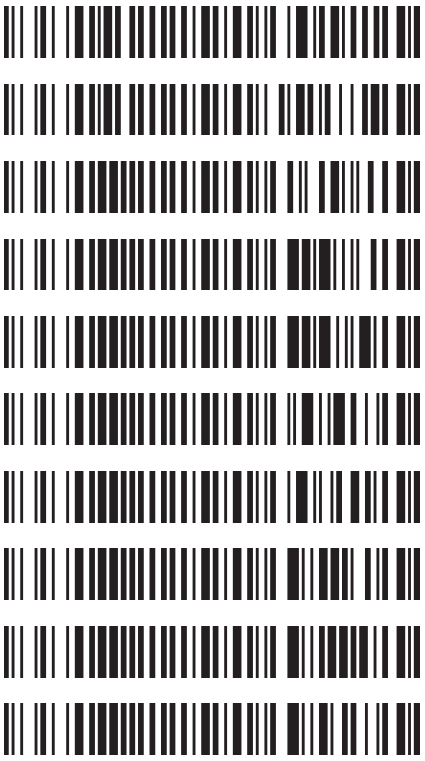
REVIEWED
By Sarah Collins at 11:46 am, Mar 18, 2021

3/15/2021

Worklist: 4842

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION
M2021-0606	2	BCK	AM 27 Blood THC Quant by LC-QQQ
M2021-0957	2	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0567	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0571	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0572	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0587	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0600	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0601	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0603	1	BCK	AM 27 Blood THC Quant by LC-QQQ
P2021-0604	1	BCK	AM 27 Blood THC Quant by LC-QQQ



P2021-0377-1 and P2012-0415-1 were also included in this run ~~(they were previously extracted and needed to be re-extracted)~~.
3/19/21

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

Extraction Date: 03/12/2021
Plate lot#: IDP-108-2-201206
Mobile phase A: 0.1% Formic Acid in LCMS Water
Blank Blood Lot: Lampire 20L20724
LCMS-QQQ ID: 069901

Analyst: Celena Shrum
Plate Expiration: 06/06/2021
Mobile phase B: 0.1% Formic acid in Acetonitrile
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.

Analytic:

☒ 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
☒ 2. Using a calibrated pipette, add **1000µl blood and urine (if applicable) (calibrated pipette)** into the appropriate wells of analytical (standards) plate. **Pipette ID: #42**
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 4. Pipette **500µL 0.1% formic acid in water blood sample** of analytical plate.
☒ 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 6. Transfer **800µL of blood+acid or urine+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)***
☒ 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.
☒ 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.
☒ 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? (if not, describe in comments section)
☒ 6 Enter QCs into control charting.
☒ 7 Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: Curve Limitations: THC 3-100, c-THC 5-250, and THC-OH 3-100. THC quantitative from 3-100 per deviation. M2021-0957-2 was inadvertently left off of the instrument worklist and was not injected with the rest of the samples. This was noticed during data analysis and the sample was injected (03/15/2021).

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, 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
Toxicology Discipline Lead
Date: 02/24/2021

	1	2	3	4	5	6
A				P2021-0604-1	M2021-0957-2	IS + QC_1
B				P2021-0603-1	M2021-0606-2	IS + Cal. 7
C				P2021-0601-1	Blood NC	IS + Cal. 6
D				P2021-0600-1	P2021-0415-1	IS + Cal. 5
E				P2021-0587-1	P2021-0377-1	IS + Cal. 4
F				P2021-0572-1	Not Used	IS + Cal. 3
G				P2021-0571-1	Not Used	IS + Cal. 2
H				P2021-0567-1	Not Used	IS + Cal. 1

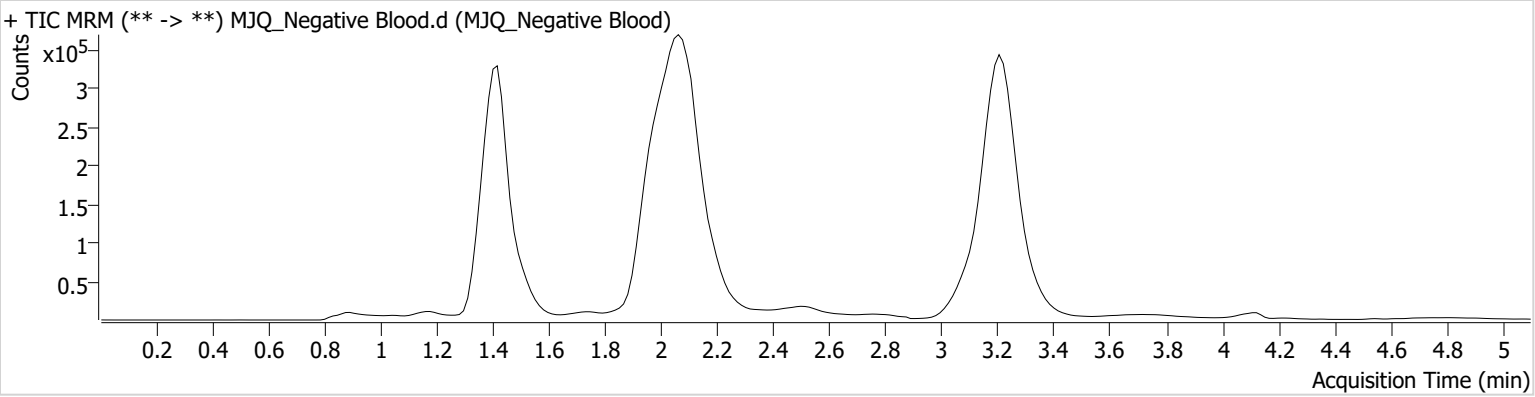
All wells to contain 100 µl of residual DMSO

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Negative Blood.d
Type	Sample	**Sample**	MJQ_Negative Blood
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-C5	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 2:35:36 PM		
Sample Info.			

