15

REVIEWED
By Sarah Pickle at 3:57 pm, May 05, 2020

4/29/2020

Worklist: 4205

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2020-1137	51	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2020-1303	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2020-1343	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-0612	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-0903	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1045	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1180	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1181	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1225	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1226	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2020-1227	1	BCK	AM 27 Blood THC Quant by LC-QQQ	

TS

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

Extraction Date: 04/30/2020
Plate lot#: IDP-108-200303

Analyst: Tamara Salazar
Plate Expiration: 09/03/2020

Mobile phase A: 0.1% Formic Acid in LCMS Water
MTBE LCMS Methanol
Blank Blood Lot: Hemostat 445283-3
LCMS-QQQ ID: 069901

Mobile phase B: 0.1% Formic acid in Acetonitrile
Hexane
Column: UCT Selectra DA 100 x 2.1mm 3um

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 (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** 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\043020 AM 27 28 wklst 4205 4206 TS*
Batch Name: *AM 27*
☒ 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*~~ 3-100 05/05/2020 TS
THC-OH not evaluated.

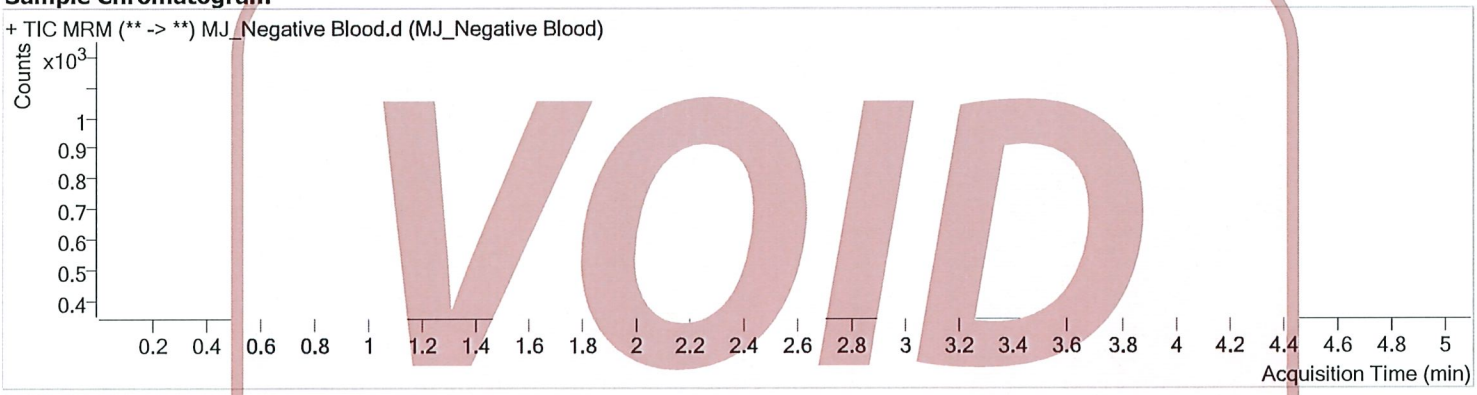
TS

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update	5/5/2020 10:18:03 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**	Sample failed to inject. The sample was reconstituted and reinjected on 05/01/2020. Please refer to the reinjection data. TS
Injection Volume	10		
Acq. Date-Time	5/1/2020 3:23:05 AM		
Sample Info.			

Sample Chromatogram



TS

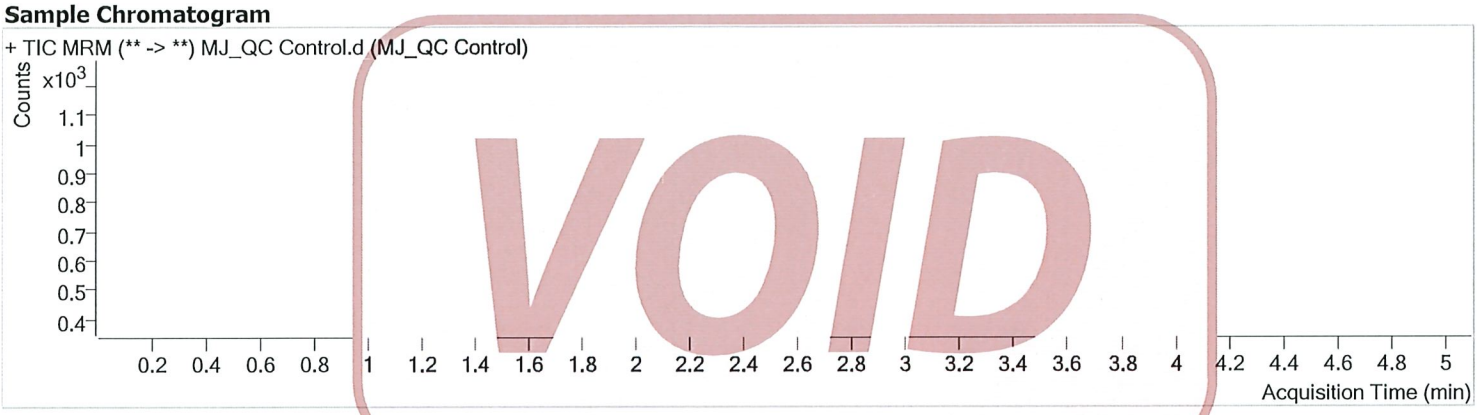
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update	5/5/2020 10:18:03 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	5/1/2020 3:07:53 AM		
Sample Info.			

