REVIEWED
By Tamara Salazar at 2:14 pm, Jun 10, 2022

6/9/2022

Worklist: 5974

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2022-1179	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
M2022-1322	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
M2022-1889	3	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2022-0879	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2022-0997	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2022-1440	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ
P2022-1466	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ

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

Extraction Date: 05/25/2022
Plate lot#: 220309
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 20L20723
LCMS-QQQ ID: 069901

Analyst: Celena Shrum
Plate Retest Date: 09/09/2022
Mobile phase B: 0.1% Formic acid in Acetonitrile
Column: UCT Selectra DA 100 x 2.1mm 3um
Blank Urine Lot: POC021022

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. Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID: #42**
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 4. Pipette **500µL 0.1% formic acid in water blood sample** 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)***
☒ 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.
☒ 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 5ng/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, 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: Only THC and THC-COOH evaluated. Urine QC had to be reinjected due to it not injecting properly the first time. The sample was reinjected on 5/31/22 with no issues. A blood sample from a previous batch was also included in this run (M2022-1304-1).

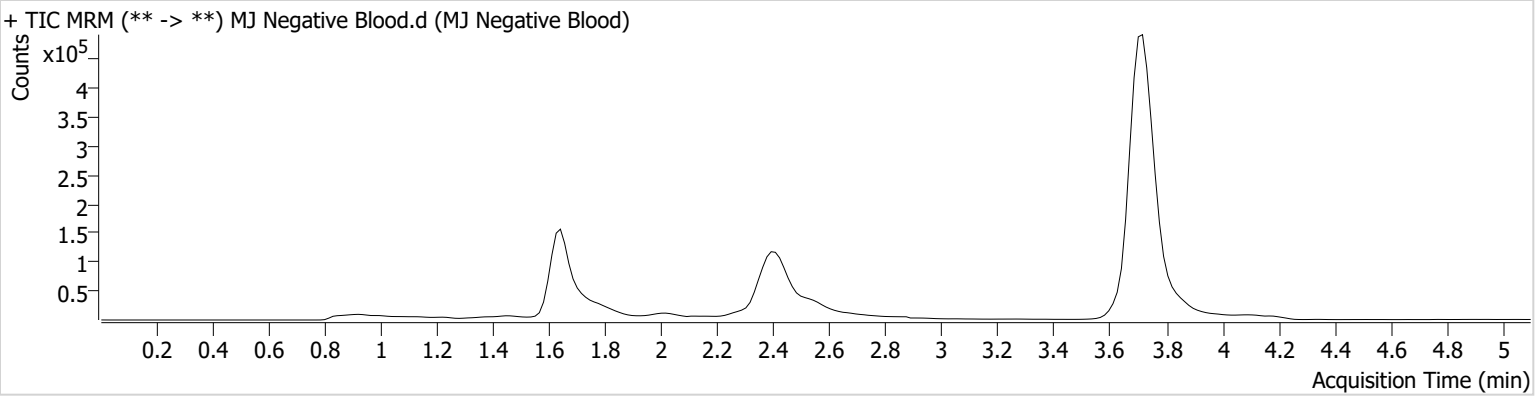
	1	2	3	4	5	6
a	cal 1ng	QC 2	P2022-1440-1			
b	cal 3 ng	NEG Blood	P2022-1466-1			
c	cal 5 ng	Neg Urine	M2022-1304-1			
d	cal 10ng	M2022-1179-2				
e	cal 25 ng	M2022-1322-2				
f	cal 50 ng	M2022-1889-3				
g	cal 100 ng	P2022-0879-4				
h	QC 1	P2022-0997-2				

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	MJ Negative Blood.d
Type	Sample	**Sample**	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 3:35:47 PM		
Sample Info.			

