TS

3/25/2020

Worklist: 4107

LAB CASE	ITEM	ITEM TYPE	DESCRIPTION	
M2019-1518	5	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
M2020-0865	4	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0574	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0656	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0705	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0780	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0801	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0803	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
P2020-0871	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖

Worklist: 4110

LAB CASE	ITEM	ITEM TYPE	DESCRIPTION
P2020-0498	4	URINE	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: 03/25/2020   Analyst: <u>Tamara Salazar</u>

Plate lot# IDP-108-2, 200303   Plate Expiration: 09-03-2020

Mobile phase A: 10mM Ammonium Formate   **Mobile phase B:** 0.1% Formic acid in MeOH
     0.1% Formic Acid in Water   MTBE   Hexane
Blank Blood Lot: 445283-3   **Column**: Phenomenex Phenyl Hexyl (4.6x50mm: 2.6 um)
Blank Urine Lot: POC031319
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 250ul 1N KOH. Shake and incubate at 40 degrees for 15 minutes.
☒   3.   Using a calibrated pipette, add **1000 µL blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID: #42**
☒   4.   Place on shaking incubator at ambient temp., 900rpm for 15 minutes.   *Shaker ID: 067105*
☒   5.   Pipette **500 µL 0.1% formic acid in water** for blood samples, **500µL saturated phosphate buffer** for urine samples, in wells of analytical plate.
☒   5.   Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒   6.   Transfer **800 µL of blood+acid or urine+acid** mixture to corresponding wells of SLE+ plate.
☒   7.   Apply positive pressure for approx. 4 seconds (or until no liquid remains on top of sorbent). *(Load at 85-100 PSI- Selector to the right)* Manifold ID: 067104
☒   8.   Wait 5 minutes.
☒   9.   Add **2.25 mL 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 **2.25 mL 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: 067103*
☒   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.
       Worklist path: *D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS*
       Batch Name: *AM 27 TS*
☒   2.   Calculated sample concentration of 3 ng/mL or greater for THC and THC-OH, a calculated sample concentration of 10 ng/mL or greater for Carboxy-THC.
☒   3.   Retention time within +/- 2% or +/-.100 min whichever is greater of the average retention time of the calibrators.
☒   4.   Did all QCs pass for each analyte? Y / N

☒ 5.　Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *Curves Limited: THC 3-100, THC-COOH 10-250, THC-OH 5-100*

Due to extenuating circumstances, P2020-0498-4 from worklist 4110 was assigned to Tamara Salazar. Tamara Salazar acted as the primary analyst on the case to open the evidence and perform the necessary extractions. Celena Shrum reviewed all of the data and central files. *TS*

Idaho State Police
Forensic Services

AM #26 Screening of THC and Metabolites Screen in Blood and Urine by LCMS-QQQ
AM #27 Quantitative Analysis of THC and Metabolites in Blood and Urine by LCMS-QQQ

Methanol External Control Solution (Lot: WS021320)
100 ul of 100 ug/mL C-THC in 9900 ul MeOH
Approximate concentration 1ug/mL.

Component	Source	Source Lot Number	Expiration Date
Methanol (LCMS)	Fisher	193941	
C-THC	Cerilliant	FE07171501	09/30/2020
Prepared:	02/13/2020		
Prepared By:	Celena Shrum		
Expires:	Per AM 21 reference materials used for qualitative purposes do not have an expiration date.		

Urine External Control Solution (Lot: 021320)
200 ul of methanol external control solution was added to 9800 ul of urine.
Approximately 20ng/mL.

| Component	Source	Source Lot Number
Negative Urine	Pocatello Lab	POC031319
Methanol External Control Solution	-	WS021320
Prepared:	02/13/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Negative Blood.d
Type	Sample	**Sample**	MJ_Negative Blood
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 4:37:52 PM		
Sample Info.			

