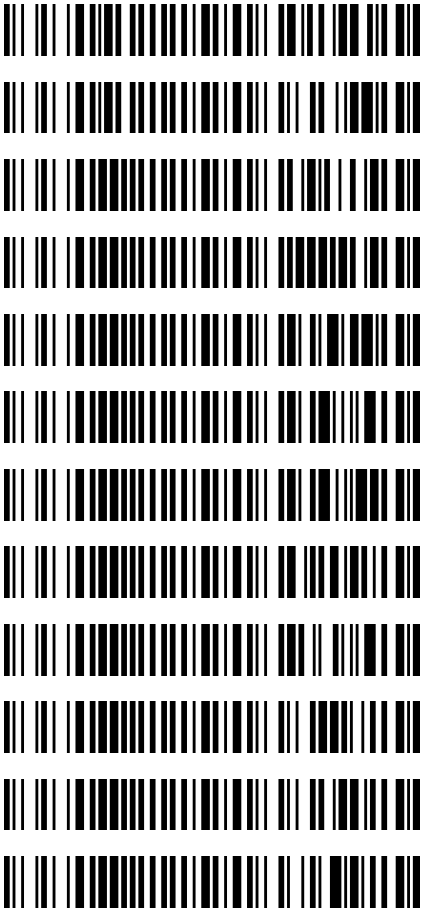
REVIEWED
By Celena Shrum at 10:27 am, Apr 09, 2021

4/8/2021

Worklist: 4890

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2021-1048	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-1387	3	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0895	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0940	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0956	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0957	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0958	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0960	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0981	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0990	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0991	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-1015	1	BCK	AM 27 Blood THC Quant by LC-QQQ



SC

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

Extraction Date: 04/07/21 Analyst: Sarah Collins
Plate lot#: IDP-108-2-201206 Plate Expiration: 06/06/21

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: Lampire 20L20724 **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. Pipette **1000 µL blood (calibrated pipette)** in wells of analytical (standards) plate. **Pipette ID: #3382167**
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 4. Pipette **500 µL 0.1% formic acid in LCMS water** in wells of analytical plate.
☒ 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 6. Transfer **800 µL 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 85-95 PSI- Selector to the right)***
☒ 8. Wait 5 minutes.
☒ 9. Add **2.25 mL MTBE. (*Add in 3 increments of 750 µL)***
☒ 10. Wait 5 minutes.
☒ 11. Apply positive pressure for approx. 15 seconds. ***(12-15 PSI- Selector to the left).***
☒ 12. Add **2.25 mL Hexane. (*Add in 3 increments of 750 µL)***
☒ 13. Wait 5 minutes.
☒ 14. Apply positive pressure for approx. 15 seconds. ***(12-15 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 **100 µL 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 sample response for THC 1ng/mL and OH-THC 3ng/mL (quantitative), Carboxy-THC: 5ng/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: *Curve range limited: THC 3-100 THC-OH 3-100*
Reinjected Calibrator 6 due low internal standard response in initial injection. Reinject data used.

AC

Idaho State Police
Forensic Services
Toxicology Discipline

Request for Departure from an Analytical Method

Date of Request
02/24/2021

Forensic Scientist
Anne Nord

Analytical Method
Toxicology AM #27: Quantitative Analysis of THC and Metabolites in Blood and Urine by LCMS-QQQ

Request

The method currently reads:
4.3.2.5 If any points are dropped from the approved quantitative range of the curve, the compound will be reported qualitatively. For calibrators and controls 10 ng and below, the accuracy must be within 30%, for calibrators and controls greater than 10 ng/mL the accuracy must be within 20%. If a control falls outside the accuracy range, at the analyst's discretion, the compound may be reported qualitatively.

I would like to add in the following exception:

If the 1ng/ml point is dropped for THC. If the 1 ng/ml point is dropped the quantitative range will be 3-50 ng/ml.

Discipline Leader Review

☒ Departure approved
Comments: This deviation is approved and will remain in effect until it is changed in the actual method.

☐ Departure Not Approved
Comments:

Celena Shrum

Celena Shrum
Toxicology Discipline Lead
Date: 02/24/2021

	1	2	3	4	5	6
A	IS + Cal. 1	negative	p2021-0960-1			IS + QC_1
B	IS + Cal. 2	m2021-1048-2	p2021-0981-1			IS + Cal. 7
C	IS + Cal. 3	m2021-1387-3	p2021-0990-1			IS + Cal. 6
D	IS + Cal. 4	p2021-0895-1	p2021-0991-1			IS + Cal. 5
E	IS + Cal. 5	p2021-0940-1	p2021-1015-1*			IS + Cal. 4
F	IS + Cal. 6	p2021-0956-1	p2021-1015-1			IS + Cal. 3
G	IS + Cal. 7	p2021-0957-1				IS + Cal. 2
H	IS + QC_1	p2021-0958-1				IS + Cal. 1

