Worklist: 3910

LAB CASE	ITEM	ITEM TYPE	DESCRIPTION	
M2019-5430	3	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
M2019-5533	3	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
M2019-5648	2	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
M2019-5651	3	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3615	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3686	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3762	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3769	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3841	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3843	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3844	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3867	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
P2019-3885	1	BCK	AM 27 Blood THC Quant by LC-QQQ	|||||||||||||||||||||||||||||||||||||||||
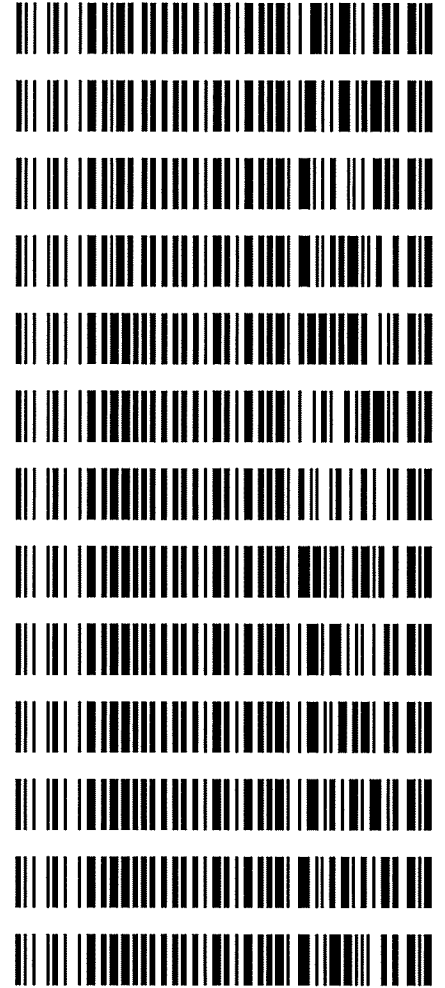
AM# 27: Quantitation of THC and Metabolites in Blood by LC-MS/MS

Extraction Date: 12/27/19   Analyst: <u>Tamara Salazar</u>

Plate lot#: IDP-108-190716   Plate Expiration: 01/16/2020

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: Hemostat 445283-3   **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.

☒ 3.   Create worklist:

Analytic:

☒ 1.   Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.

☒ 2.   Pipette **1000µL blood/urine (calibrated pipette) Pipette ID: 27** in wells of analytical (standards) plate.

☒ 3.   Place on shaking incubator at ambient temp., 900rpm for 15 minutes.   *Shaker ID: 067105*

☒ 4.   Pipette **500µL 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 **800µL of blood+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: 067104

☒ 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: 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\2019\AM 27\122719 THCQ wklst 3910 TS*
     Batch Name: *THCQ TS*

☒ 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 10ng/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?   Y / N

☒ 6   Enter QCs into control charting.

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

COMMENTS: *Curves limited: THC-OH 3-100*
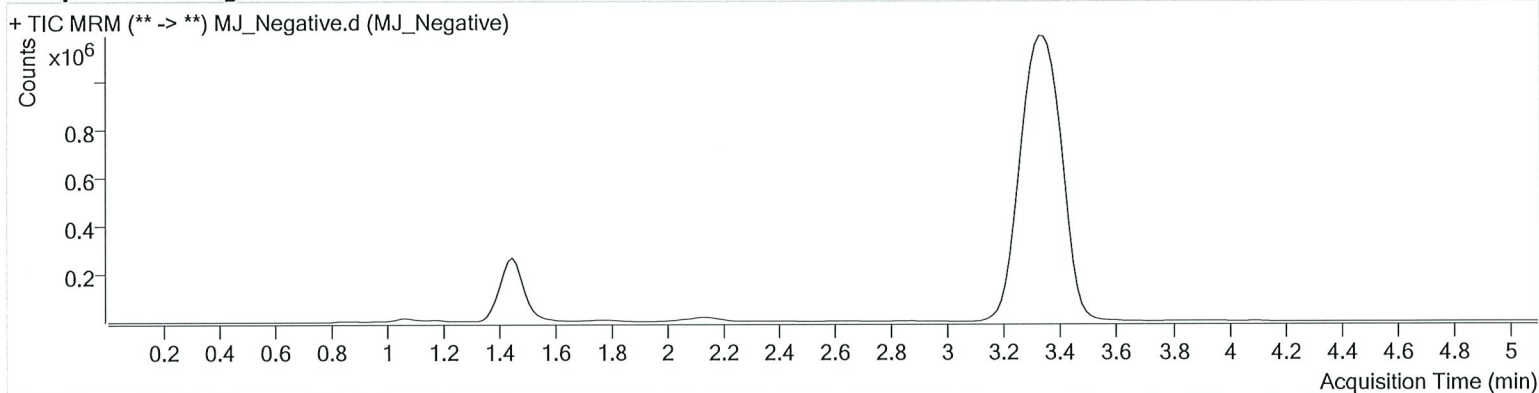
AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 AM

Instrument	Falco	**Data File**	MJ_Negative.d
Type	Sample	**Sample**	MJ_Negative
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 11:06:20 AM		
Sample Info.			