Sample Chromatogram

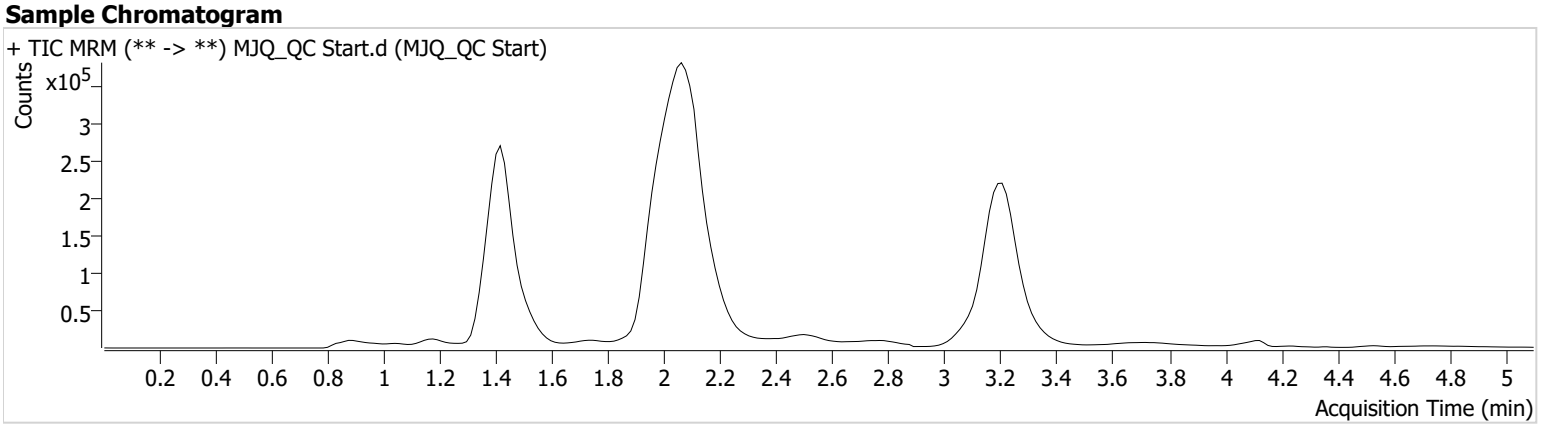


AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_QC Start.d
Type	Sample	**Sample**	MJQ_QC Start
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 2:50:50 PM		
Sample Info.			

Sample Chromatogram



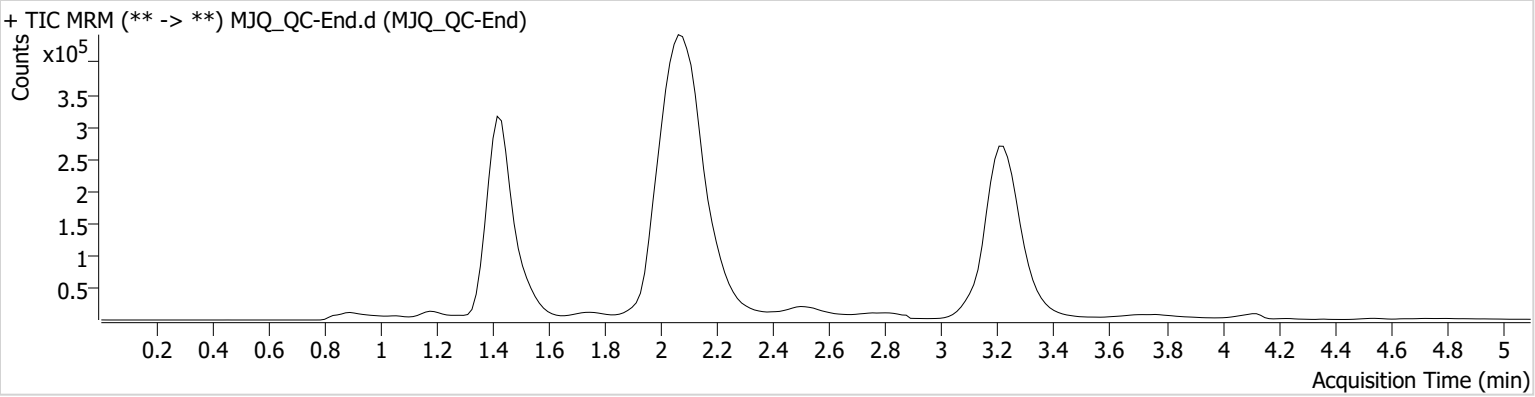
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.444	130365	∞	50.2	∞	359862	14.1191	ng/ml
THC-OH	1.423	90968	∞	9.9	51.23	1177610	4.6833	ng/ml
THC	3.209	67590	112.19	35.8	53.01	1959367	4.3156	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_QC-End.d
Type	Sample	**Sample**	MJQ_QC-End
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-A6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 5:54:05 PM		
Sample Info.			

