REVIEWED
By Sarah Collins at 2:58 pm, Mar 23, 2022

3/23/2022 TS

Worklist: 5700

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2022-0566	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2022-0687	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
M2022-0826	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0432	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0433	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0585	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0593	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0670	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	
P2022-0830	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	

TS

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.

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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

TS

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

Extraction Date: 03/22/2022
Analyst: Tamara Salazar
Plate lot#: 211018
Plate Retest Date: 04/18/2022
Mobile phase A: 0.1% Formic Acid in LCMS Water
Mobile phase B: 0.1% Formic acid in Acetonitrile
Blank Blood Lot: Lampire 22B52016-2
Blank Urine Lot: POC021022
Column: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 069901

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 1.5mL urine to blank plate, add 250µl 1N KOH. Shake and incubate at 40 degrees for 15 minutes. 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, 500 µL saturated phosphate buffer in urine** in wells of analytical plate.
☒ 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 6. Transfer **700-800µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate. Amount transferred: 800µL
☒ 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 samples with calculated concentrations for THC at 1ng/mL or greater and OH-THC at 3ng/mL or greater may be reported quantitatively (blood only). Calculated concentrations for carboxy-THC of 5ng/mL may be reported qualitatively. 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-COOH evaluated with this run.

TS

	1	2	3	4	5	6
A	IS + Cal. 1	IS + QC_1	IS + Sample	IS + Sample	P2022-0433-1	IS + QC_1
B	IS + Cal. 2	IS + Sample	IS + Sample	IS + Sample	P2022-0432-1	IS + Cal. 7
C	IS + Cal. 3	IS + Sample	IS + Sample	IS + Sample	M2022-0826-2	IS + Cal. 6
D	IS + Cal. 4	IS + Sample	IS + Sample	IS + Sample	M2022-0687-4	IS + Cal. 5
E	IS + Cal. 5	IS + Sample	IS + Sample	P2022-0830-1	M2022-0566-4	IS + Cal. 4
F	IS + Cal. 6	IS + Sample	IS + Sample	P2022-0670-1	Neg Urine	IS + Cal. 3
G	IS + Cal. 7	IS + Sample	IS + Sample	P2022-0593-1	Neg Blood	IS + Cal. 2
H	IS + QC_1	IS + Sample	IS + Sample	P2022-0585-1	IS + QC_1	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

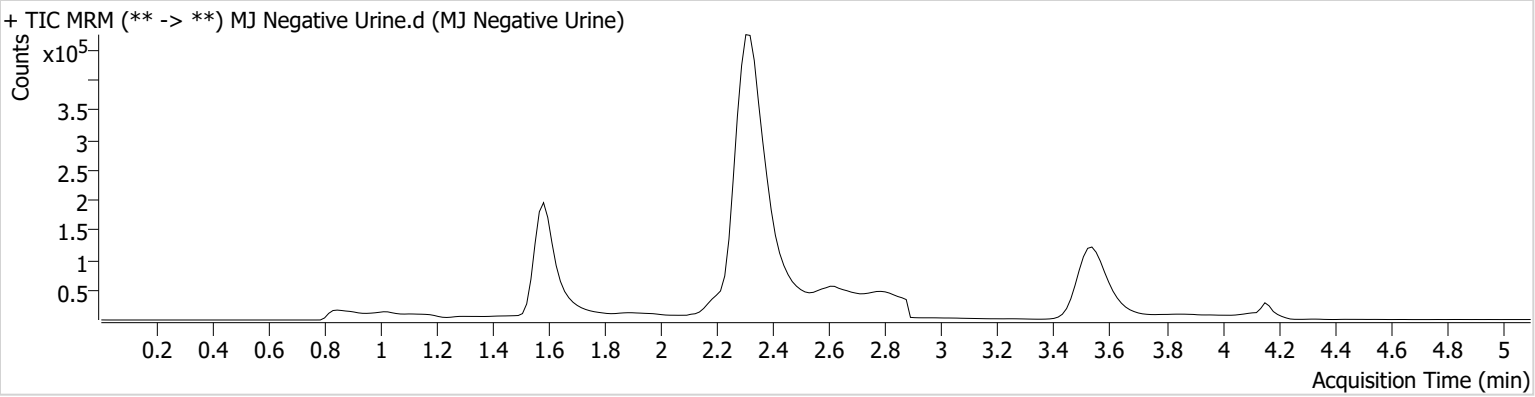
TS

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update	3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Negative Urine.d
Type	Sample	**Sample**	MJ Negative Urine
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-F5	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 3:33:43 PM		
Sample Info.			