Sample Chromatogram
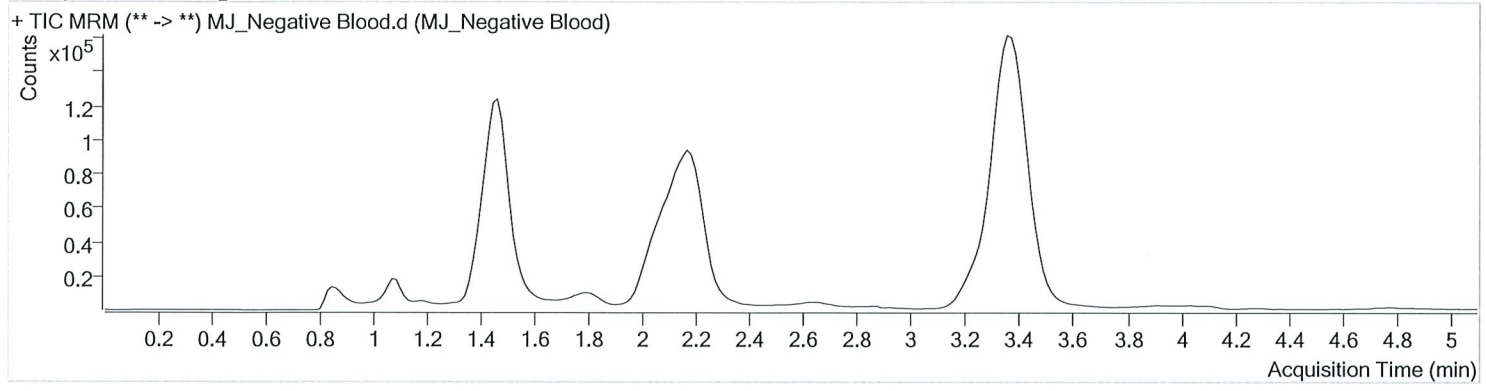
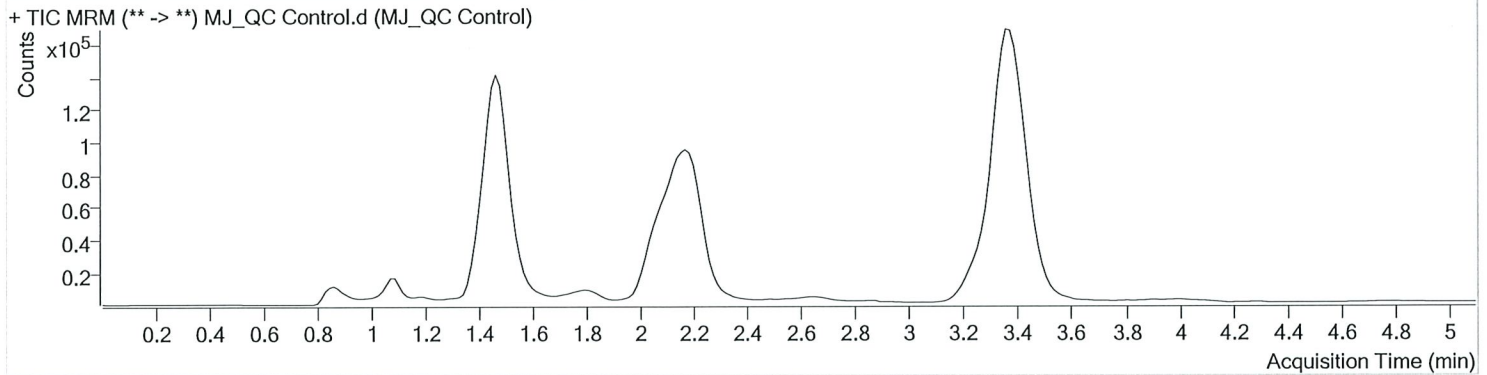+ TIC MRM (** -> **) MJ_Negative Blood.d (MJ_Negative Blood)

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_QC Control.d
Type	Sample	**Sample**	MJ_QC Control
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 4:22:41 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_QC Control.d (MJ_QC Control)

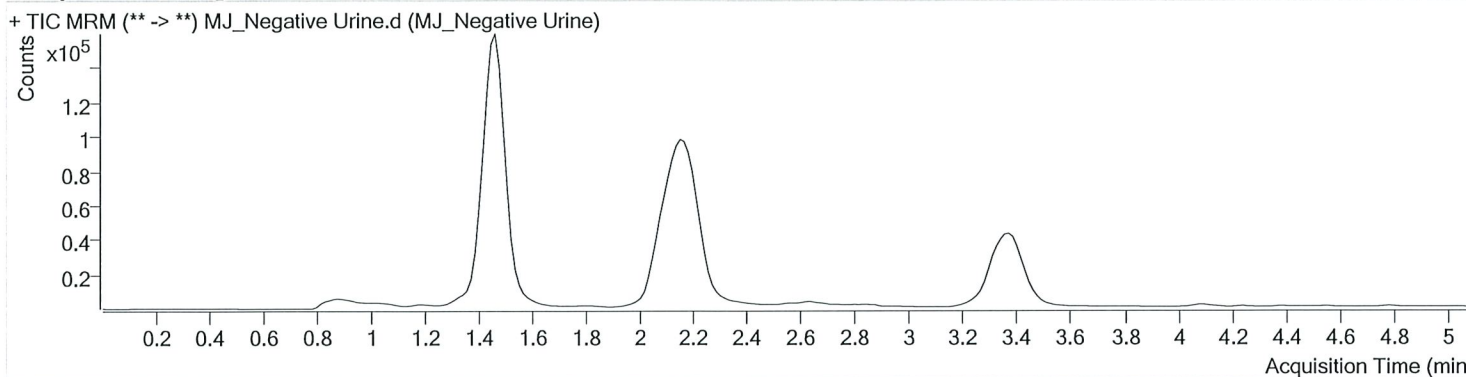| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	98930	∞	8.8	77.15	571814	4.4467	ng/ml
THC-COOH	1.504	73835	266.39	52.2	209.64	190478	15.6040	ng/ml
THC	3.390	58208	145.12	27.4	18.92	1514888	4.4256	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Negative Urine.d
Type	Sample	**Sample**	MJ_Negative Urine
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-C2	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 5:08:13 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Negative Urine.d (MJ_Negative Urine)

