REVIEWED
By Anne Nord at 9:23 am, Mar 02, 2021

&C 2/24/2021

Worklist: 4802

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
M2021-0182	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-0404	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
M2021-0434	3	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0095	2	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0101	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0245	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0278	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0279	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0282	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0288	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0290	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0294	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0315	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0317	1	BCK	AM 27 Blood THC Quant by LC-QQQ	
P2021-0325	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: 02/23/21 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: 16**
☒ 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 6 due to low internal standard response in initial injection. Reinject data used.*

Calibrator 1 dropped for THC due to high ratio

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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

8C

	1	2	3	4	5	6
A	IS + Cal. 1	negative	p2021-0279-1			IS + QC_1
B	IS + Cal. 2	m2021-0182-1	p2021-0282-1			IS + Cal. 7
C	IS + Cal. 3	m2021-0404-2	p2021-0288-1			IS + Cal. 6
D	IS + Cal. 4	m2021-0434-3	p2021-0290-1			IS + Cal. 5
E	IS + Cal. 5	p2021-0095-2	p2021-0294-1			IS + Cal. 4
F	IS + Cal. 6	p2021-0101-1	p2021-0315-1			IS + Cal. 3
G	IS + Cal. 7	p2021-0245-1	p2021-0317-1			IS + Cal. 2
H	IS + QC_1	p2021-0278-1	p2021-0325-1			IS + Cal. 1

All wells to contain 100 µl of residual DMSO

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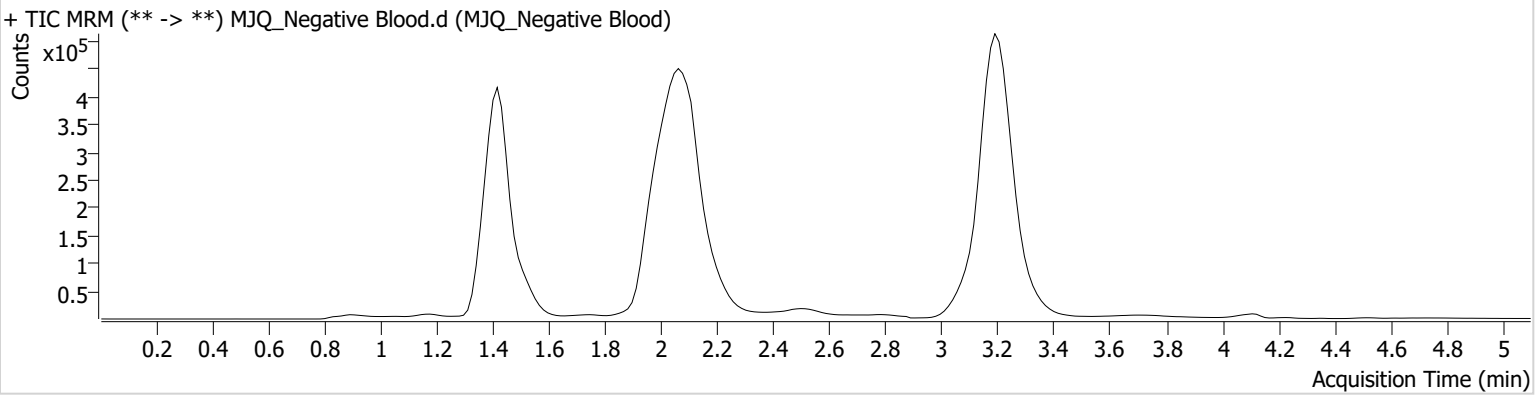
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	2/24/2021 7:48:33 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	2/23/2021 7:11:17 PM		
Sample Info.			