Sample Chromatogram



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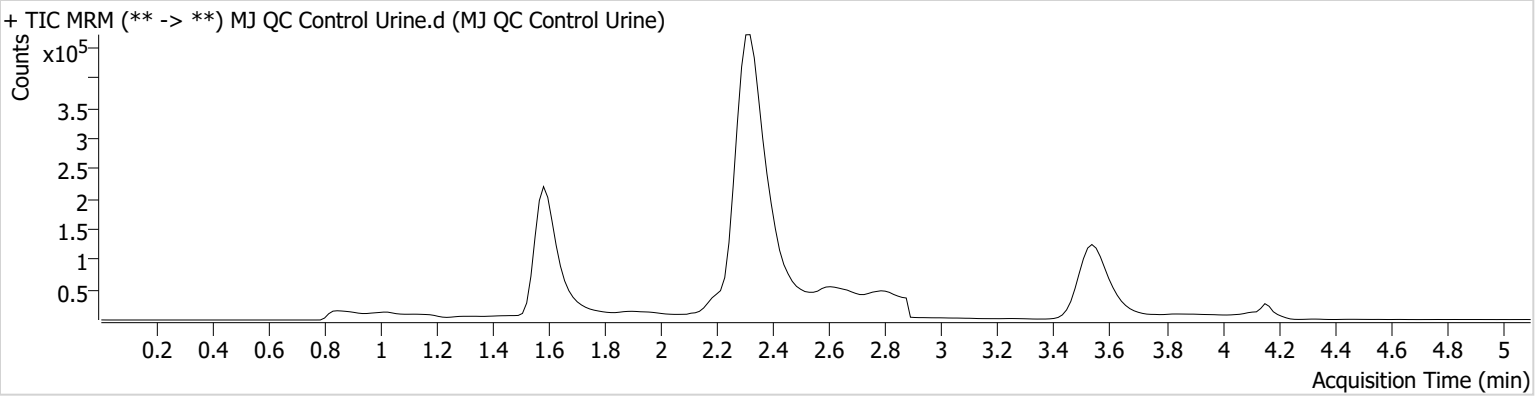
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update	3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ QC Control Urine.d
Type	QC	**Sample**	MJ QC Control Urine
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 3:18:30 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	69462	∞	62.8	138.20	195228	16.4109	ng/ml

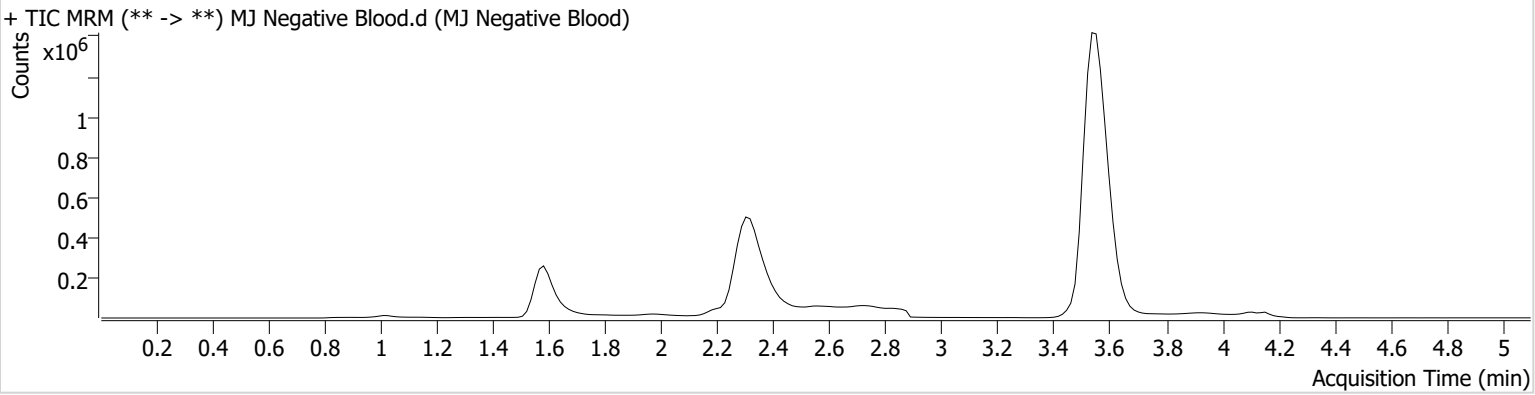
TS

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update	3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Negative Blood.d
Type	Sample	**Sample**	MJ Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-G5	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 3:10:54 PM		
Sample Info.			