Sample failed to inject. The sample was reconstituted and reinjected on 05/01/2020. Please refer to the reinjection data. TS

Sample Chromatogram



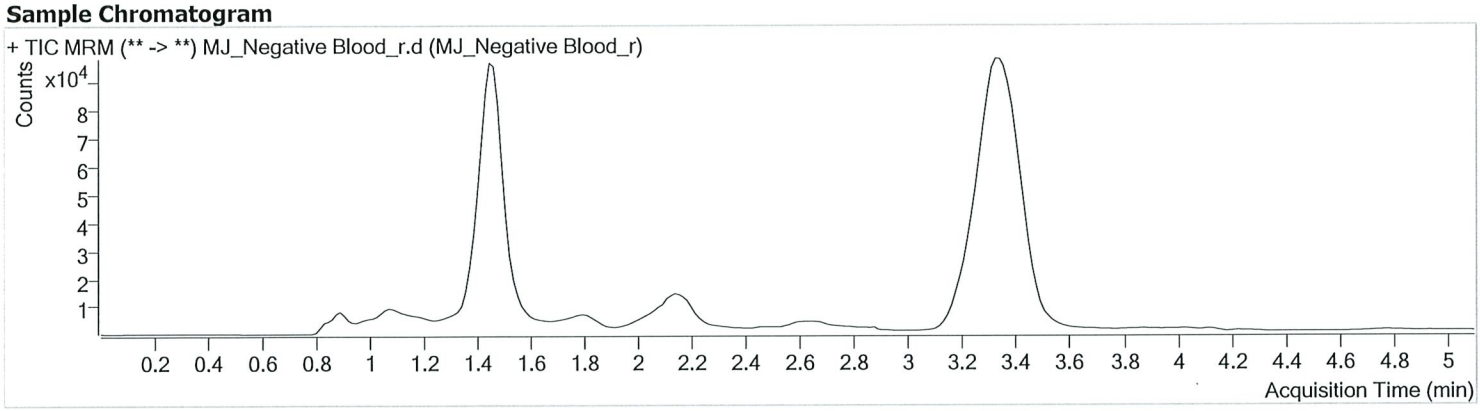
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 AM

Instrument	Falco	**Data File**	MJ_Negative Blood_r.d
Type	Sample	**Sample**	MJ_Negative Blood_r
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 7:17:08 PM		
Sample Info.			

Sample Chromatogram



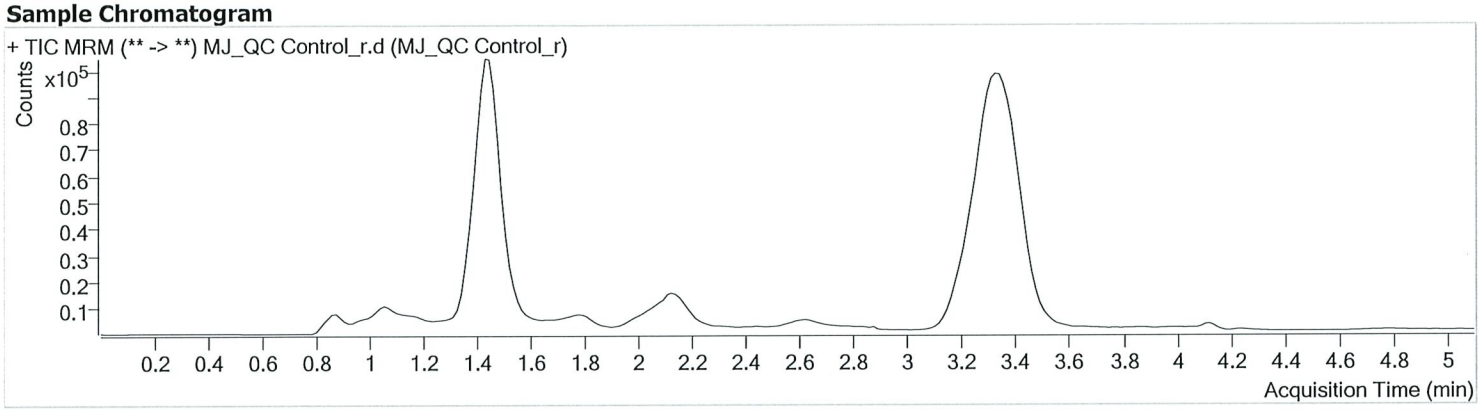
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 AM

Instrument	Falco	**Data File**	MJ_QC Control_r.d
Type	Sample	**Sample**	MJ_QC Control_r
Acq. Method	AM 27 THC quant.m	**Operator**	Tamara Salazar
Sample Position	P3-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 7:01:47 PM		
Sample Info.			

