REVIEWED
By Sarah Collins at 8:10 pm, Aug 09, 2022

8/8/2022

Worklist: 6056

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
C2022-1561	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1566	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1568	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1611	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1612	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
C2022-1621	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1635	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
C2022-1660	1	BCK	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 8/4/22 Analyst: Anne Nord
Plate lot#: 220309 Plate re-test: 9/9/2022

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 not evaluated due ratio lowing as concentration increased.*
Calibrator level 7 did not inject on the first injection, reinjected it still did not inject, added more reconstitution solvent and injected, (this sample was evaluated). The urine positive control, end of run blood control, and case sample C2022-1660-1 did not inject, they were re-constituted and injected. The re-injections were evaluated.

	1	2	3	4	5	6
a	cal 1	Internal urine	negative urine			
b	cal 2	negative blood	1612-2			
c	cal 3	1611-1				
d	cal 4	1635-1				
e	Cal 5	1561-1				
f	cal 6	1566-1				
g	cal 7	1621-1				
h	Internal control (blood)	1660-1				

c2022-____-_

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

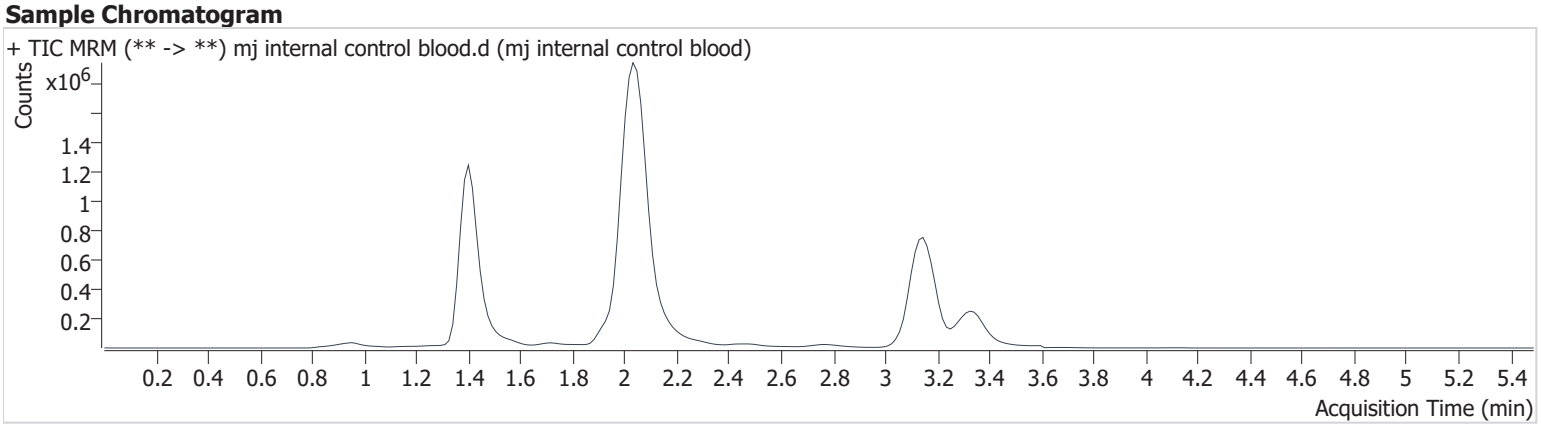
Quality Approver: Jason Crowe
Title: Quality Manager
Date: 3/2/2022

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 3:21:37 PM		
Sample Info.			

Sample Chromatogram



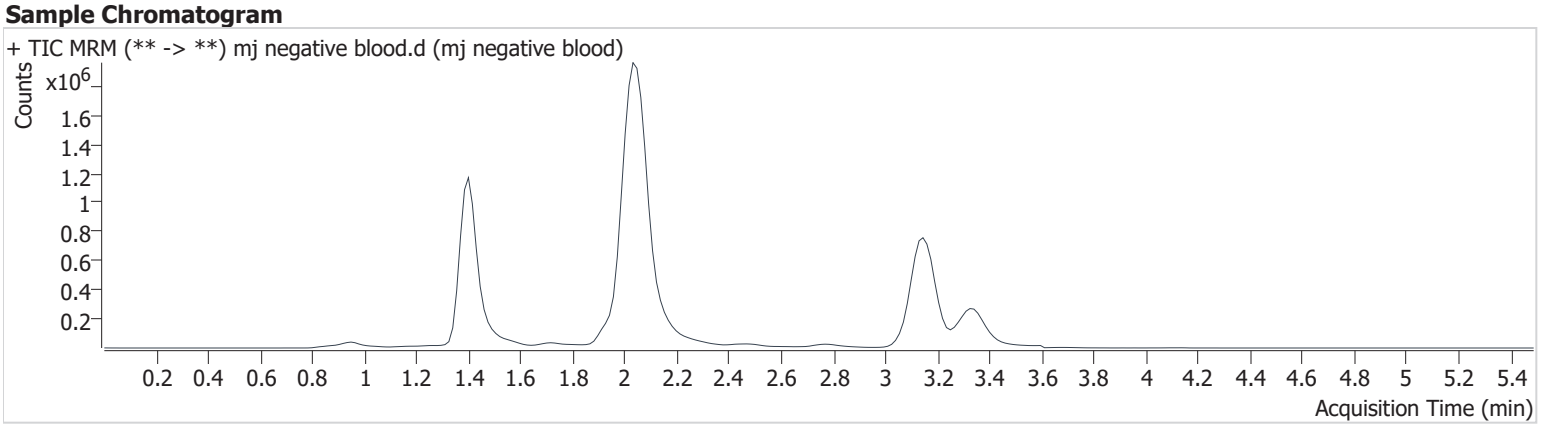
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	69481	∞	270.5	∞	991892	14.004 ng/ml
THC	3.167	431289	∞	24.6	∞	3798443	4.727 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update	8/5/2022 9:07:25 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	8/4/2022 3:28:21 PM		
Sample Info.			