Sample Chromatogram

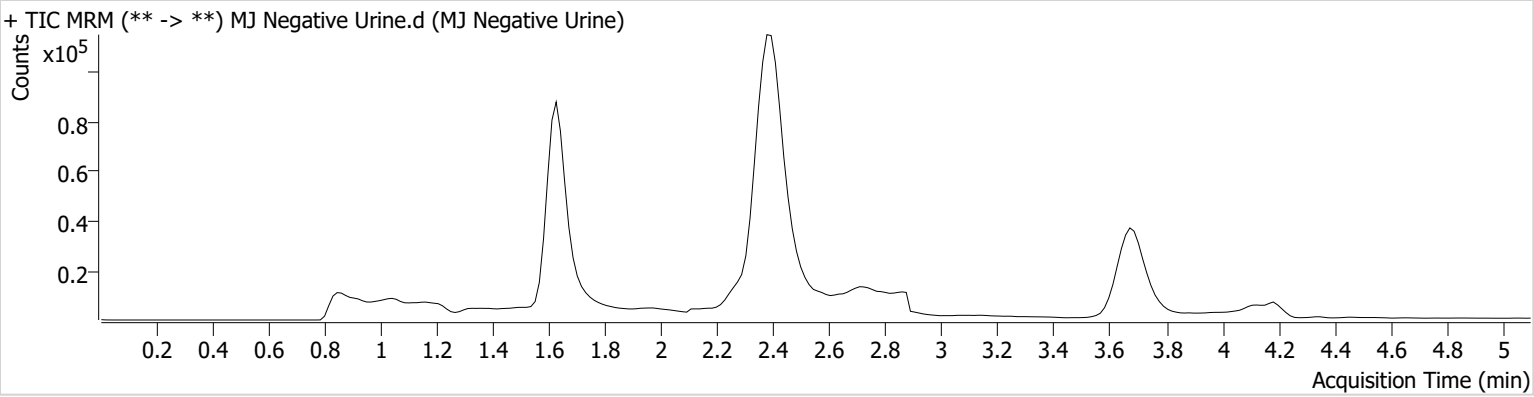


AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	MJ Negative Urine.d
Type	Sample	**Sample**	MJ Negative Urine
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-C2	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 3:43:24 PM		
Sample Info.			

Sample Chromatogram

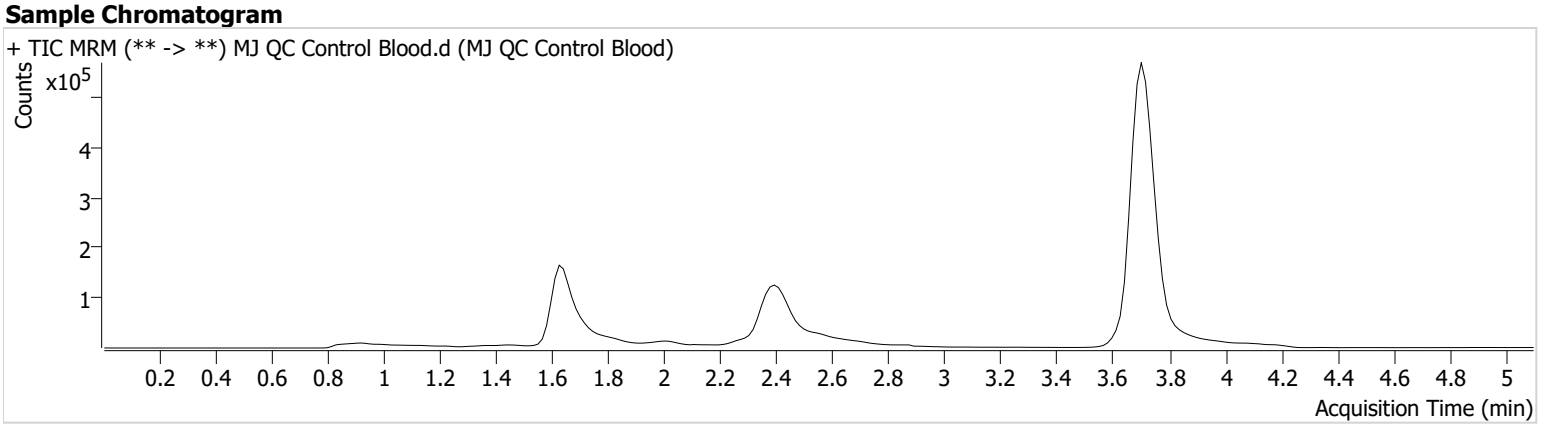


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	MJ QC Control Blood.d
Type	QC	**Sample**	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 3:20:35 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	56012	142.50	53.2	349.17	174457	15.7948	ng/ml
THC	3.721	130047	1431.24	29.4	∞	3517016	4.7324	ng/ml

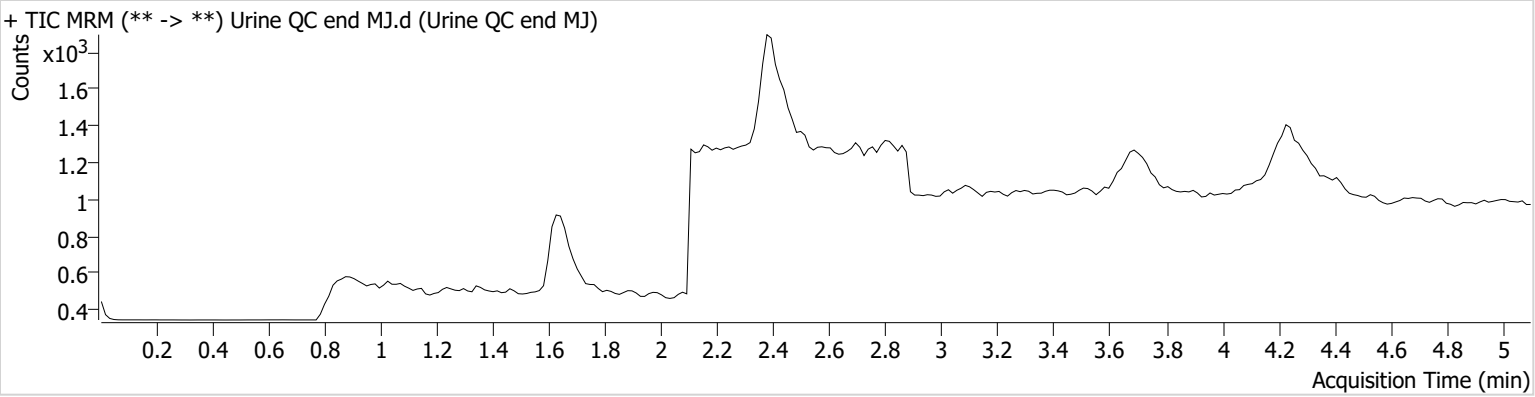
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Urine QC end MJ.d
Type	QC	**Sample**	Urine QC end MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 6:00:26 PM		
Sample Info.			