Sample Chromatogram



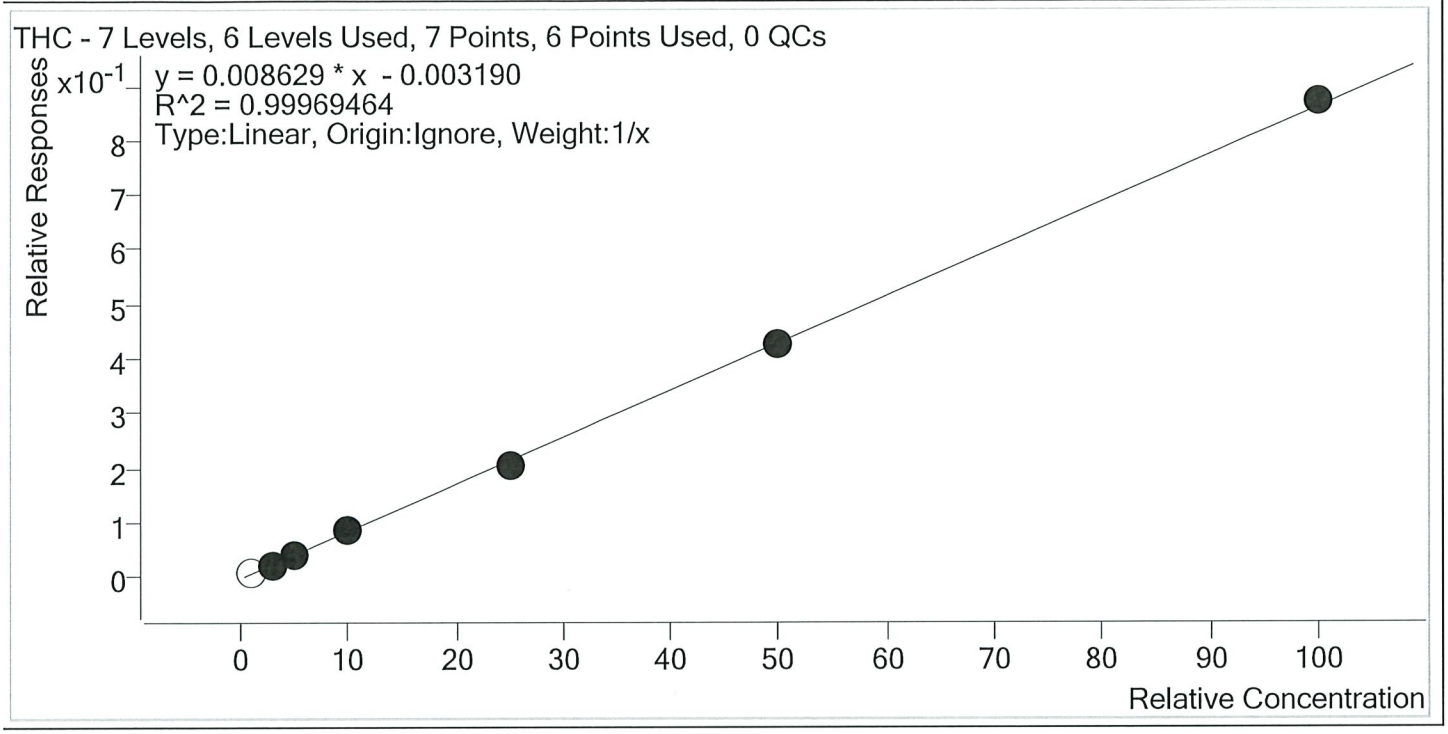
	Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
*	THC-OH	1.468	85679	∞	6.8 **Low**	23.18	446559	5.6170	ng/ml
	THC-COOH	1.474	39684	∞	50.5	∞	108389	16.4044	ng/ml
	THC	3.345	41978	133.01	27.8	36.37	1119824	4.7138	ng/ml

* Compound not evaluated - 5/5/2020 TS

TS

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	5/5/2020 10:18 AM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3

