BC 3/10/2021

REVIEWED
By Britany Wylie at 12:33 pm, Mar 12, 2021

Worklist: 4834

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2020-5027	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-0533	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-0671	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0324	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0390	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0437	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0438	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0471	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0481	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0482	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0513	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0527	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0528	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: 03/09/21 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 Reconstituted and reinjected Calibrator 1 due retention time shift in initial injection. Reinject data used.*

8C

	1	2	3	4	5	6
A	IS + Cal. 1				p2021-0438-1	IS + QC_1
B	IS + Cal. 2			m2021-0533-1	p2021-0437-1	IS + Cal. 7
C	IS + Cal. 3			p2021-0528-1	p2021-0390-1	IS + Cal. 6
D	IS + Cal. 4			p2021-0527-1	p2021-0324-1	IS + Cal. 5
E	IS + Cal. 5			p2021-0513-1	m2021-0671-2	IS + Cal. 4
F	IS + Cal. 6			p2021-0482-1	m2021-0533-1*	IS + Cal. 3
G	IS + Cal. 7			p2021-0481-1	m2020-5027-1	IS + Cal. 2
H	IS + QC_1			p2021-0471-1	negative	IS + Cal. 1

All wells to contain 100 µl of residual DMSO

*Sample moved during analytical step 6 due to blood clot

SC

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

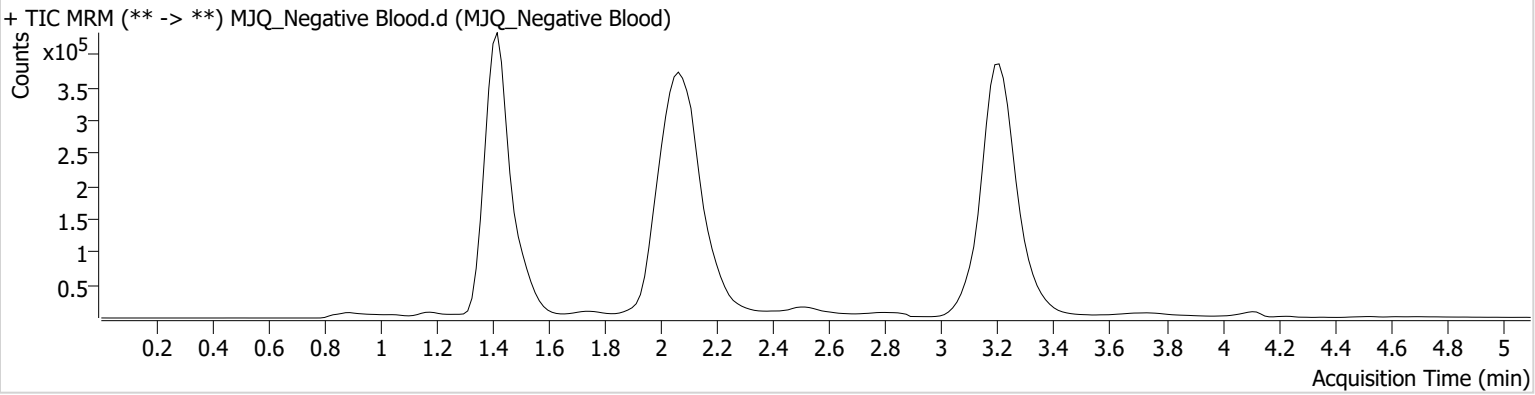
SC

AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	3/10/2021 8:49:17 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-H5	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 4:02:24 PM		
Sample Info.			

Sample Chromatogram



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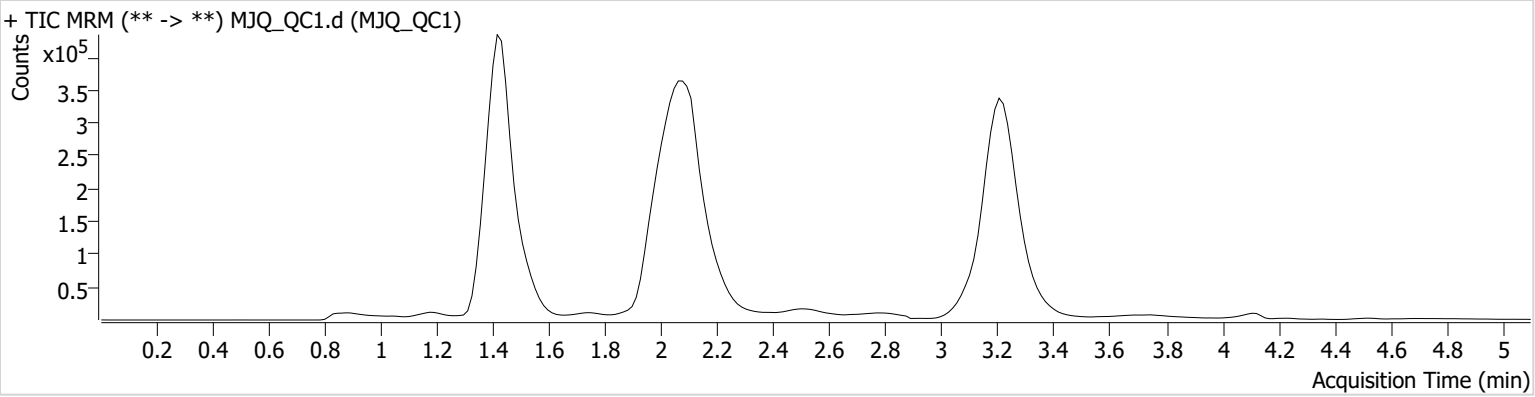
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 AM