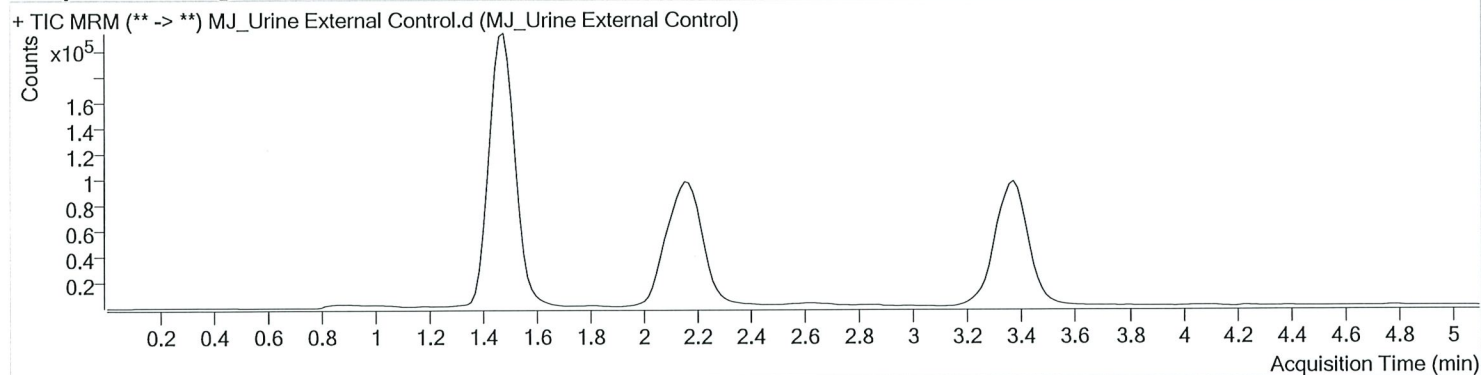| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.339 **Low**	29417	∞			204947	5.0439	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update   3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Urine External Control.d
Type	Sample	**Sample**	MJ_Urine External Control
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-B2	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 4:53:03 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Urine External Control.d (MJ_Urine External Control)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.504	229405	∞	59.8	1153.23	219078	44.1357	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Last Cal. Update	3/26/2020 9:56 AM
Analyst Name	ISP\datastor

Analyte	THC	**Internal Standard**	THC-D3

THC - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 0 QCs
$y = 0.009302 * x - 0.002746$
$R^2 = 0.99889927$
Type:Linear, Origin:Ignore, Weight:1/x
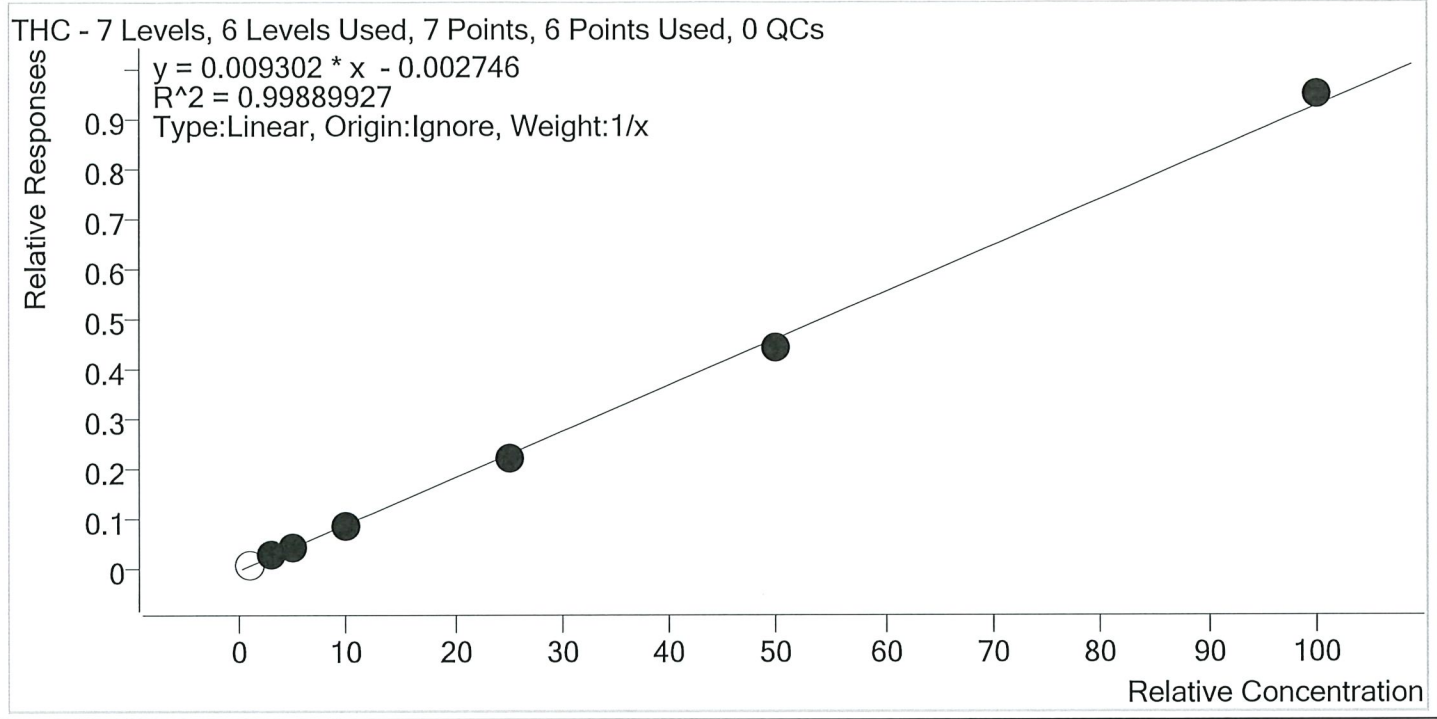
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✗	1.0	1.3	128.8
MJ_Cal 2	2	✓	3.0	3.2	105.1
MJ_Cal 3	3	✓	5.0	5.1	102.1
MJ_Cal 4	4	✓	10.0	9.6	95.7
MJ_Cal 5	5	✓	25.0	24.6	98.2
MJ_Cal 6	6	✓	50.0	48.2	96.5
MJ_Cal 7	7	✓	100.0	102.4	102.4