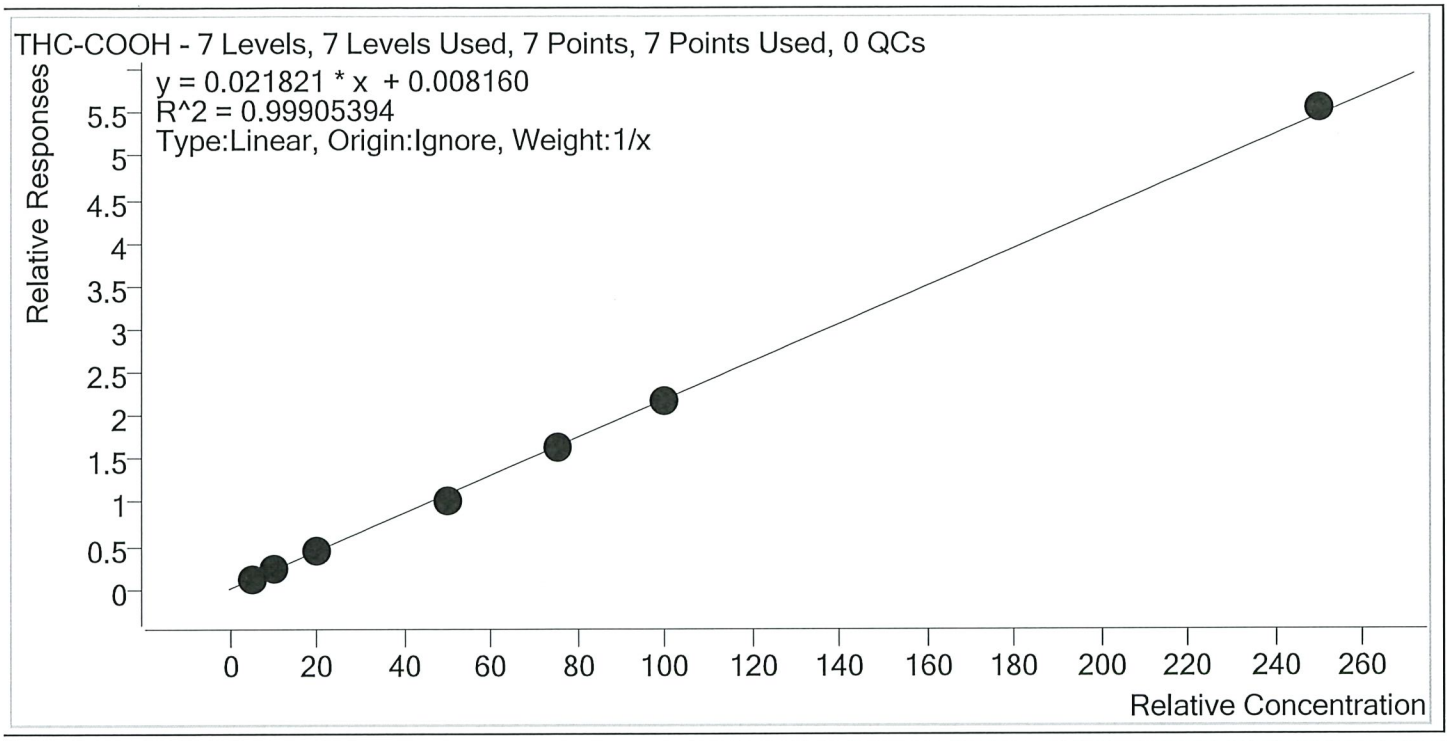


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	×	1.0	1.3	132.4
MJ_Cal 2	2	✓	3.0	3.0	101.0
MJ_Cal 3	3	✓	5.0	5.0	100.9
MJ_Cal 4	4	✓	10.0	10.1	101.1
MJ_Cal 5	5	✓	25.0	24.3	97.0
MJ_Cal 6	6	✓	50.0	49.4	98.9
MJ_Cal 7	7	✓	100.0	101.1	101.1

TS

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	5/5/2020 10:18 AM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9