Instrument	Instrument 1	**Data File**	MJQ_QC1.d
Type	Sample	**Sample**	MJQ_QC1
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 4:17:38 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.438	122672	∞	10.4	∞	2006622	3.9432	ng/ml
THC-COOH	1.459	184769	720.90	54.4	360.51	537398	13.7469	ng/ml
THC	3.224	108881	∞	32.5	∞	2969039	4.4373	ng/ml

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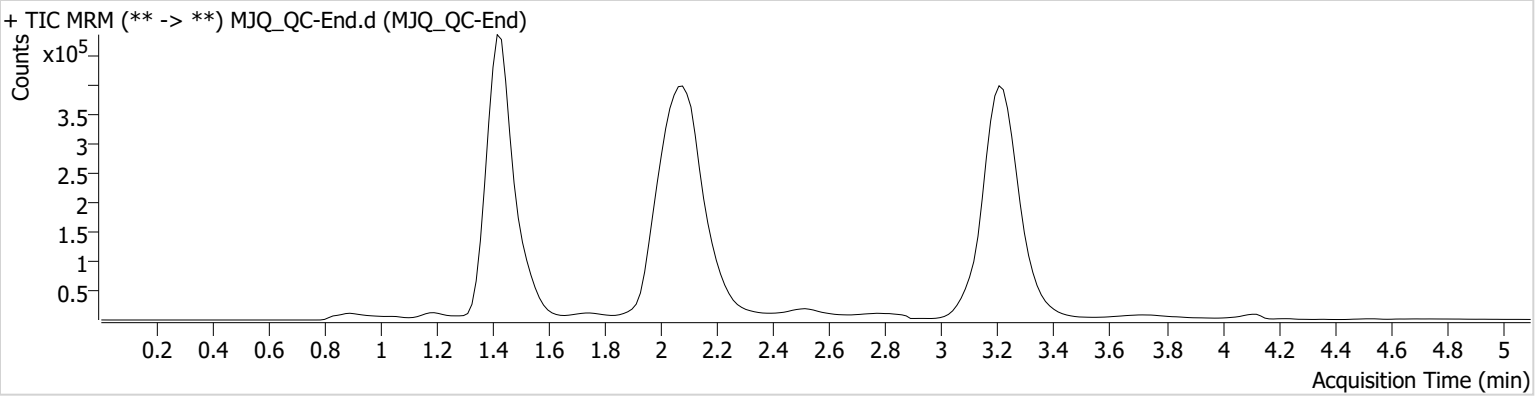
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 7:50:47 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.438	131261	∞	10.6	∞	2187842	3.8536	ng/ml
THC-COOH	1.459	200398	334.04	55.2	259.56	583757	13.7244	ng/ml
THC	3.209	117438	764.14	34.1	∞	3467077	4.1291	ng/ml