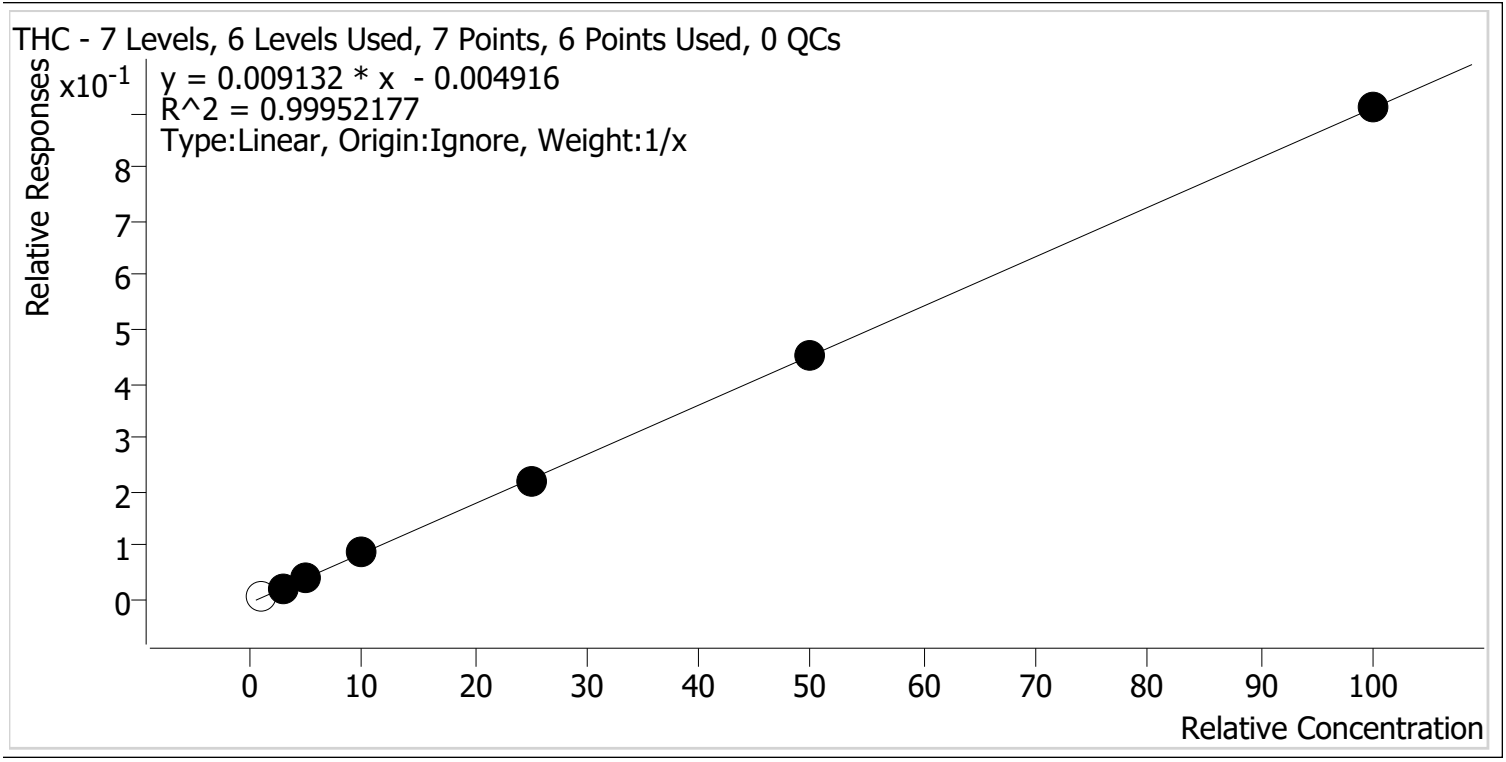
Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	146175	802.55	49.8	693.25	406543	14.0090	ng/ml
THC-OH	1.438	107964	∞	9.3	134.56	1320448	4.9997	ng/ml
THC	3.224	83469	178.20	34.9	23.97	2343893	4.4377	ng/ml

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update	3/17/2021 2:39 PM
Analyst Name	ISP\Datastor
Analyte	THC
Internal Standard	THC-D3

