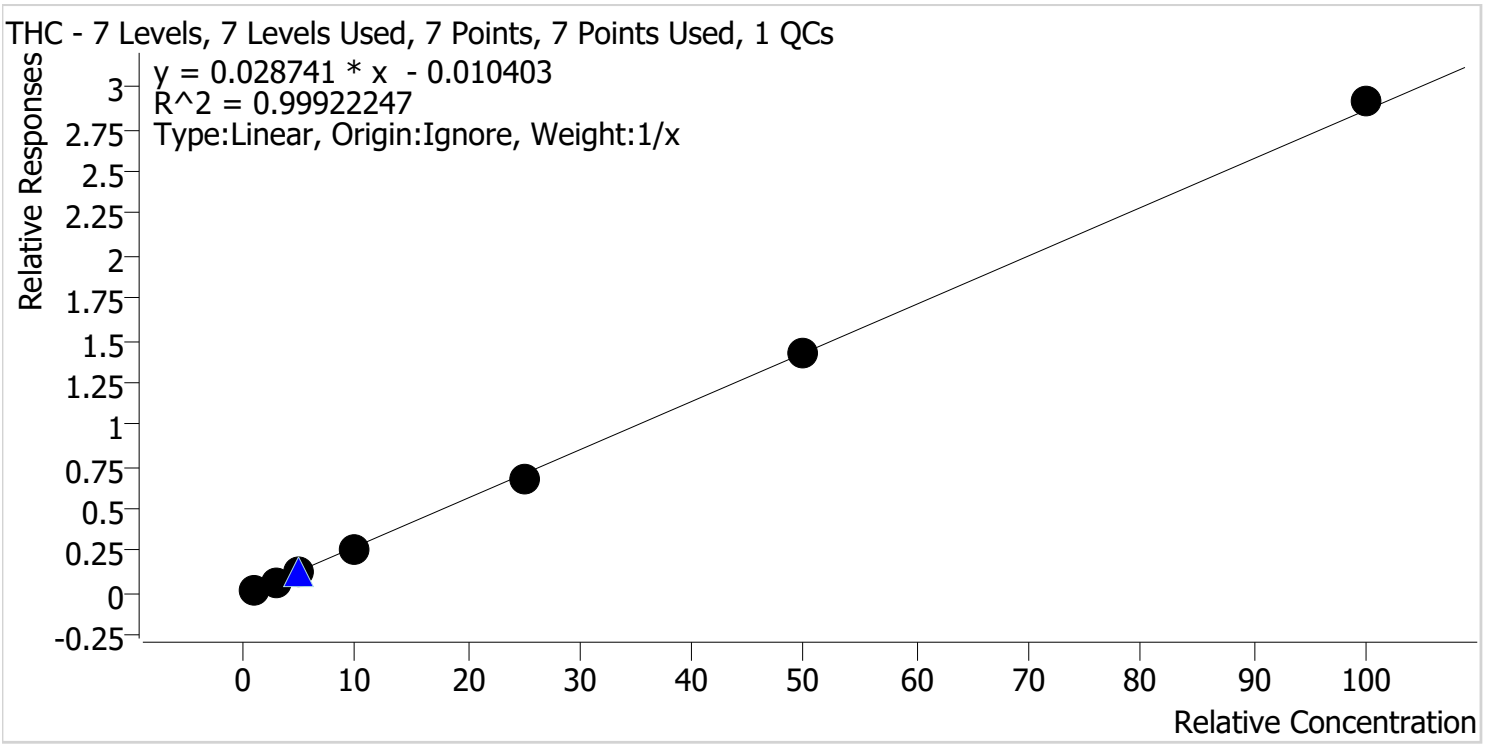
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	40387	23929.5	903.2	7970.2	2694534	4.735 ng/ml
THC-COOH	1.403	60388	∞	272.0	∞	817897	13.752 ng/ml
THC	3.061	248019	2408.9	24.5	∞	2033745	4.605 ng/ml