All wells to contain 100 µl of residual DMSO

*Sample moved during analytical step 6 due to blood clot

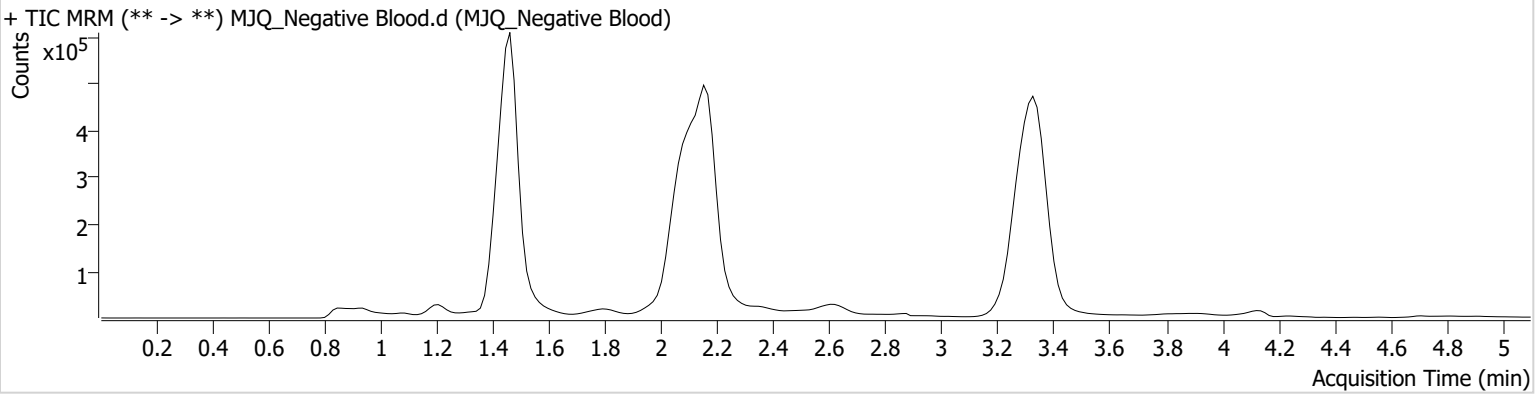
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Negative Blood.d
Type	Sample	**Sample**	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-A2	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 12:09:58 PM		
Sample Info.			

Sample Chromatogram



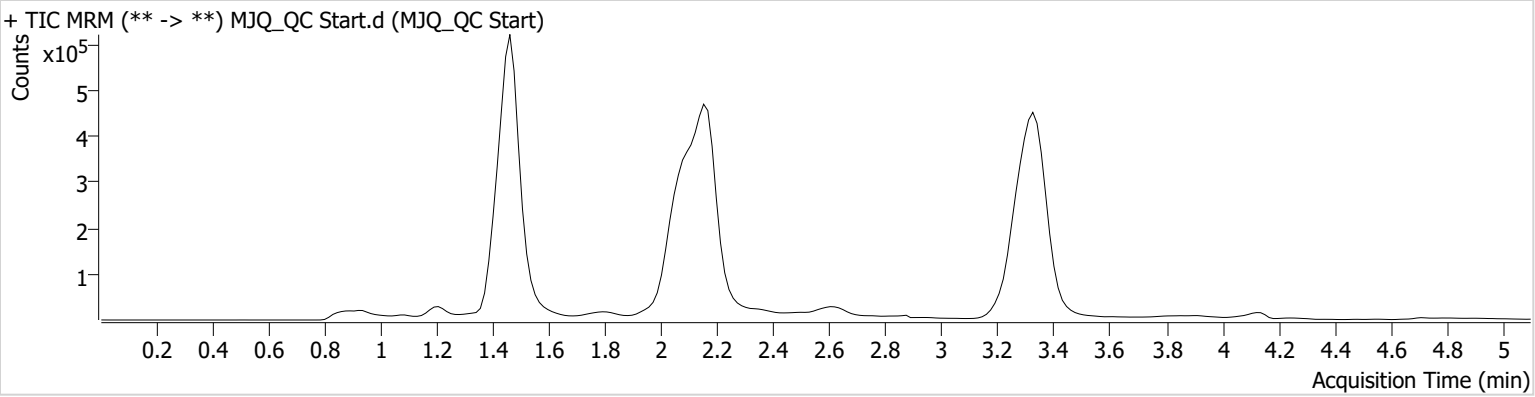
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_QC Start.d
Type	Sample	**Sample**	MJQ_QC Start
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 12:25:10 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	181094	∞	8.2	59.57	2360605	4.0092	ng/ml
THC-COOH	1.489	196710	∞	54.0	606.05	523262	14.6748	ng/ml
THC	3.345	117510	493.75	30.3	44.75	3436313	4.0773	ng/ml