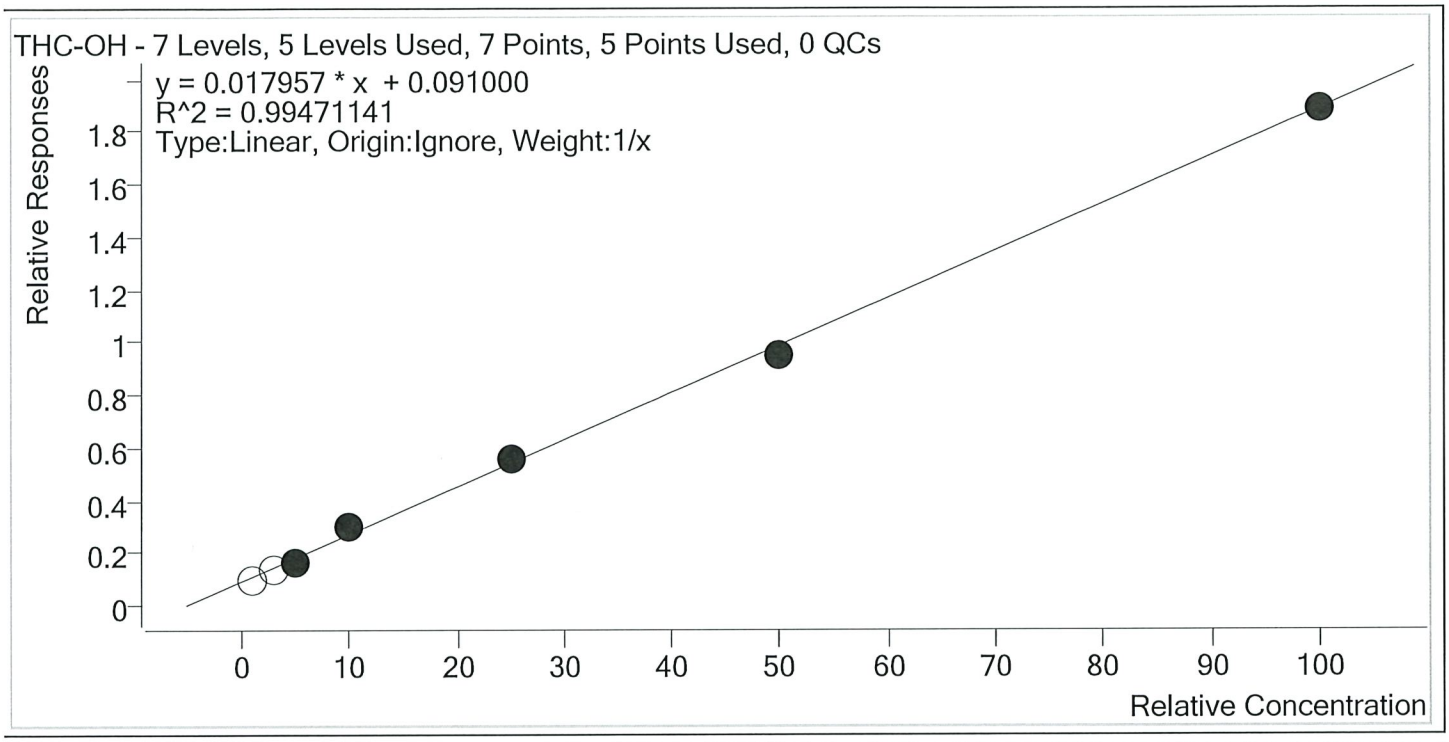


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	5.0	100.9
MJ_Cal 2	2	✓	10.0	10.5	104.9
MJ_Cal 3	3	✓	20.0	20.2	100.9
MJ_Cal 4	4	✓	50.0	46.3	92.5
MJ_Cal 5	5	✓	75.0	75.1	100.2
MJ_Cal 6	6	✓	100.0	99.2	99.2
MJ_Cal 7	7	✓	250.0	253.7	101.5

TS

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Last Cal. Update	5/5/2020 10:18 AM
Analyst Name	ISP\Datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	×	1.0	0.4	38.4
MJ_Cal 2	2	×	3.0	2.9	95.2
MJ_Cal 3	3	✓	5.0	4.0	80.8
MJ_Cal 4	4	✓	10.0	11.8	118.4
MJ_Cal 5	5	✓	25.0	26.2	104.7
MJ_Cal 6	6	✓	50.0	48.1	96.2
MJ_Cal 7	7	✓	100.0	99.8	99.8

Compound not evaluated. Multiple ratios out of range.
5/5/2020 TS

Generated at 10:18 AM on 5/5/2020

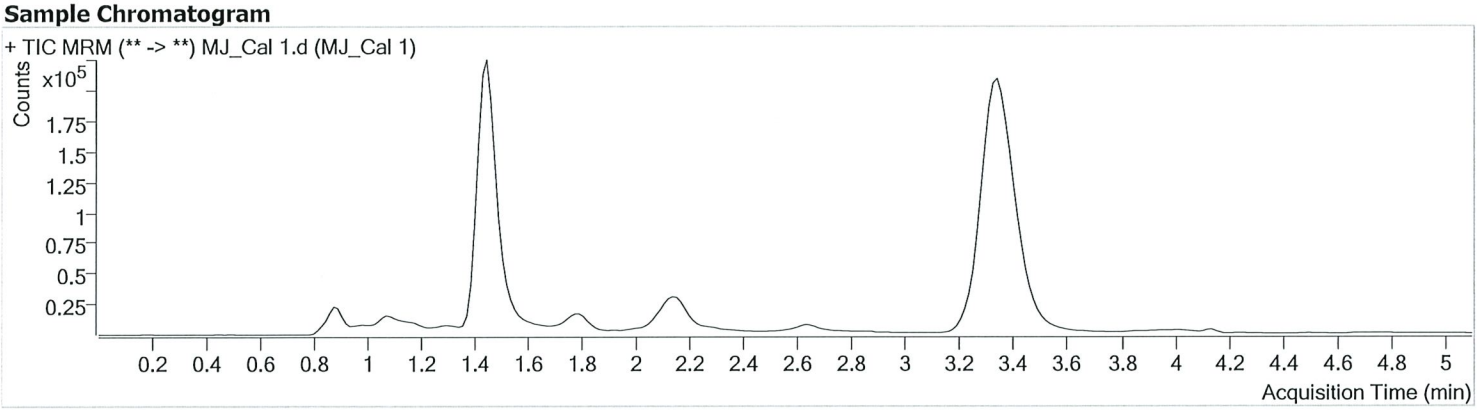
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:07:03 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.513 **High**	81799	∞	5.0 **Low**	∞	835613	0.3838	ng/ml **Low**
THC-COOH	1.474	24264	∞	45.6	116.73	205276	5.0429	ng/ml **Low**
THC	3.345	15168	80.10	27.5	6.24 **Low**	1842046	1.3239	ng/ml **Low**