Sample Chromatogram



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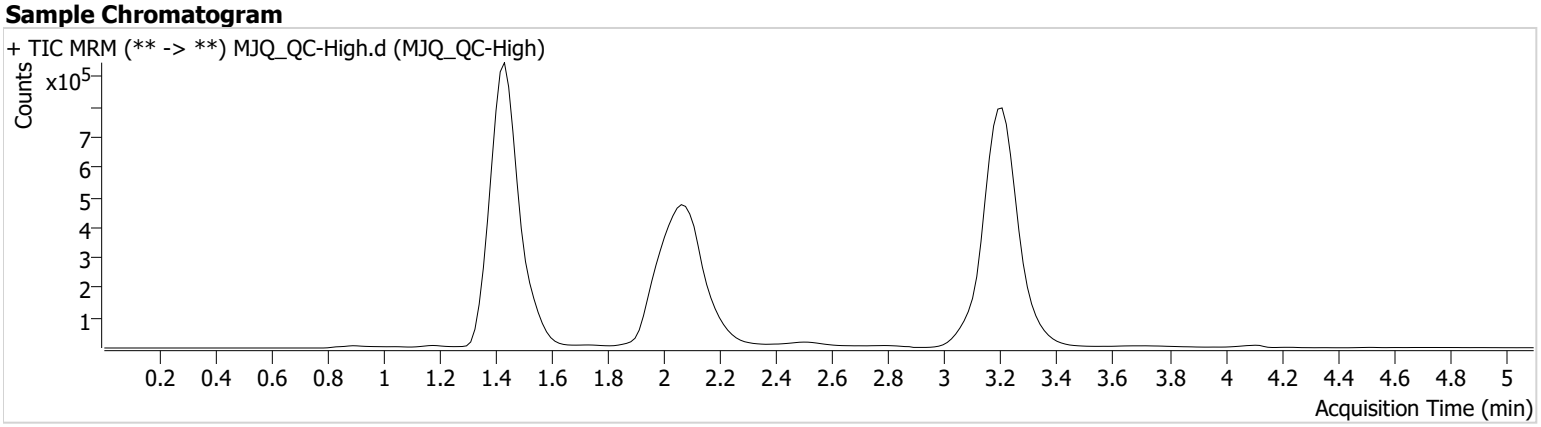
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 AM

Instrument	Instrument 1	**Data File**	MJQ_QC-High.d
Type	Sample	**Sample**	MJQ_QC-High
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/23/2021 7:26:27 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	1419720	∞	11.6	∞	2188384	49.8632	ng/ml
THC-COOH	1.444	1358348	∞	59.1	5778.93	577231	99.7047	ng/ml
THC	3.209	2035183	6308.31	24.6	1312.32	4425433	50.0946	ng/ml

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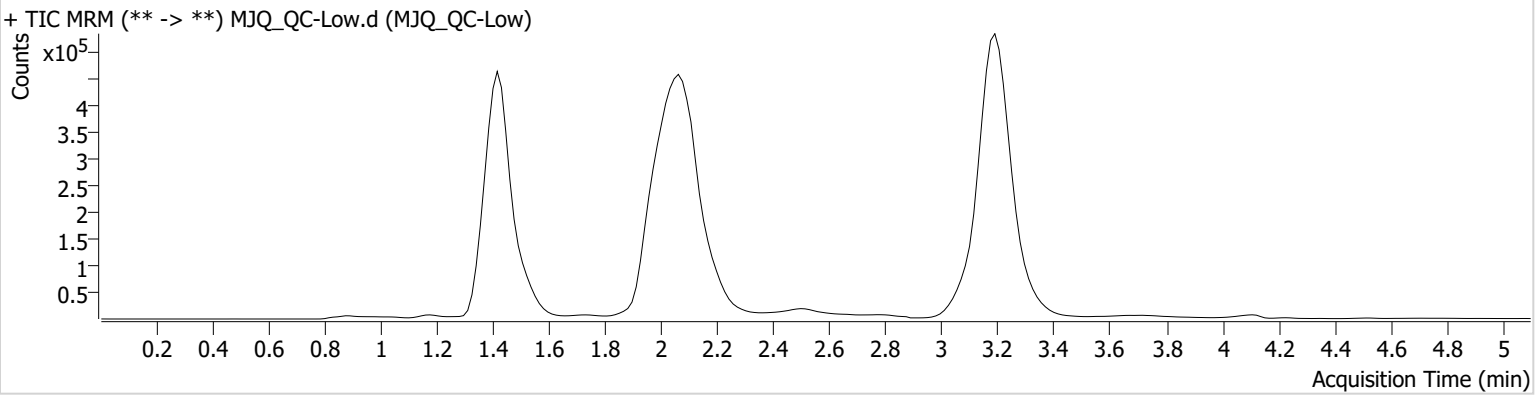
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 AM