Sample Chromatogram
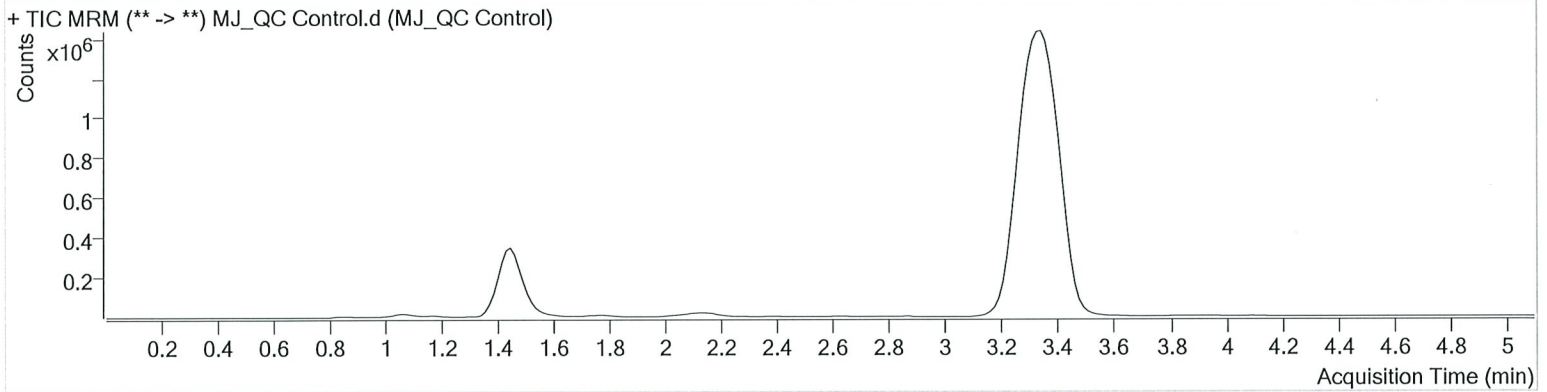
AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:51:09 AM		
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	1.453	132447	∞	9.4	∞	1378087	4.5926	ng/ml
THC-COOH	1.489	136847	130.24	59.6	1179.33	404868	13.7869	ng/ml
THC	3.360	452111	1205.03	27.1	383.34	13837773	4.2421	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin

Last Cal. Update   1/6/2020 10:12 AM

Analyst Name   ISP\Datastor

Analyte   THC   **Internal Standard**   THC-D3

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

$y = 0.008011 * x - 0.001312$

$R^2 = 0.99979546$

Type:Linear, Origin:Ignore, Weight:1/x
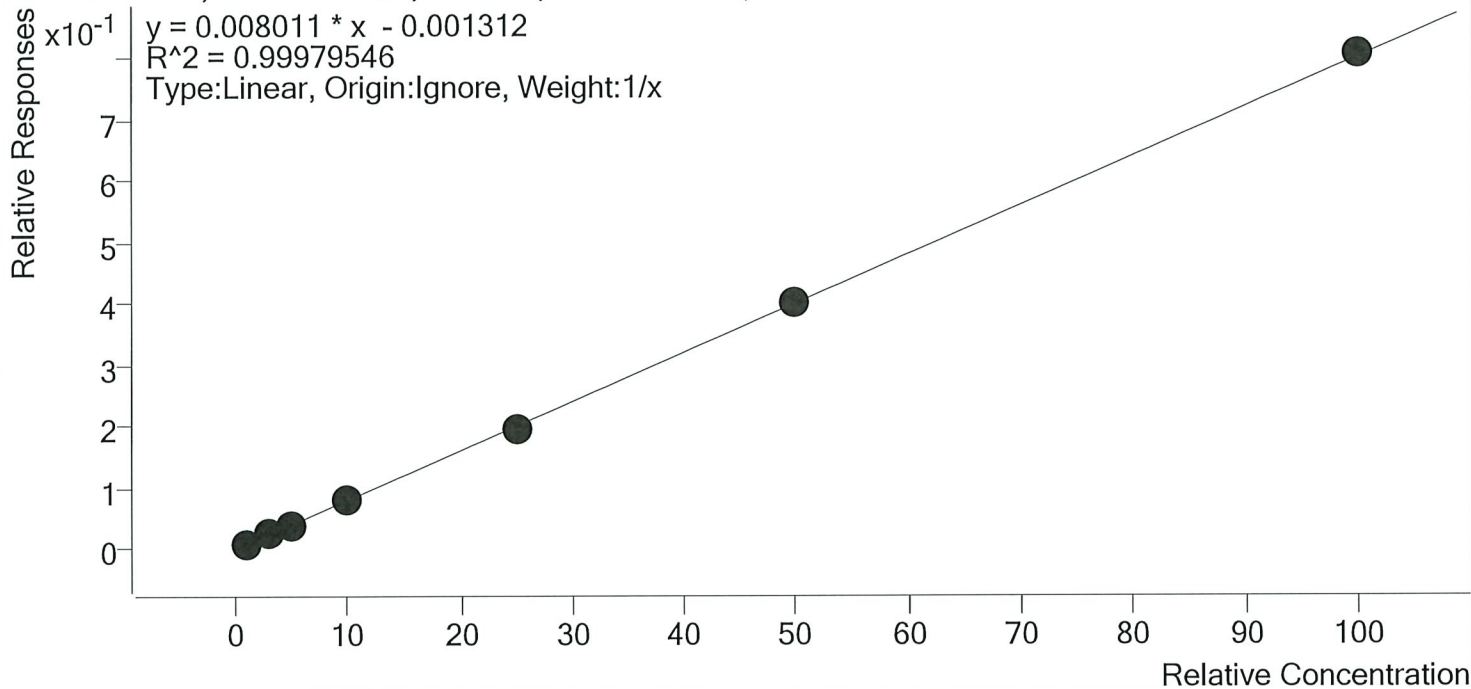
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	1.0	1.1	108.5
MJ_Cal 2	2	✓	3.0	3.0	99.0
MJ_Cal 3	3	✓	5.0	4.7	94.7
MJ_Cal 4	4	✓	10.0	9.9	98.5
MJ_Cal 5	5	✓	25.0	24.6	98.6
MJ_Cal 6	6	✓	50.0	50.0	100.0
MJ_Cal 7	7	✓	100.0	100.7	100.7

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin

Last Cal. Update 1/6/2020 10:12 AM

Analyst Name ISP\Datastor

Analyte THC-COOH   **Internal Standard** THC-COOH-D9

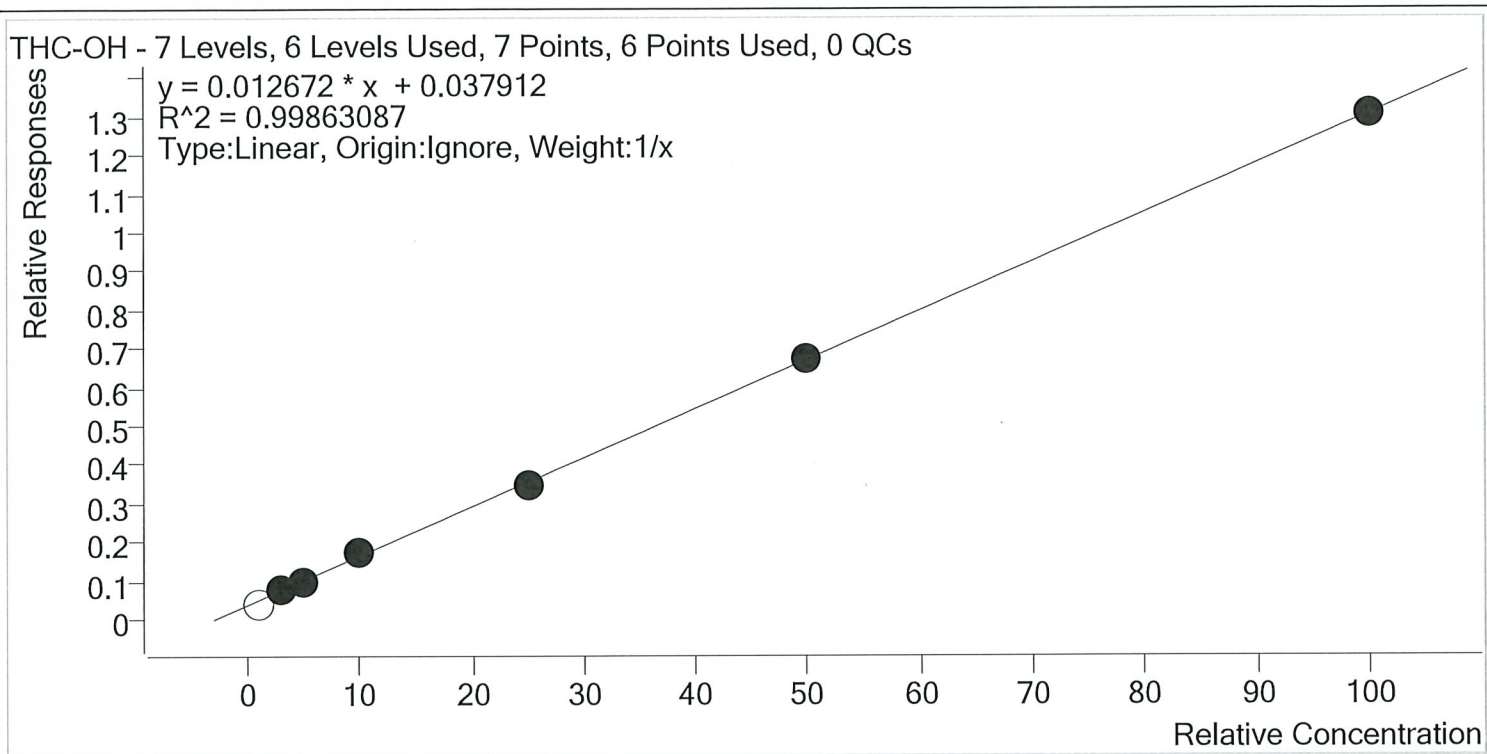THC-COOH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 0 QCs