Sample Chromatogram



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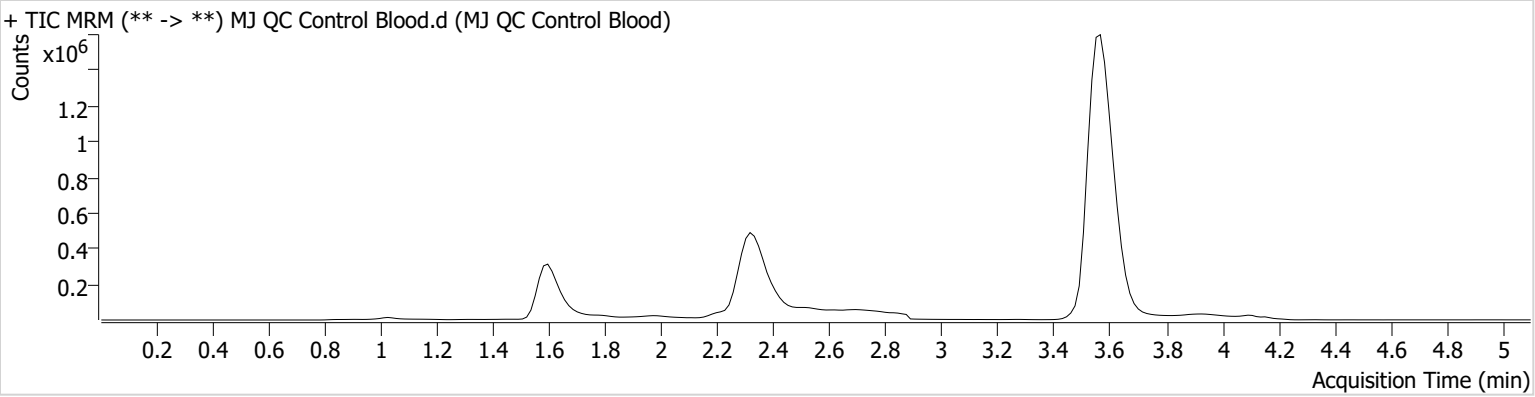
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ QC Control Blood.d
Type	QC	**Sample**	MJ QC Control Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:55:39 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	121580	∞	59.3	586.80	353755	15.8545	ng/ml

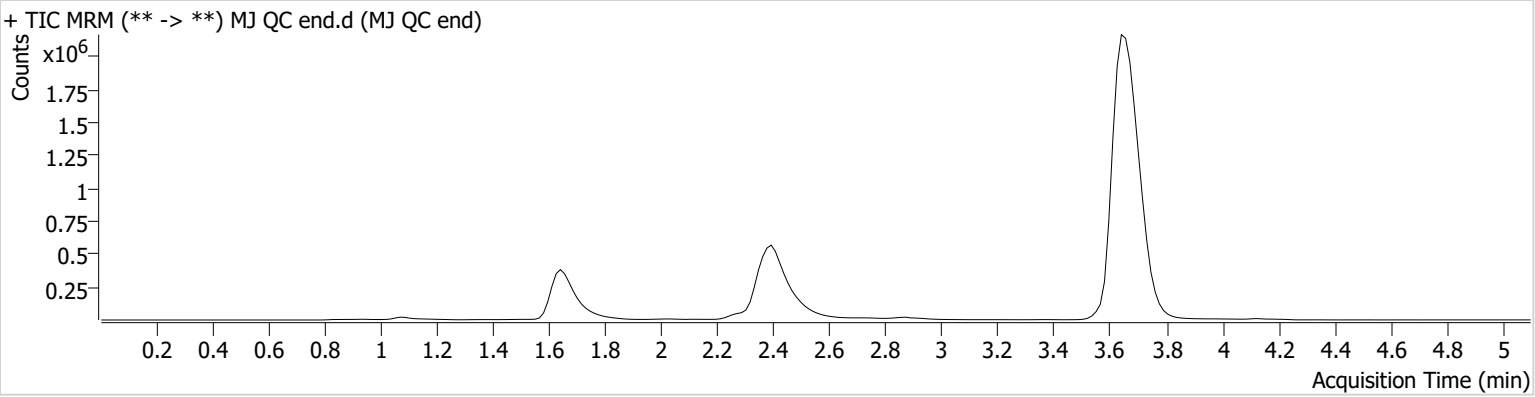
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ QC end.d
Type	QC	**Sample**	MJ QC end
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 6:06:05 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.685	176751	987.93	53.8	1205.79	440010	18.5197	ng/ml