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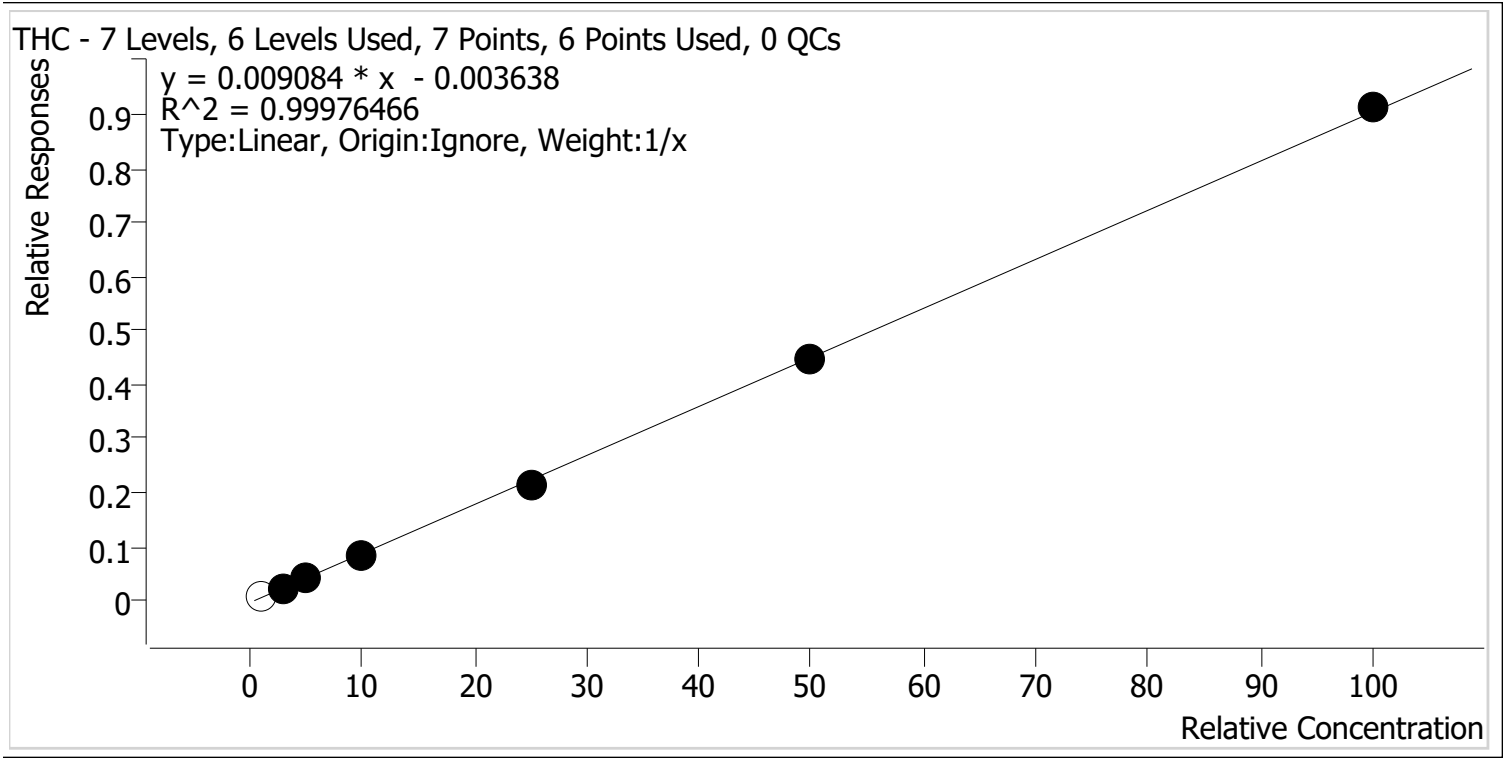
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Last Cal. Update	3/12/2021 9:16 AM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3



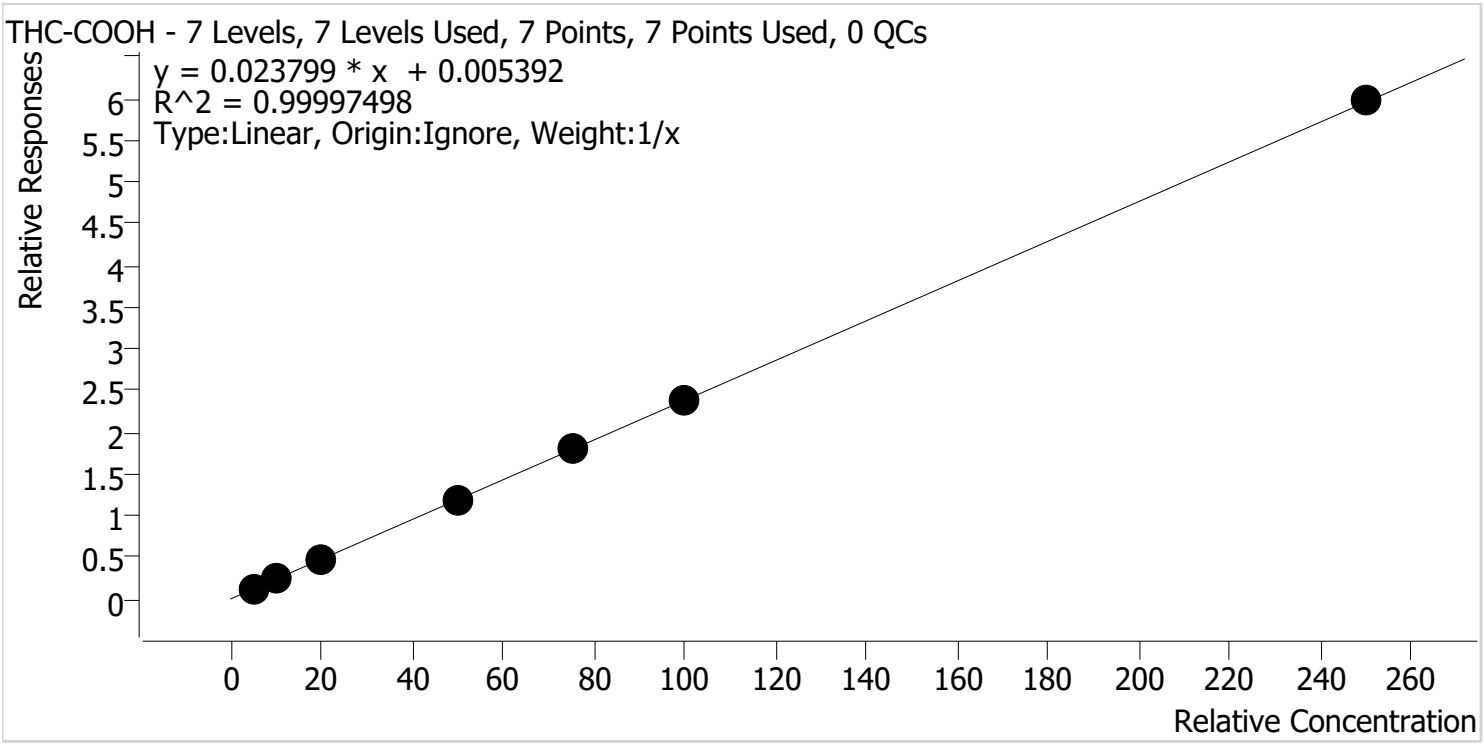
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1R	1	✗	1.0	1.4	141.5
MJQ_Cal 2	2	✔	3.0	3.0	100.8
MJQ_Cal 3	3	✔	5.0	5.1	102.5
MJQ_Cal 4	4	✔	10.0	9.9	99.2
MJQ_Cal 5	5	✔	25.0	24.3	97.1
MJQ_Cal 6	6	✔	50.0	49.8	99.5
MJQ_Cal 7	7	✔	100.0	100.9	100.9