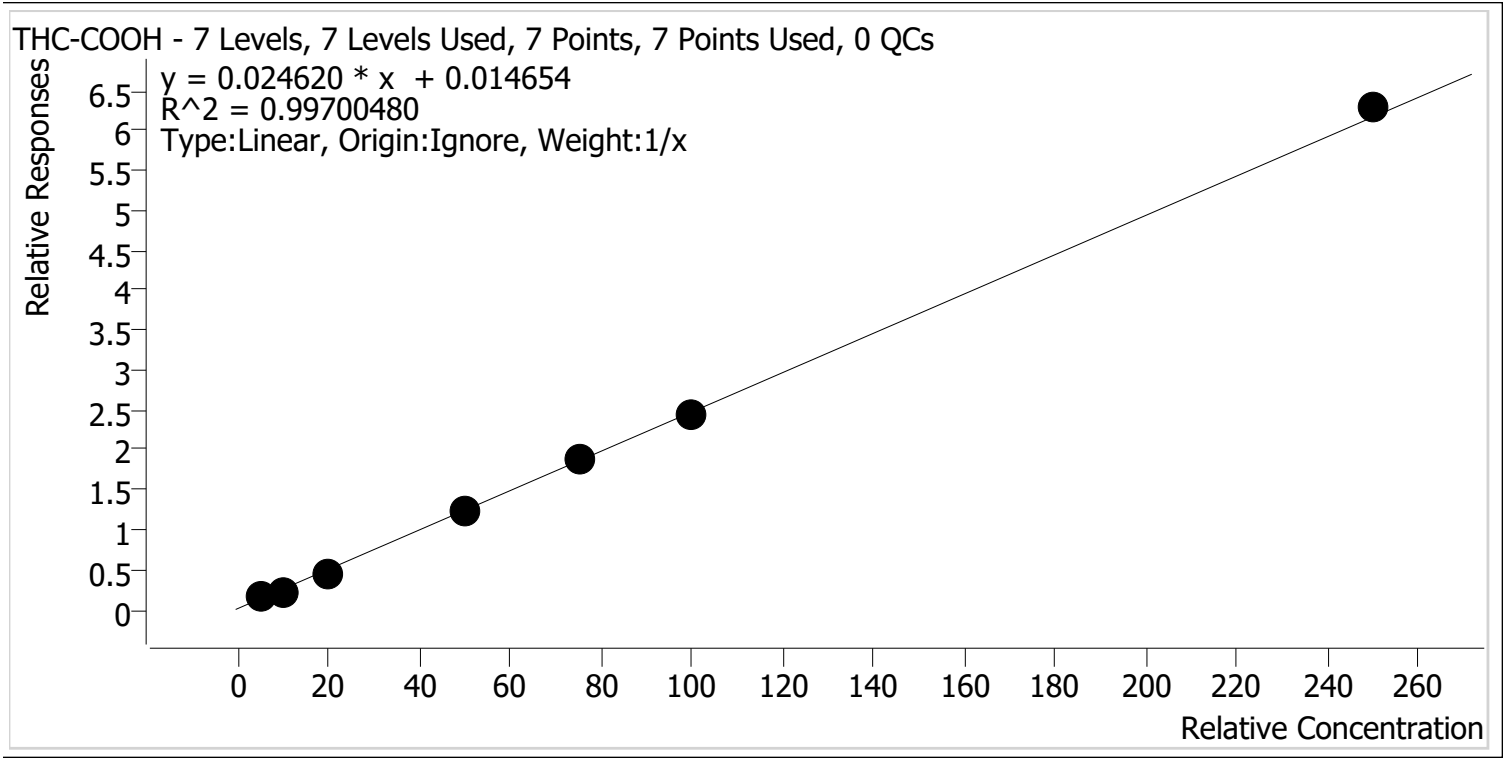


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✗	1.0	1.4	144.8
MJQ_Cal 2	2	✔	3.0	3.0	100.1
MJQ_Cal 3	3	✔	5.0	4.8	95.4
MJQ_Cal 4	4	✔	10.0	10.7	106.6
MJQ_Cal 5	5	✔	25.0	24.4	97.7
MJQ_Cal 6	6	✔	50.0	50.0	100.1
MJQ_Cal 7	7	✔	100.0	100.1	100.1

Calibrator 1 dropped due to not meeting accuracy requirement.