QC was reinjected due to an injection issue.

Sample Chromatogram



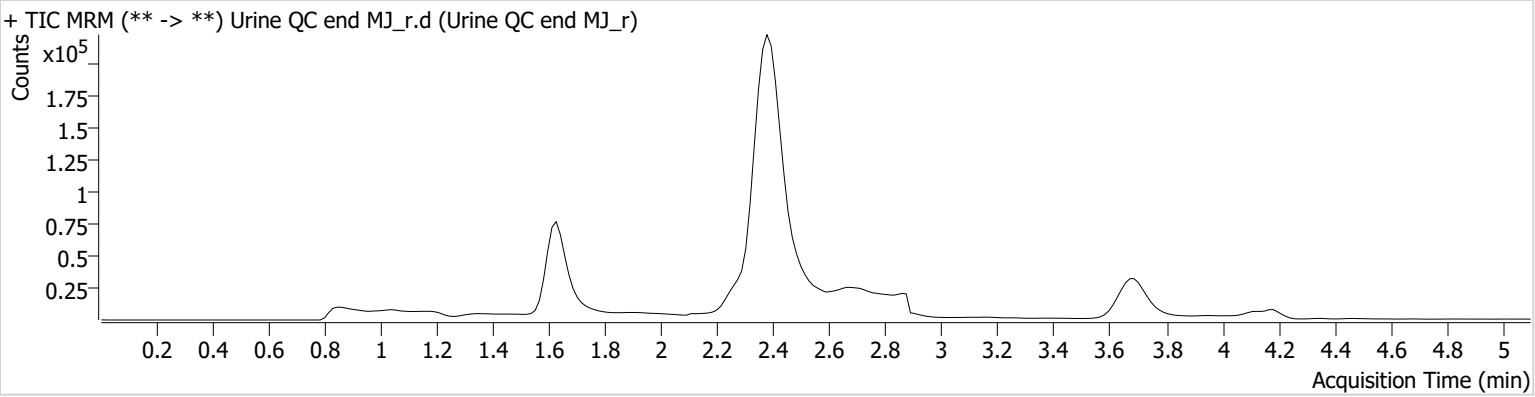
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Urine QC end MJ_r.d
Type	QC	**Sample**	Urine QC end MJ_r
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/31/2022 1:10:11 PM		
Sample Info.			

Reinjection data.

