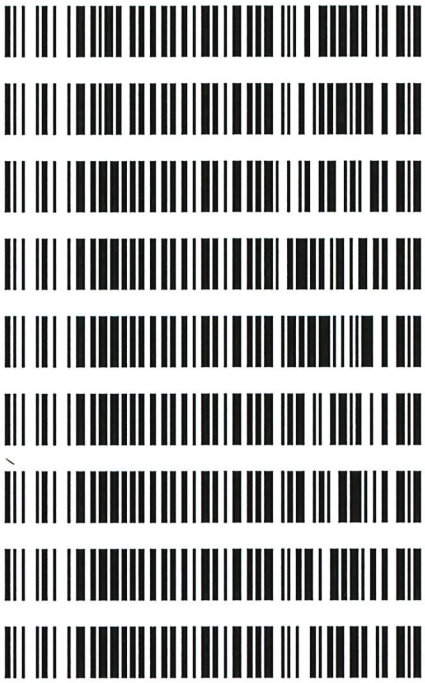
REVIEWED
By Anne Nord at 2:11 pm, Feb 13, 2020

TS 2/10/2020

Worklist: 3988

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2020-0081	1	BCK	AM 27 Blood THC Quant by LC-QQQ
M2020-0174	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2019-3446	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2019-3894	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0053	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0282	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0284	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0334	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2020-0373	1	BCK	AM 27 Blood THC Quant by LC-QQQ



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Idaho State Police
Forensic Services
Toxicology Discipline

Request for Departure from an Analytical Method

Date of Request
01/13/2020

Forensic Scientist
Celena Shrum

Analytical Methods
Toxicology AM #25, Toxicology AM #26/27, and AM #28

Deviation

The expiration dates listed for the current batch of PinPoint ToxBox extraction plates are as follows:
*MDS (batch IDP-107-190725)- Expiration is 1/25/2020
*THC (batch IDP-108-190716)- Expiration is 1/16/2020
*MDQ P1 (batch IDP-111-190729)- Expiration is 1/29/2020
*MDQ P2 (batch IDP-112-190730)- Expiration is 1/30/2020

I am issuing a deviation to allow for the use of the remaining plates of these batches. The controls will be used to evaluate if the plate is working as intended. In addition, at least one external control must be included for each run.

Celena Shrum

Date: 01/13/2020
Celena Shrum
Toxicology Discipline Lead

TS

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

Extraction Date: 02/12/2020
Plate lot#: IDP-108-190716

Analyst: Tamara Salazar
Plate Expiration: 01/16/2020

Mobile phase A: 0.1% Formic Acid in LCMS Water
MTBE LCMS Methanol

Mobile phase B: 0.1% Formic acid in Acetonitrile
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: 42** 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\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 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: ~~1-100~~, THC-COOH: 10-250, THC-OH: 3-100*
THC: 3-100 --02/13/2020
TS

Idaho State Police
Forensic Services

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

Methanol External Control Solution (Lot: WS011620)

10 ul of 1mg/mL THC, 100 ul of 100 ug/mL THC-OH, C-THC in 9790 ul MeOH
Approximate concentration 1ug/mL.

Component	*Source*	*Source Lot Number*	*Expiration Date*
Methanol (LCMS)	Fisher	193941	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2020
THC-OH	Cerilliant	FE07221601	07/31/2021
Prepared:	01/16/2020		
Prepared By:	Tamara Salazar		
Expires:	09/30/2020		

Blood External Control Solution (Lot: 011620)

100 ul of methanol external control solution was added to 9900 ul of blood.
Approximately 10ng/mL of each compound.

Component	*Source*	*Source Lot Number*
Negative Blood	Hemostat	445283-3
Methanol External Control Solution	-	WS011620
Prepared:	01/16/2020	
Prepared by:	Tamara Salazar	
Expires:	09/30/2020	

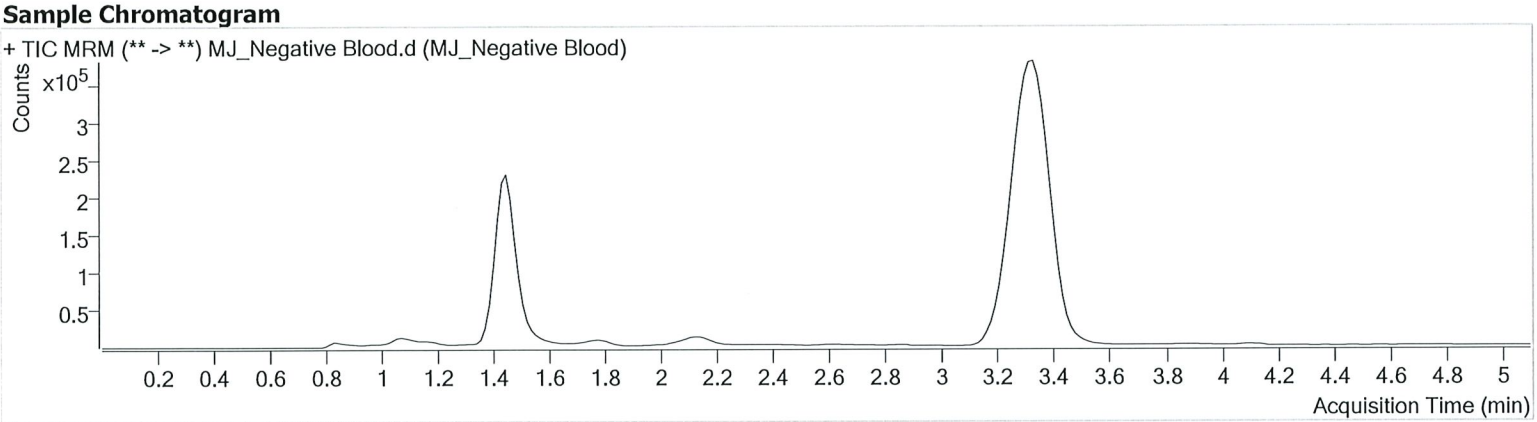
TS

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 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-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/12/2020 11:11:23 AM		
Sample Info.			