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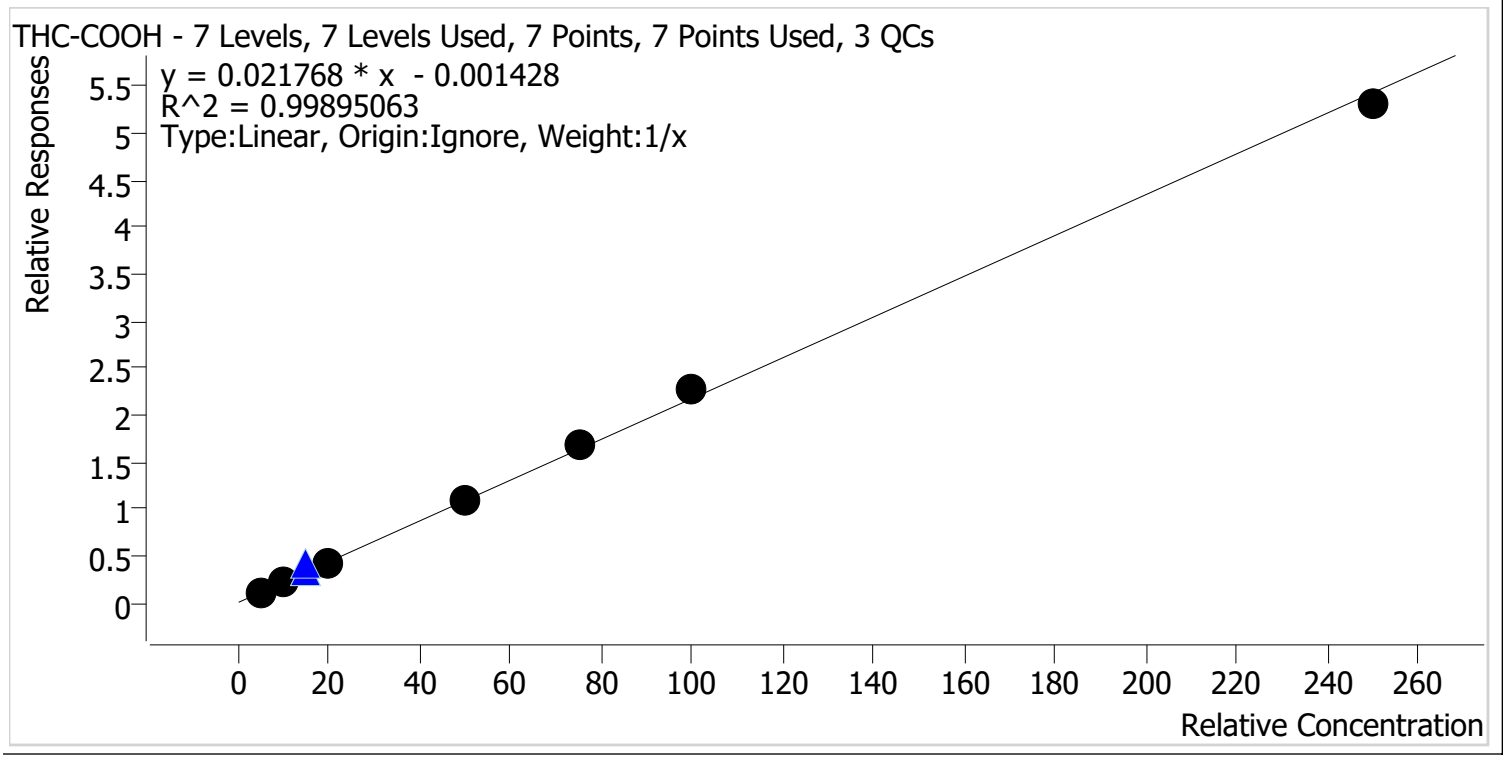
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Last Cal. Update	3/23/2022 12:32 PM
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	4.9	98.5
MJ Cal 2	2	✔	10.0	9.9	99.0
MJ Cal 3	3	✔	20.0	19.6	97.9
MJ Cal 4	4	✔	50.0	50.0	99.9
MJ Cal 5	5	✔	75.0	76.6	102.1
MJ Cal 6	6	✔	100.0	104.8	104.8
MJ Cal 7	7	✔	250.0	244.2	97.7