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Last Cal. Update	3/26/2020 9:56 AM
Analyst Name	ISP\datastor
Analyte	THC-COOH

Internal Standard   THC-COOH-D9

THC-COOH - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 0 QCs

$y = 0.023115 * x + 0.026943$
$R^2 = 0.99937382$
Type:Linear, Origin:Ignore, Weight:1/x
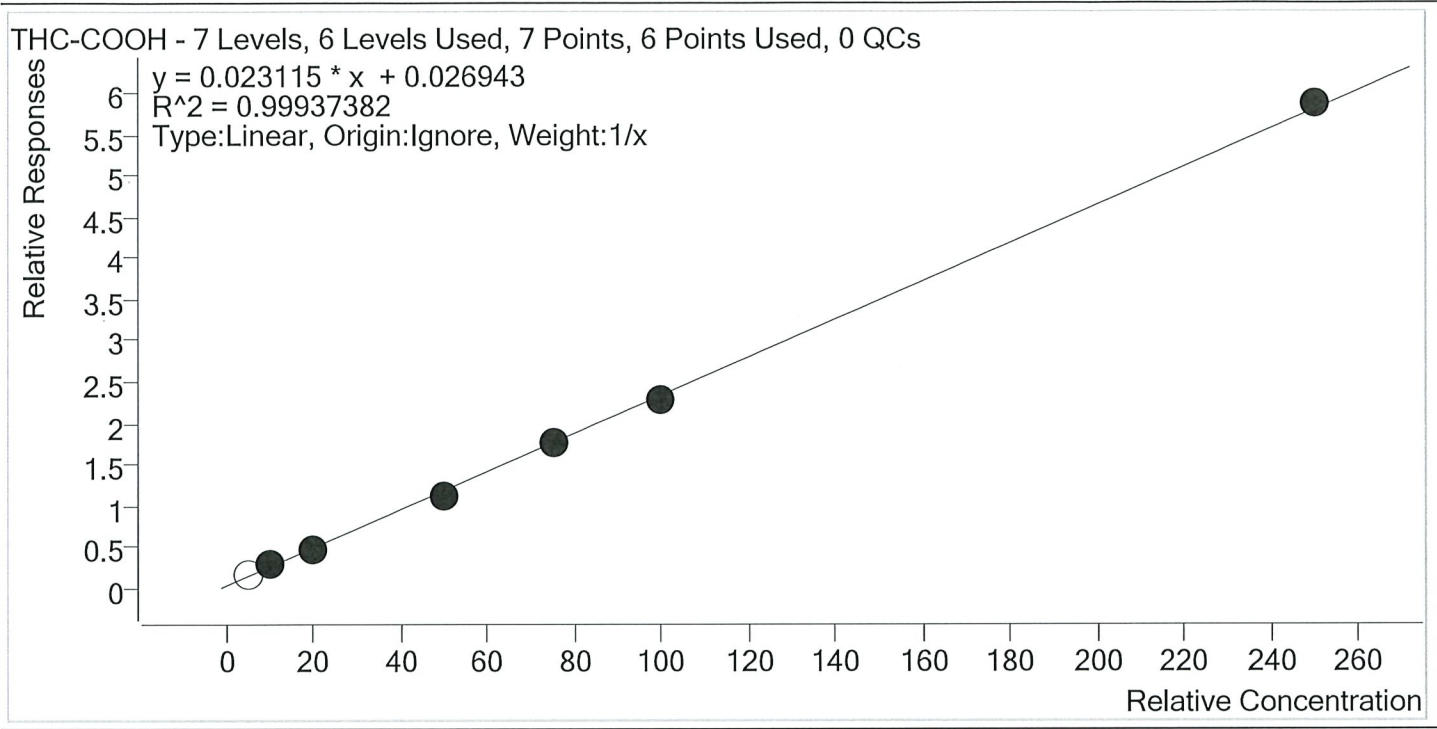
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✗	5.0	6.1	121.0
MJ_Cal 2	2	✓	10.0	10.6	105.9
MJ_Cal 3	3	✓	20.0	19.6	97.9
MJ_Cal 4	4	✓	50.0	47.9	95.8
MJ_Cal 5	5	✓	75.0	75.7	101.0
MJ_Cal 6	6	✓	100.0	98.3	98.3
MJ_Cal 7	7	✓	250.0	252.9	101.2