Sample Chromatogram

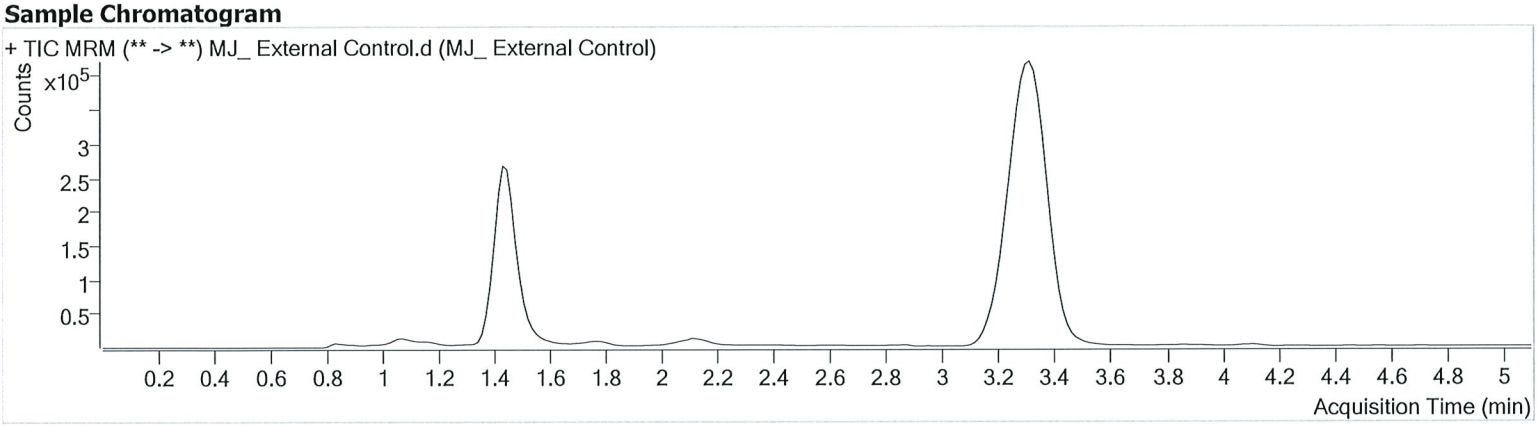


AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 2/13/2020 11:01:05 AM

Instrument	Falco	**Data File**	MJ_ External Control.d
Type	Sample	**Sample**	MJ_ External Control
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-G5	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/12/2020 11:26:34 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.		
THC-OH	1.453	189109	∞	9.9	163.64	1027568	10.3658	ng/ml	
THC-COOH	1.474	49424	202.26	58.6	∞	231684	8.4439	ng/ml	Low
THC	3.330	245210	885.94	27.5	123.16	3778216	8.3877	ng/ml	

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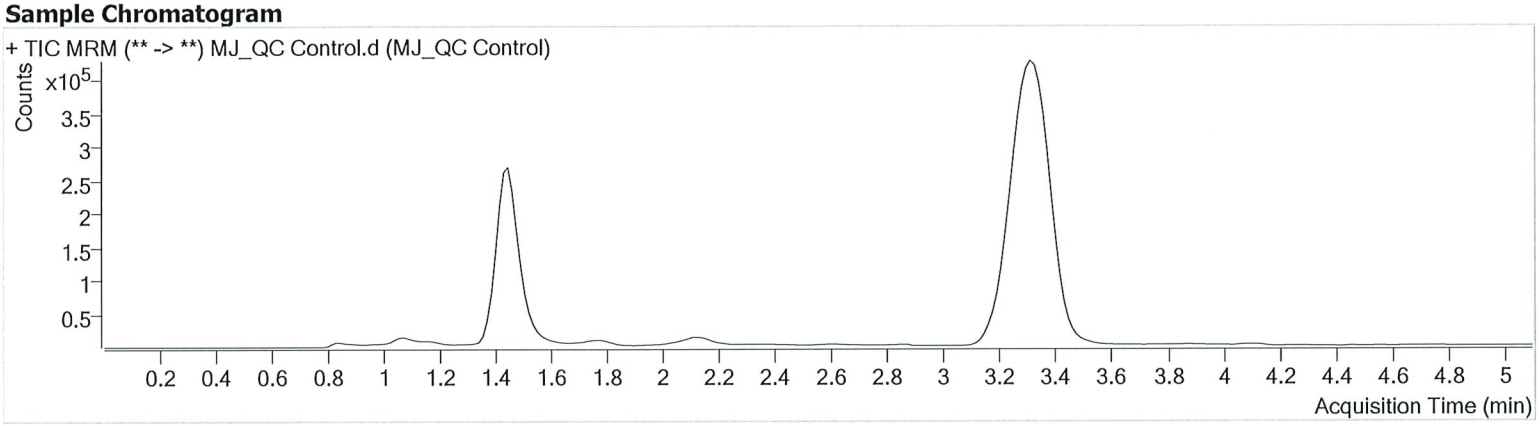
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 2/13/2020 11:01:05 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	2/12/2020 10:56:10 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	108395	∞	9.2	47.05	1065471	4.0243	ng/ml
THC-COOH	1.474	80797	115.35	55.1	239.28	240756	13.7376	ng/ml
THC	3.330	134385	294.71	28.0	44.19	4103670	4.4106	ng/ml