Generated at 12:43 PM on 3/23/2022

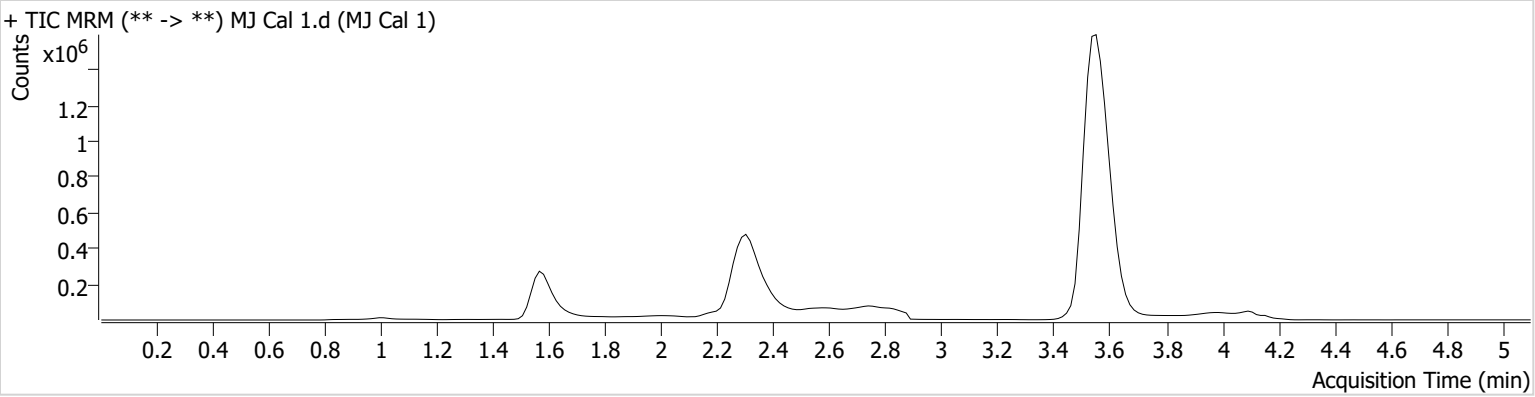
TS

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update	3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 1.d
Type	Cal	**Sample**	MJ Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:02:16 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.610	37442	∞	72.5	∞	353809	4.9272	ng/ml Low