Instrument	Instrument 1	**Data File**	MJQ_QC-Low.d
Type	Sample	**Sample**	MJQ_QC-Low
Acq. Method	AM 27 THCQ.m	**Operator**	Sarah Collins
Sample Position	P1-H1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/23/2021 11:37:11 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	120409	57.40	11.3	∞	2096918	3.8769	ng/ml
THC-COOH	1.444	195750	∞	55.4	∞	576911	14.0025	ng/ml
THC	3.194	149846	296.96	30.4	53.33	4375916	3.9949	ng/ml

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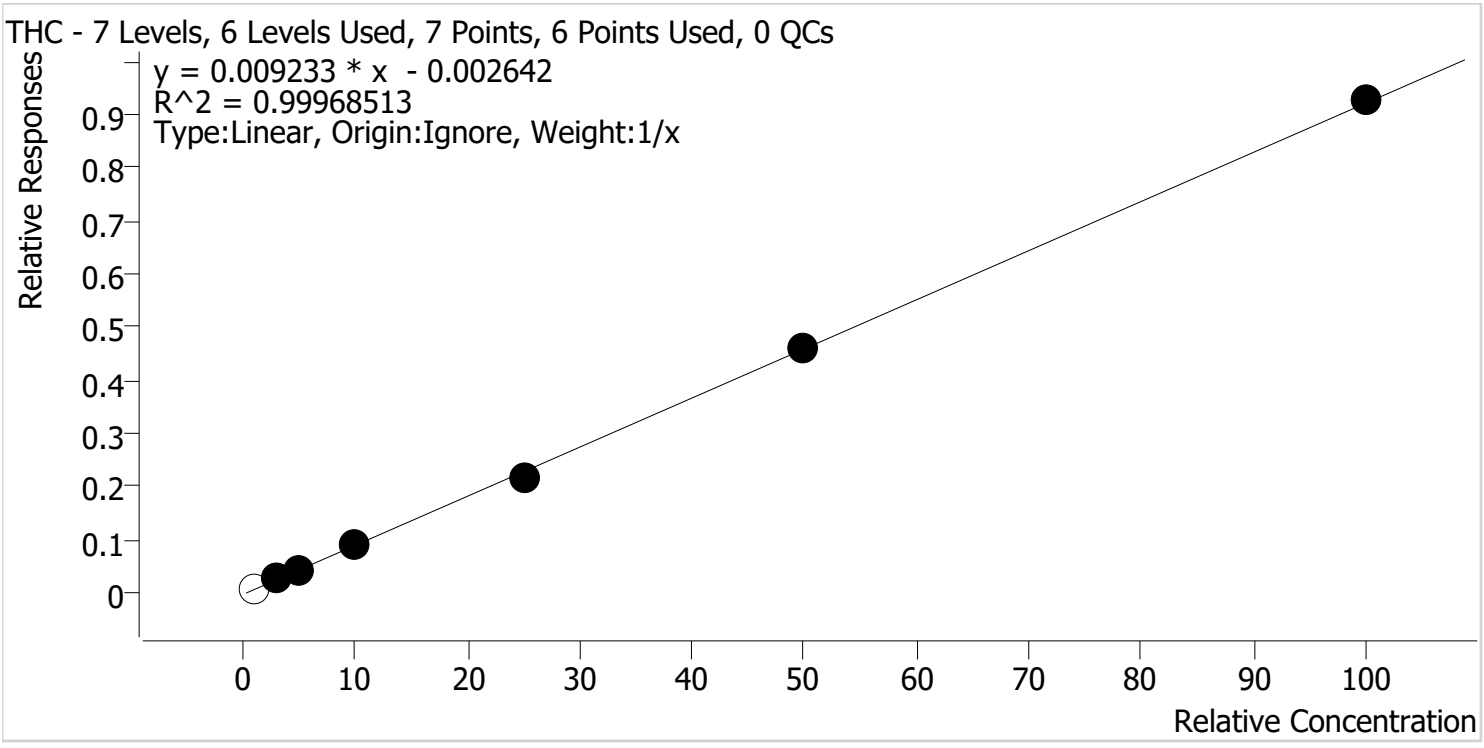
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Last Cal. Update	2/24/2021 7:48 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	1.3	125.9
MJQ_Cal 2	2	✔	3.0	3.0	101.4
MJQ_Cal 3	3	✔	5.0	5.1	102.7
MJQ_Cal 4	4	✔	10.0	9.8	98.3
MJQ_Cal 5	5	✔	25.0	24.1	96.4
MJQ_Cal 6R	6	✔	50.0	50.3	100.7
MJQ_Cal 7	7	✔	100.0	100.6	100.6