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin

Last Cal. Update   3/26/2020 9:56 AM

Analyst Name   ISP\datastor

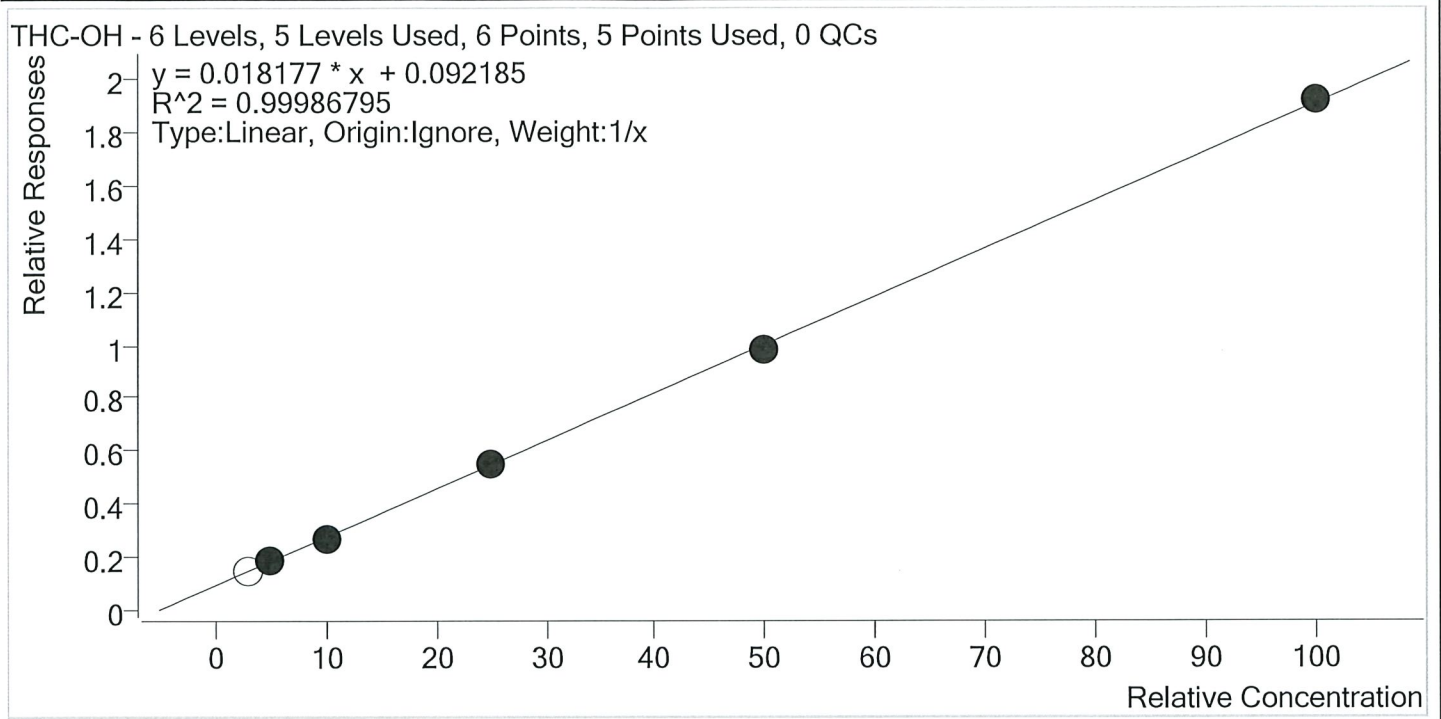**Analyte**   THC-OH   **Internal Standard**   THC-OH-D3

THC-OH - 6 Levels, 5 Levels Used, 6 Points, 5 Points Used, 0 QCs
$y = 0.018177 * x + 0.092185$
$R^2 = 0.99986795$
Type:Linear, Origin:Ignore, Weight:1/x

| Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 2	2	✗	3.0	3.1	103.6
MJ_Cal 3	3	✓	5.0	5.0	100.7
MJ_Cal 4	4	✓	10.0	10.0	99.6
MJ_Cal 5	5	✓	25.0	25.1	100.5
MJ_Cal 6	6	✓	50.0	49.3	98.5
MJ_Cal 7	7	✓	100.0	100.6	100.6