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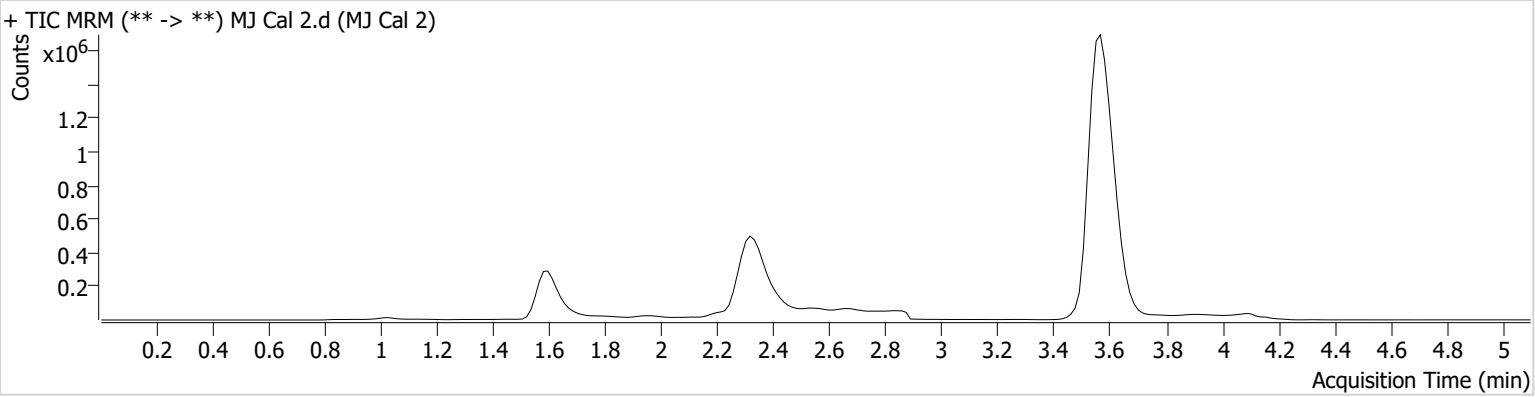
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 2.d
Type	Cal	**Sample**	MJ Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:10:03 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	76519	∞	75.1	893.89	357398	9.9014	ng/ml

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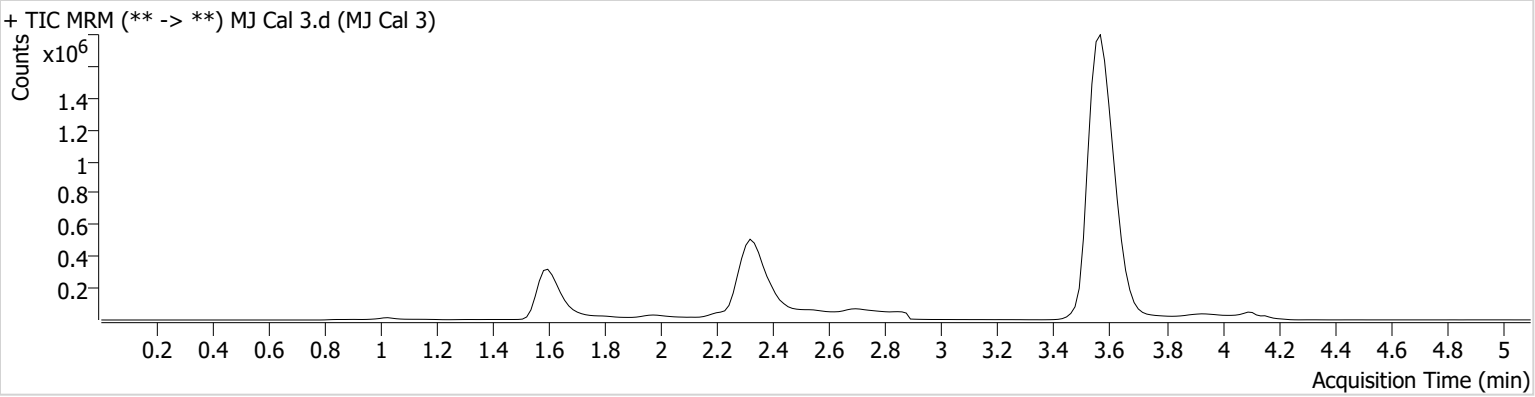
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 3.d
Type	Cal	**Sample**	MJ Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:17:39 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	156328	∞	60.7	∞	367998	19.5813	ng/ml