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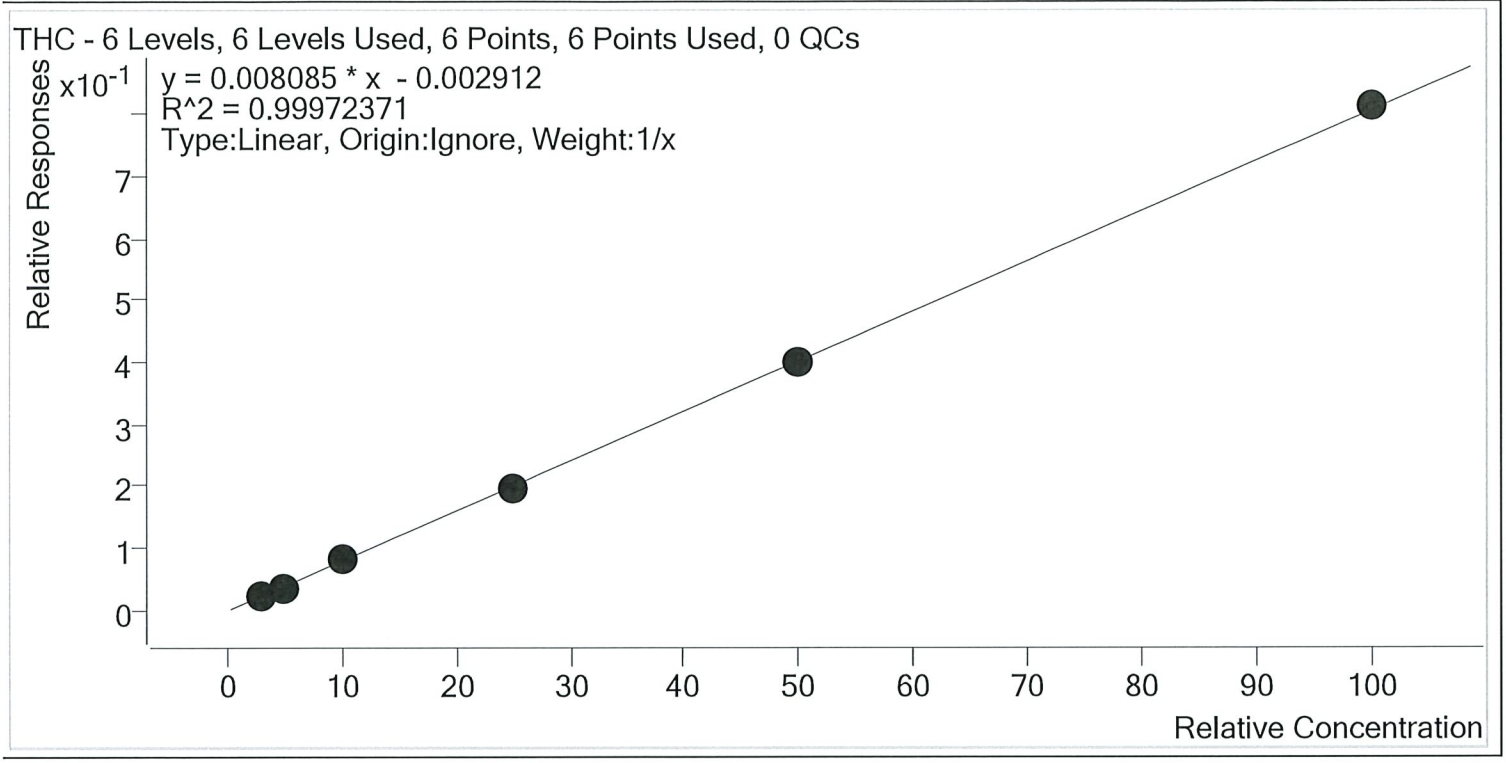
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin

Last Cal. Update 2/13/2020 11:01 AM

Analyst Name ISP\datastor

Analyte THC **Internal Standard** THC-D3

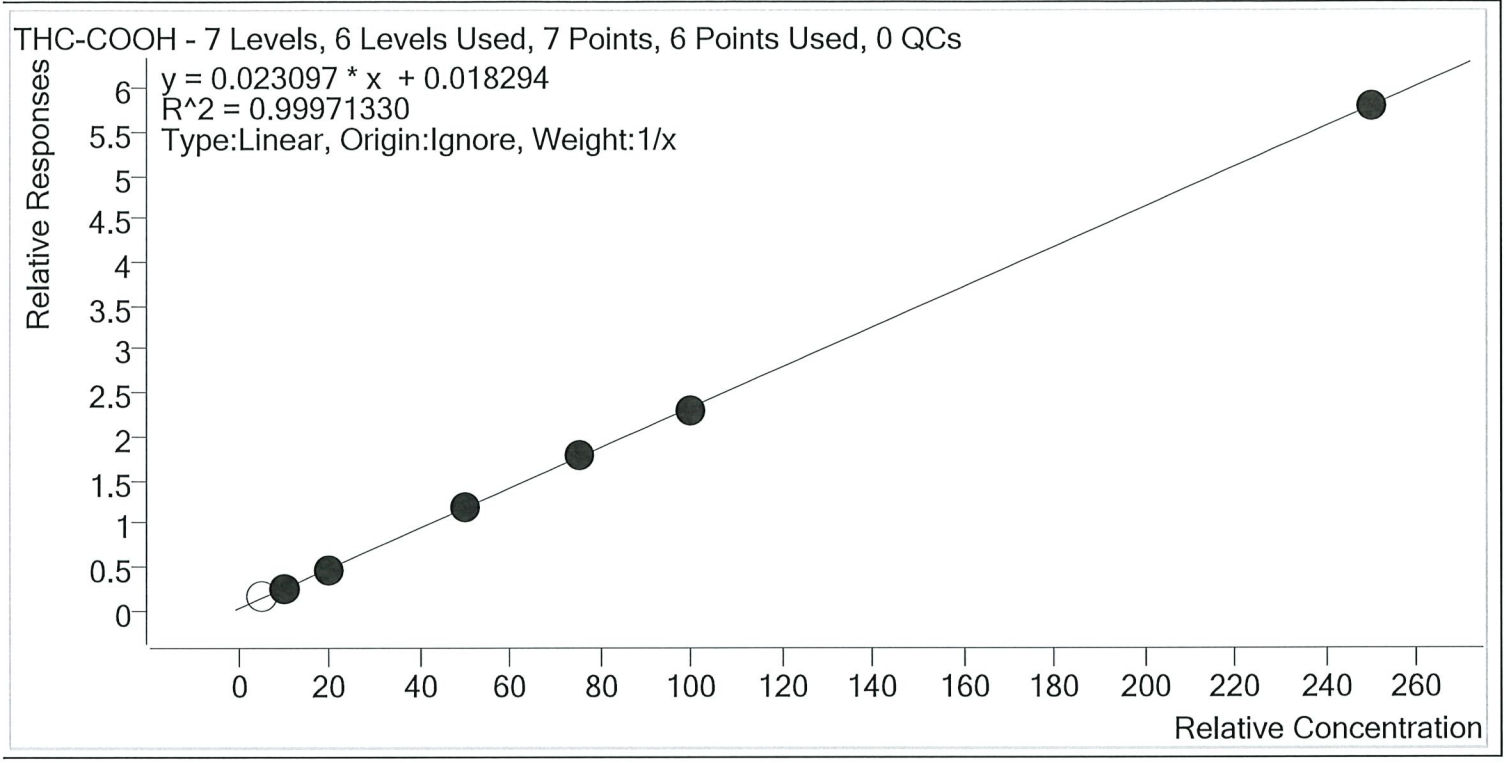


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 2	2	✓	3.0	3.1	104.8
MJ_Cal 3	3	✓	5.0	4.8	96.9
MJ_Cal 4	4	✓	10.0	10.1	100.7
MJ_Cal 5	5	✓	25.0	24.3	97.2
MJ_Cal 6	6	✓	50.0	49.9	99.7
MJ_Cal 7	7	✓	100.0	100.8	100.8

TS

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Last Cal. Update	2/13/2020 11:01 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	6.2	124.4
MJ_Cal 2	2	✓	10.0	10.2	102.3
MJ_Cal 3	3	✓	20.0	19.1	95.4
MJ_Cal 4	4	✓	50.0	51.0	102.1
MJ_Cal 5	5	✓	75.0	76.0	101.3
MJ_Cal 6	6	✓	100.0	99.0	99.0
MJ_Cal 7	7	✓	250.0	249.7	99.9