Sample Chromatogram

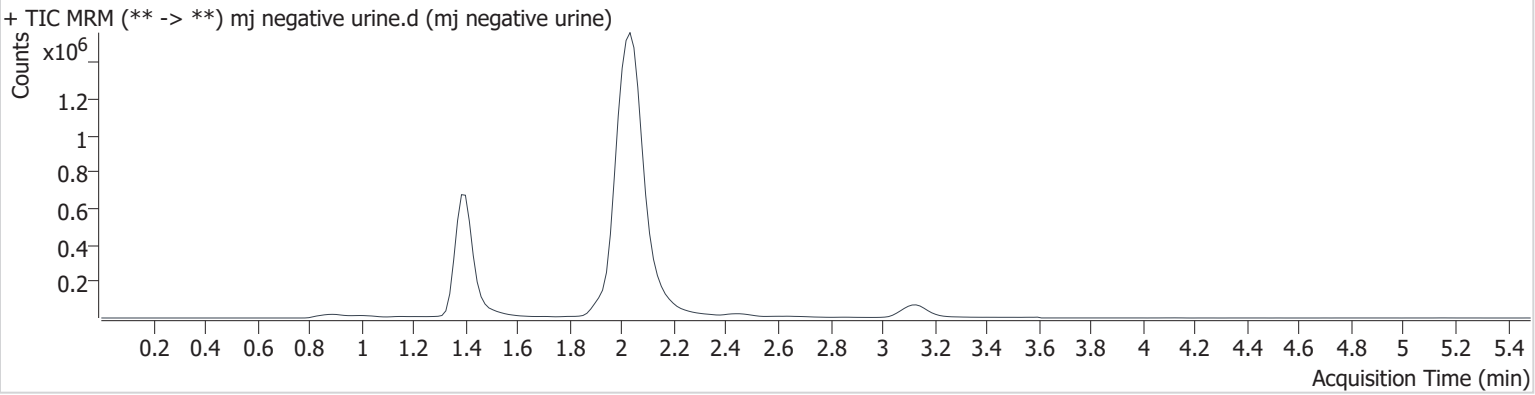


AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update	8/5/2022 9:07:25 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-A3	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/4/2022 5:15:29 PM		
Sample Info.			

Sample Chromatogram



AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update	8/5/2022 9:07:25 AM

Instrument	69679	**Data File**	mj urine control.d
Type	Sample	**Sample**	mj urine control
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/4/2022 5:35:36 PM		
Sample Info.			

Sample Chromatogram

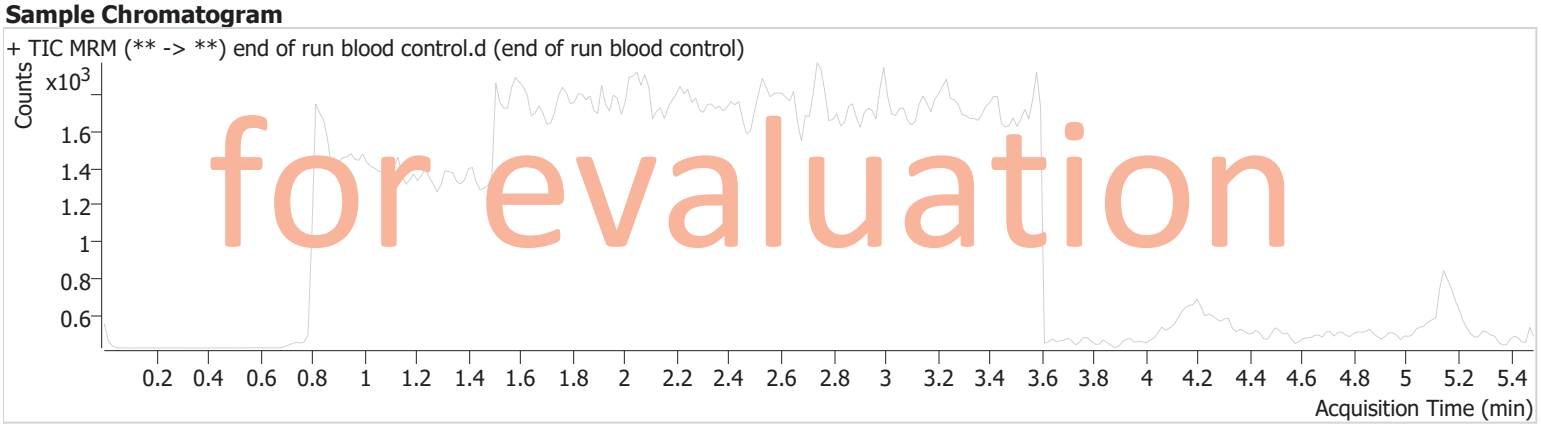
sample did not inject

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 5:42:20 PM		
Sample Info.			