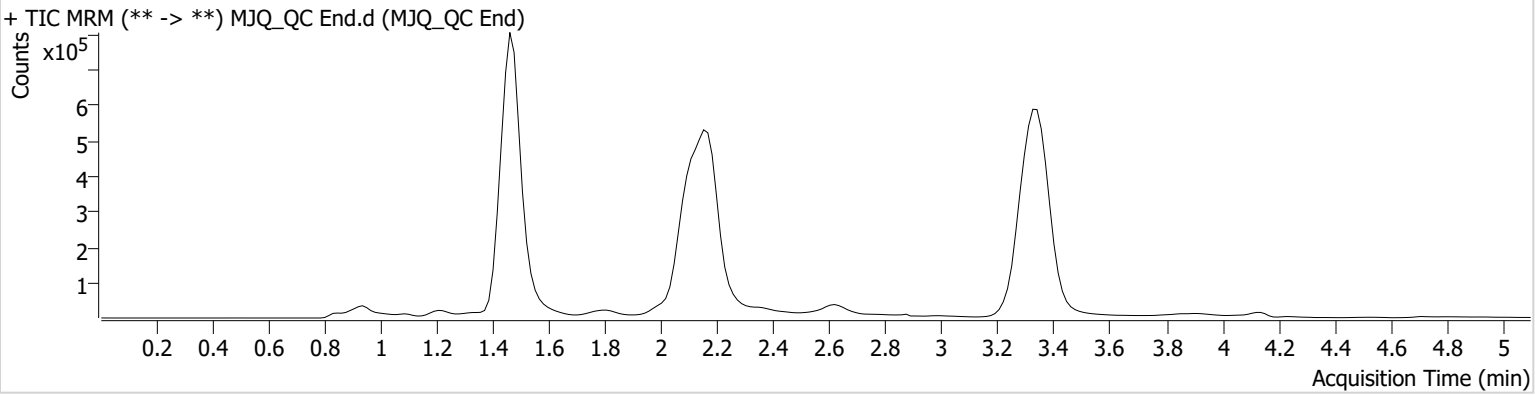
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_QC End.d
Type	Sample	**Sample**	MJQ_QC End
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 3:50:32 PM		
Sample Info.			

Sample Chromatogram

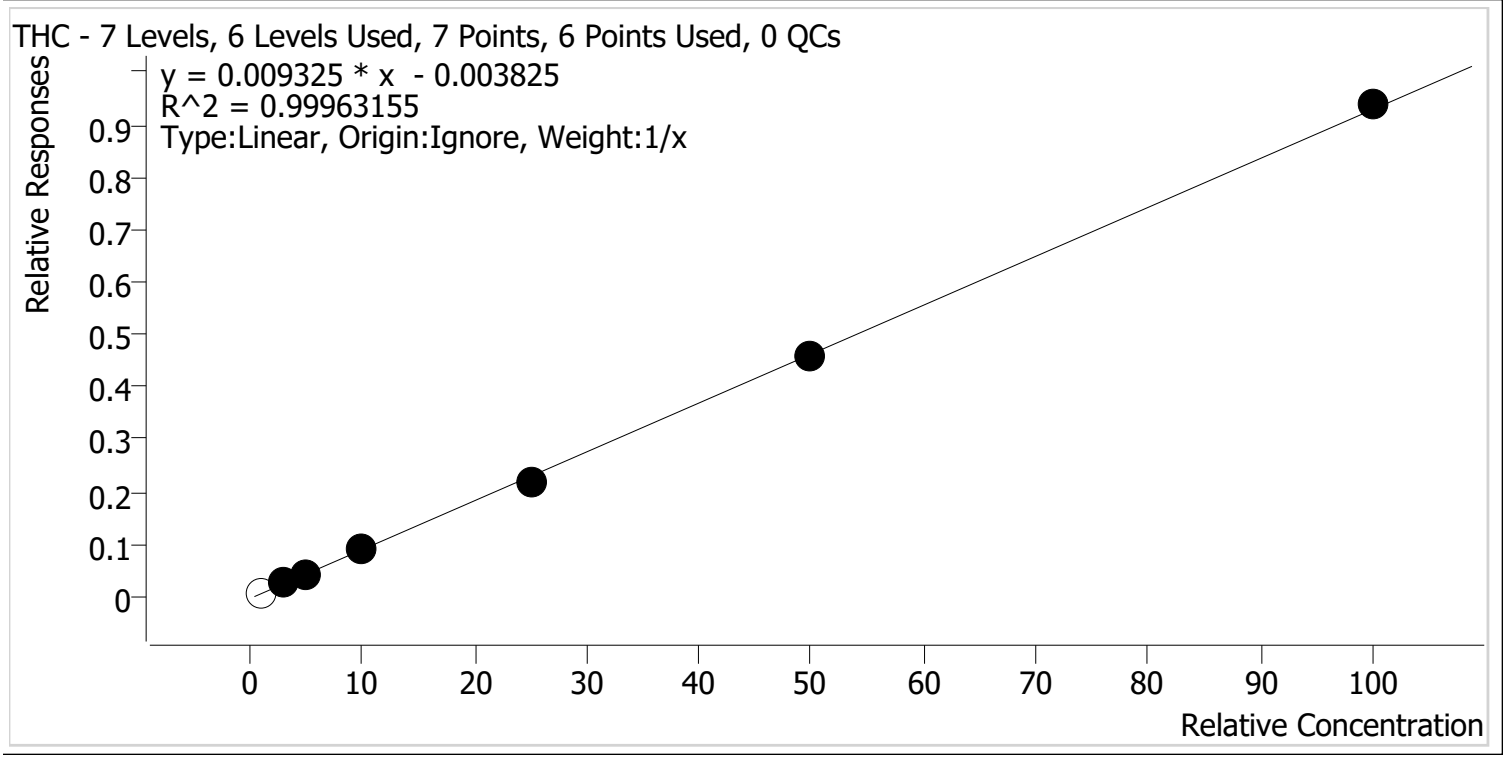


Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.483	207121	∞	8.3	78.71	2837023	3.7189	ng/ml
THC-COOH	1.504	234805	215.22	55.2	699.63	659112	13.8907	ng/ml
THC	3.345	146658	702.07	32.4	∞	4232621	4.1258	ng/ml