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update   3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 1.d
Type	Cal	**Sample**	MJ_Cal 1
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:21:52 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 1.d (MJ_Cal 1)
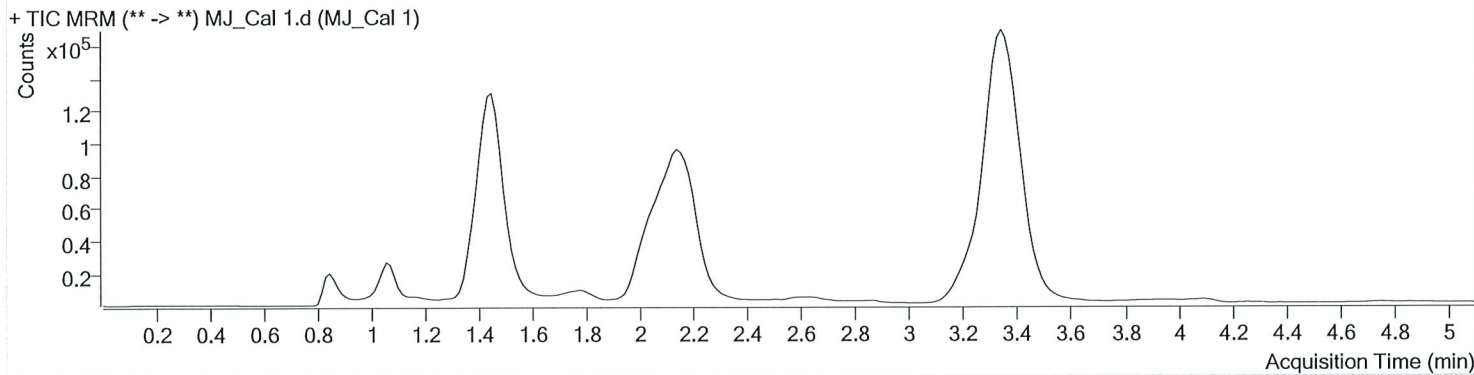
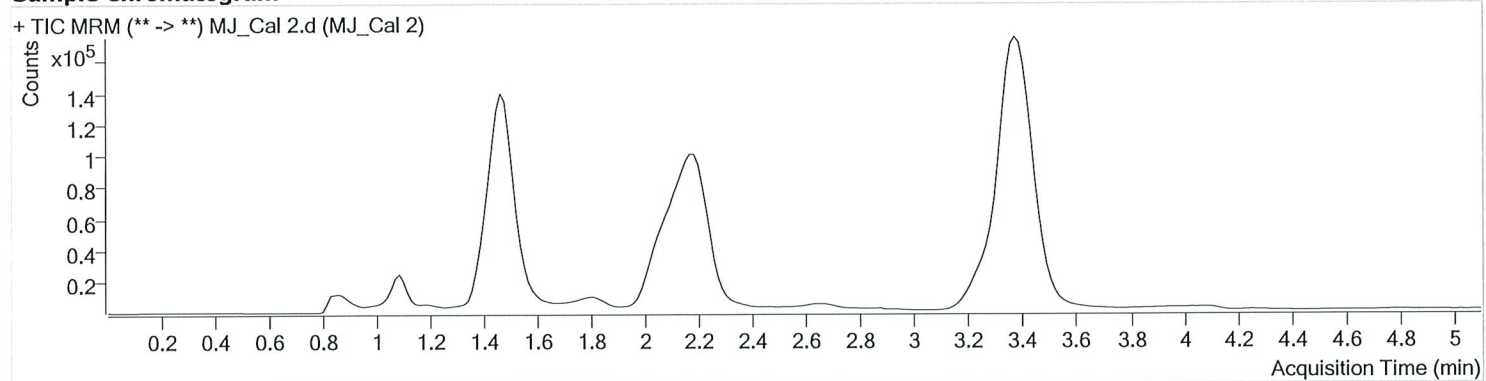| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.474	33502	91.83	39.2 **Low**	248.14	200807	6.0520	ng/ml **Low**
THC	3.360	15145	7.63 **Low**	27.5	3.07 **Low**	1639086	1.2884	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update   3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 2.d
Type	Cal	**Sample**	MJ_Cal 2
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:29:37 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 2.d (MJ_Cal 2)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.528 **High**	89556	∞	5.8 **Low**	14.61	602432	3.1069	ng/ml
THC-COOH	1.504	53388	68.88	46.9	194.41	196499	10.5884	ng/ml
THC	3.390	43203	202.94	25.5	13.70	1625211	3.1528	ng/ml

Batch results D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 3.d
Type	Cal	**Sample**	MJ_Cal 3
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:37:12 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 3.d (MJ_Cal 3)