Sample Chromatogram



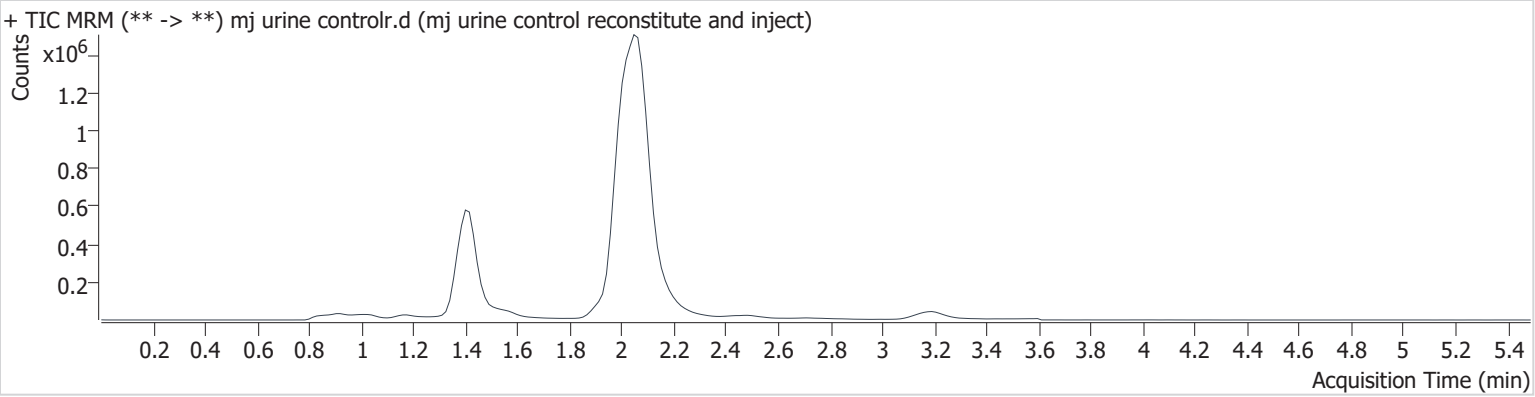
sample did not inject

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 AM

Instrument	69679	**Data File**	mj urine controlr.d
Type	Sample	**Sample**	mj urine control reconstitute and inject
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/5/2022 8:11:35 AM		
Sample Info.			

Sample Chromatogram



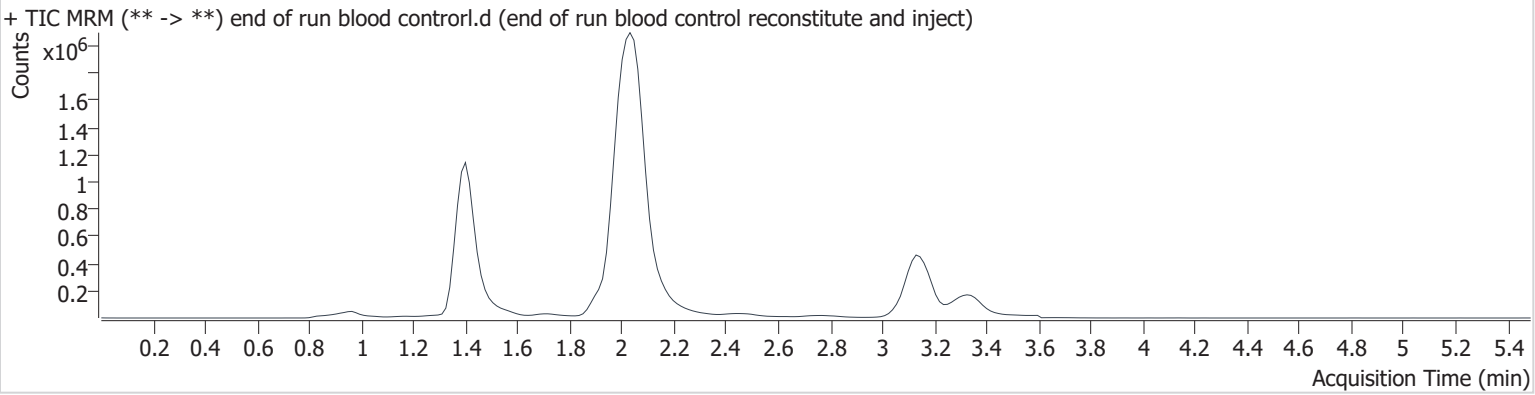
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	26238	∞	231.4	∞	355963	14.684 ng/ml
THC	3.212	33698	1516.4	22.0	124.0	282705	4.945 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 AM

Instrument	69679	**Data File**	end of run blood controrl.d
Type	Sample	**Sample**	end of run blood control reconstitute and inject
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/5/2022 8:31:42 AM		
Sample Info.			