SC

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	4/8/2021 9:07 AM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3



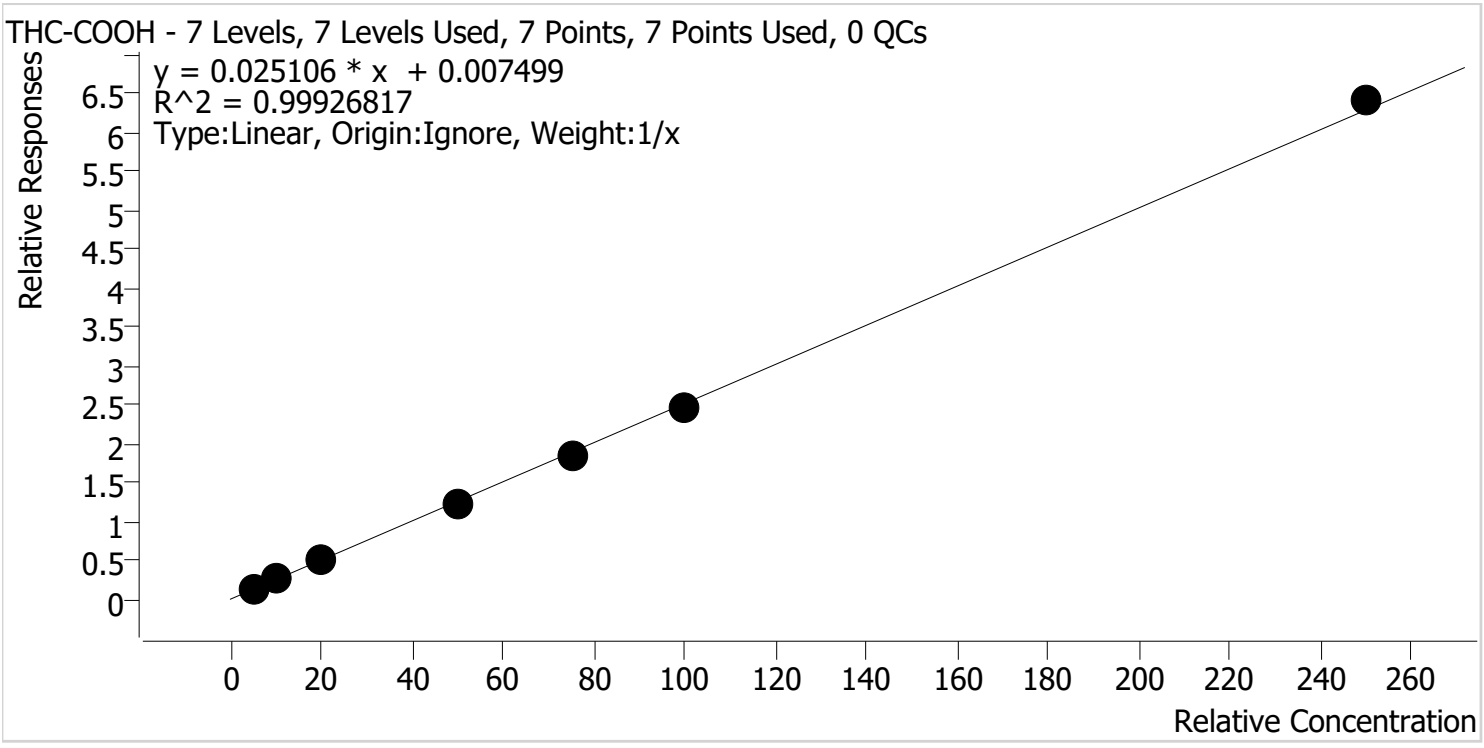
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✗	1.0	1.2	121.2
MJQ_Cal 2	2	✔	3.0	3.0	101.2
MJQ_Cal 3	3	✔	5.0	5.1	101.5
MJQ_Cal 4	4	✔	10.0	10.1	100.5
MJQ_Cal 5	5	✔	25.0	24.0	96.0
MJQ_Cal 6R	6	✔	50.0	49.9	99.7
MJQ_Cal 7	7	✔	100.0	101.0	101.0

Calibrator 1 dropped due to not meeting ratio requirement

SC

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	4/8/2021 9:07 AM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9