AM #27 Cannabinoids Quant. Calibration Curve Report

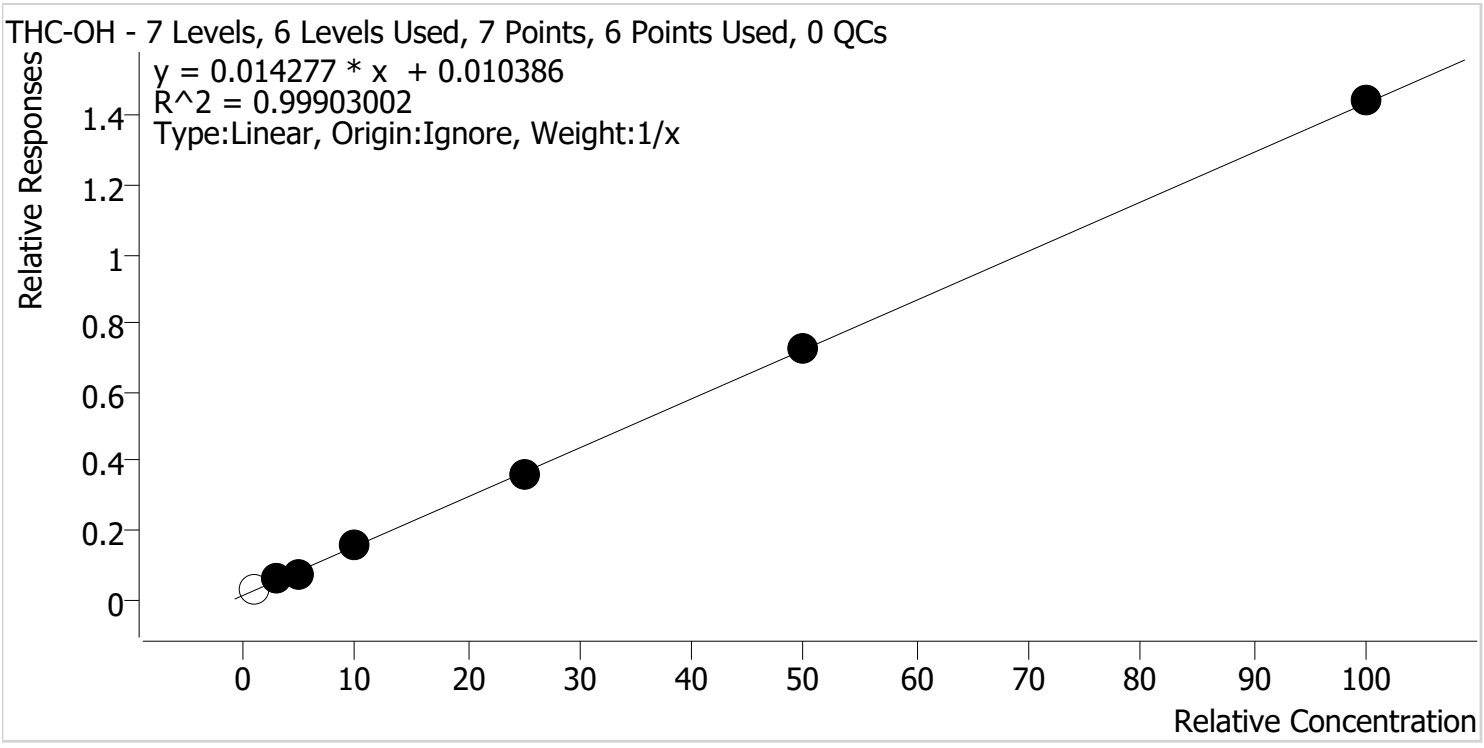
Batch results	G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update	3/17/2021 2:39 PM
Analyst Name	ISP\Datastor
Analyte	THC-COOH
Internal Standard	THC-COOH-D9



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJQ_Cal 1	1	✔	5.0	6.4	128.2
MJQ_Cal 2	2	✔	10.0	8.8	87.9
MJQ_Cal 3	3	✔	20.0	16.9	84.3
MJQ_Cal 4	4	✔	50.0	49.3	98.6
MJQ_Cal 5	5	✔	75.0	75.4	100.6
MJQ_Cal 6	6	✔	100.0	98.5	98.5
MJQ_Cal 7	7	✔	250.0	254.7	101.9

AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Last Cal. Update	3/17/2021 2:39 PM
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	1.4	138.8
MJQ_Cal 2	2	✔	3.0	3.3	111.4
MJQ_Cal 3	3	✔	5.0	4.4	87.4
MJQ_Cal 4	4	✔	10.0	10.3	102.7
MJQ_Cal 5	5	✔	25.0	24.5	98.0
MJQ_Cal 6	6	✔	50.0	50.0	100.0
MJQ_Cal 7	7	✔	100.0	100.5	100.5

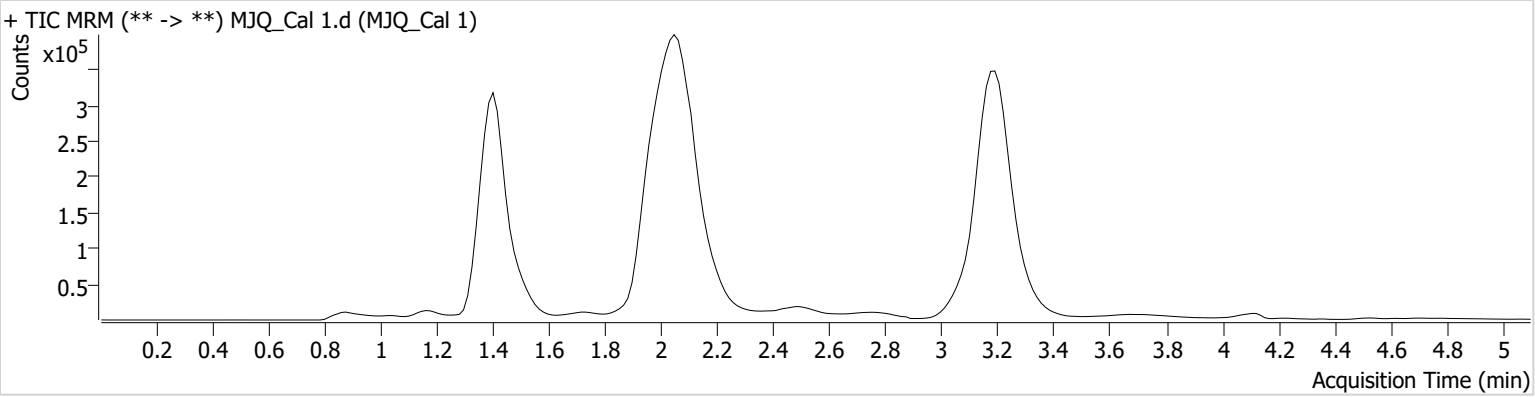
Calibrator 1 dropped due to not meeting accuracy requirement.

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 1.d
Type	Cal	**Sample**	MJQ_Cal 1
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-H6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 1:34:38 PM		
Sample Info.			