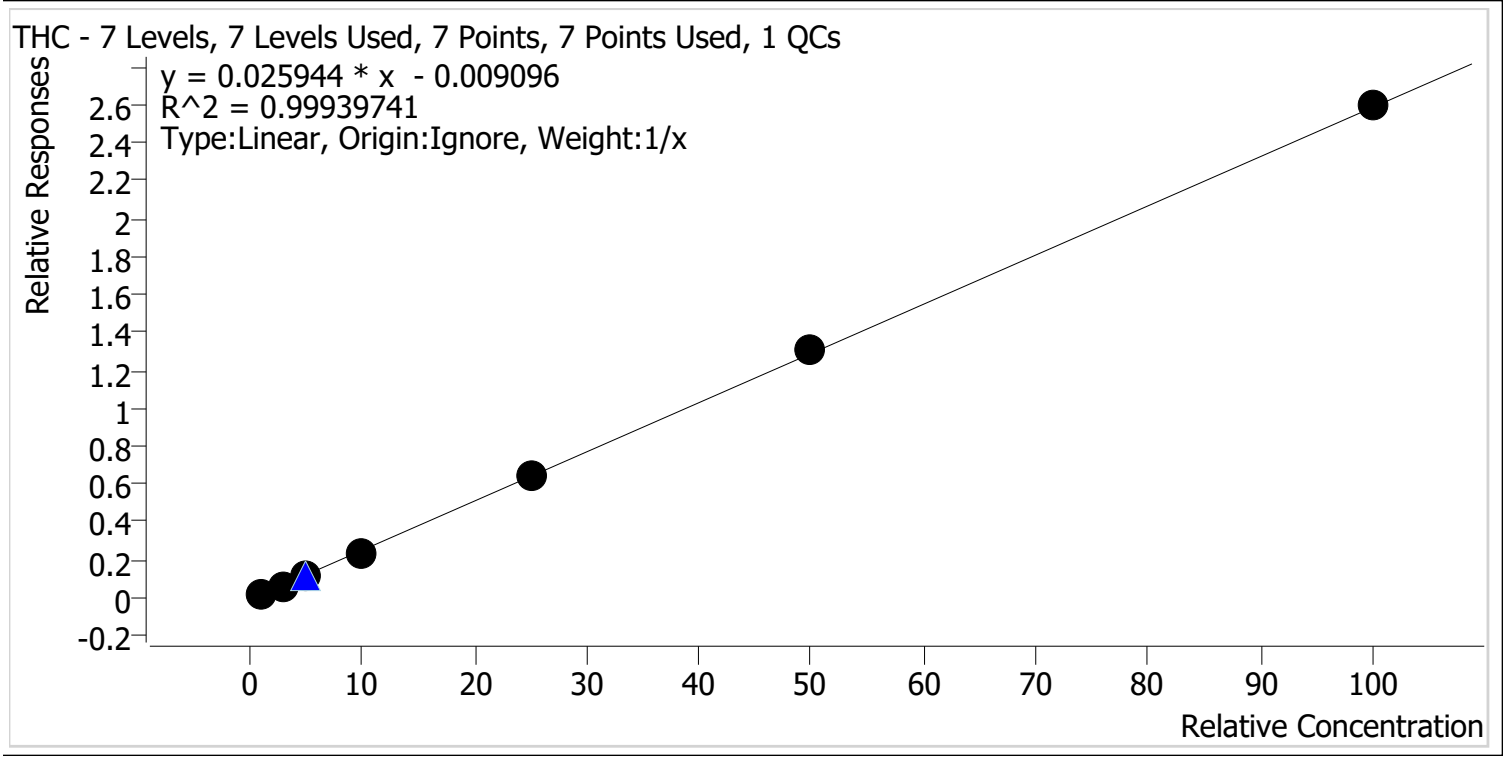
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	65457	∞	277.6	∞	950422	13.785 ng/ml
THC	3.152	253375	∞	24.8	∞	2296779	4.603 ng/ml

Compound Calibration Report

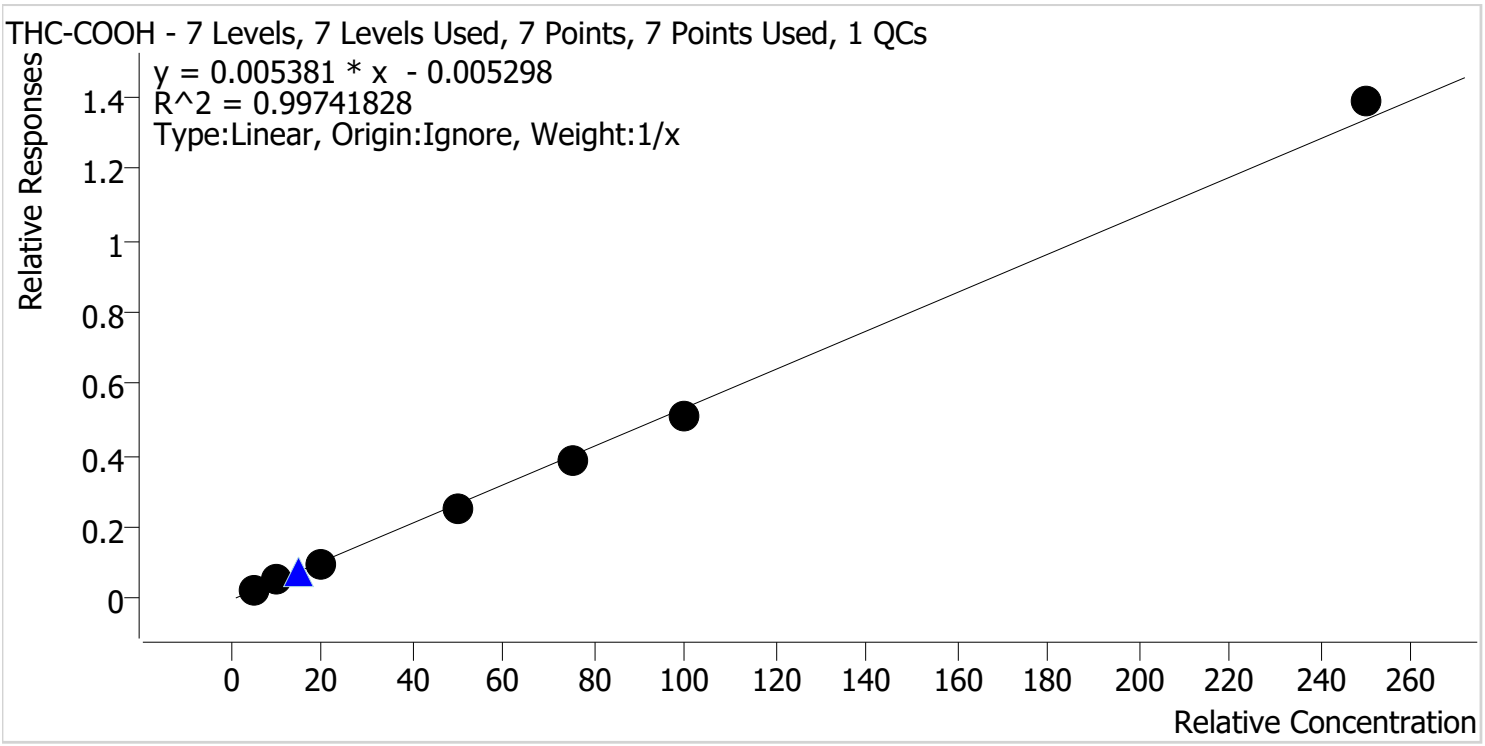
Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Last Cal. Update	8/5/2022 9:07 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-d3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	1.0	1.2	118.0
mj cal 2	2	✔	3.0	2.8	91.7
mj cal 3	3	✔	5.0	4.7	93.0
mj cal 4	4	✔	10.0	9.6	96.2
mj cal 5	5	✔	25.0	24.9	99.5
mj cal 6	6	✔	50.0	50.6	101.2
mj cal 7 added 50 ul solvent	7	✔	100.0	100.3	100.3