Cal 1 dropped due to not meeting retention time requirement

SC

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Last Cal. Update	3/12/2021 9:16 AM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1R	1	✔	5.0	5.0	100.3
MJQ_Cal 2	2	✔	10.0	10.0	100.2
MJQ_Cal 3	3	✔	20.0	20.1	100.7
MJQ_Cal 4	4	✔	50.0	49.5	98.9
MJQ_Cal 5	5	✔	75.0	74.8	99.7
MJQ_Cal 6	6	✔	100.0	99.9	99.9
MJQ_Cal 7	7	✔	250.0	250.7	100.3

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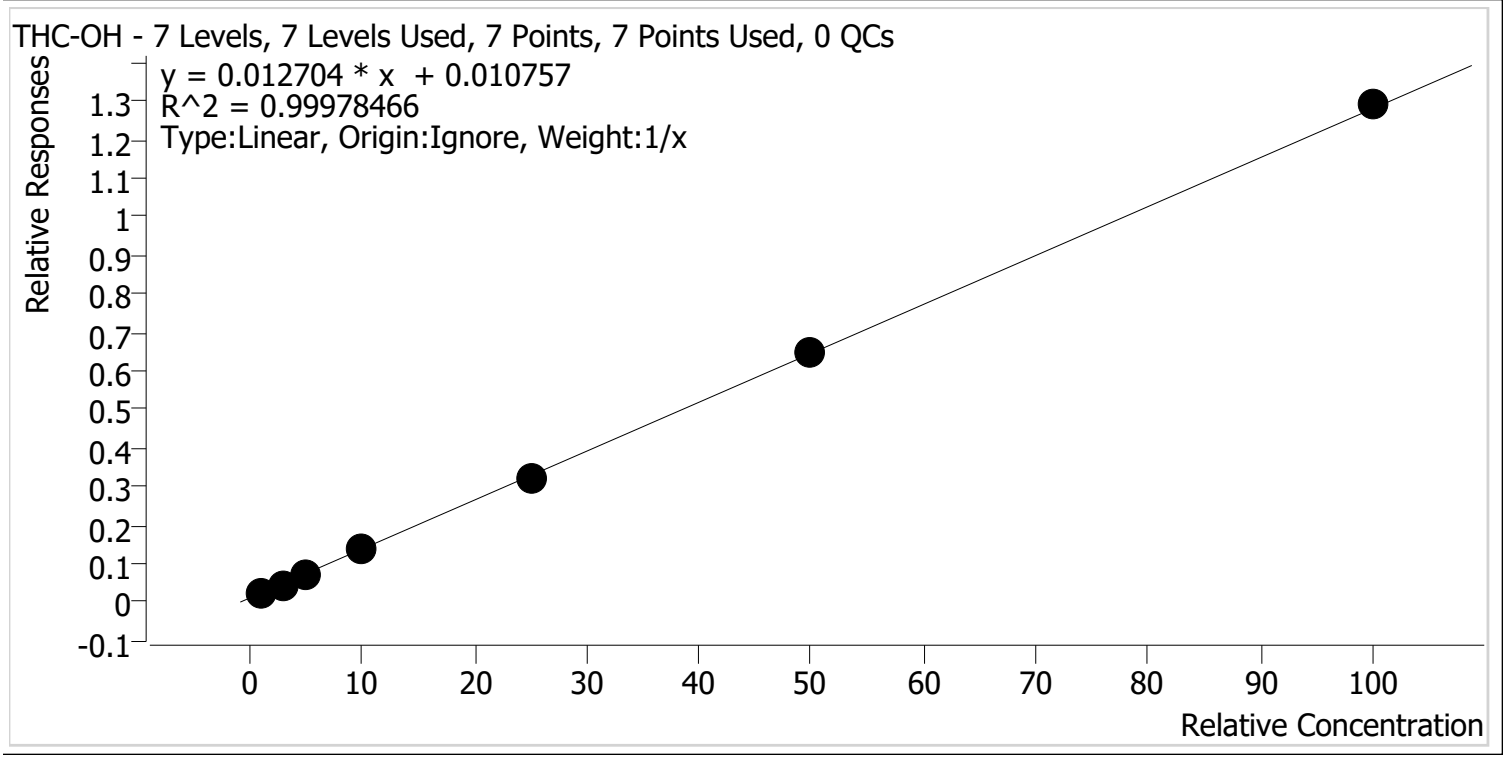
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Last Cal. Update	3/12/2021 9:16 AM
Analyst Name	ISP\Datastor
Analyte	THC-OH
Internal Standard	THC-OH-D3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1R	1	✔	1.0	1.1	107.3
MJQ_Cal 2	2	✔	3.0	2.8	94.4
MJQ_Cal 3	3	✔	5.0	5.0	99.2
MJQ_Cal 4	4	✔	10.0	10.1	100.9
MJQ_Cal 5	5	✔	25.0	24.4	97.7
MJQ_Cal 6	6	✔	50.0	49.8	99.6
MJQ_Cal 7	7	✔	100.0	100.8	100.8