Cal 1 dropped due to not meeting ratio requirement

Generated at 8:06 AM on 3/2/2021

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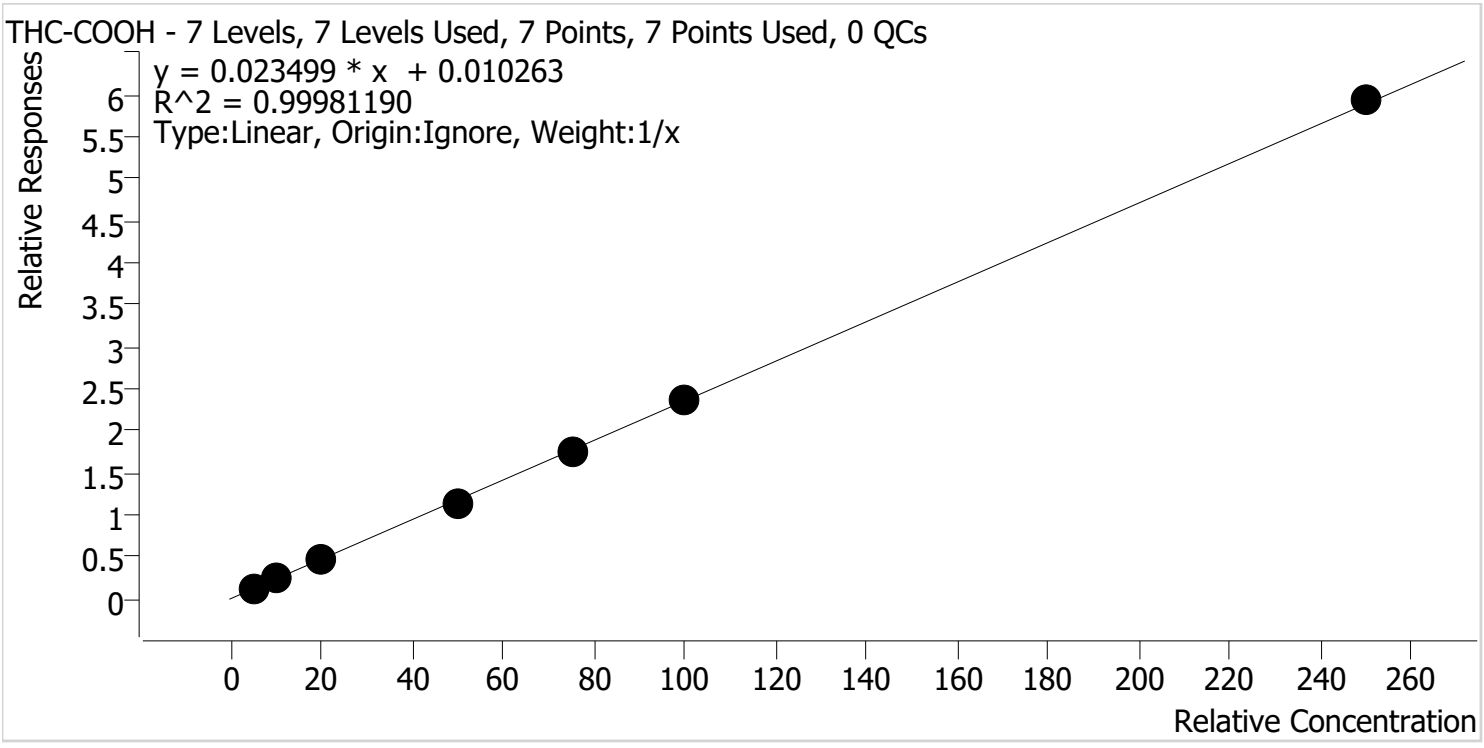
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Last Cal. Update	2/24/2021 7:48 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	5.1	101.2
MJQ_Cal 2	2	✔	10.0	10.0	100.0
MJQ_Cal 3	3	✔	20.0	20.4	102.0
MJQ_Cal 4	4	✔	50.0	48.7	97.4
MJQ_Cal 5	5	✔	75.0	73.9	98.5
MJQ_Cal 6R	6	✔	100.0	100.1	100.1
MJQ_Cal 7	7	✔	250.0	251.9	100.7