Compound Calibration Report

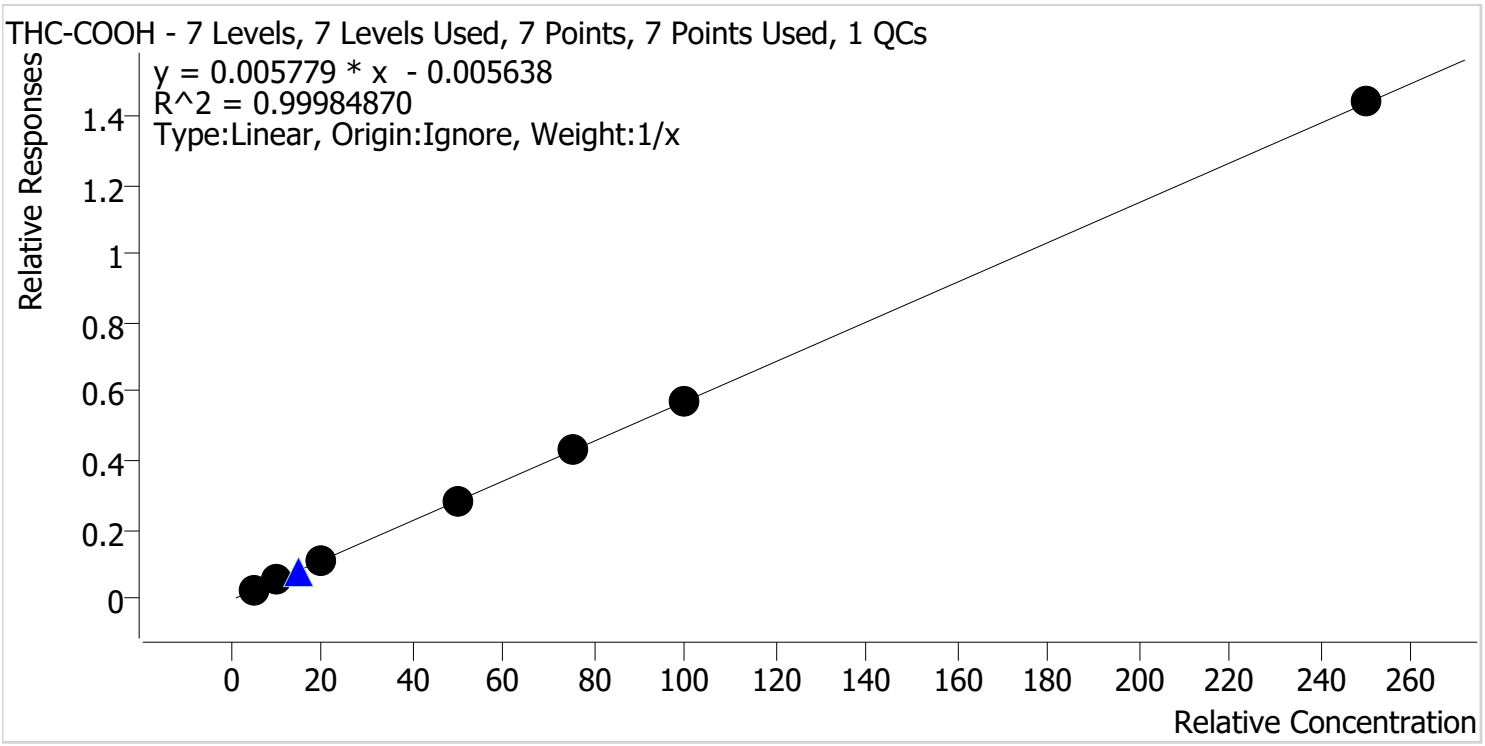
Batch results	D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Last Cal. Update	9/29/2022 10:46 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-d3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	1.0	1.2	116.4
mj cal 2	2	✔	3.0	2.9	96.4
mj cal 3	3	✔	5.0	4.7	93.7
mj cal 4	4	✔	10.0	9.5	95.4
mj cal 5	5	✔	25.0	24.2	96.8
mj cal 6	6	✔	50.0	49.8	99.5
mj cal 7	7	✔	100.0	101.8	101.8