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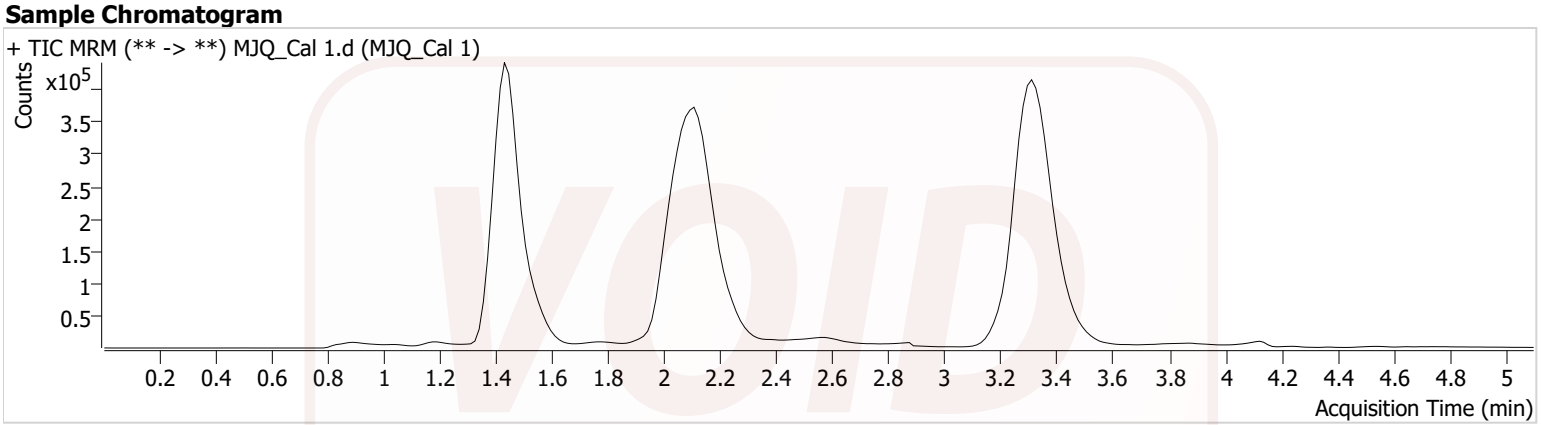
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:01:25 PM		
Sample Info.	Due to retention time shifts, sample was reconstituted and reinjected. See reinject data.		

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.468 **High**	55799	∞	7.4 **Low**	19.87	2301293	1.0376	ng/ml **Low**
THC-COOH	1.474	94390	∞	45.5	156.61	596652	5.9013	ng/ml
THC	3.315	35844	25.43	69.7 **High**	117.49	4052319	1.3742	ng/ml

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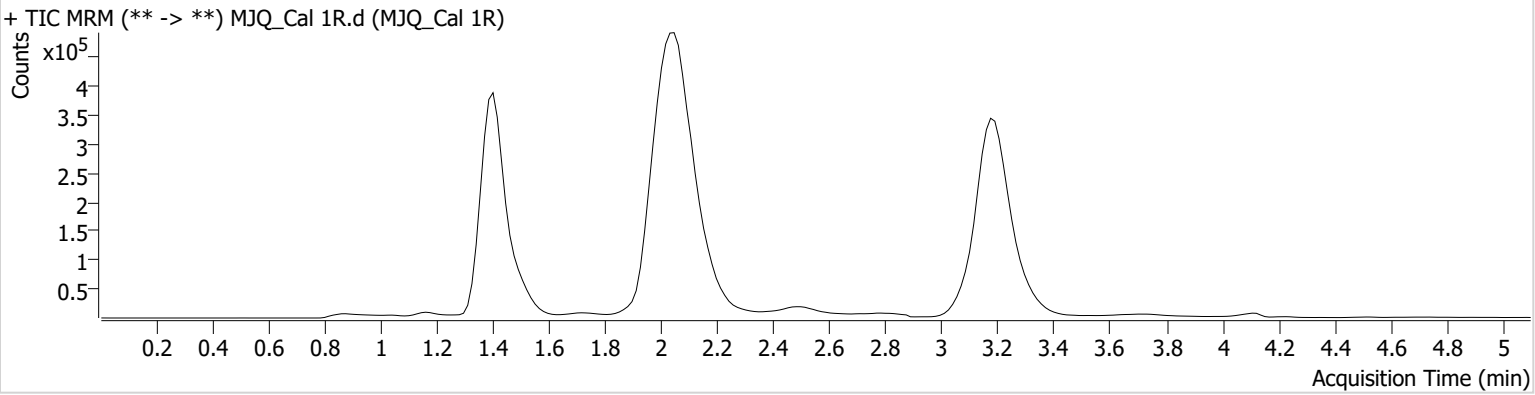
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 1R.d
Type	Cal	**Sample**	MJQ_Cal 1R
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/10/2021 8:39:17 AM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.438	45284	∞	8.3	28.17	1856312	1.0492	ng/ml Low
THC-COOH	1.429	61783	185.97	49.2	337.37	495120	4.4890	ng/ml Low
THC	3.179 Low	28220	10.83	29.1	∞	3061419	1.4152	ng/ml