$y = 0.024717 * x - 0.002772$
$R^2 = 0.99928356$
Type:Linear, Origin:Ignore, Weight:1/x
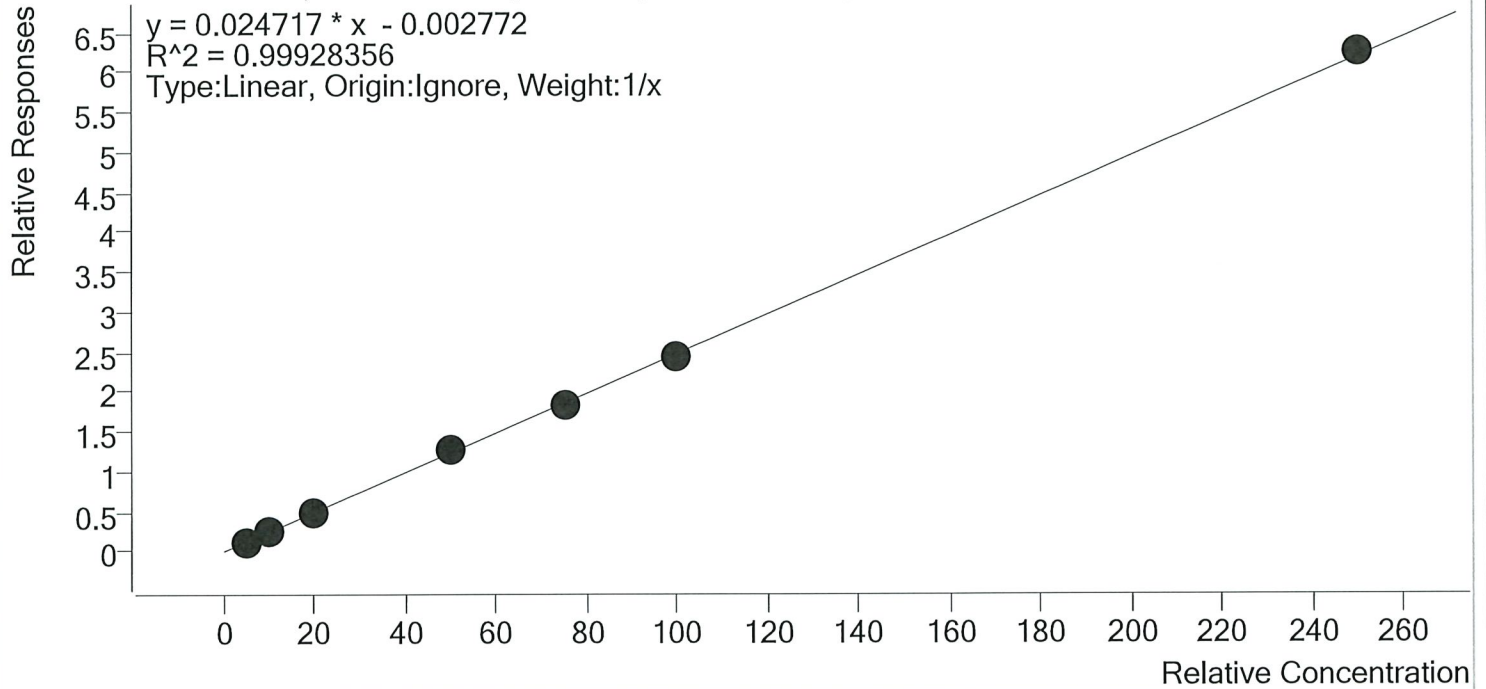
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	4.8	96.2
MJ_Cal 2	2	✓	10.0	11.1	110.6
MJ_Cal 3	3	✓	20.0	18.8	94.0
MJ_Cal 4	4	✓	50.0	50.7	101.4
MJ_Cal 5	5	✓	75.0	74.1	98.8
MJ_Cal 6	6	✓	100.0	98.0	98.0
MJ_Cal 7	7	✓	250.0	252.5	101.0