Compound Calibration Report

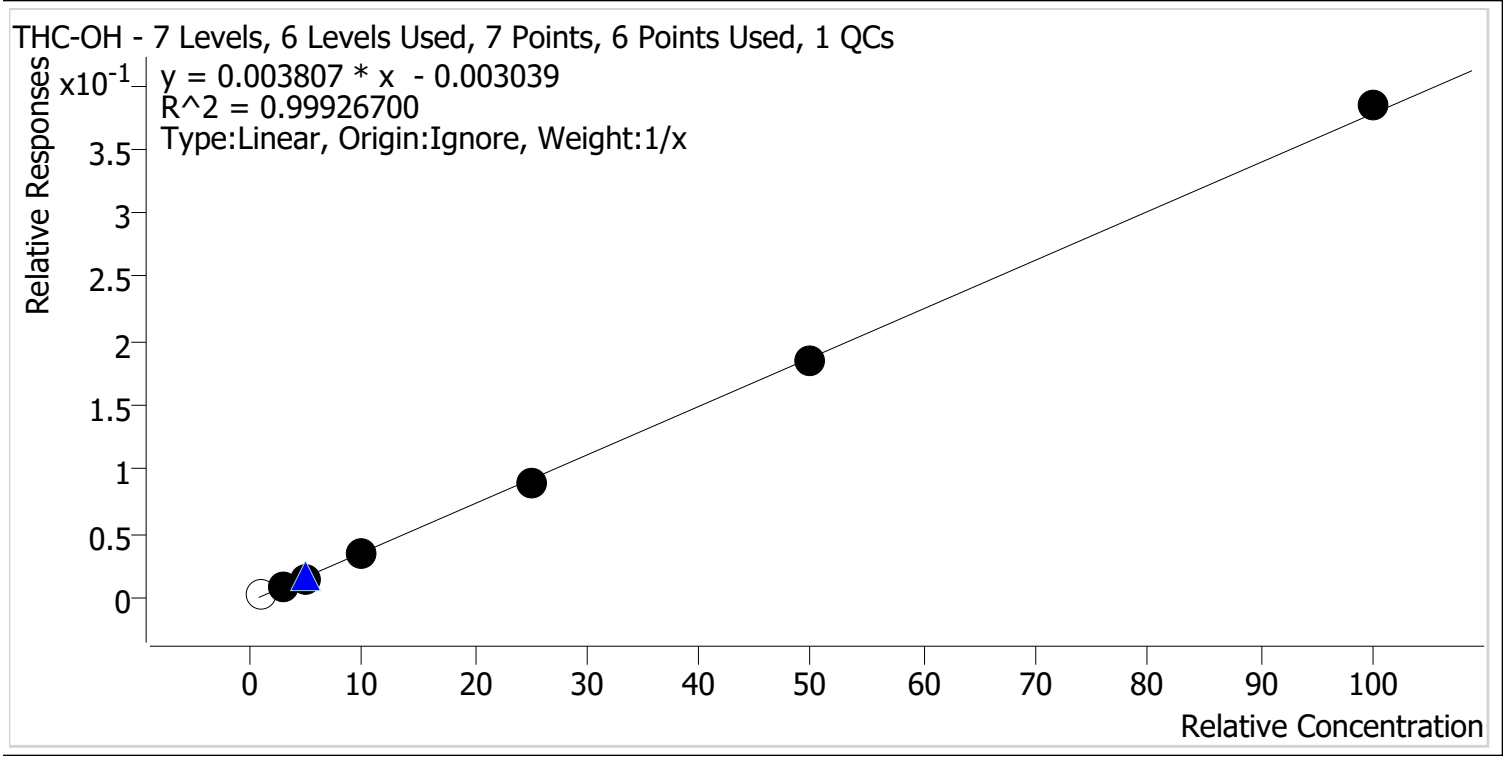
Batch results	D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Last Cal. Update	9/29/2022 10:46 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-d9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	5.0	5.2	103.9
mj cal 2	2	✔	10.0	10.1	101.0
mj cal 3	3	✔	20.0	19.1	95.4
mj cal 4	4	✔	50.0	49.5	99.0
mj cal 5	5	✔	75.0	75.1	100.1
mj cal 6	6	✔	100.0	100.2	100.2
mj cal 7	7	✔	250.0	250.8	100.3