Compound Calibration Report

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Last Cal. Update	8/5/2022 9:07 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-d9



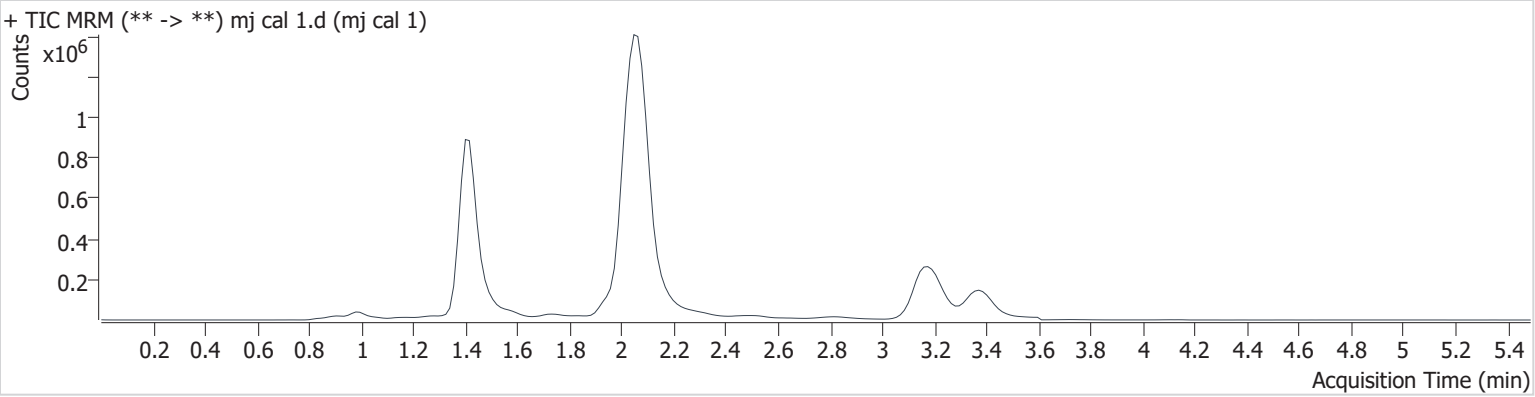
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	5.0	5.3	105.9
mj cal 2	2	✔	10.0	11.1	111.3
mj cal 3	3	✔	20.0	18.3	91.3
mj cal 4	4	✔	50.0	47.7	95.5
mj cal 5	5	✔	75.0	71.9	95.9
mj cal 6	6	✔	100.0	96.4	96.4
mj cal 7 added 50 ul solvent	7	✔	250.0	259.3	103.7

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 2:27:48 PM		
Sample Info.			

Sample Chromatogram



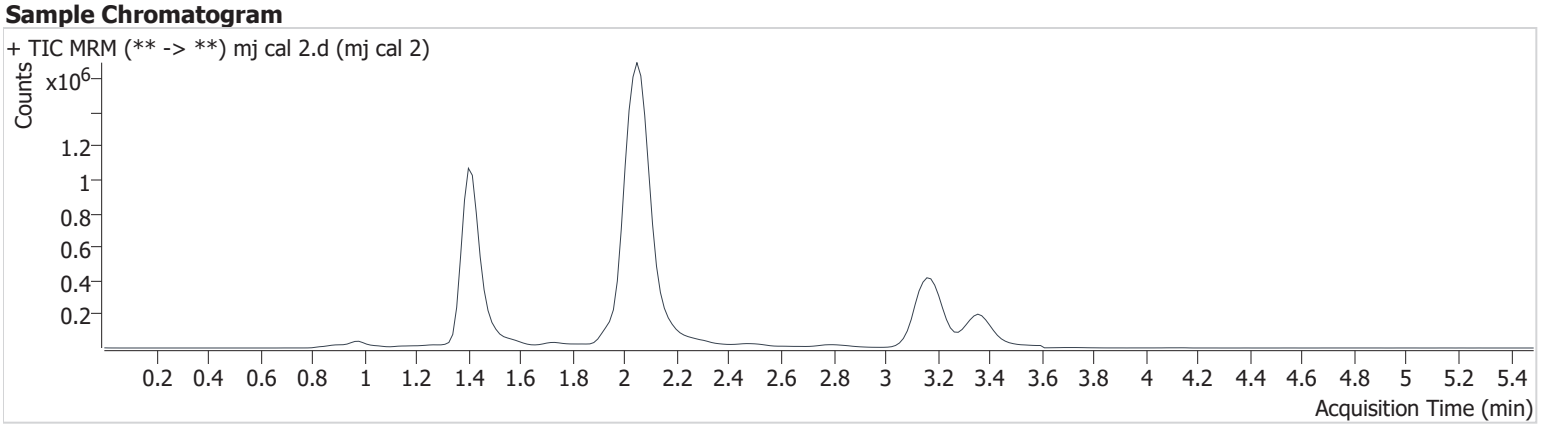
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.448	14125	∞	247.0	∞	608852	5.296 ng/ml
THC	3.212	29640	2798476134 2635.6	24.2	90.8	1378223	1.180 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 2:34:33 PM		
Sample Info.			