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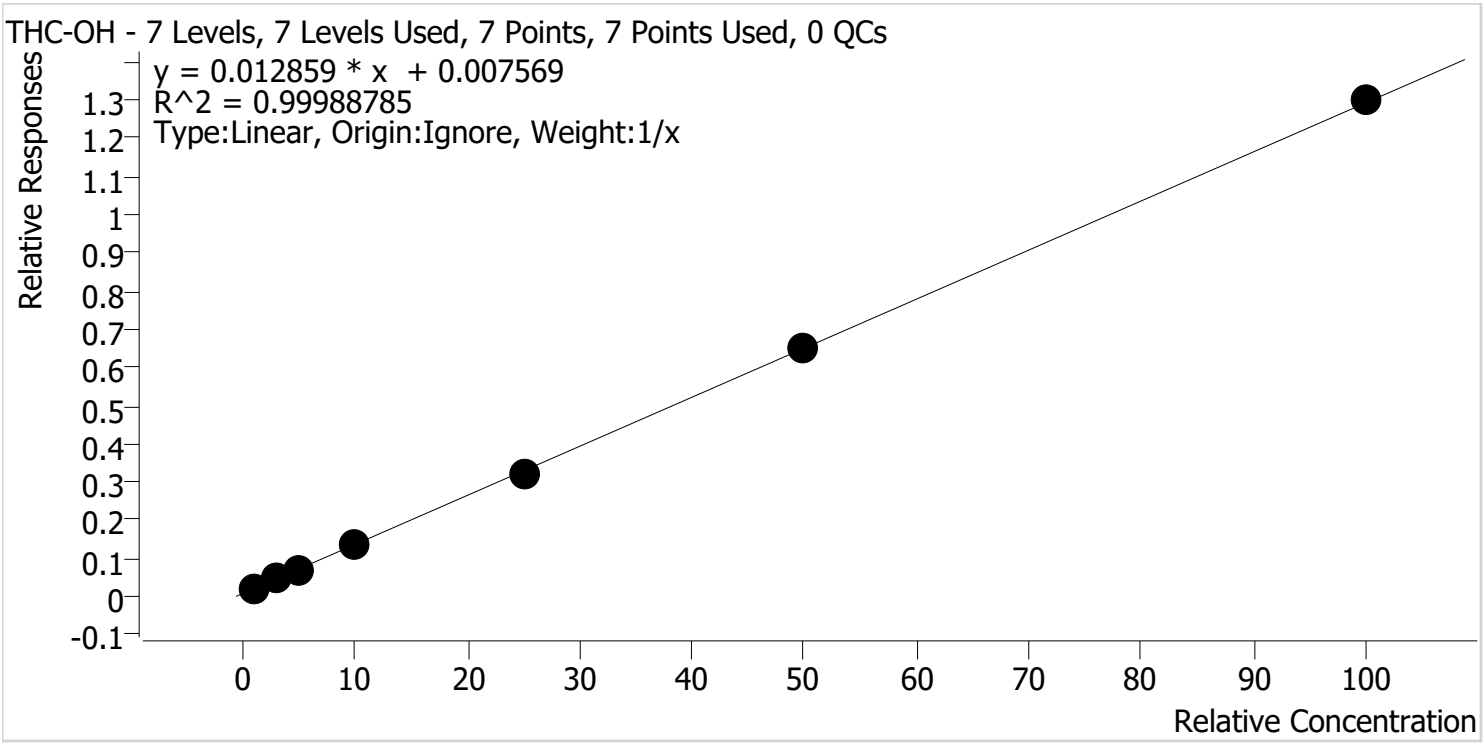
AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin

Last Cal. Update 2/24/2021 7:48 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	1.0	104.2
MJQ_Cal 2	2	✔	3.0	2.9	97.1
MJQ_Cal 3	3	✔	5.0	5.0	100.2
MJQ_Cal 4	4	✔	10.0	10.0	100.1
MJQ_Cal 5	5	✔	25.0	24.5	97.8
MJQ_Cal 6R	6	✔	50.0	50.1	100.2
MJQ_Cal 7	7	✔	100.0	100.5	100.5

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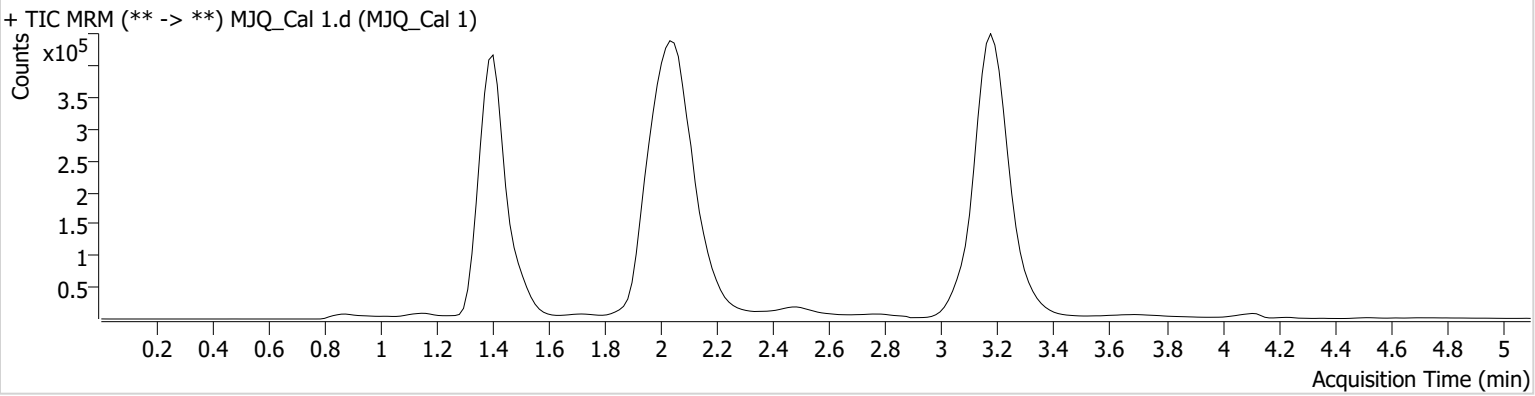
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 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	2/23/2021 6:10:22 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	44018	∞	8.8	∞	2099842	1.0416	ng/ml **Low**
THC-COOH	1.429	72222	∞	46.3	∞	559021	5.0611	ng/ml
THC	3.179	35708	15.19	65.7 **High**	∞	3975176	1.2590	ng/ml

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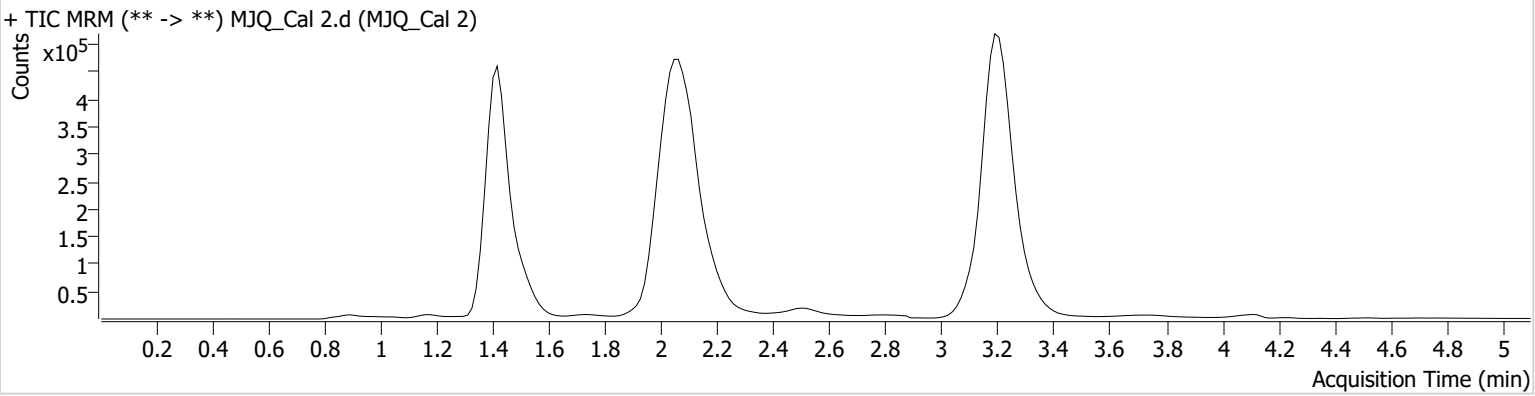
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 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	2/23/2021 6:18:09 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	90282	∞	10.0	∞	2005478	2.9123	ng/ml Low
THC-COOH	1.444	134118	∞	51.1	243.55	546835	10.0005	ng/ml
THC	3.209	104549	265.49	30.8	∞	4109011	3.0418	ng/ml