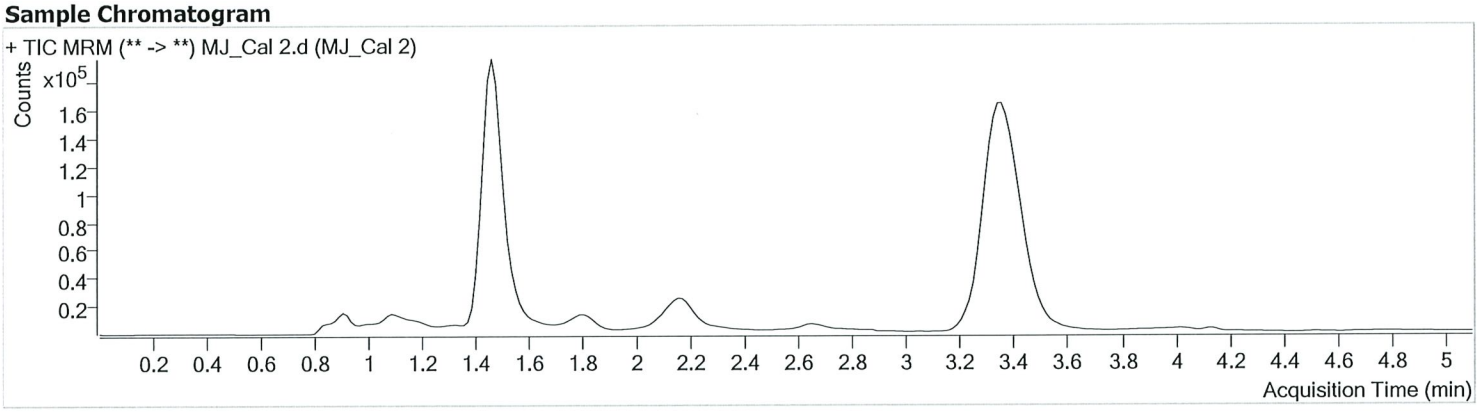
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:14:47 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.528 **High**	104610	∞	6.4 **Low**	∞	735244	2.8557	ng/ml **Low**
THC-COOH	1.489	42134	∞	48.7	41.15	177772	10.4876	ng/ml
THC	3.375	35968	102.72	30.6	10.13	1567471	3.0288	ng/ml

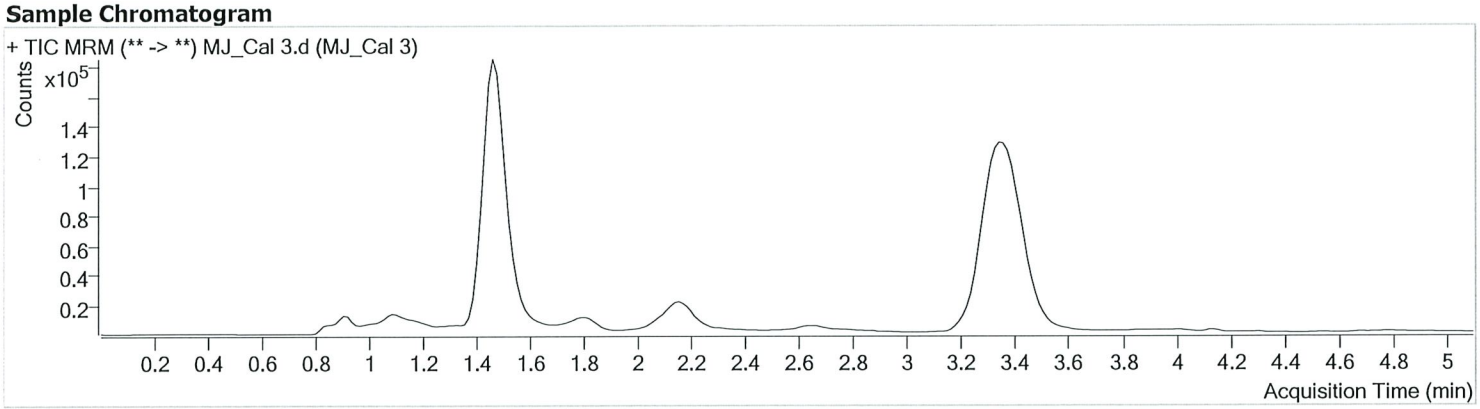
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:22:21 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	113315	∞	8.4	103.59	692839	4.0404	ng/ml
THC-COOH	1.504	73445	∞	53.5	146.57	163778	20.1767	ng/ml
THC	3.360	51658	131.07	28.7	16.35	1279838	5.0472	ng/ml