Sample Chromatogram



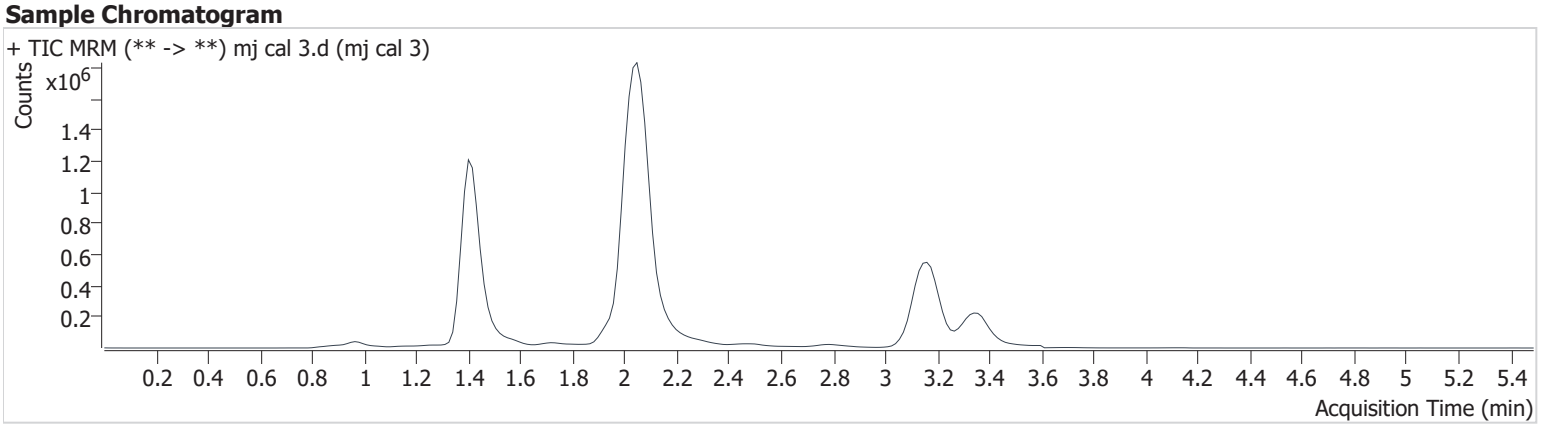
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.448	41961	∞	225.6	2302.8	768686	11.130 ng/ml
THC	3.197	134802	1080080139 28517.0	23.7	557.4	2163500	2.752 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 2:41:17 PM		
Sample Info.			

Sample Chromatogram



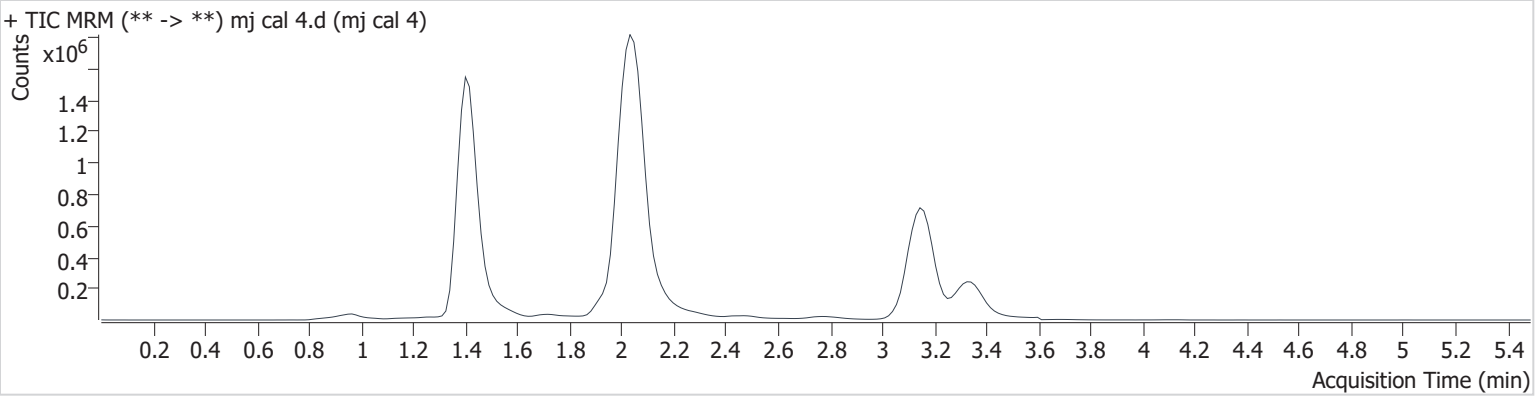
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	81655	∞	269.7	∞	878185	18.266 ng/ml
THC	3.182	308052	∞	25.6	21815 44788 3063.1	2760320	4.652 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 2:48:02 PM		
Sample Info.			