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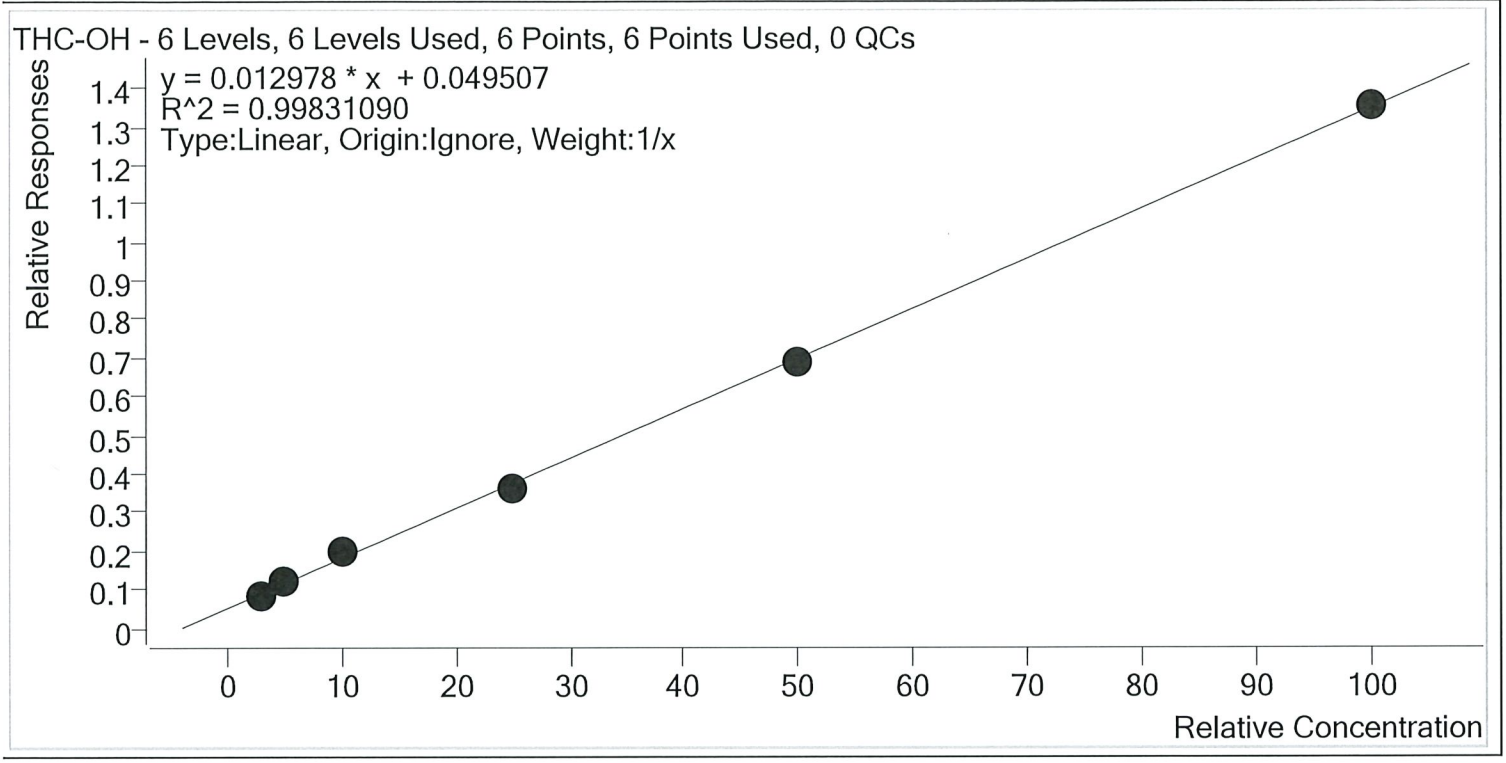
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Last Cal. Update	2/13/2020 11:01 AM
Analyst Name	ISP\datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 2	2	✓	3.0	2.6	87.6
MJ_Cal 3	3	✓	5.0	5.2	105.0
MJ_Cal 4	4	✓	10.0	11.2	111.8
MJ_Cal 5	5	✓	25.0	24.0	96.1
MJ_Cal 6	6	✓	50.0	49.6	99.1
MJ_Cal 7	7	✓	100.0	100.3	100.3

AM #27 Cannabinoid Quant. Results

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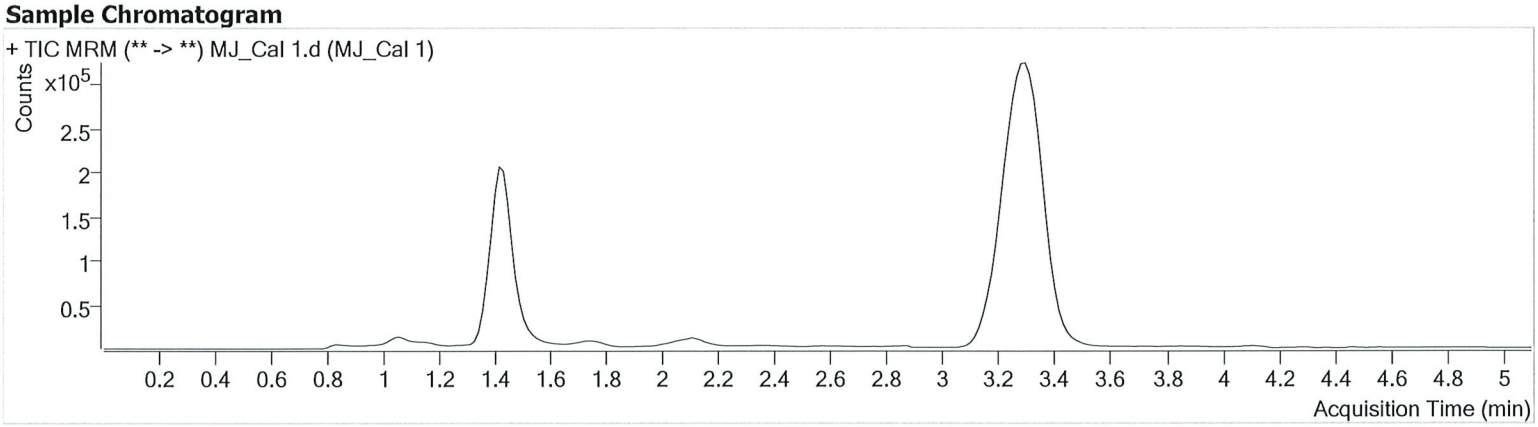
Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 AM

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	2/12/2020 9:55:18 AM		
Sample Info.			