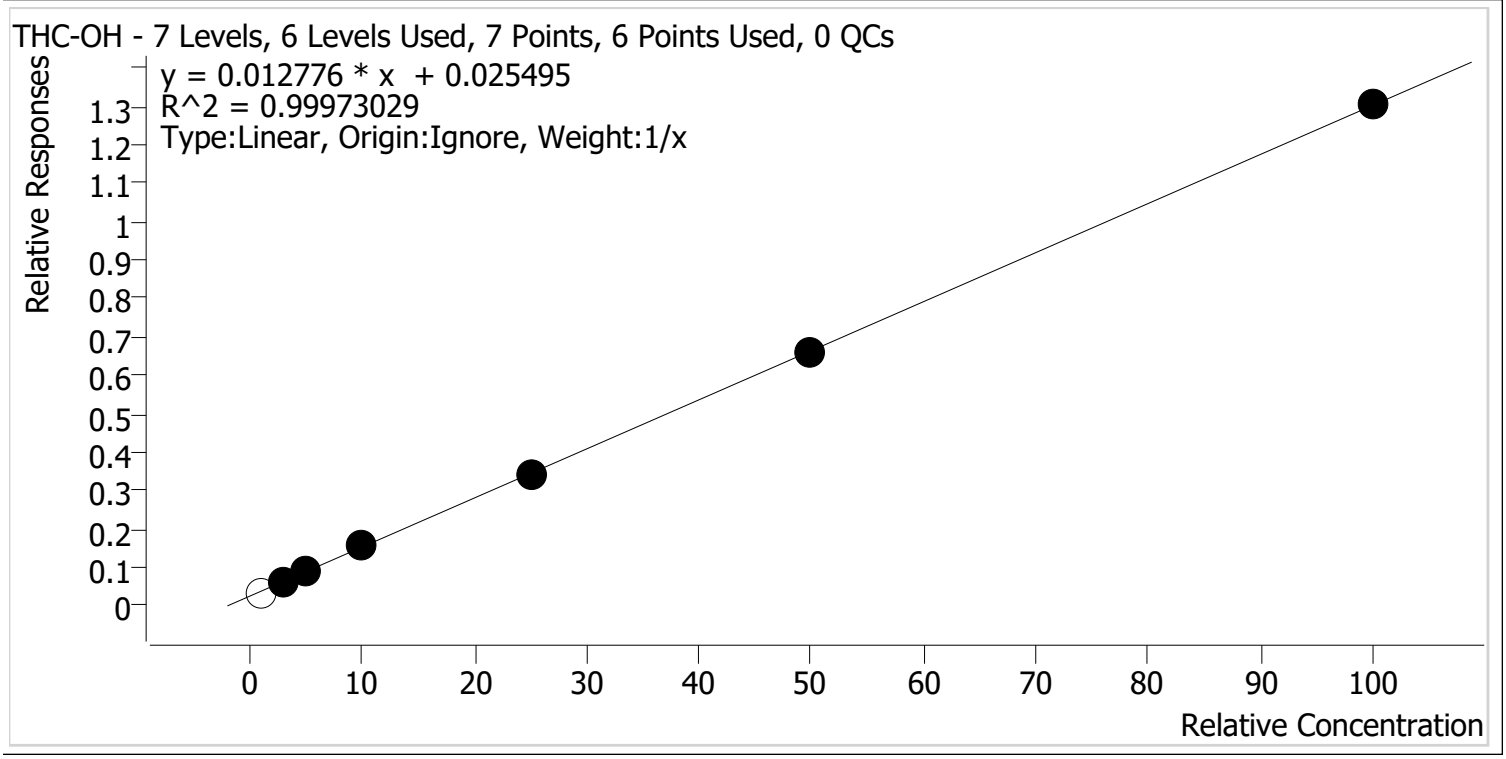


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	5.0	5.2	103.1
MJQ_Cal 2	2	✔	10.0	9.9	98.7
MJQ_Cal 3	3	✔	20.0	20.9	104.3
MJQ_Cal 4	4	✔	50.0	48.3	96.7
MJQ_Cal 5	5	✔	75.0	73.1	97.5
MJQ_Cal 6R	6	✔	100.0	97.9	97.9
MJQ_Cal 7	7	✔	250.0	254.8	101.9

SC

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Last Cal. Update	4/8/2021 9:07 AM
Analyst Name	ISP\Datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✗	1.0	0.7	70.0
MJQ_Cal 2	2	✔	3.0	2.9	96.4
MJQ_Cal 3	3	✔	5.0	5.0	100.9
MJQ_Cal 4	4	✔	10.0	10.5	104.7
MJQ_Cal 5	5	✔	25.0	24.5	97.8
MJQ_Cal 6R	6	✔	50.0	50.0	100.0
MJQ_Cal 7	7	✔	100.0	100.1	100.1

Calibrator 1 dropped due to not meeting ratio requirement

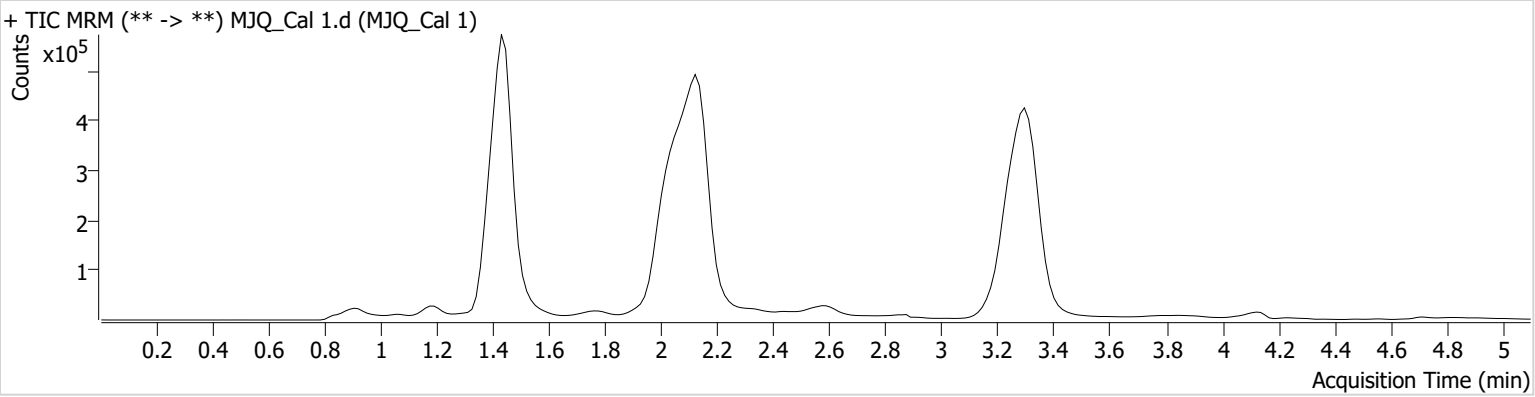
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 1.d
Type	Cal	**Sample**	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-A1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:09:04 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.513 High	85761	25.31	5.3 Low	27.80	2490498	0.6998	ng/ml Low
THC-COOH	1.474	74871	∞	49.3	∞	546813	5.1550	ng/ml
THC	3.315	26172	65.33	53.5 High	34.45	3500706	1.2119	ng/ml