Sample Chromatogram



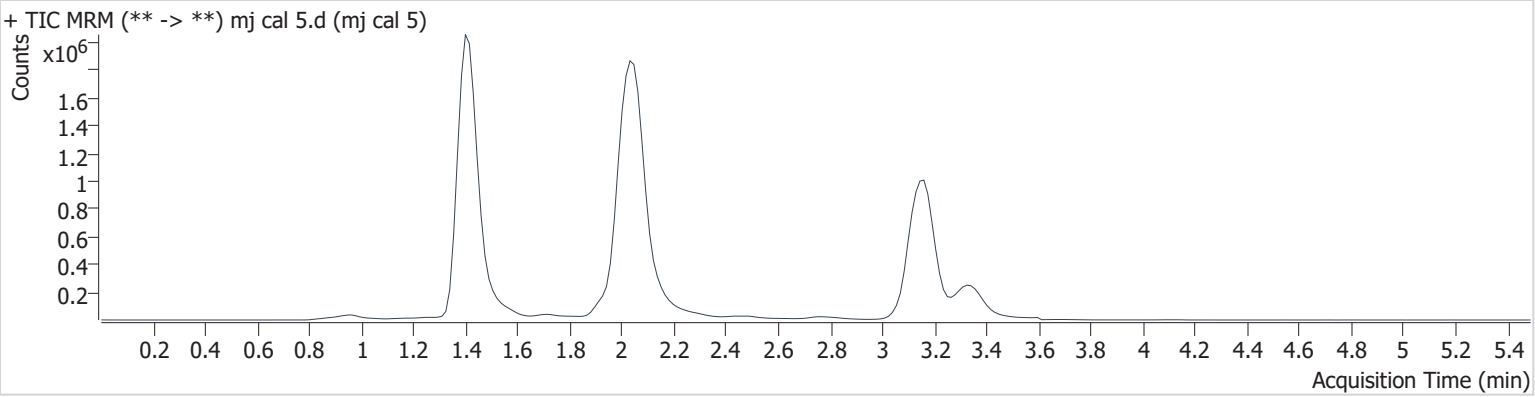
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	239559	13048.9	263.4	∞	952477	47.729 ng/ml
THC	3.167	781474	∞	22.8	∞	3248179	9.624 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 2:54:46 PM		
Sample Info.			

Sample Chromatogram



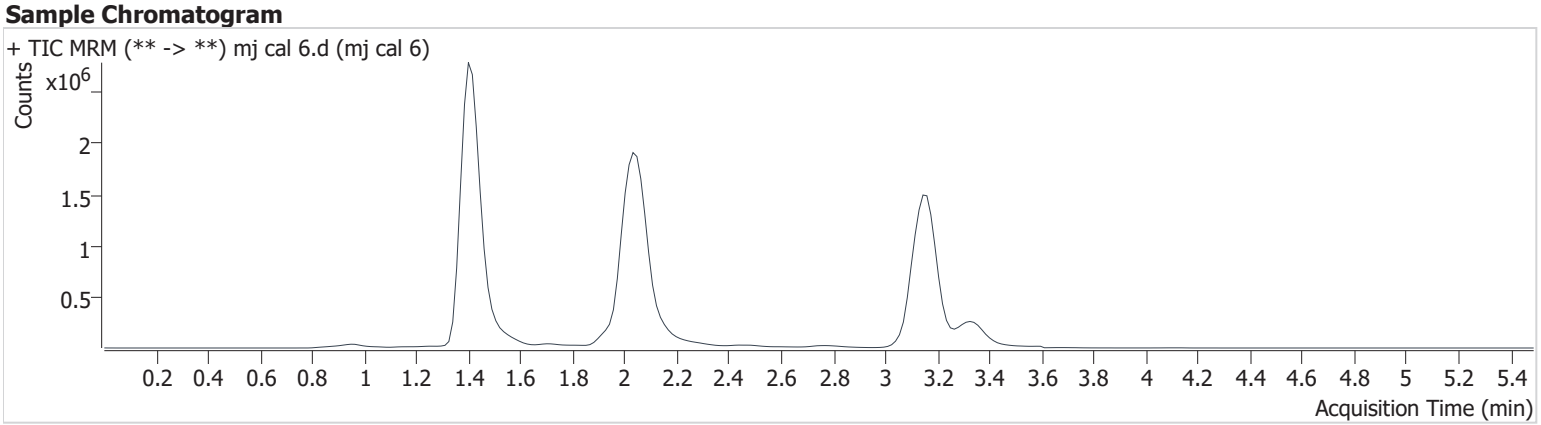
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	362916	∞	265.0	2479.7	950498	71.947 ng/ml
THC	3.167	2202644	∞	24.0	∞	3461396	24.879 ng/ml

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 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	8/4/2022 3:01:30 PM		
Sample Info.			

Sample Chromatogram



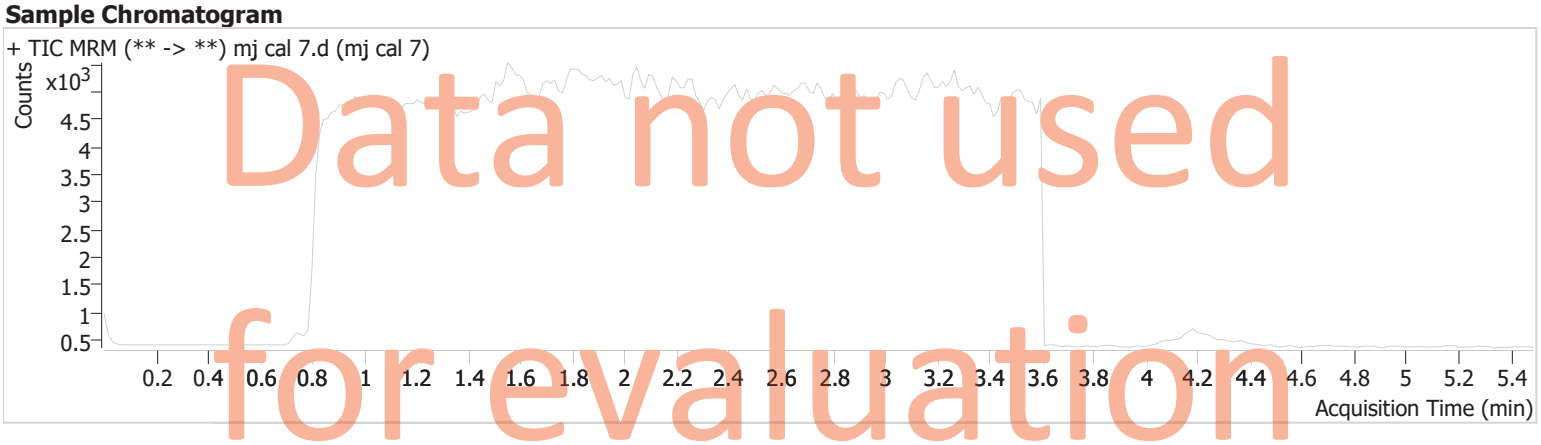
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	475414	∞	259.6	∞	926513	96.350 ng/ml
THC	3.167	4570053	∞	24.5	∞	3506232	50.590 ng/ml

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update	8/5/2022 9:07:25 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	8/4/2022 3:08:14 PM		
Sample Info.			