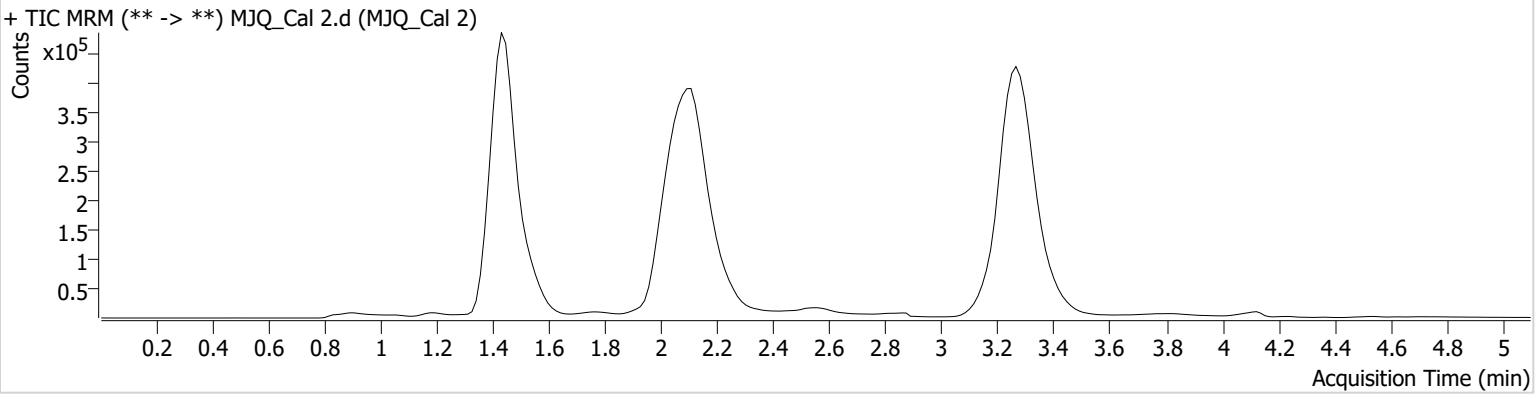
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:09:11 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.453	108920	∞	9.6	78.77	2330087	2.8098	ng/ml Low
THC-COOH	1.474	146946	∞	52.2	655.52	602390	9.5252	ng/ml
THC	3.285	90707	138.22	33.1	46.84	3804802	3.0248	ng/ml

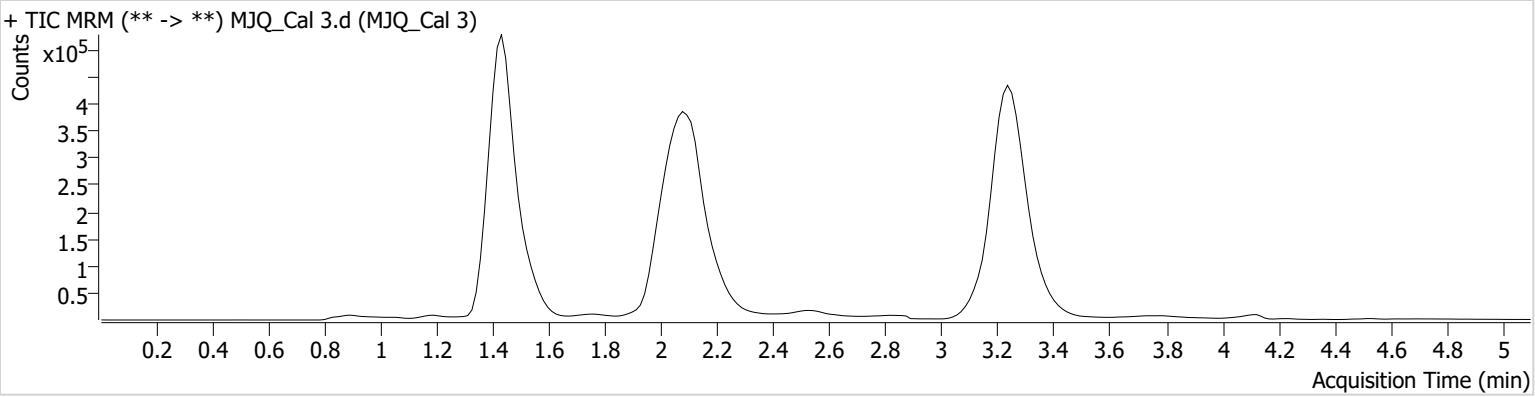
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:16:47 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.438	173221	69.88	10.2	∞	2348201	4.9384	ng/ml
THC-COOH	1.459	291460	∞	56.8	2939.30	601615	19.6913	ng/ml
THC	3.254	161162	260.54	30.1	48.36	3756944	5.1226	ng/ml