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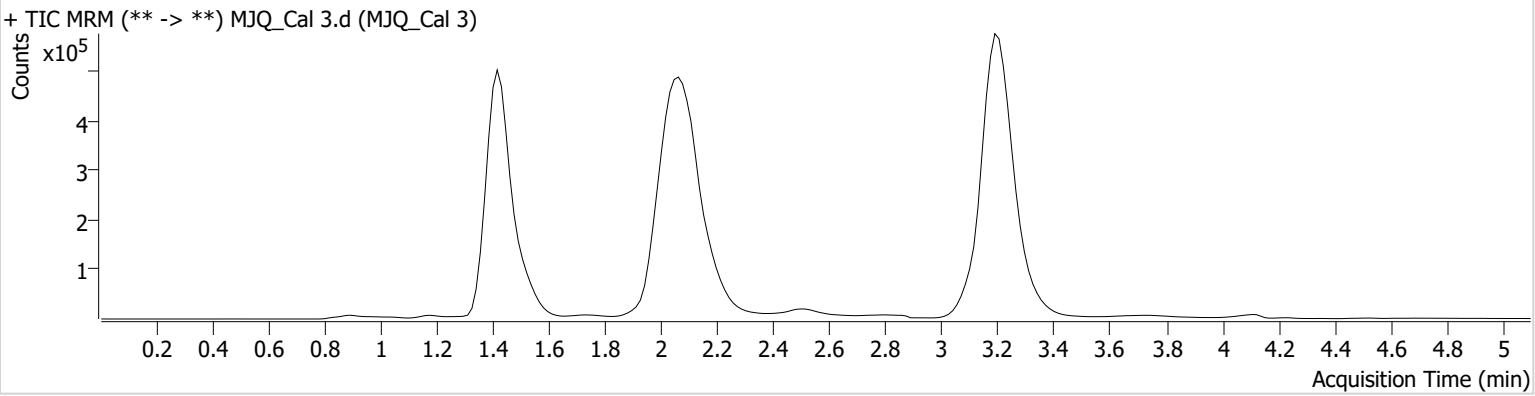
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 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	2/23/2021 6:25:44 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	149361	∞	11.1	∞	2074835	5.0096	ng/ml
THC-COOH	1.444	274203	∞	55.5	1510.04	559921	20.4033	ng/ml
THC	3.209	200139	1955.98	29.6	75.38	4471712	5.1336	ng/ml