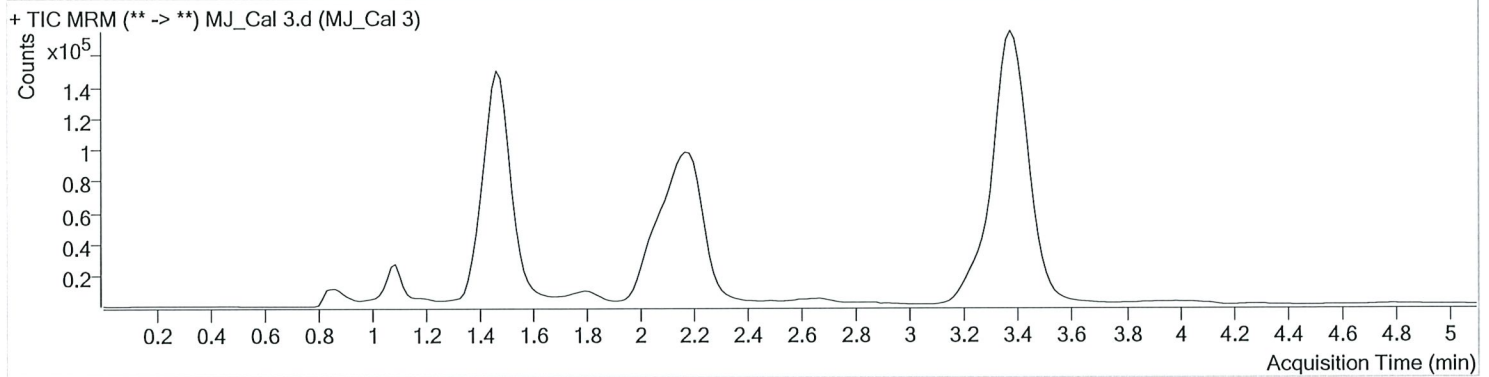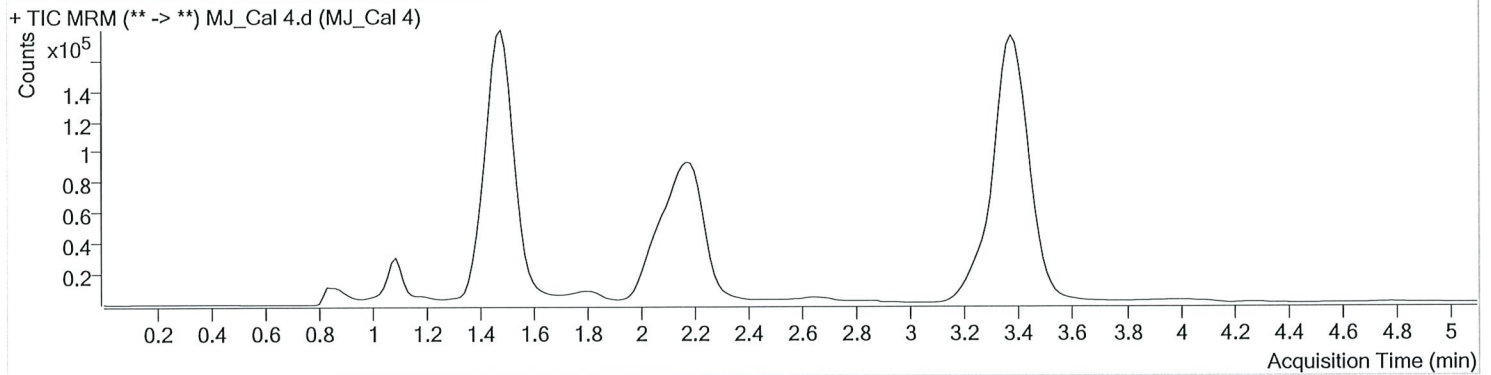| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	111119	∞	8.8	∞	604786	5.0366	ng/ml
THC-COOH	1.504	94860	327.53	54.6	355.10	197820	19.5796	ng/ml
THC	3.390	70904	468.24	29.6	295.86	1583858	5.1074	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update   3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 4.d
Type	Cal	**Sample**	MJ_Cal 4
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:44:47 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 4.d (MJ_Cal 4)

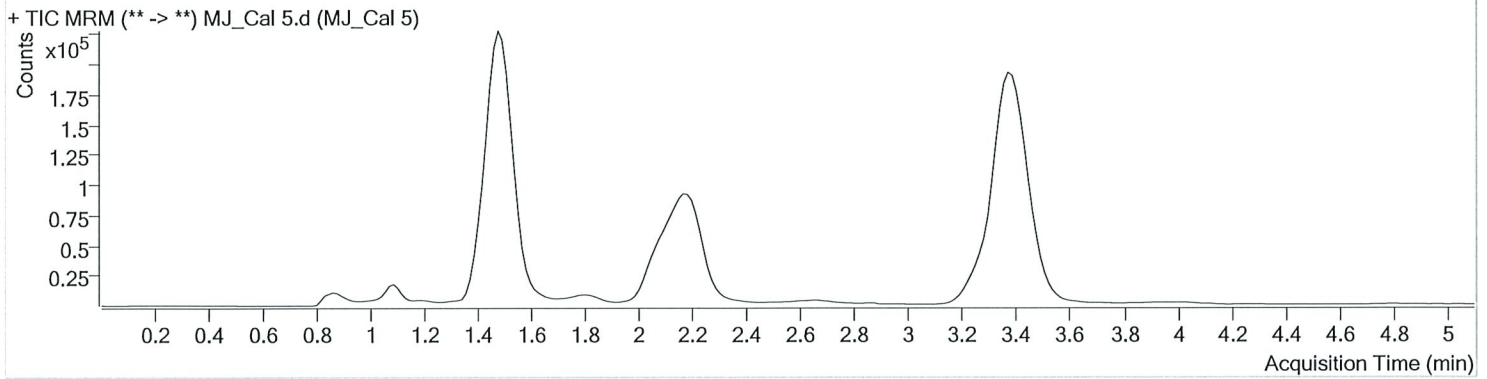| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	158185	∞	9.9	∞	578900	9.9614	ng/ml
THC-COOH	1.504	212902	918.00	57.5	1199.11	187754	47.8908	ng/ml
THC	3.375	128968	375.46	27.8	369.96	1494649	9.5708	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 5.d
Type	Cal	**Sample**	MJ_Cal 5
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:52:21 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 5.d (MJ_Cal 5)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	307887	∞	10.8	266.52	560777	25.1340	ng/ml
THC-COOH	1.504	318505	∞	57.2	1714.75	179202	75.7257	ng/ml
THC	3.390	323173	1102.39	26.0	∞	1432271	24.5507	ng/ml