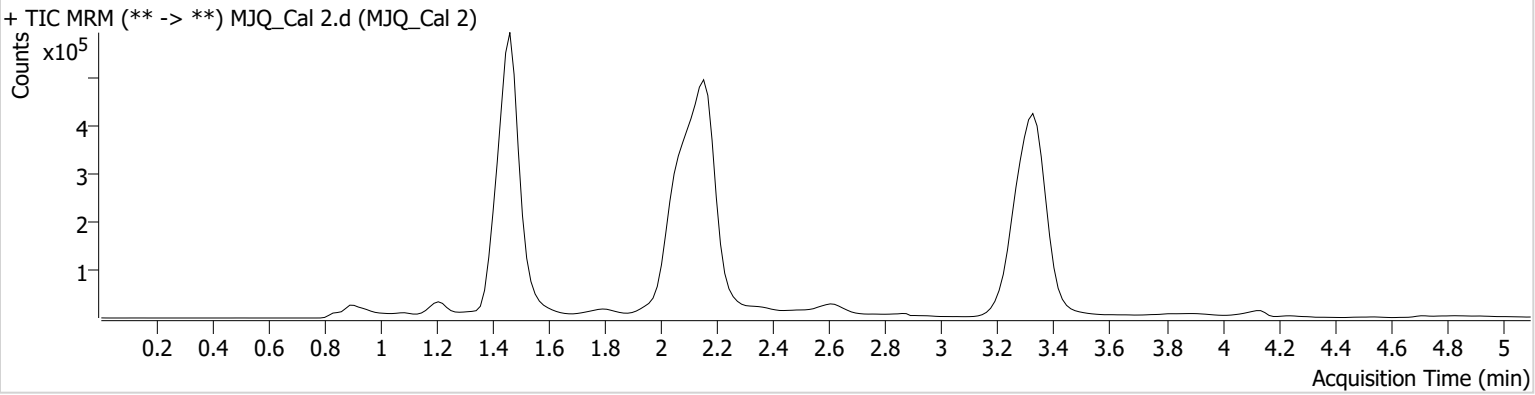
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 2.d
Type	Cal	**Sample**	MJQ_Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-B1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:16:49 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	144933	∞	8.0	52.01	2320574	2.8930	ng/ml Low
THC-COOH	1.489	138880	∞	51.8	711.18	544043	9.8690	ng/ml
THC	3.345	81136	305.03	33.2	61.53	3311924	3.0372	ng/ml

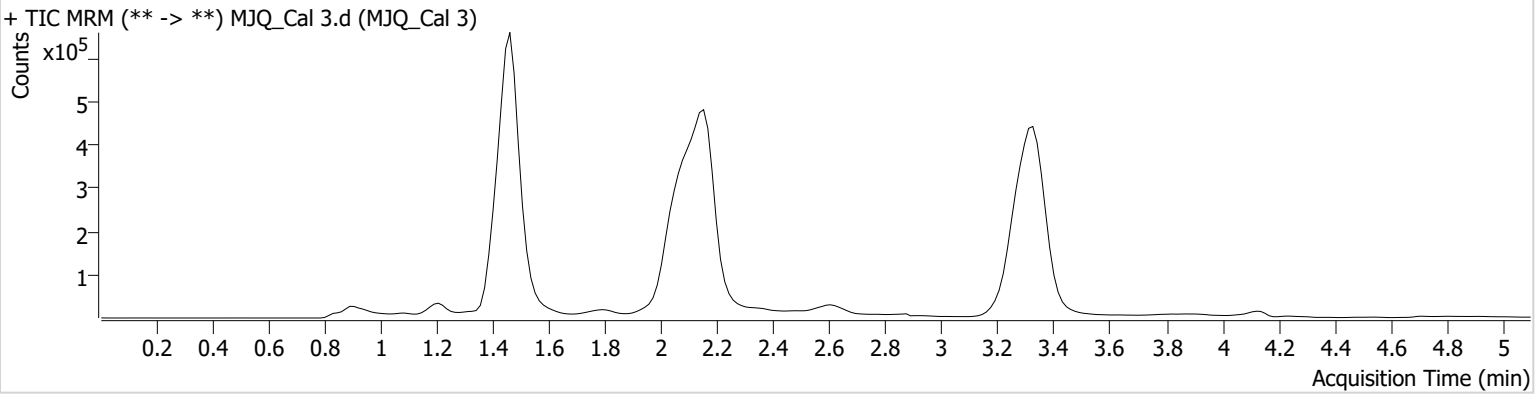
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 3.d
Type	Cal	**Sample**	MJQ_Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-C1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:24:24 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	219201	50.65	8.3	304.62	2437274	5.0441	ng/ml
THC-COOH	1.489	280564	∞	54.2	∞	528317	20.8535	ng/ml
THC	3.330	145616	626.98	31.3	∞	3345786	5.0773	ng/ml