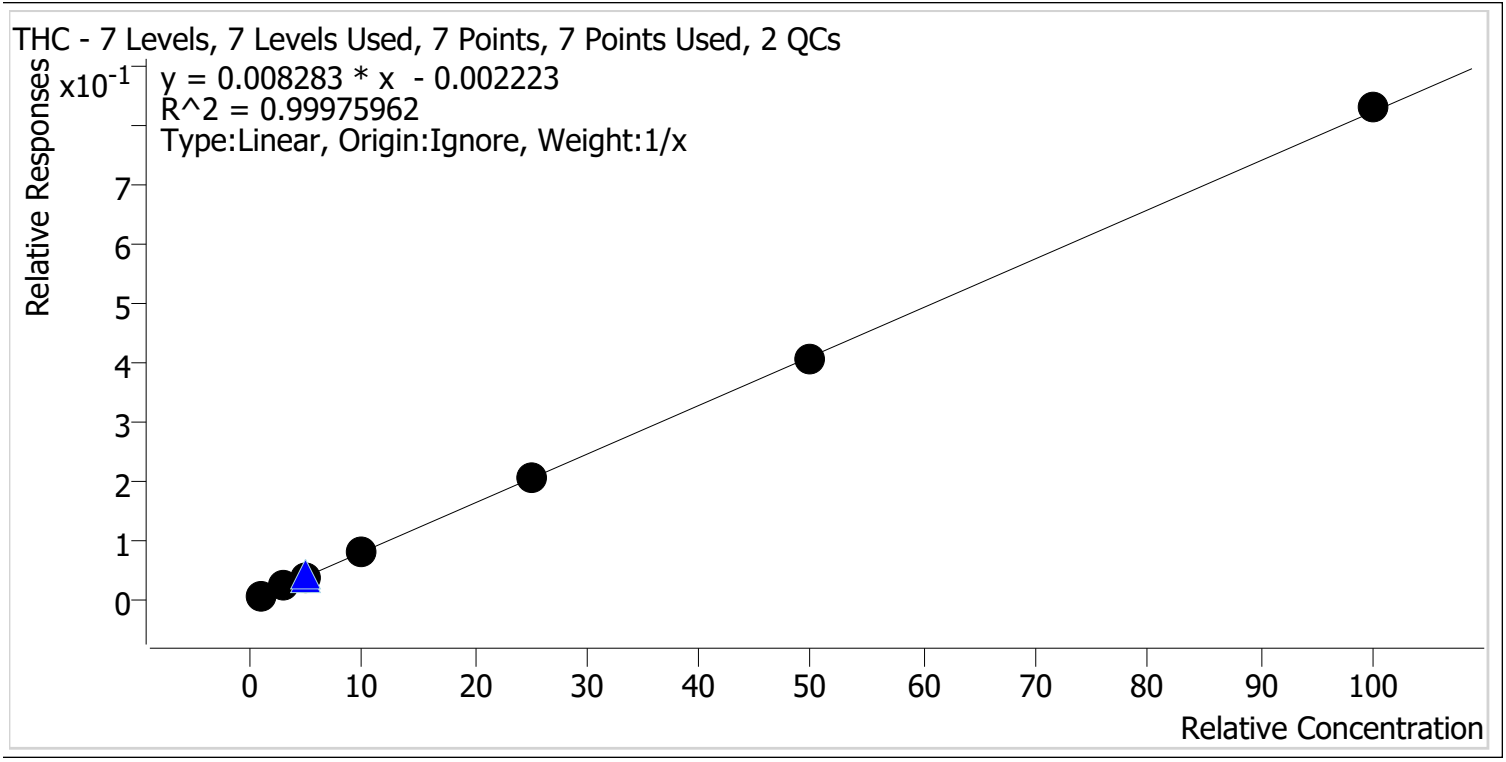
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.655	26379	122.04	50.7	97.09	76139	16.9646	ng/ml
THC	3.706	8997	∞	32.8	10.18	221272	5.1771	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

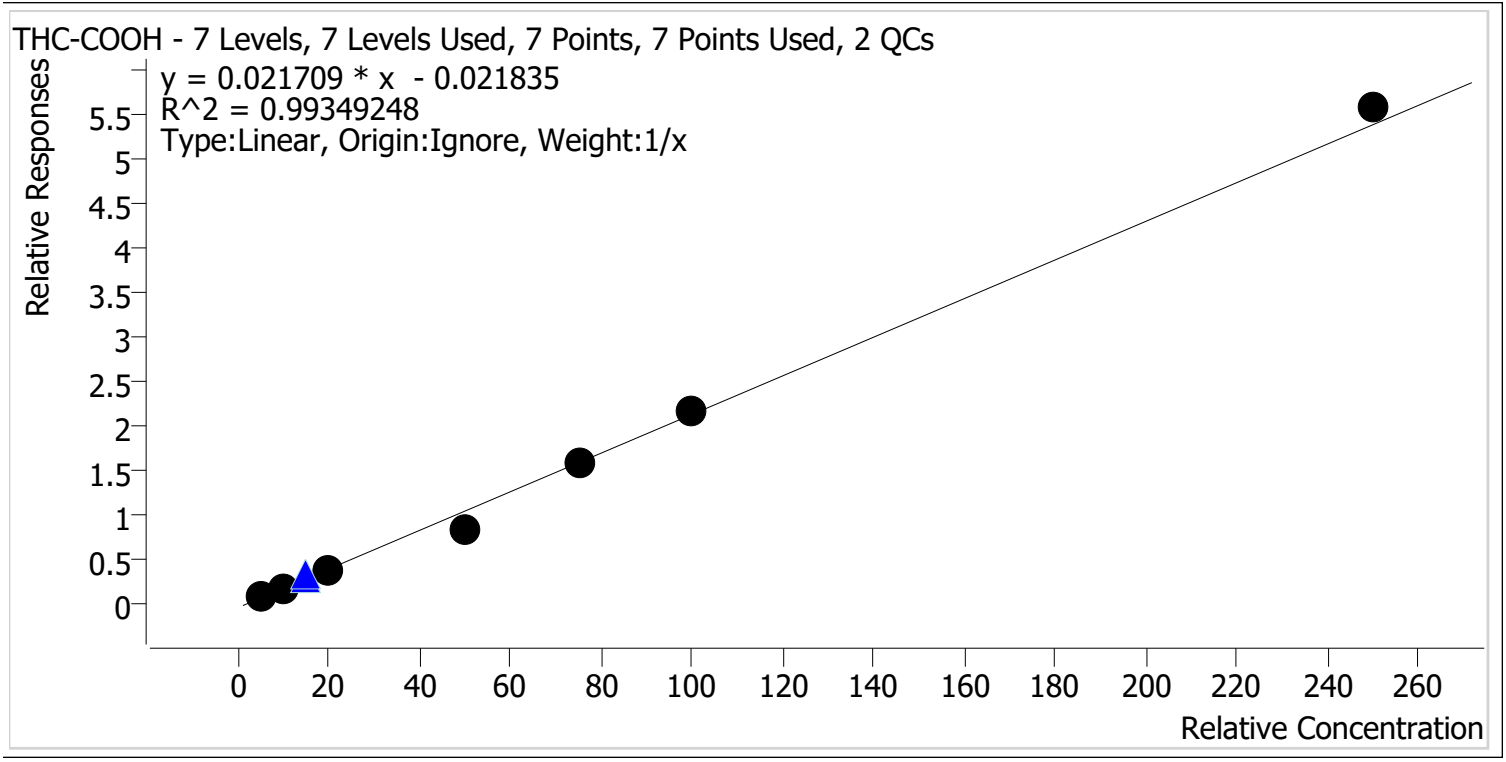
Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	6/9/2022 9:16 AM
Analyst Name	ISP\datastor
Analyte	THC
Internal Standard	THC-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	1.0	1.1	109.3
Cal 2 MJ	2	✔	3.0	3.0	98.4
Cal 3 MJ	3	✔	5.0	4.7	94.8
Cal 4 MJ	4	✔	10.0	9.7	97.4
Cal 5 MJ	5	✔	25.0	25.0	100.0
Cal 6 MJ	6	✔	50.0	49.6	99.3
Cal 7 MJ	7	✔	100.0	100.8	100.8