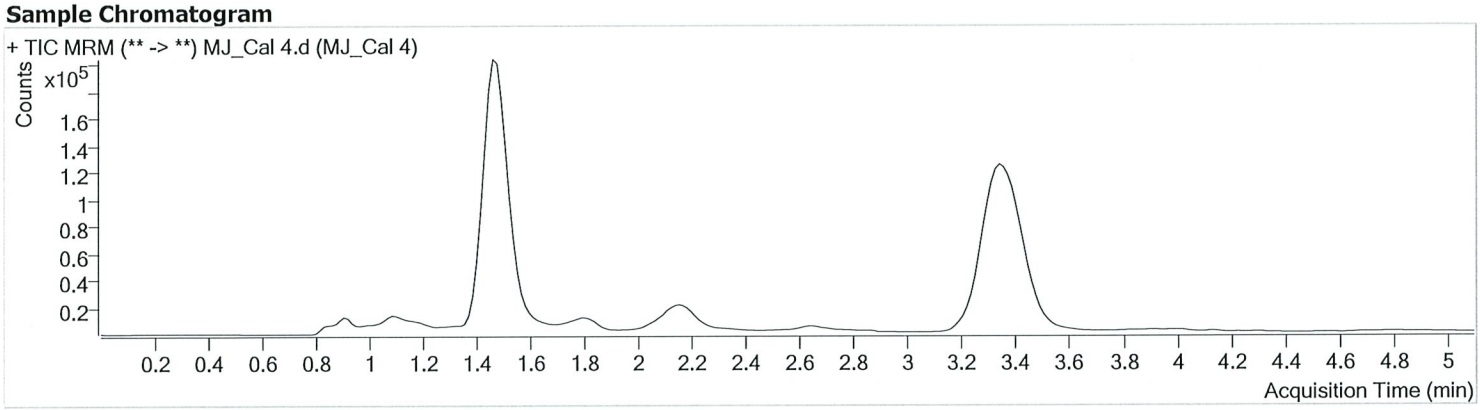
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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	5/1/2020 2:29:55 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	197420	∞	8.5	155.06	650176	11.8418	ng/ml
THC-COOH	1.504	158446	∞	57.5	1528.33	155687	46.2649	ng/ml
THC	3.360	100739	96.53	27.0	27.79	1198783	10.1080	ng/ml

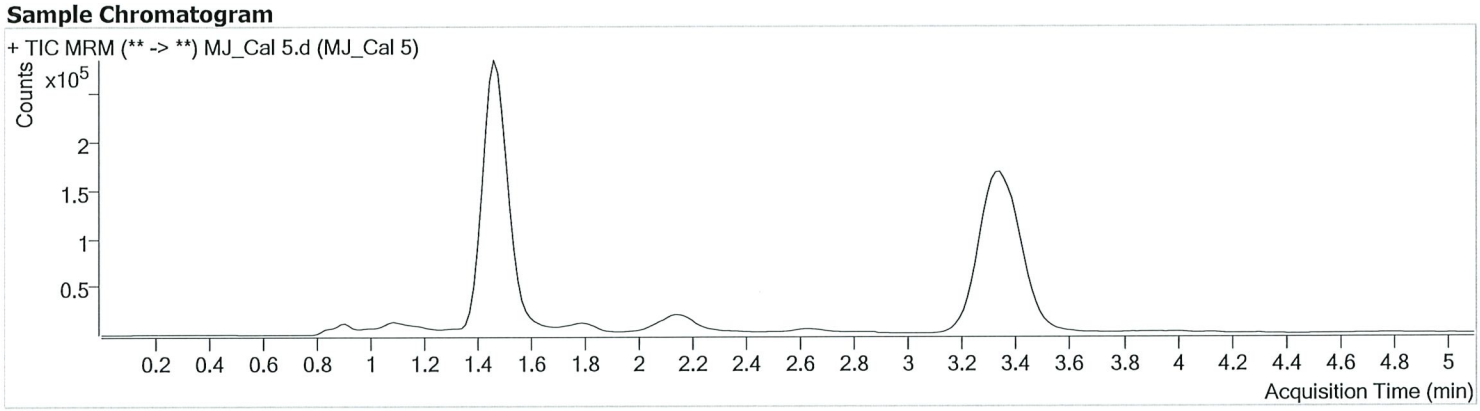
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:37:30 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	407735	∞	10.6	403.59	726613	26.1819	ng/ml
THC-COOH	1.489	287060	∞	57.4	∞	174217	75.1353	ng/ml
THC	3.345	303277	1572.21	26.9	197.73	1471241	24.2580	ng/ml