Sample Chromatogram



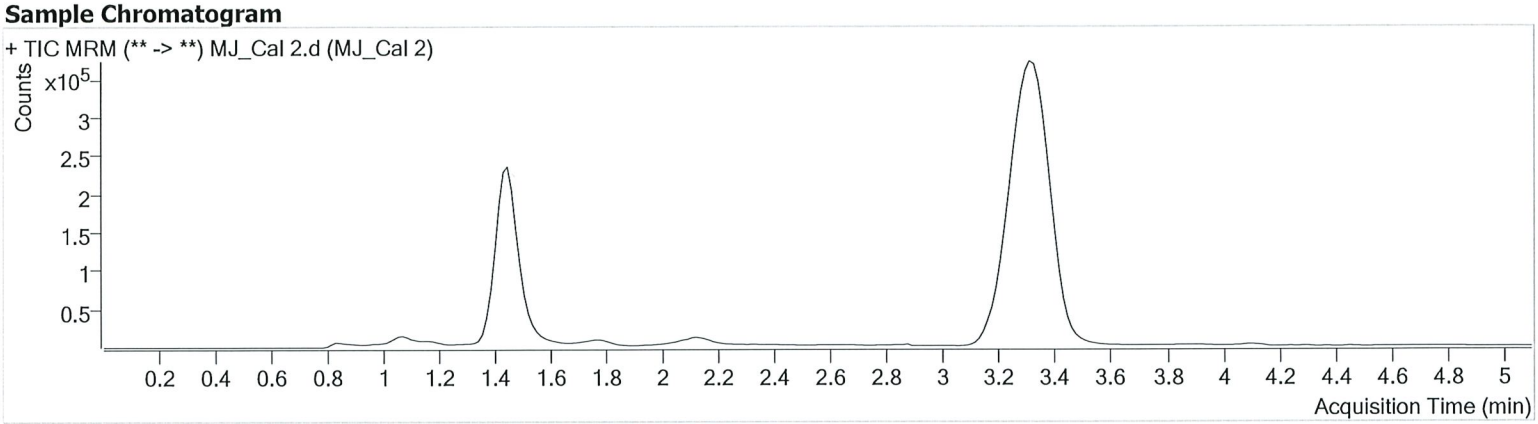
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	33740	∞	40.9 **Low**	99.73	208255	6.2223	ng/ml **Low**

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 2/13/2020 11:01:05 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	2/12/2020 10:03:03 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	81663	57.53	9.5	∞	976571	2.6287	ng/ml **Low**
THC-COOH	1.489	58485	107.83	48.5	207.01	229694	10.2318	ng/ml
THC	3.330	81929	175.44	28.6	397.33	3640856	3.1435	ng/ml

AM #27 Cannabinoid Quant. Results

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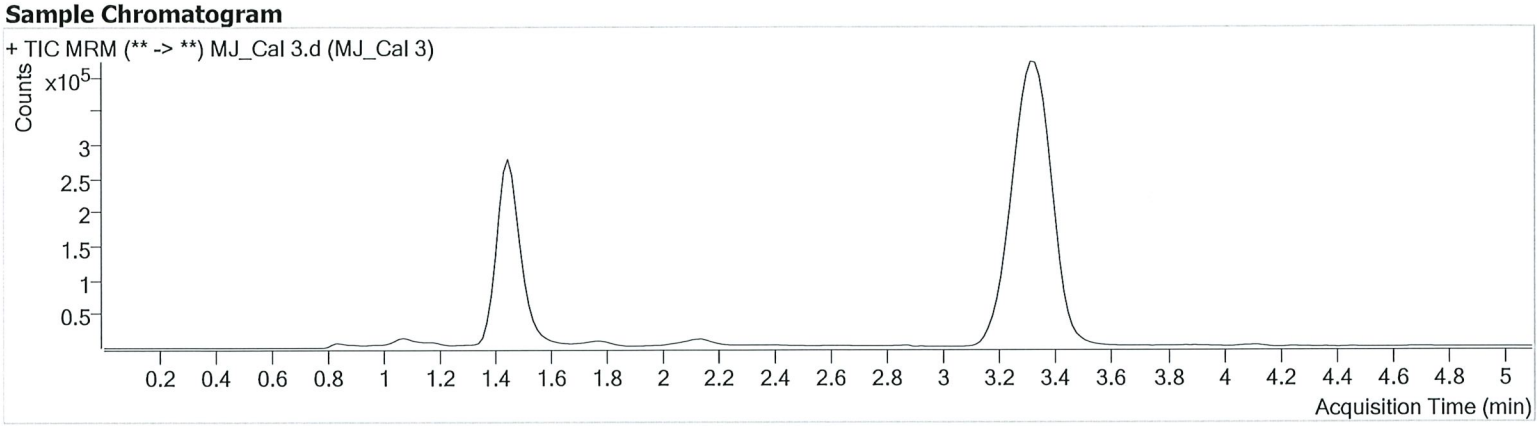
Batch results D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update 2/13/2020 11:01:05 AM

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	2/12/2020 10:10:39 AM		
Sample Info.			