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Last Cal. Update	6/9/2022 9:16 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



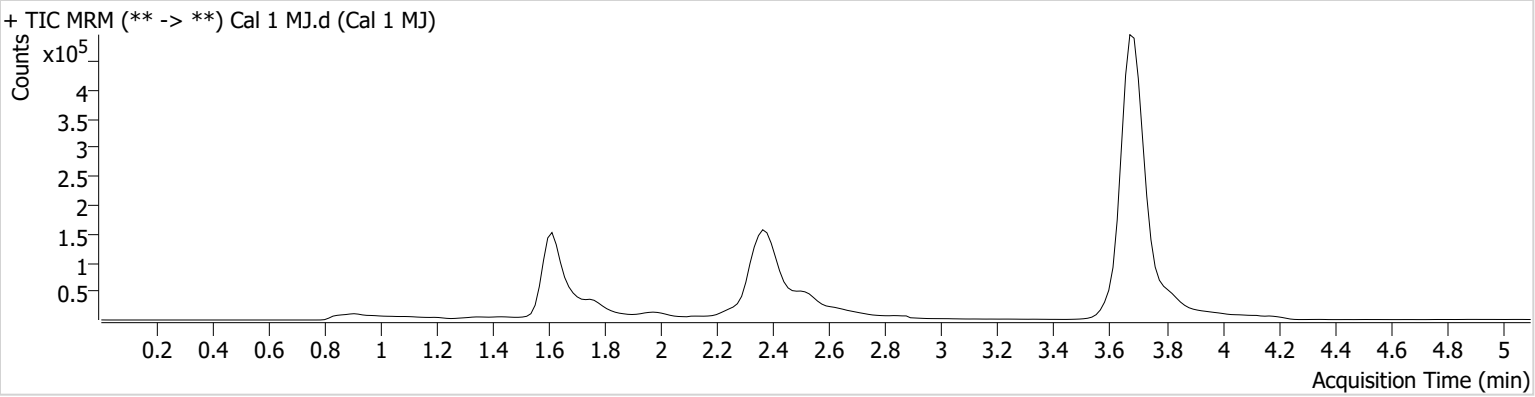
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
Cal 1 MJ	1	✔	5.0	6.1	122.2
Cal 2 MJ	2	✔	10.0	9.4	93.9
Cal 3 MJ	3	✔	20.0	19.9	99.5
Cal 4 MJ	4	✔	50.0	40.2	80.4
Cal 5 MJ	5	✔	75.0	74.6	99.5
Cal 6 MJ	6	✔	100.0	101.0	101.0
Cal 7 MJ	7	✔	250.0	258.8	103.5

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 1 MJ.d
Type	Cal	**Sample**	Cal 1 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:19:34 PM		
Sample Info.			