Sample Chromatogram



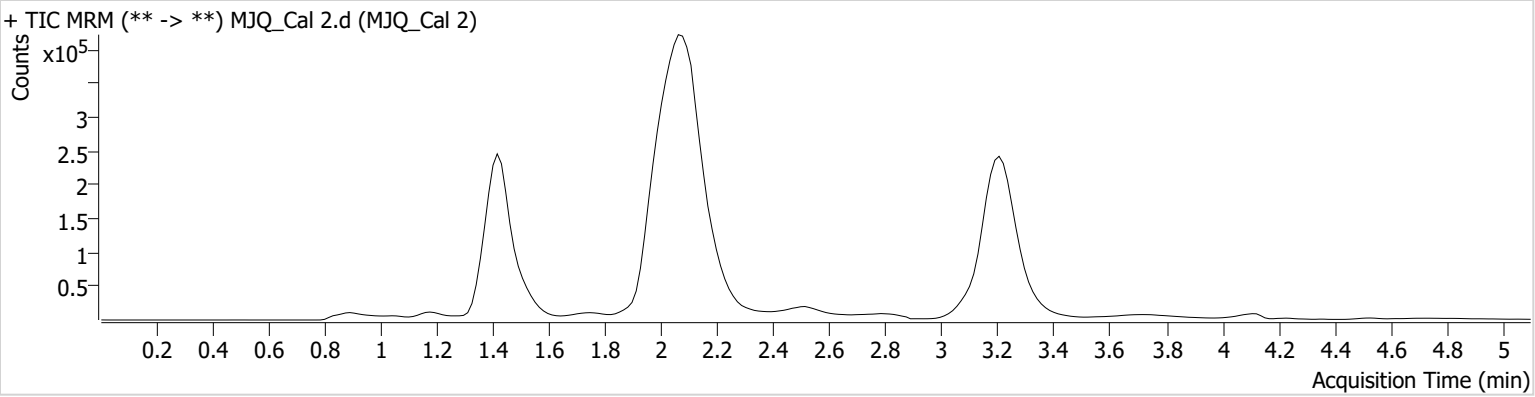
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.429	75467	∞	42.1	∞	437477	6.4115	ng/ml
THC-OH	1.438	47448	∞	7.6 **Low**	25.36	1571062	1.3880	ng/ml **Low**
THC	3.194	26793	79.70	80.1 **High**	18.75	3223258	1.4485	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 2.d
Type	Cal	**Sample**	MJQ_Cal 2
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-G6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 1:42:24 PM		
Sample Info.			

Sample Chromatogram



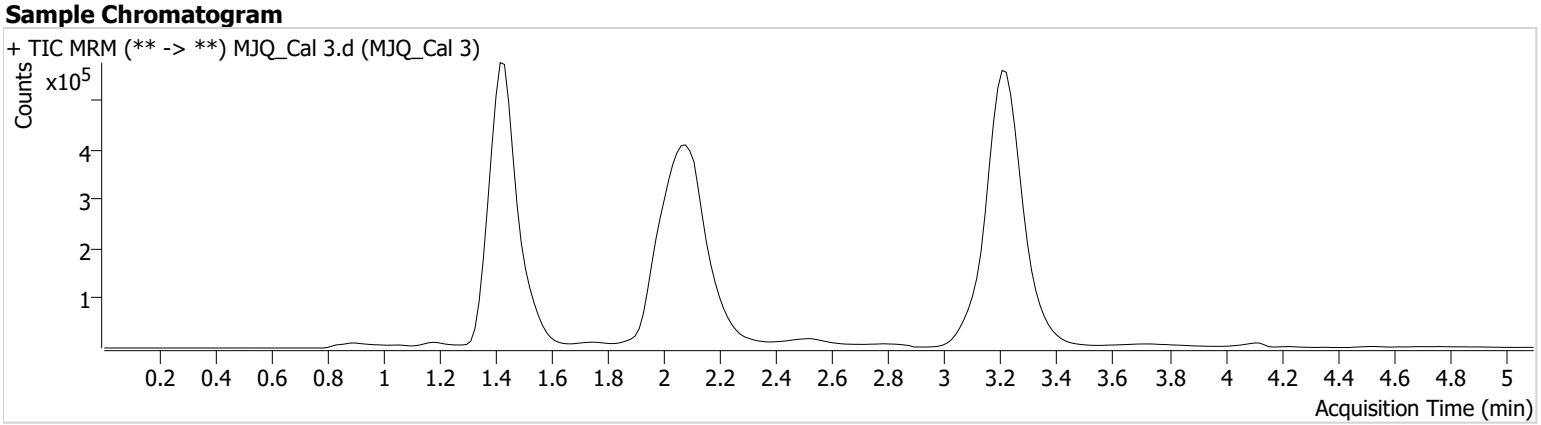
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.444	73939	157.82	47.1	874.68	319954	8.7912	ng/ml
THC-OH	1.438	65827	∞	9.0	∞	1133420	3.3406	ng/ml
THC	3.209	47782	∞	34.1	∞	2123252	3.0025	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results	G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update	3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 3.d
Type	Cal	**Sample**	MJQ_Cal 3
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-F6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 1:50:00 PM		
Sample Info.			