Sample Chromatogram



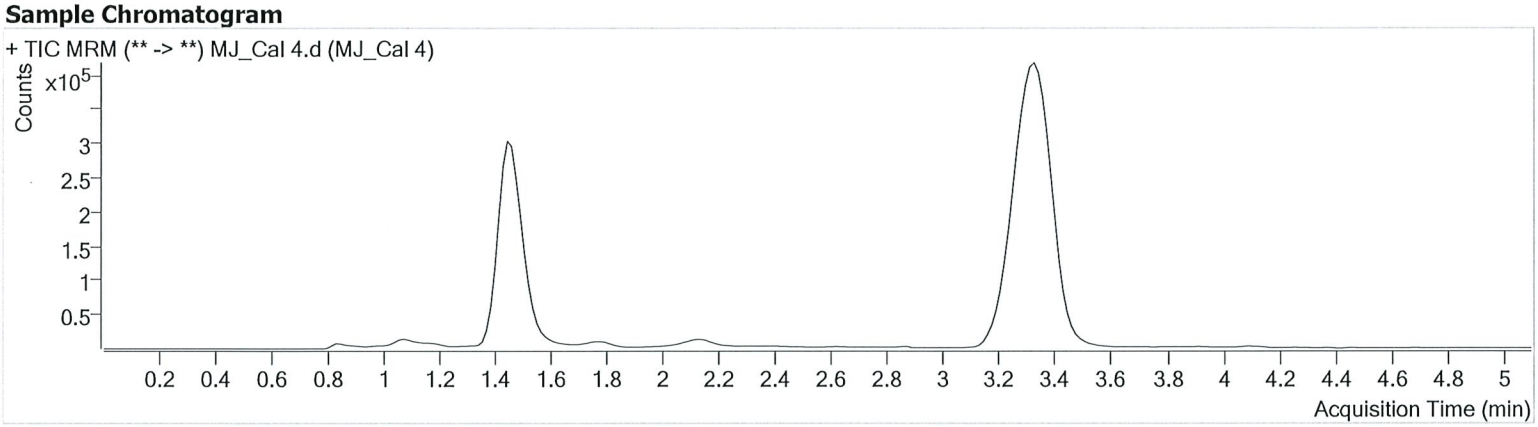
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	128198	∞	9.4	66.00	1089853	5.2490	ng/ml
THC-COOH	1.489	111358	∞	57.1	246.68	242543	19.0859	ng/ml
THC	3.330	145130	238.56	28.8	128.86	4004090	4.8433	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 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	2/12/2020 10:18:13 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	190304	87.20	10.0	243.37	977952	11.1794	ng/ml
THC-COOH	1.489	261970	1749.05	57.3	569.93	218863	51.0304	ng/ml
THC	3.345	287604	934.59	27.5	252.52	3664371	10.0680	ng/ml

AM #27 Cannabinoid Quant. Results

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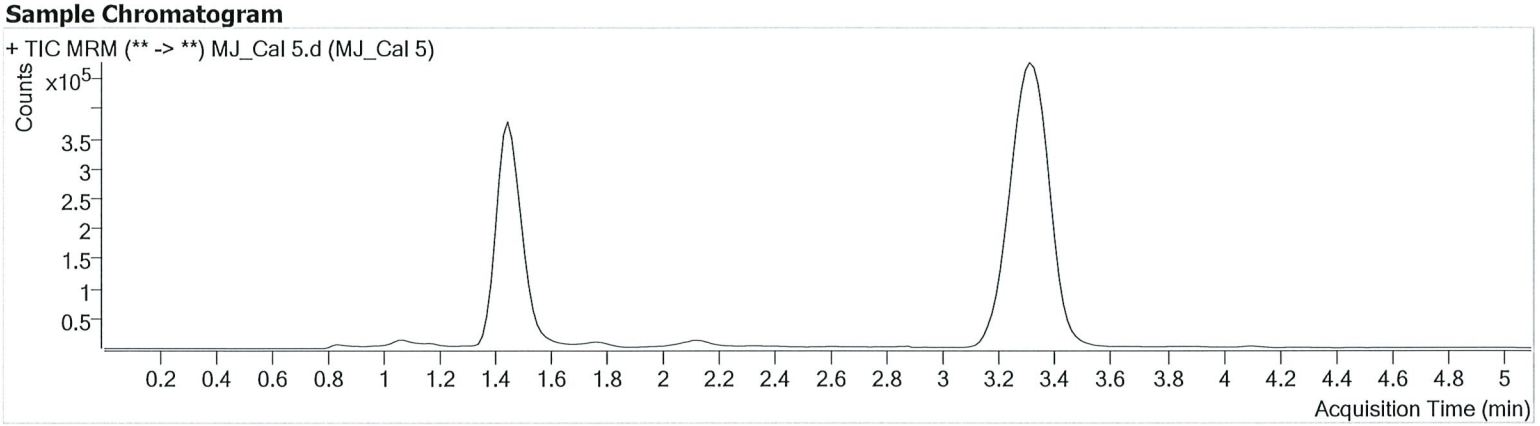
Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 AM

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	2/12/2020 10:25:50 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.438	366540	∞	12.7	∞	1014307	24.0299	ng/ml
THC-COOH	1.474	398287	742.36	57.7	1261.32	224541	76.0039	ng/ml
THC	3.330	734691	∞	26.4	∞	3797325	24.2910	ng/ml