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Last Cal. Update	1/6/2020 10:12 AM
Analyst Name	ISP\Datastor
Analyte	THC-OH

Internal Standard   THC-OH-D3

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

$y = 0.012672 * x + 0.037912$
$R^2 = 0.99863087$
Type:Linear, Origin:Ignore, Weight:1/x

| Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✗	1.0	0.3	26.1
MJ_Cal 2	2	✓	3.0	3.1	103.9
MJ_Cal 3	3	✓	5.0	4.4	88.2
MJ_Cal 4	4	✓	10.0	11.0	110.3
MJ_Cal 5	5	✓	25.0	24.4	97.6
MJ_Cal 6	6	✓	50.0	50.0	100.0
MJ_Cal 7	7	✓	100.0	100.0	100.0

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 9:50:18 AM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) MJ_Cal 1.d (MJ_Cal 1)
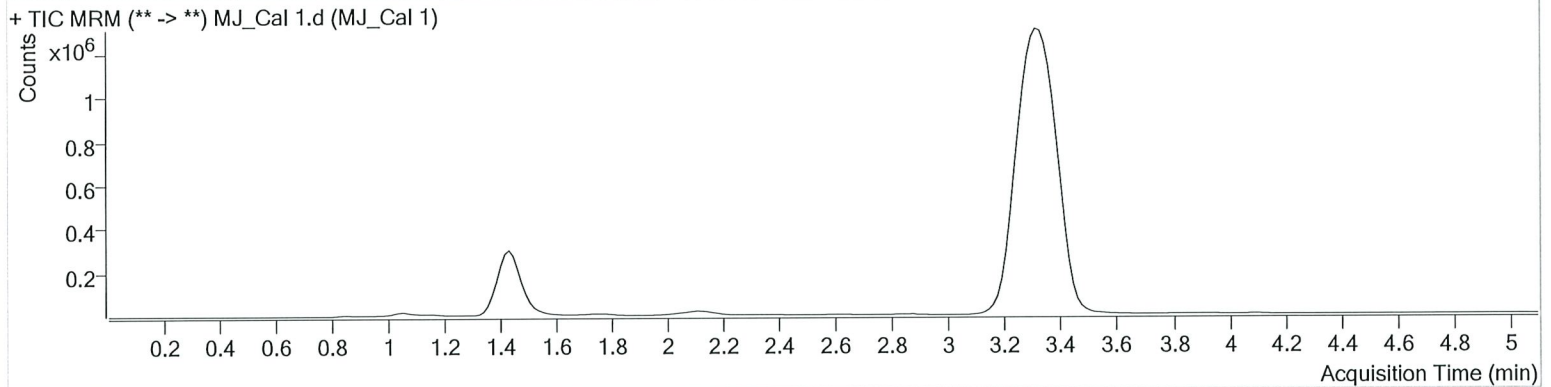
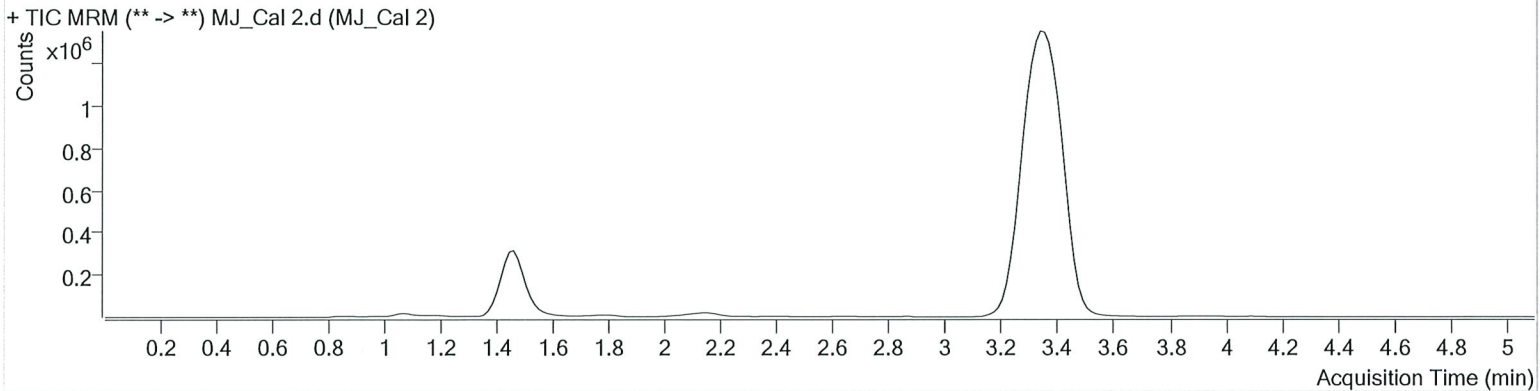| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	1.498 **High**	52757	∞	6.3 **Low**	14.95	1279828	0.2612 ng/ml **Low**
THC-COOH	1.474	44791	81.79	62.2	153.96	385926	4.8077 ng/ml **Low**
THC	3.330	95588	350.95	29.9	∞	12952218	1.0850 ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 9:58:03 AM		
Sample Info.			