Sample Chromatogram



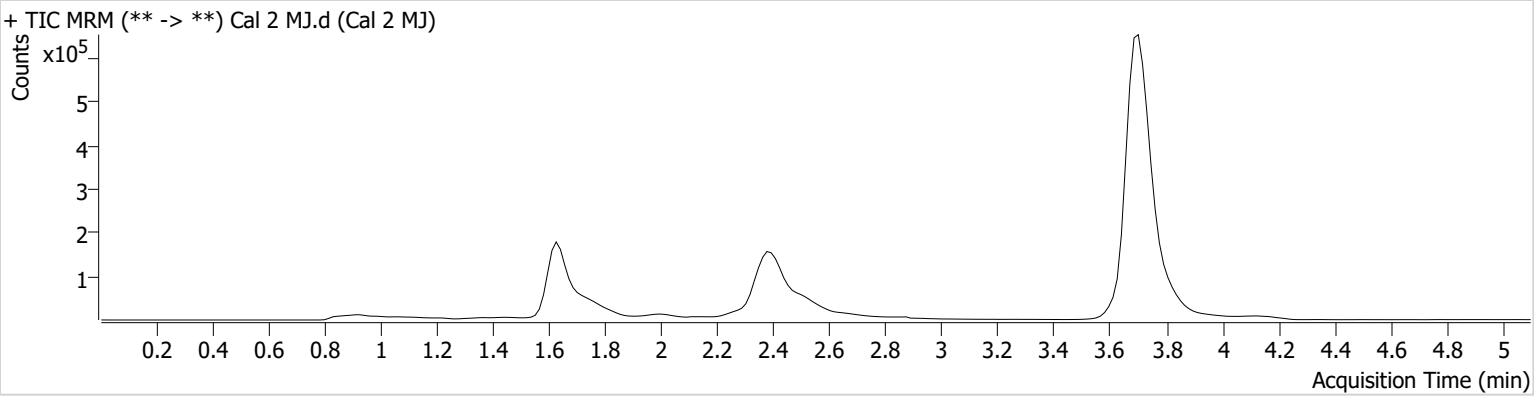
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.655	17588	∞	49.1	∞	158718	6.1100	ng/ml
THC	3.691	22676	123.18	32.0	22.03	3318620	1.0932	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 2 MJ.d
Type	Cal	**Sample**	Cal 2 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:27:21 PM		
Sample Info.			

Sample Chromatogram



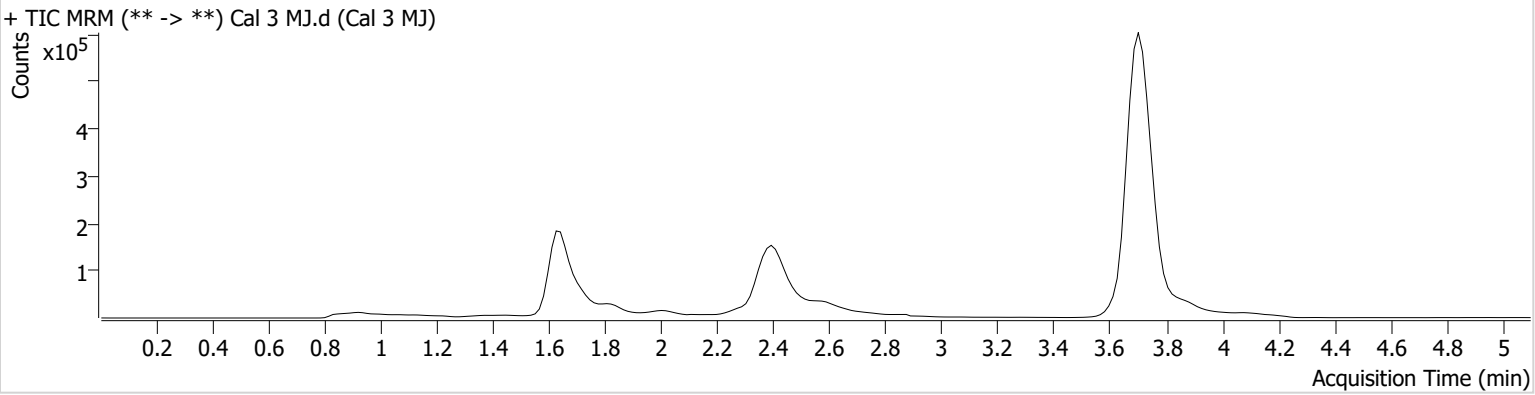
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	38675	75.55	58.7	331.38	212467	9.3906	ng/ml
THC	3.706	97248	1048.08	29.7	∞	4375423	2.9516	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 3 MJ.d
Type	Cal	**Sample**	Cal 3 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:34:57 PM		
Sample Info.			