AM #27 Cannabinoid Quant. Results

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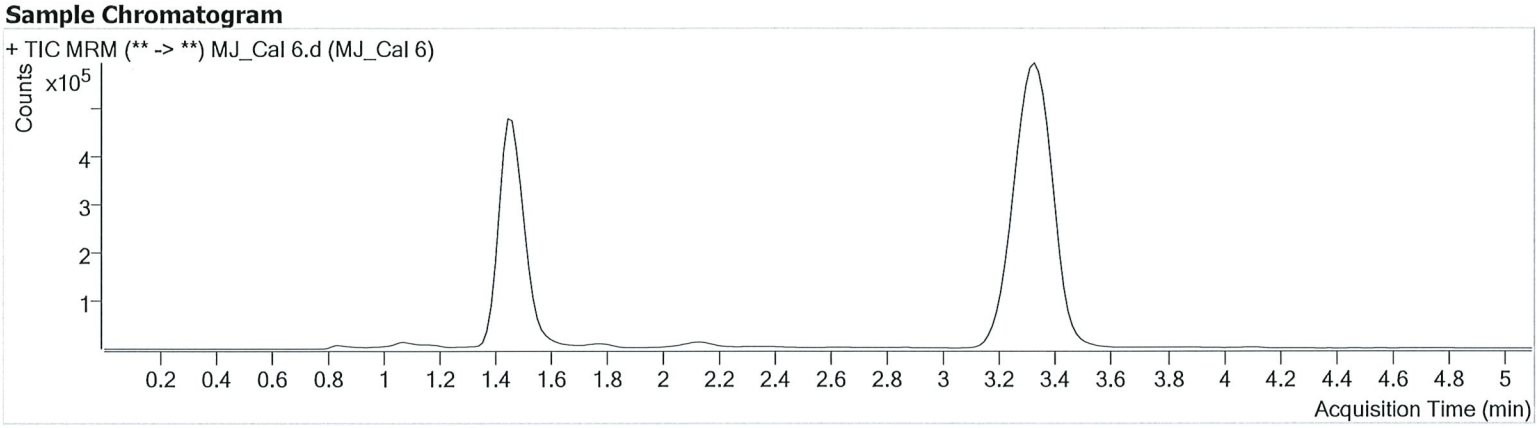
Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 AM

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	2/12/2020 10:33:24 AM		
Sample Info.			

Sample Chromatogram



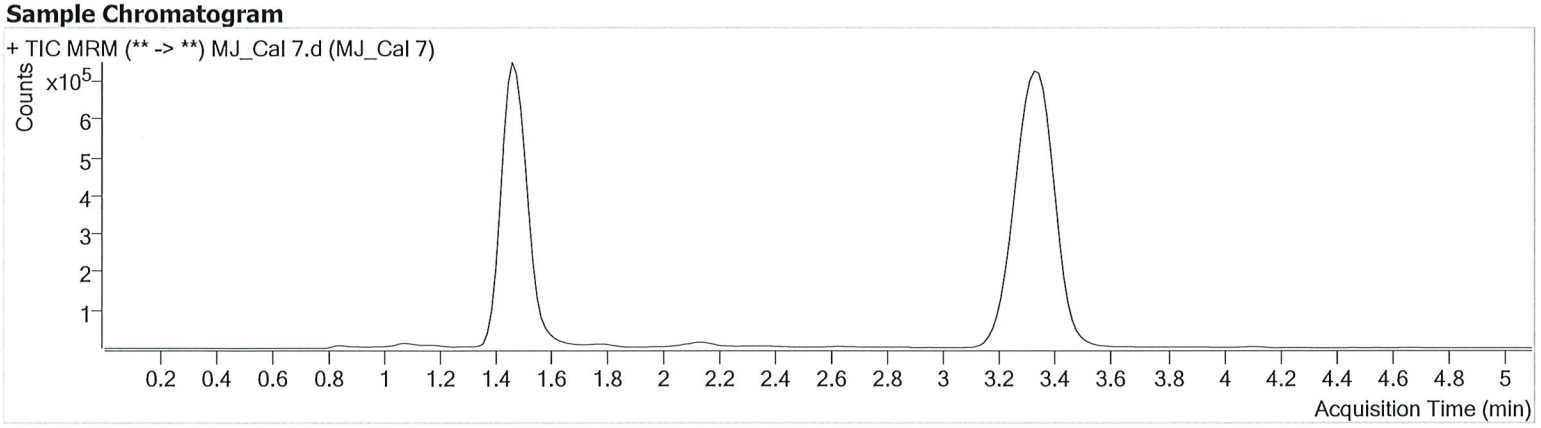
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	711442	∞	13.0	2304.32	1026848	49.5706	ng/ml
THC-COOH	1.489	514723	∞	59.8	1316.46	223338	98.9893	ng/ml
THC	3.345	1547156	5856.91	25.9	∞	3866521	49.8532	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\021220 AM 27 28 wklst 3988 3989 TS\QuantResults\THCQ TS.batch.bin
Calibration Last Update	2/13/2020 11:01:05 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	2/12/2020 10:40:58 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	1296355	∞	13.0	4353.32	959008	100.3425	ng/ml
THC-COOH	1.489	1196829	5358.91	59.1	∞	206894	249.6587	ng/ml
THC	3.345	2880997	3764.47	26.3	∞	3547834	100.8010	ng/ml