Sample Chromatogram

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

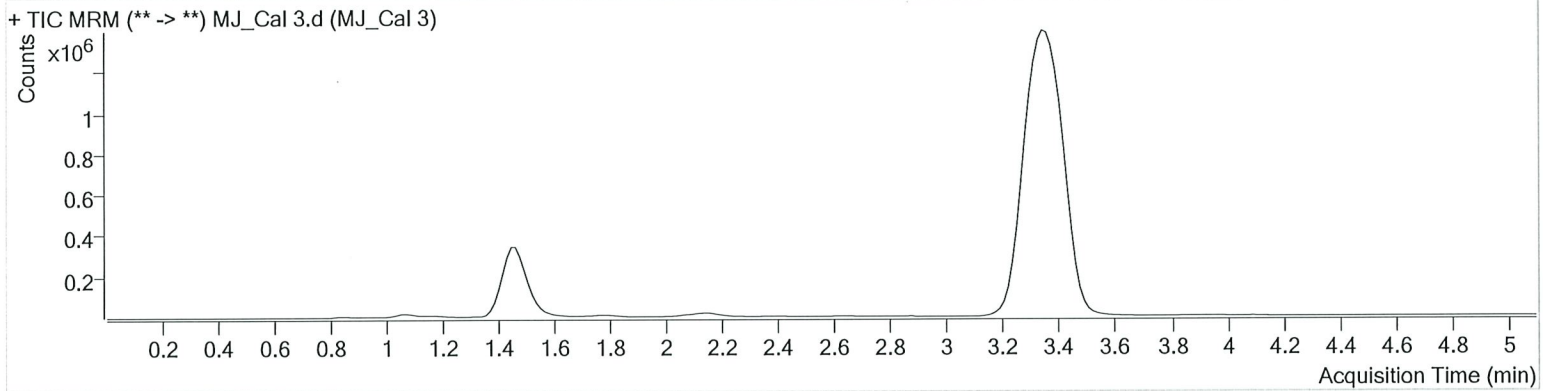| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	99016	∞	9.6	173.74	1279264	3.1162	ng/ml
THC-COOH	1.504	104500	∞	51.9	∞	386068	11.0631	ng/ml
THC	3.375	291952	∞	28.1	∞	12989775	2.9693	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 1/6/2020 10:12:48 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-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:05:38 AM		
Sample Info.			