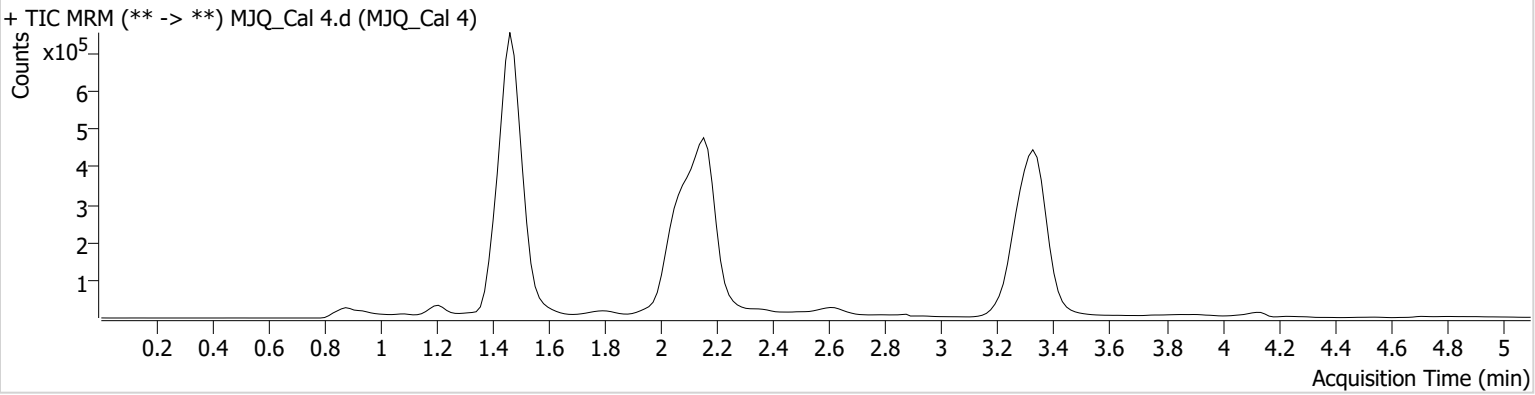
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 4.d
Type	Cal	**Sample**	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-D1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:32:00 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	372639	∞	9.4	303.60	2339864	10.4700	ng/ml
THC-COOH	1.489	653320	1464.58	56.2	∞	535138	48.3283	ng/ml
THC	3.345	289509	∞	27.3	∞	3219465	10.0533	ng/ml

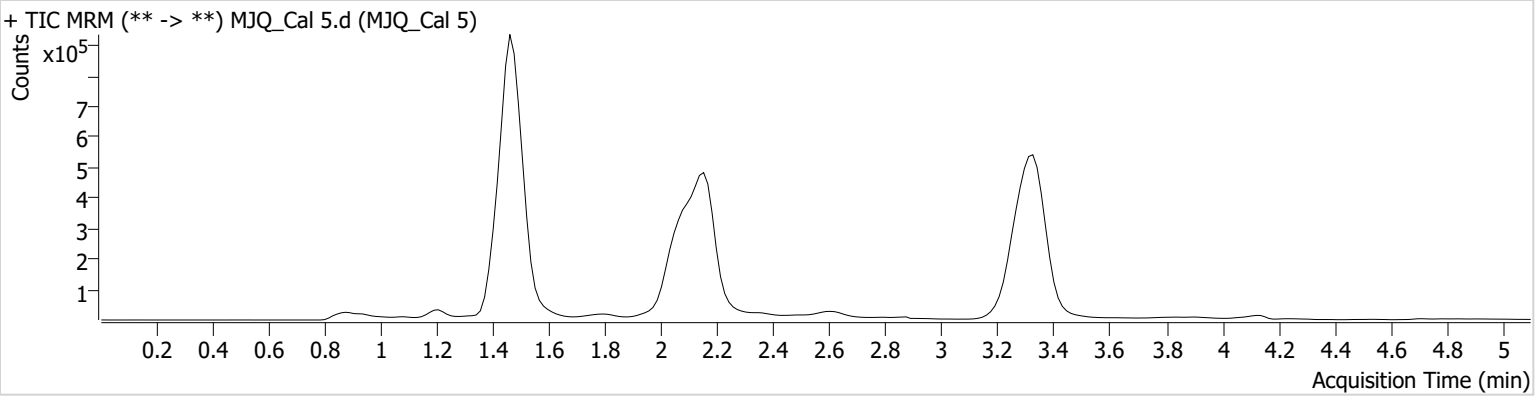
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 5.d
Type	Cal	**Sample**	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-E1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:39:35 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	797666	∞	10.7	303.85	2360364	24.4562	ng/ml
THC-COOH	1.489	981000	∞	56.8	∞	532288	73.1086	ng/ml
THC	3.330	752835	∞	27.2	∞	3423240	23.9934	ng/ml