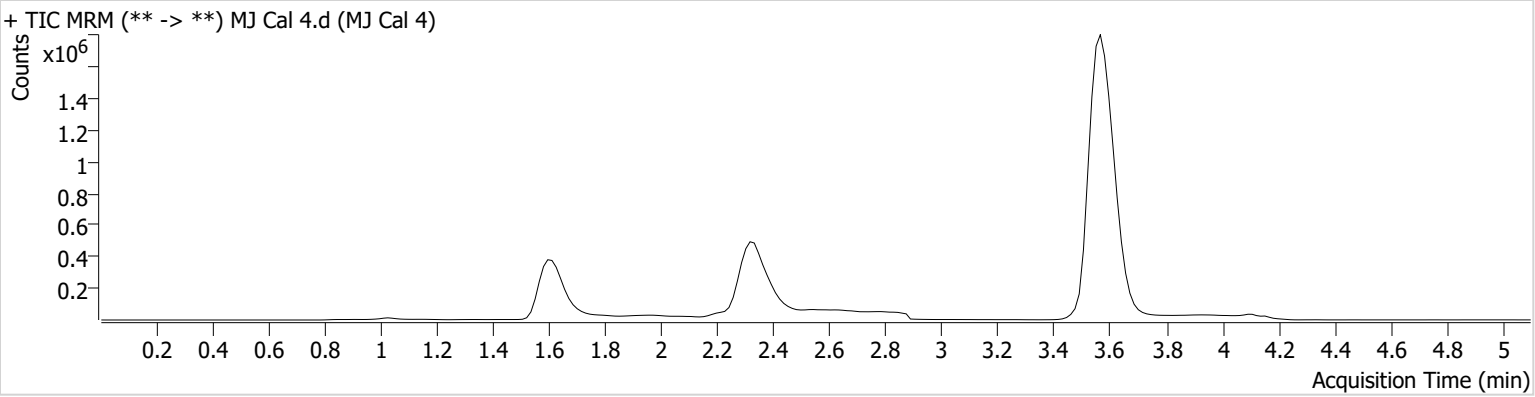
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 4.d
Type	Cal	**Sample**	MJ Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:25:14 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	386137	∞	62.1	4295.46	355505	49.9641	ng/ml

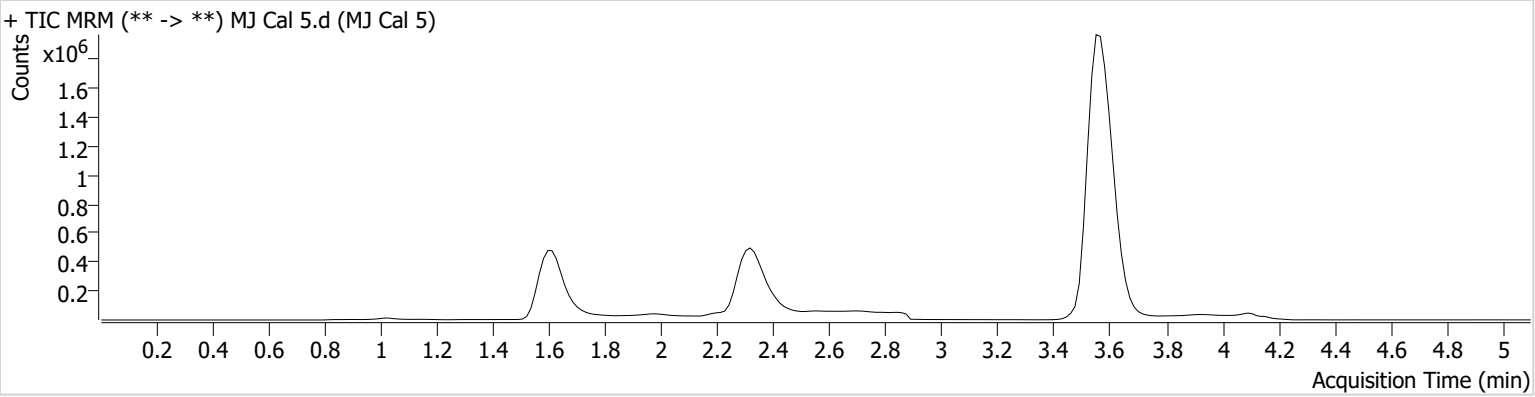
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 5.d
Type	Cal	**Sample**	MJ Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:32:50 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	592359	∞	70.3	12343.46	355620	76.5882	ng/ml