Compound Calibration Report

Batch results	D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Last Cal. Update	9/29/2022 10:46 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-d3



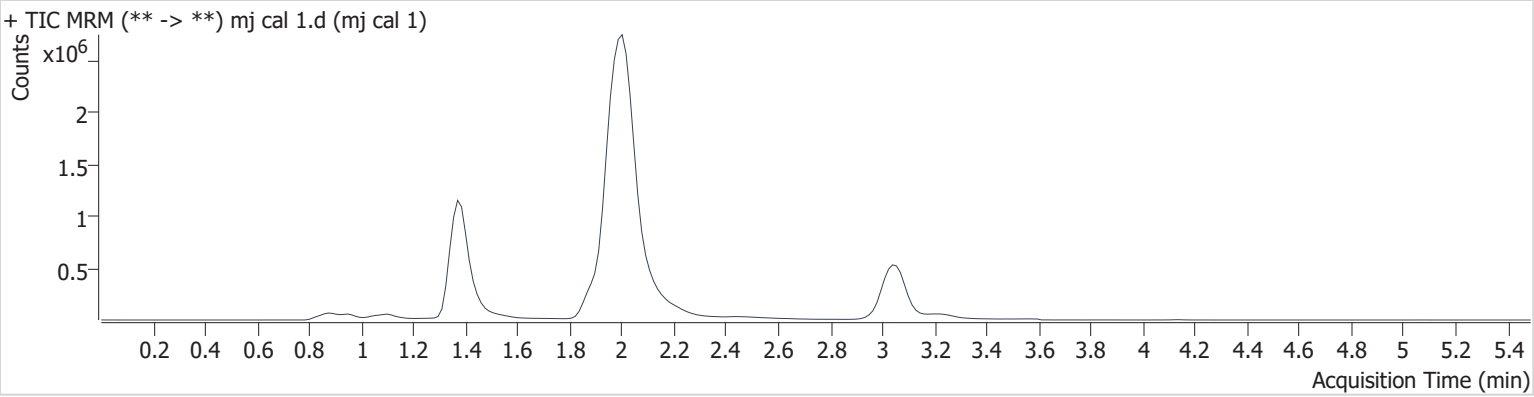
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✗	1.0	1.6	157.3
mj cal 2	2	✔	3.0	3.3	108.9
mj cal 3	3	✔	5.0	4.8	96.8
mj cal 4	4	✔	10.0	9.7	97.5
mj cal 5	5	✔	25.0	24.1	96.2
mj cal 6	6	✔	50.0	49.4	98.9
mj cal 7	7	✔	100.0	101.6	101.6

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 1.d
Type	Cal	**Sample**	mj cal 1
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 6:47:54 PM		
Sample Info.			