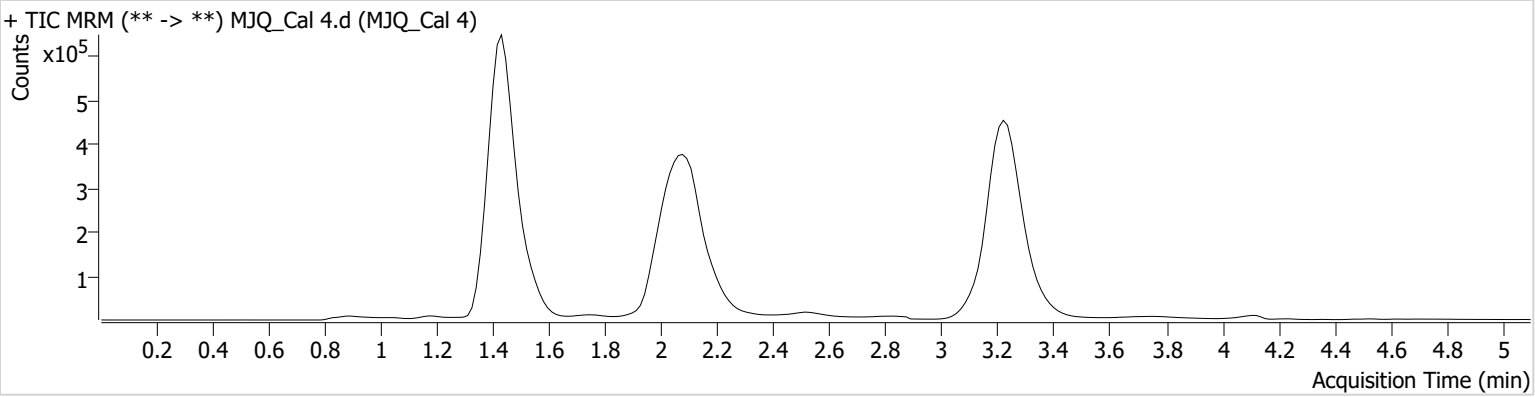
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:24:24 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	330458	∞	10.3	374.66	2379222	10.0685	ng/ml
THC-COOH	1.459	702817	∞	57.8	683.94	594220	49.2056	ng/ml
THC	3.224	324379	∞	32.0	∞	3752124	9.9171	ng/ml

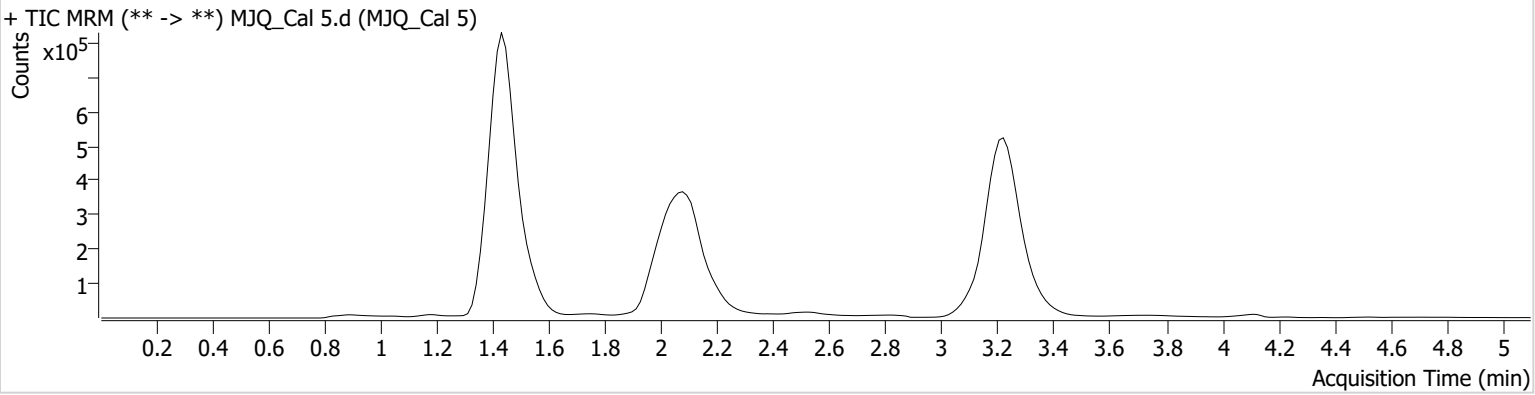
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:32:00 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	798670	∞	11.1	∞	2486918	24.4249	ng/ml
THC-COOH	1.459	1085447	2194.08	58.5	4260.65	608271	74.6383	ng/ml
THC	3.224	814294	∞	28.1	∞	3753490	24.2814	ng/ml