Sample Chromatogram

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

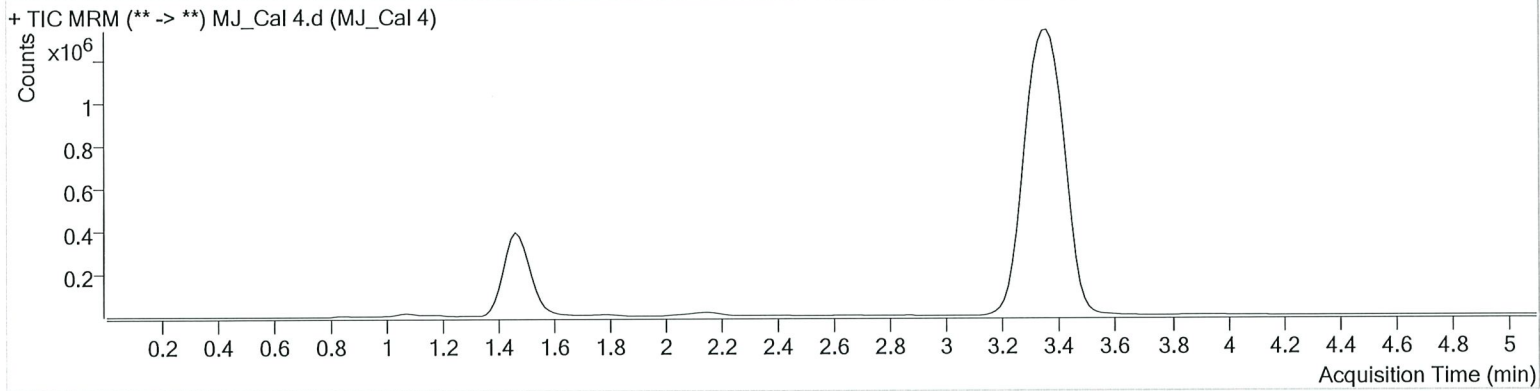| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	121208	∞	9.9	491.33	1292332	4.4096	ng/ml
THC-COOH	1.489	178999	245.34	60.9	1040.54	387585	18.7967	ng/ml
THC	3.375	475334	2980.27	27.3	404.75	12981481	4.7344	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:13:12 AM		
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	216072	∞	10.4	446.09	1216241	11.0278	ng/ml
THC-COOH	1.489	447624	∞	59.9	4070.97	358024	50.6946	ng/ml
THC	3.375	931528	3855.10	26.9	∞	11999087	9.8543	ng/ml

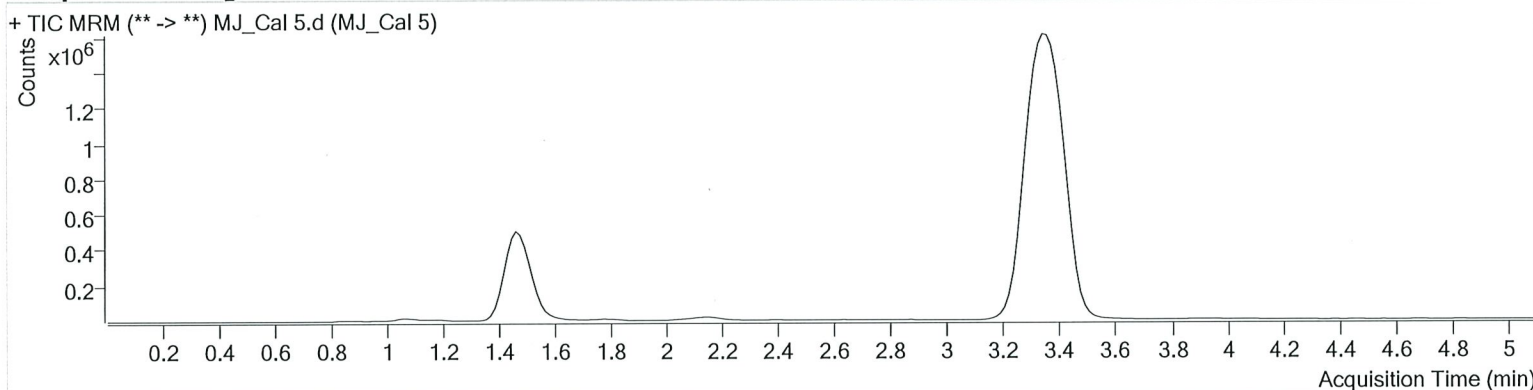MJ_Cal 4   Generated at 10:13 AM on 1/6/2020

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:20:47 AM		
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	1.453	431979	∞	13.0	1766.89	1244658	24.3967	ng/ml
THC-COOH	1.489	662981	∞	59.5	2222.86	362429	74.1200	ng/ml
THC	3.375	2435727	5326.32	26.4	2881.87	12418687	24.6461	ng/ml