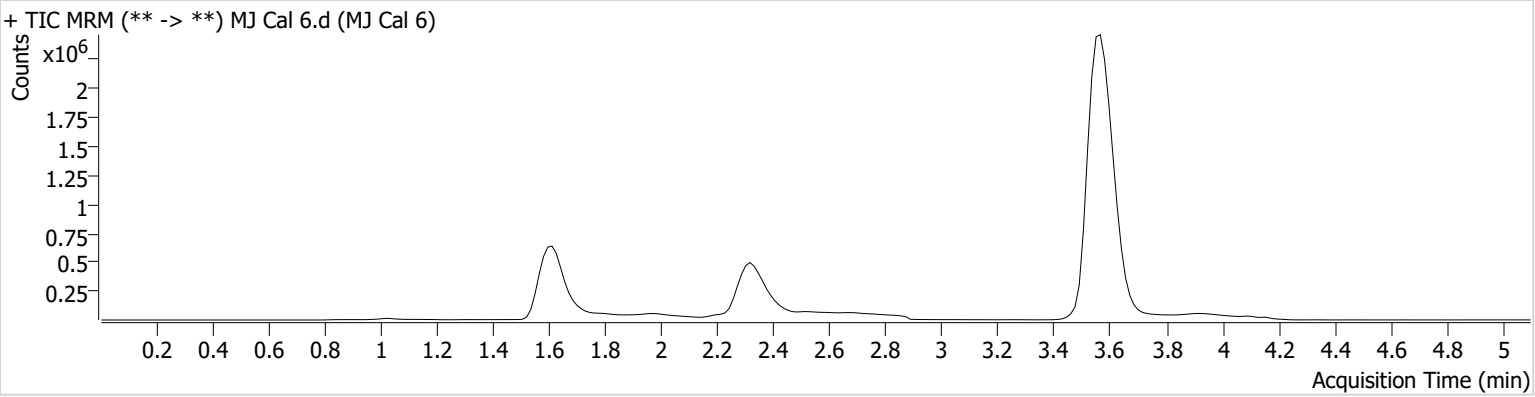
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 6.d
Type	Cal	**Sample**	MJ Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:40:26 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	809183	∞	57.3	∞	354970	104.7896	ng/ml

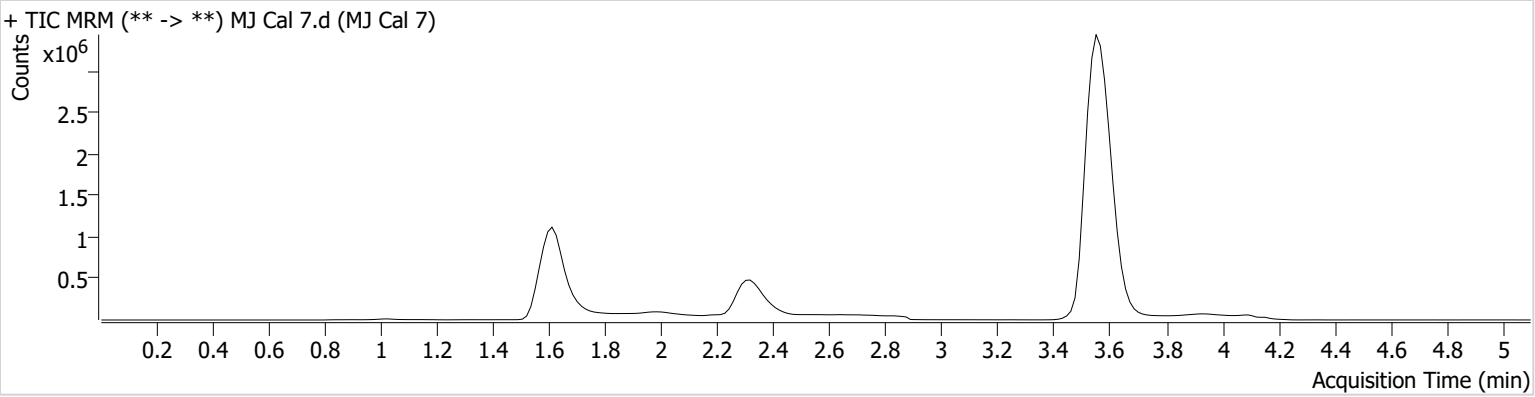
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2022\AM 27-28\032222 AM 27 28 Urines TS\QuantResults\AM 27_COOH only.batch.bin
Calibration Last Update 3/23/2022 12:32:05 PM

Instrument	Falco (069901)	**Data File**	MJ Cal 7.d
Type	Cal	**Sample**	MJ Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Tamara Salazar
Sample Position	P5-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/22/2022 2:48:03 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.625	1784748	∞	61.8	14918.85	335779	244.2481	ng/ml