Batch results   D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 6.d
Type	Cal	**Sample**	MJ_Cal 6
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 3:59:56 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 6.d (MJ_Cal 6)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	577184	∞	12.1	947.26	584549	49.2508	ng/ml
THC-COOH	1.504	436073	2247.55	58.8	2410.94	189659	98.3044	ng/ml
THC	3.375	706556	2593.14	26.9	1022.35	1584510	48.2301	ng/ml

MJ_Cal 6   Generated at 10:12 AM on 3/26/2020
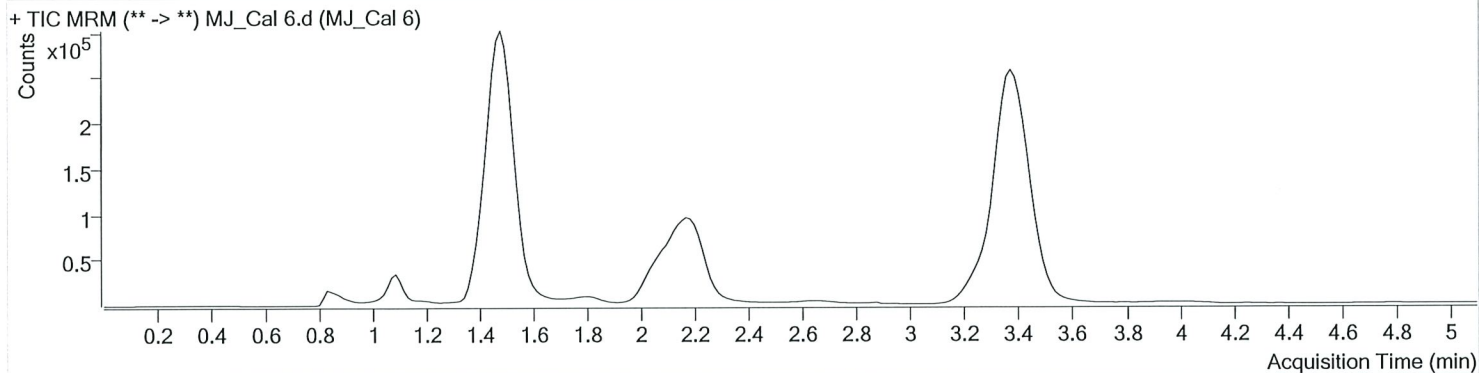
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\032520 AM 27 wklst 4107 TS\QuantResults\AM 27 TS.batch.bin
Calibration Last Update 3/26/2020 9:56:38 AM

Instrument	Falco	**Data File**	MJ_Cal 7.d
Type	Cal	**Sample**	MJ_Cal 7
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/25/2020 4:07:30 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 7.d (MJ_Cal 7)
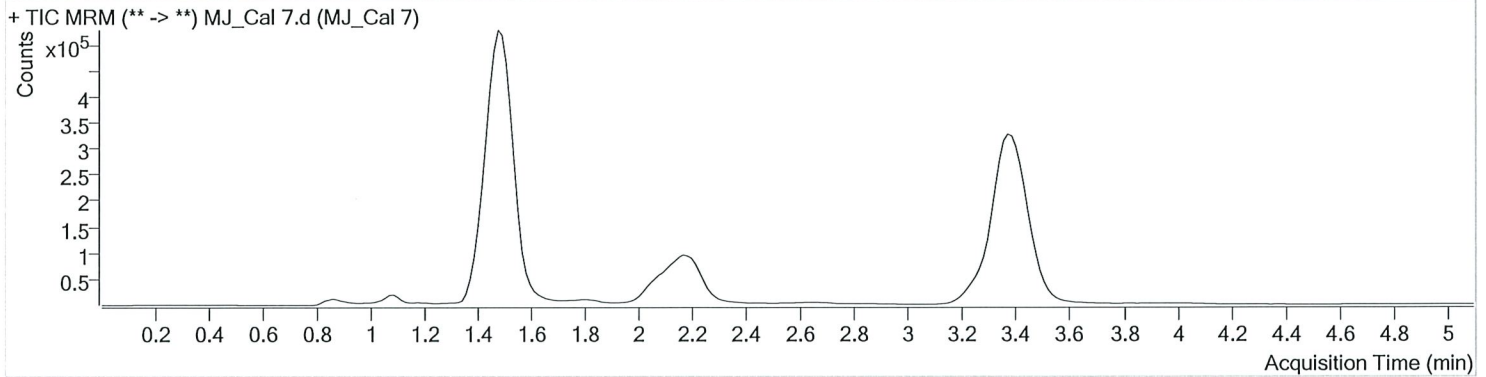
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	1070002	∞	12.7	1602.05	556982	100.6172	ng/ml
THC-COOH	1.504	1035728	∞	59.3	4144.09	176354	252.9112	ng/ml
THC	3.390	1351953	5389.66	25.2	923.38	1423528	102.3882	ng/ml