MJ_Cal 5
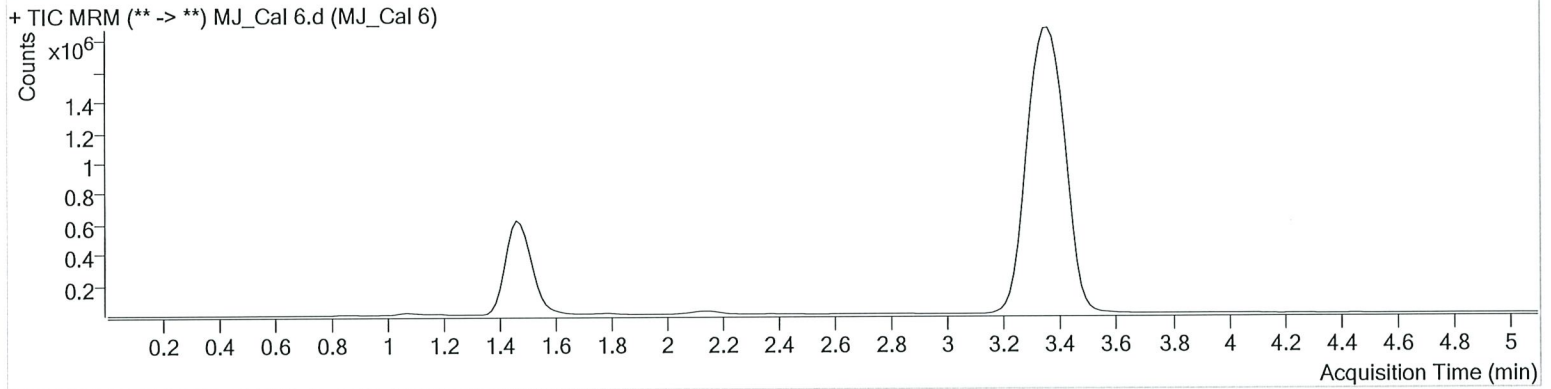
Generated at 10:13 AM on 1/6/2020

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:28:22 AM		
Sample Info.			

Sample Chromatogram

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

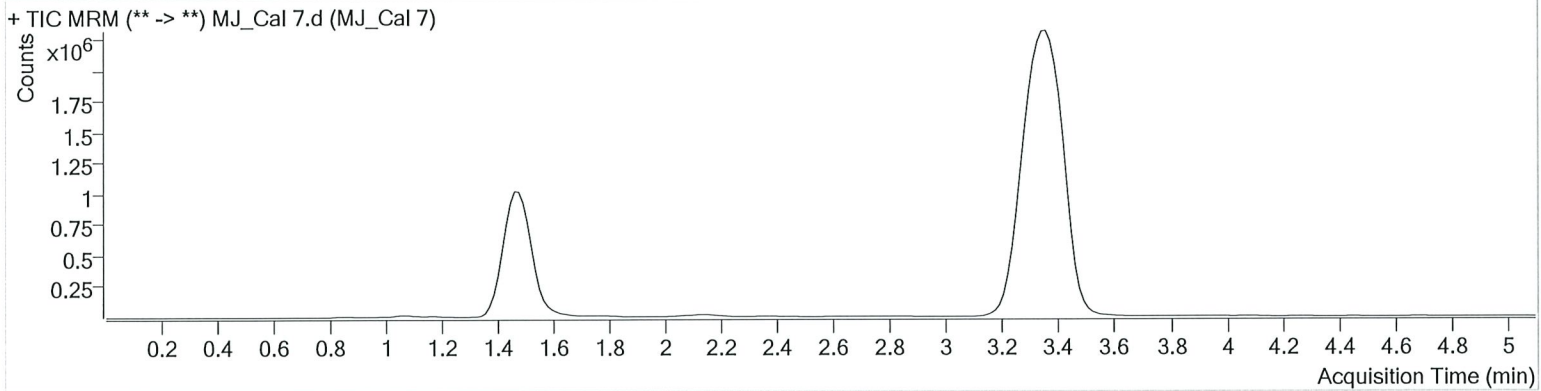| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	805817	∞	13.6	1198.42	1199536	50.0208	ng/ml
THC-COOH	1.489	834535	∞	59.9	4899.74	344864	98.0153	ng/ml
THC	3.375	4672268	18653.83	26.7	8250.90	11702135	50.0021	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results   D:\MassHunter\Data\2019\AM 27\122719 THCQ wklst 3910 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update   1/6/2020 10:12:48 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-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	12/27/2019 10:35:57 AM		
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	1.453	1579079	∞	13.9	3356.07	1209581	100.0290	ng/ml
THC-COOH	1.489	2056703	∞	60.2	18121.14	329684	252.5027	ng/ml
THC	3.360	9300738	24403.97	26.4	84331.94	11546673	100.7089	ng/ml