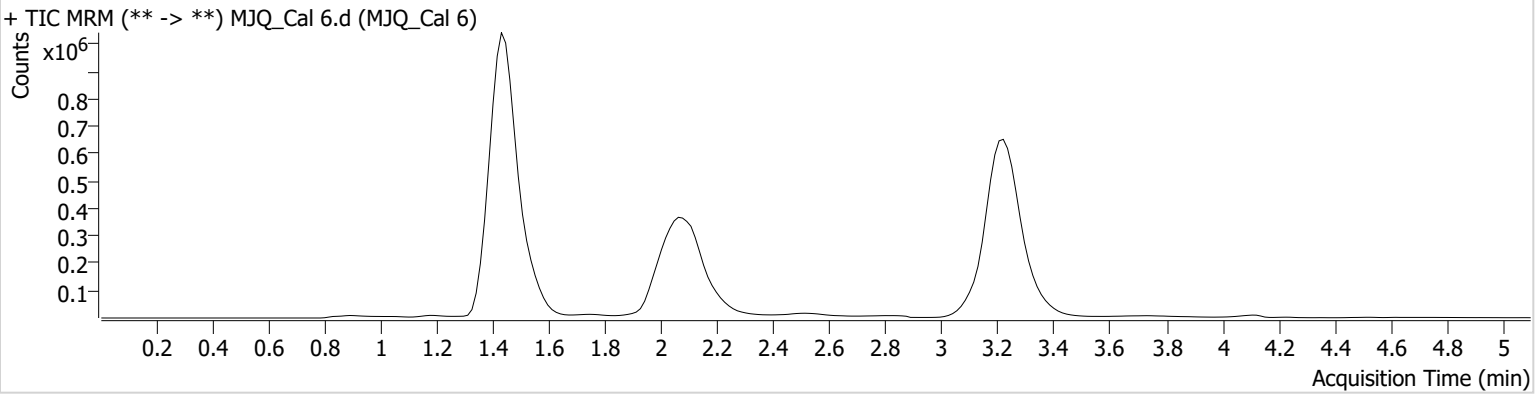
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 AM

Instrument	Instrument 1	**Data File**	MJQ_Cal 6.d
Type	Cal	**Sample**	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:39:35 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	1584917	∞	11.4	∞	2462232	49.8319	ng/ml
THC-COOH	1.459	1438075	2288.72	59.0	∞	603707	99.8965	ng/ml
THC	3.224	1701278	∞	24.6	1584.53	3793197	49.7717	ng/ml

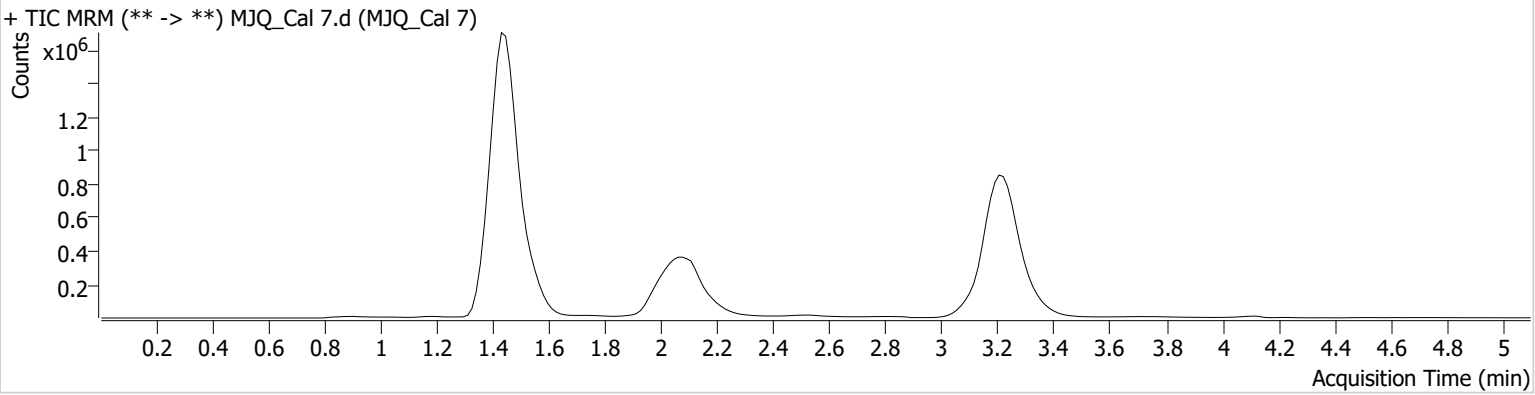
SC

AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\030821 AM 25 26 SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 3/10/2021 8:49:17 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-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/9/2021 3:47:11 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	3084816	∞	11.6	∞	2389046	100.8396	ng/ml
THC-COOH	1.459	3395415	26112.38	58.8	5367.90	568503	251.6529	ng/ml
THC	3.224	3335490	∞	24.5	∞	3654060	100.8824	ng/ml