Sample Chromatogram



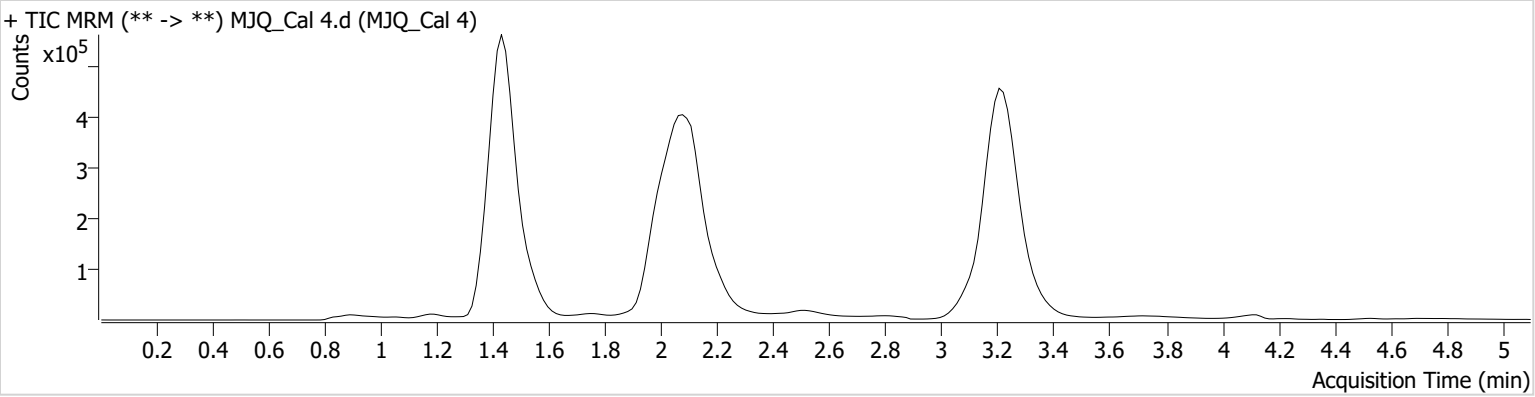
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	297849	1254.28	53.8	∞	692849	16.8658	ng/ml
THC-OH	1.423	188903	∞	10.4	297.67	2595126	4.3712	ng/ml
THC	3.224	190881	∞	35.7	∞	4937868	4.7712	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 4.d
Type	Cal	**Sample**	MJQ_Cal 4
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-E6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 1:57:36 PM		
Sample Info.			

Sample Chromatogram



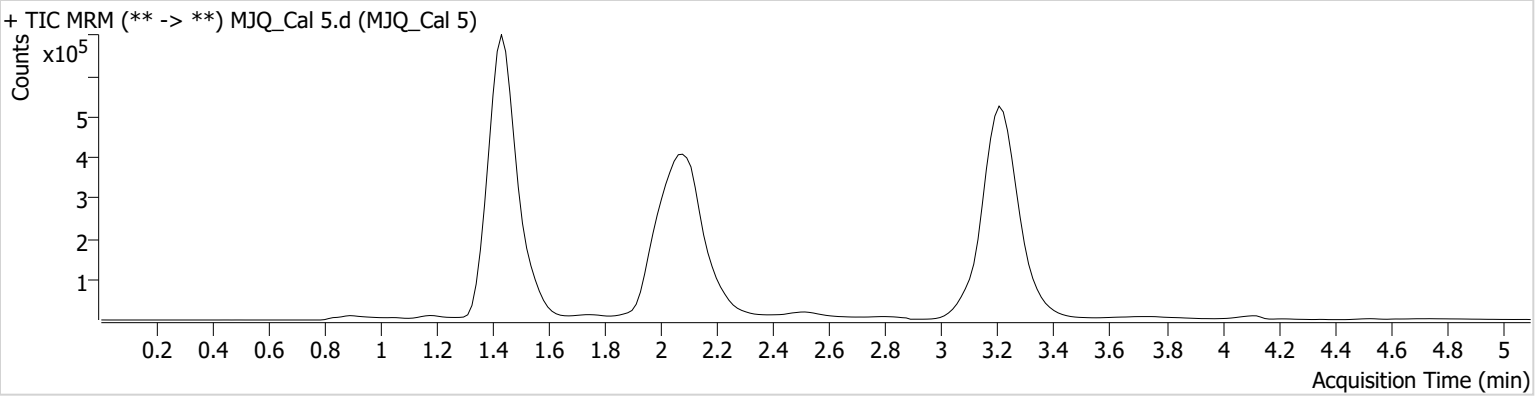
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	643873	∞	53.8	1886.80	524054	49.3089	ng/ml
THC-OH	1.423	305703	∞	10.6	783.17	1946957	10.2707	ng/ml
THC	3.224	347769	1340.78	25.2	94.53	3761564	10.6620	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 5.d
Type	Cal	**Sample**	MJQ_Cal 5
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-D6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 2:05:12 PM		
Sample Info.			