Sample Chromatogram



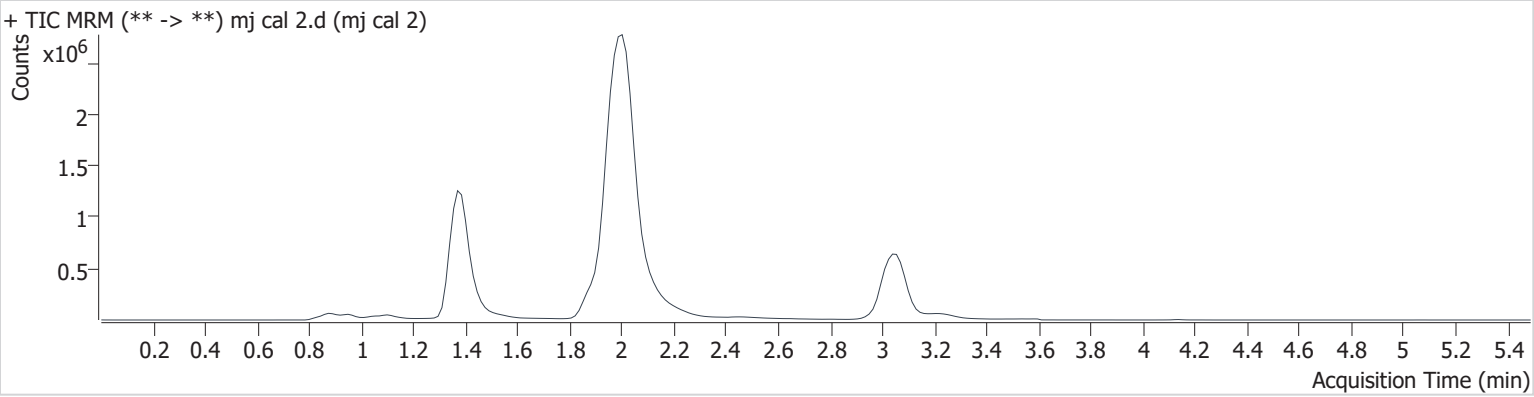
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.379	11946	36.4	1117.1 **High**	∞	4050696	1.573 ng/ml	**Low**
THC-COOH	1.403	26302	239.3	269.3	∞	1078119	5.197 ng/ml	
THC	3.061	73224	1109.5	27.0	82.1	3176982	1.164 ng/ml	

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 2.d
Type	Cal	**Sample**	mj cal 2
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 6:54:39 PM		
Sample Info.			

Sample Chromatogram



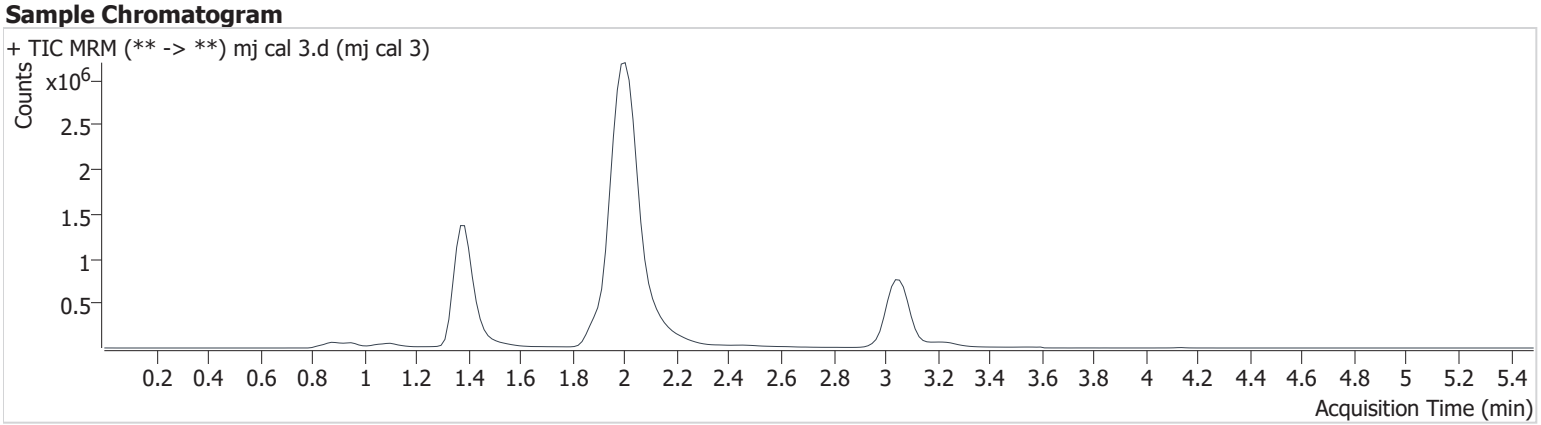
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	39112	∞	982.3	∞	4160364	3.268 ng/ml
THC-COOH	1.403	62083	∞	258.4	607.4	1177747	10.097 ng/ml
THC	3.061	267980	8390.0	24.1	621.7	3685263	2.892 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 3.d
Type	Cal	**Sample**	mj cal 3
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:01:23 PM		
Sample Info.			