Sample Chromatogram



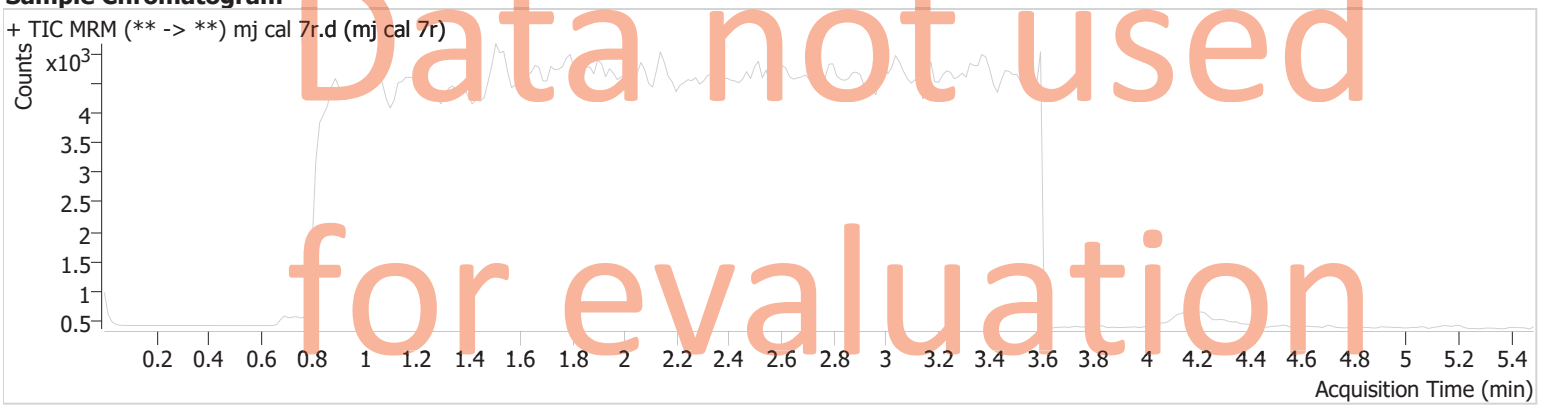
sample did not inject

AM #27 Cannabinoids

Batch results	D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update	8/5/2022 9:07:25 AM

Instrument	69679	**Data File**	mj cal 7r.d
Type	Cal	**Sample**	mj cal 7r
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/4/2022 3:48:28 PM		
Sample Info.			

Sample Chromatogram



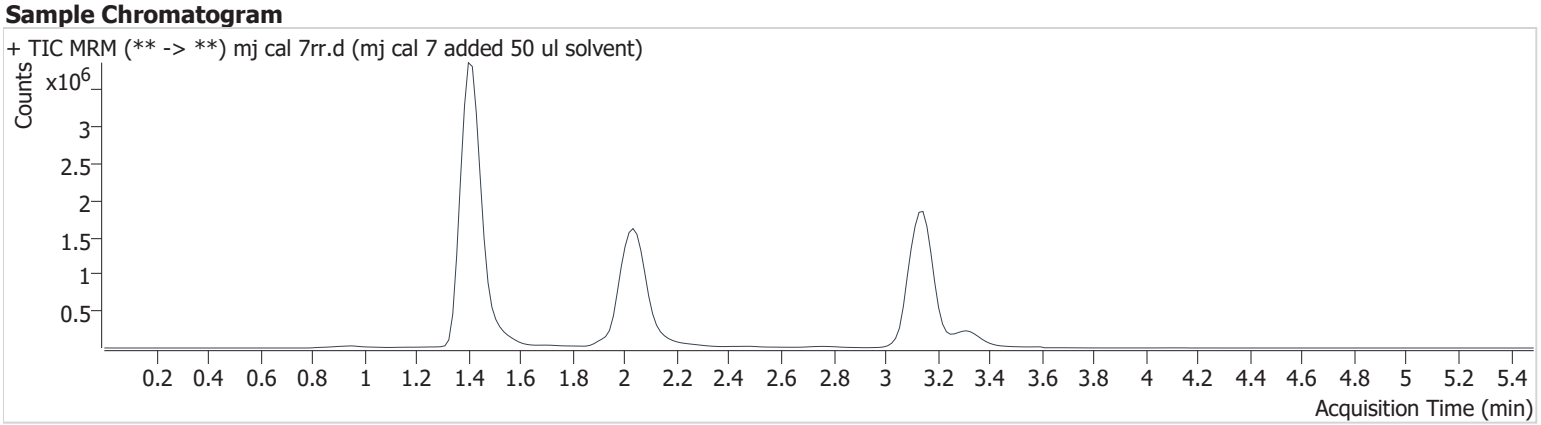
did not inject

AM #27 Cannabinoids

Batch results D:\MassHunter\Data\2022\am 27-28\080422\QuantResults\cann.batch.bin
Calibration Last Update 8/5/2022 9:07:25 AM

Instrument	69679	**Data File**	mj cal 7rr.d
Type	Cal	**Sample**	mj cal 7 added 50 ul solvent
Acq. Method	AM 27 THC quant.m	**Operator**	Anne Nord
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	8/4/2022 4:08:35 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-COOH	1.433	992068	∞	263.5	∞	713827	259.282 ng/ml
THC	3.152	7107925	∞	24.6	∞	2740503	100.323 ng/ml