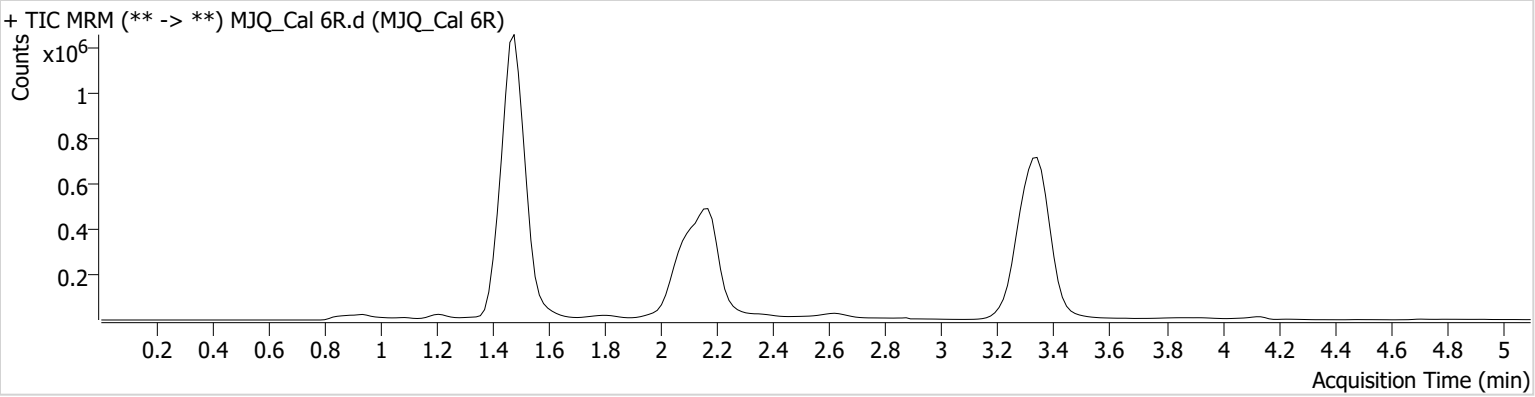
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 6R.d
Type	Cal	**Sample**	MJQ_Cal 6R
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 1:48:50 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	1661117	∞	11.1	5074.42	2499535	50.0225	ng/ml
THC-COOH	1.504	1395246	∞	57.3	4708.20	565997	97.8882	ng/ml
THC	3.345	1678588	455.29	25.1	242.79	3639645	49.8670	ng/ml

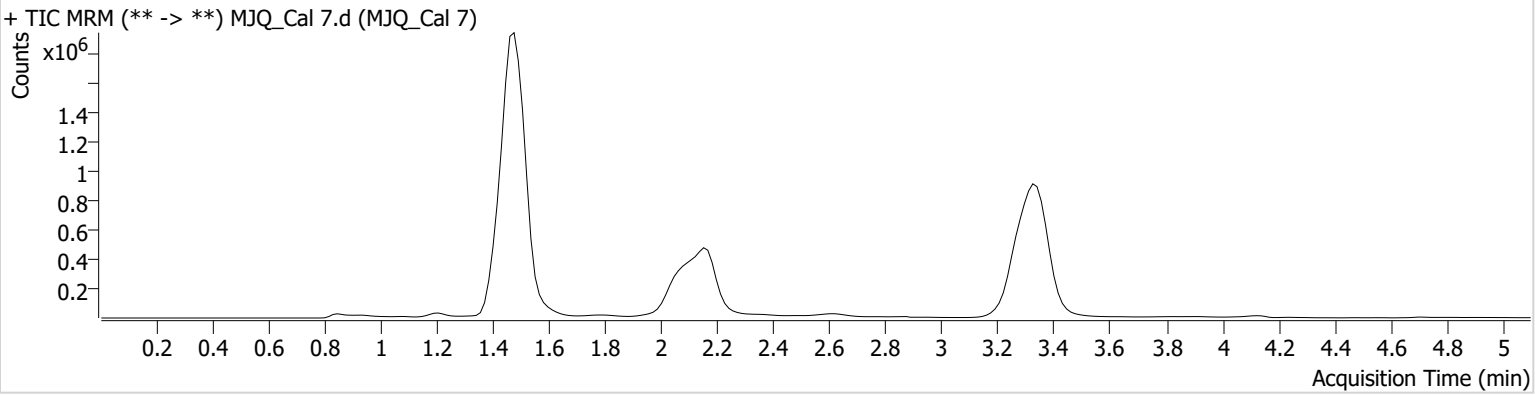
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 27-28\040721 AM 27 28 SC\QuantResults\AM 27.batch.bin
Calibration Last Update 4/8/2021 9:07:24 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 7.d
Type	Cal	**Sample**	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-G1	**Comment**	
Injection Volume	10		
Acq. Date-Time	4/7/2021 11:54:46 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468	3095918	∞	11.7	1966.21	2373207	100.1142	ng/ml
THC-COOH	1.489	3189628	8122.00	58.3	∞	498028	254.7974	ng/ml
THC	3.345	3231347	∞	25.1	∞	3445818	100.9718	ng/ml