Sample Chromatogram



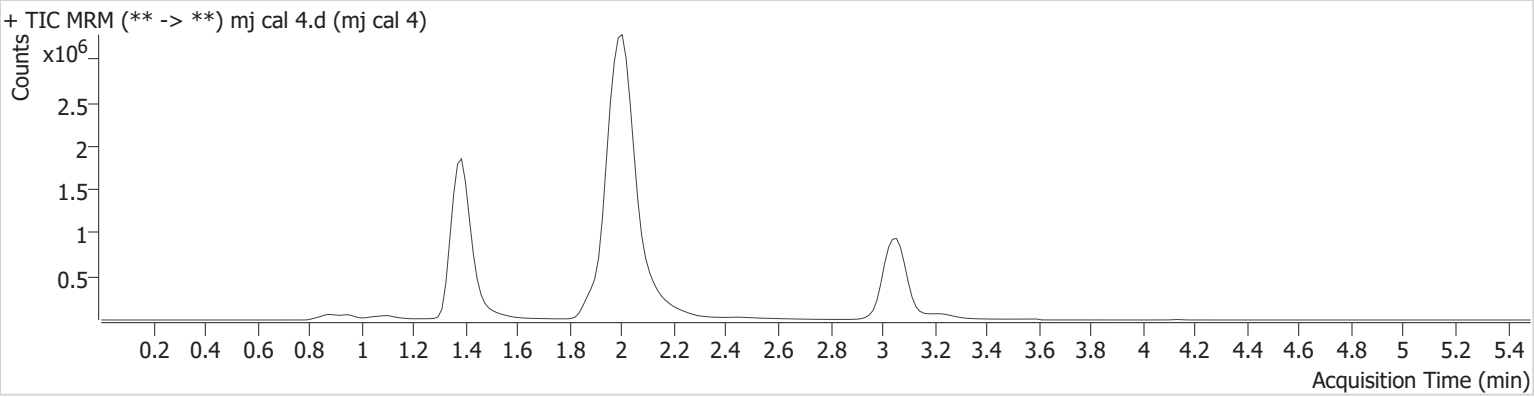
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	63453	∞	865.8	∞	4122435	4.841 ng/ml
THC-COOH	1.403	125719	∞	269.8	1231.7	1201318	19.084 ng/ml
THC	3.061	500781	19754.0	24.4	1091.6	4030447	4.685 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 4.d
Type	Cal	**Sample**	mj cal 4
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:08:07 PM		
Sample Info.			