Sample Chromatogram



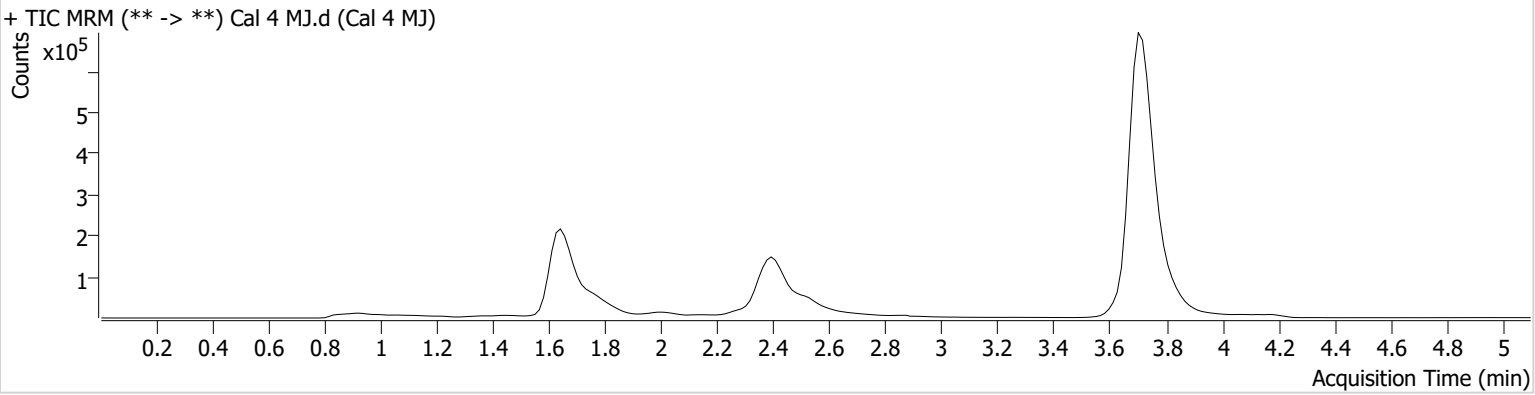
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	74441	69.94	53.9	∞	181418	19.9067	ng/ml
THC	3.706	140741	750.43	29.1	142.31	3799766	4.7400	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 4 MJ.d
Type	Cal	**Sample**	Cal 4 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:42:33 PM		
Sample Info.			

Sample Chromatogram



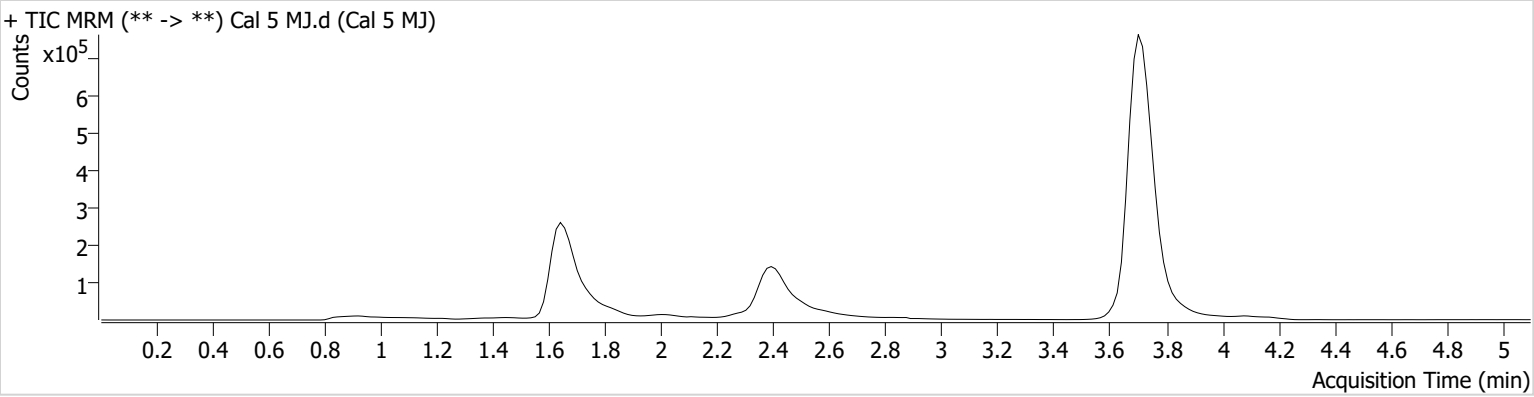
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	172888	∞	57.2	∞	203260	40.1857	ng/ml
THC	3.721	333836	∞	28.4	394.61	4257269	9.7351	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 5 MJ.d
Type	Cal	**Sample**	Cal 5 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:50:08 PM		
Sample Info.			