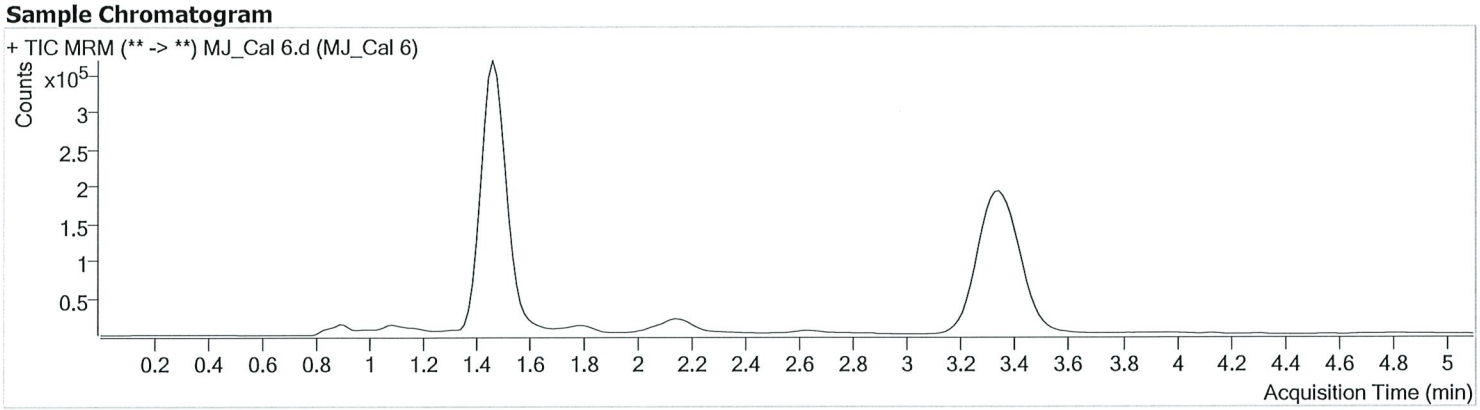
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:45:06 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	698832	∞	12.7 **High**	∞	731817	48.1112	ng/ml
THC-COOH	1.489	379666	∞	58.6	∞	174743	99.1943	ng/ml
THC	3.360	591472	1734.37	27.4	∞	1397231	49.4260	ng/ml

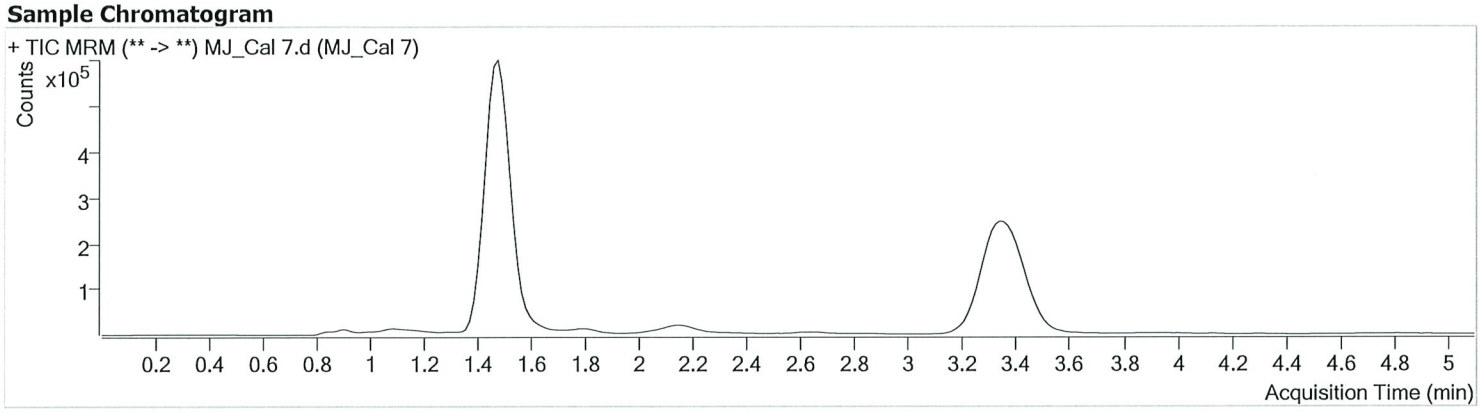
TS

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2020\AM 27-28\043020 AM 27 28 wklst 4205 4206 TS\QuantResults\AM 27.batch.bin
Calibration Last Update 5/5/2020 10:18:03 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-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	5/1/2020 2:52:41 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	1327538	∞	13.1 **High**	∞	704810	99.8247	ng/ml
THC-COOH	1.504	901283	∞	58.8	∞	162563	253.6984	ng/ml
THC	3.360	1158257	3578.90	26.1	∞	1332097	101.1320	ng/ml