Sample Chromatogram



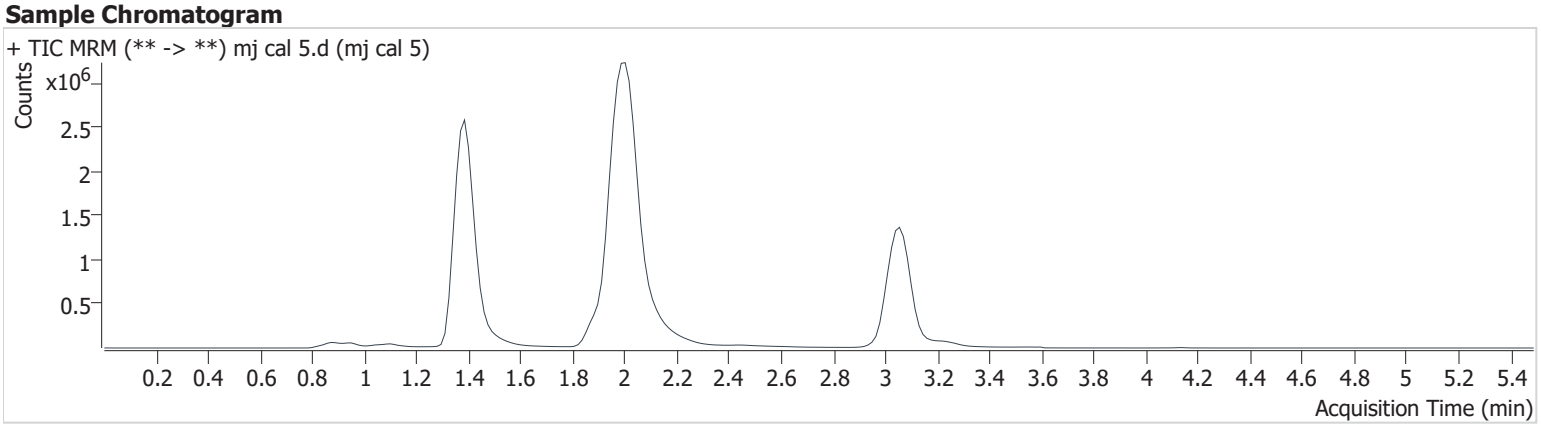
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	145194	∞	925.0	∞	4260694	9.750 ng/ml
THC-COOH	1.403	354882	∞	262.2	3029.3	1265013	49.520 ng/ml
THC	3.061	1141972	∞	24.2	∞	4329130	9.540 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 5.d
Type	Cal	**Sample**	mj cal 5
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:14:52 PM		
Sample Info.			

Sample Chromatogram



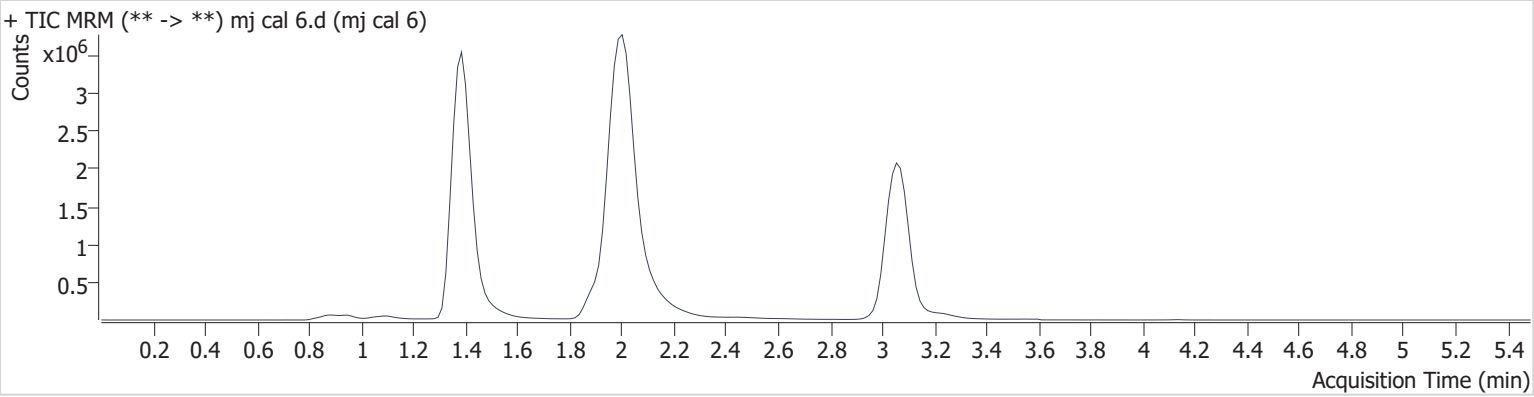
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	397885	∞	857.7	∞	4494066	24.054 ng/ml
THC-COOH	1.403	554265	∞	265.2	∞	1294709	75.054 ng/ml
THC	3.061	3152970	1158341.8	24.4	∞	4599766	24.212 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 6.d
Type	Cal	**Sample**	mj cal 6
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:21:36 PM		
Sample Info.			