Sample Chromatogram



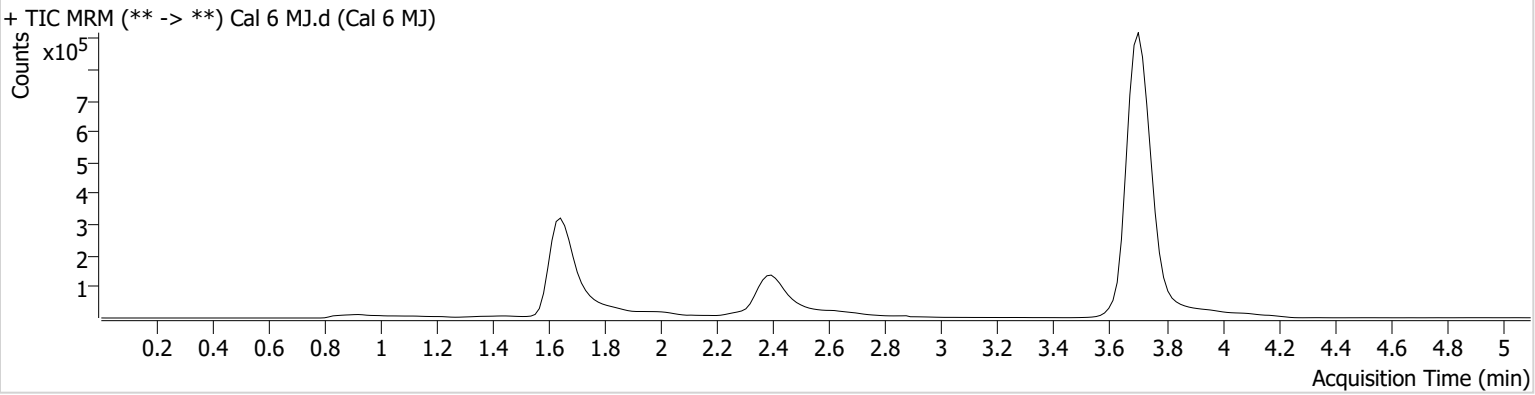
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	304881	365.61	56.9	∞	190795	74.6121	ng/ml
THC	3.721	807837	∞	27.9	852.41	3941988	25.0089	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update	6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 6 MJ.d
Type	Cal	**Sample**	Cal 6 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 2:57:44 PM		
Sample Info.			

Sample Chromatogram



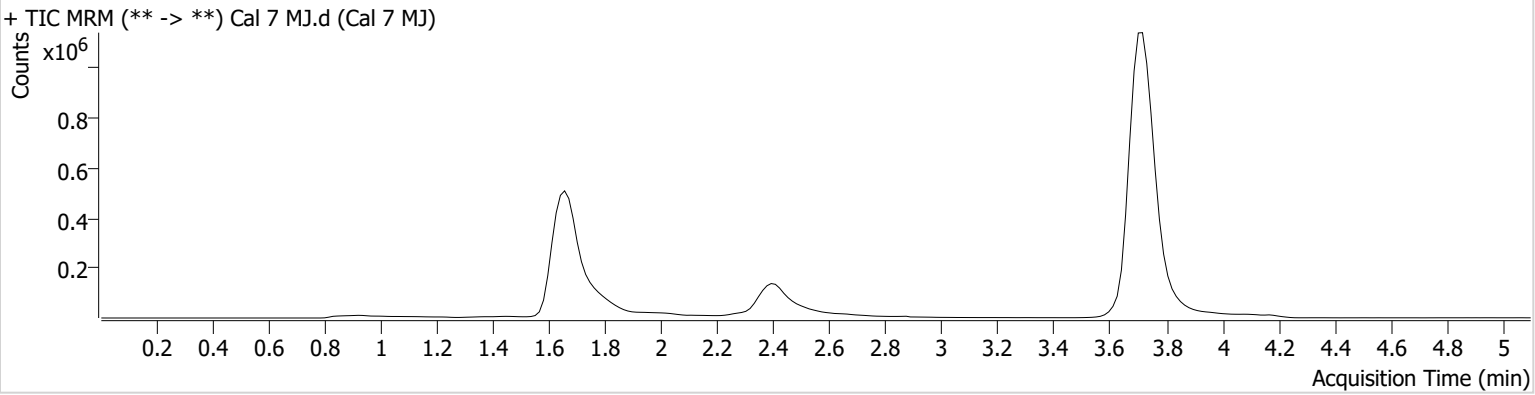
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	396122	846.45	57.1	2078.27	182512	100.9804	ng/ml
THC	3.706	1537977	∞	27.9	∞	3761257	49.6333	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\052522 AM 27 28 CS\QuantResults\AM 27.batch.bin
Calibration Last Update 6/9/2022 9:16:20 AM

Instrument	Falco (069901)	**Data File**	Cal 7 MJ.d
Type	Cal	**Sample**	Cal 7 MJ
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P5-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/25/2022 3:05:20 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.670	940617	∞	56.9	2730.20	168061	258.8145	ng/ml
THC	3.721	2966566	∞	28.4	3999.21	3561140	100.8379	ng/ml

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