Sample Chromatogram



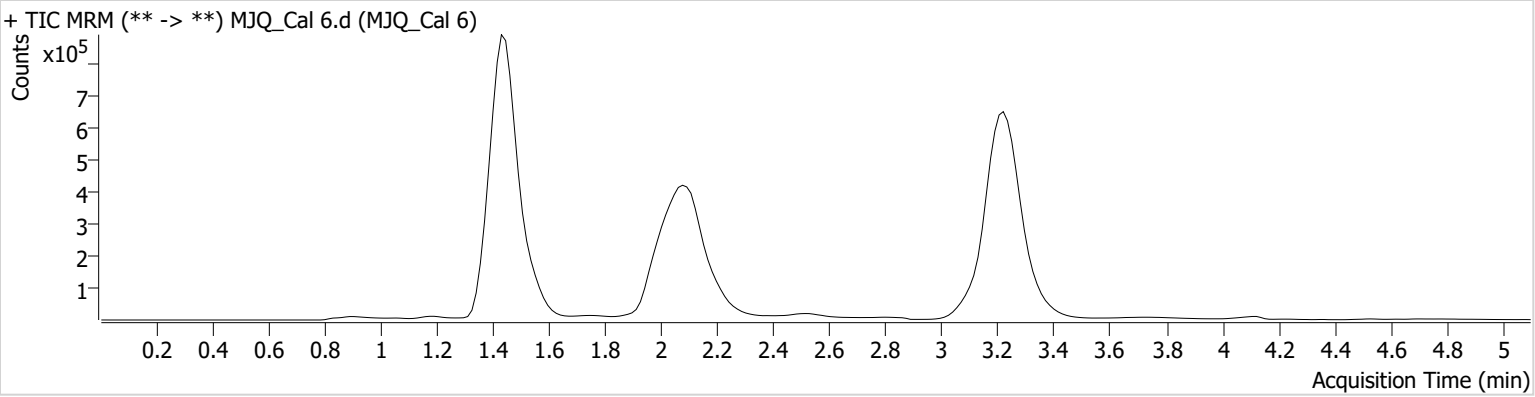
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	969960	∞	54.6	∞	518307	75.4162	ng/ml
THC-OH	1.423	710002	∞	11.2	∞	1970945	24.5052	ng/ml
THC	3.224	822487	∞	29.4	∞	3771087	24.4208	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 6.d
Type	Cal	**Sample**	MJQ_Cal 6
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-C6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 2:12:47 PM		
Sample Info.			

Sample Chromatogram



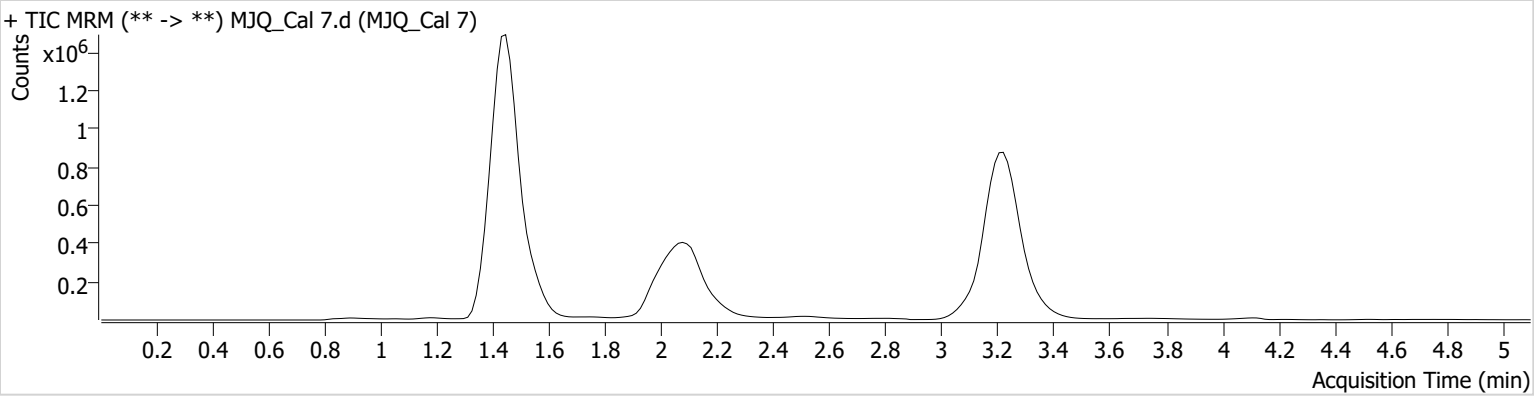
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	1299922	∞	55.0	2894.55	533049	98.4565	ng/ml
THC-OH	1.438	1441278	∞	11.4	830.03	1990840	49.9820	ng/ml
THC	3.224	1721684	∞	28.5	∞	3808022	50.0458	ng/ml

AM #27 Cannabinoid Quant. Results

Batch results G:\TOX\Pocatello\Falco\2021\AM 27-28\031221 AM 27 28 CS\QuantResults\AM 27 THCQ.batch.bin
Calibration Last Update 3/17/2021 2:39:07 PM

Instrument	Instrument 1	**Data File**	MJQ_Cal 7.d
Type	Cal	**Sample**	MJQ_Cal 7
Acq. Method	AM 27 THCQ.m	**Operator**	Celena Shrum
Sample Position	P1-B6	**Comment**	
Injection Volume	10		
Acq. Date-Time	3/12/2021 2:20:23 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-COOH	1.459	3069720	∞	54.2	∞	488297	254.7498	ng/ml
THC-OH	1.423	2774968	∞	11.6	1756.55	1919583	100.5303	ng/ml
THC	3.224	3416200	∞	28.6	∞	3757322	100.0977	ng/ml