Sample Chromatogram



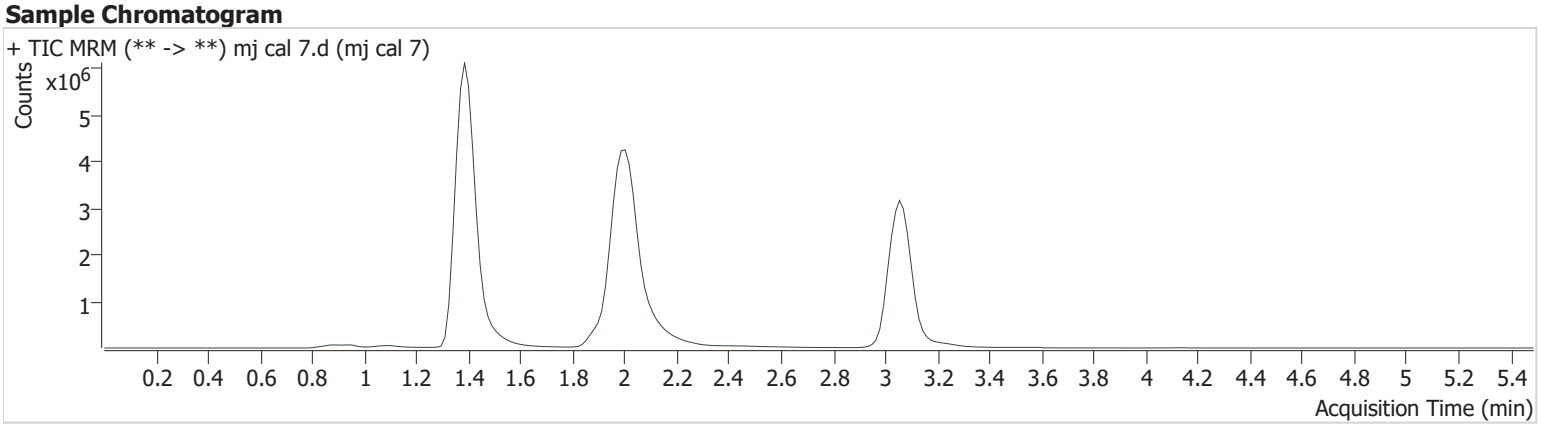
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	804553	∞	836.3	∞	4344209	49.446 ng/ml
THC-COOH	1.403	717070	4535.3	263.8	∞	1250327	100.216 ng/ml
THC	3.077	6527952	170619.5	25.2	5124.3	4598674	49.753 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\092822\QuantResults\cann.batch.bin
Calibration Last Update 9/29/2022 10:46:50 AM

Instrument	69679	**Data File**	mj cal 7.d
Type	Cal	**Sample**	mj cal 7
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	9/28/2022 7:28:20 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.379	1604406	69214.5	817.3	∞	4179152	101.641 ng/ml
THC-COOH	1.403	1665040	∞	270.2	12426.3	1153146	250.831 ng/ml
THC	3.061	12146332	126521.0	24.8	24938.8	4168142	101.754 ng/ml