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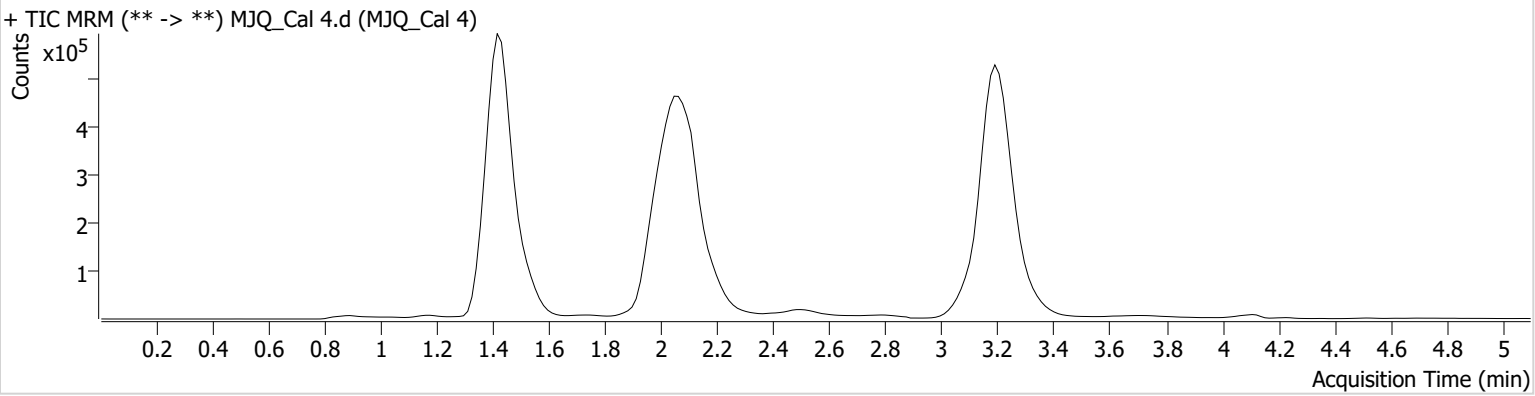
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	2/24/2021 7:48:33 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	2/23/2021 6:33:20 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	287465	853.85	11.1	672.52	2109451	10.0091	ng/ml
THC-COOH	1.444	649709	∞	58.7	3717.82	562401	48.7248	ng/ml
THC	3.209	359272	7132.51	25.5	336.67	4076775	9.8308	ng/ml

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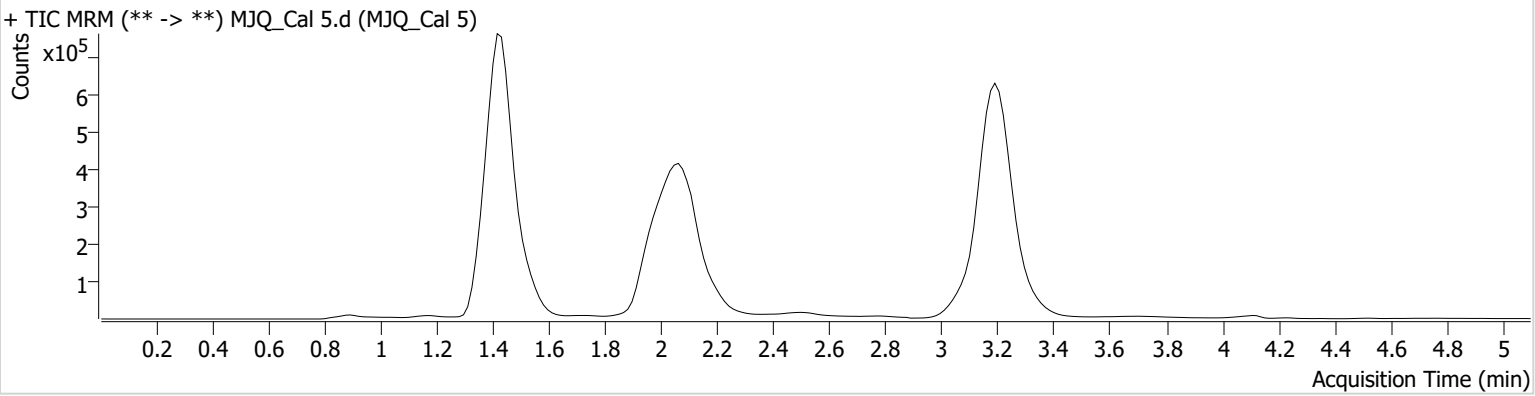
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	2/24/2021 7:48:33 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	2/23/2021 6:40:55 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	737192	∞	11.3	∞	2288836	24.4588	ng/ml
THC-COOH	1.444	1021053	997.27	59.1	∞	584810	73.8628	ng/ml
THC	3.194	967723	740.34	24.6	409.28	4401032	24.1012	ng/ml

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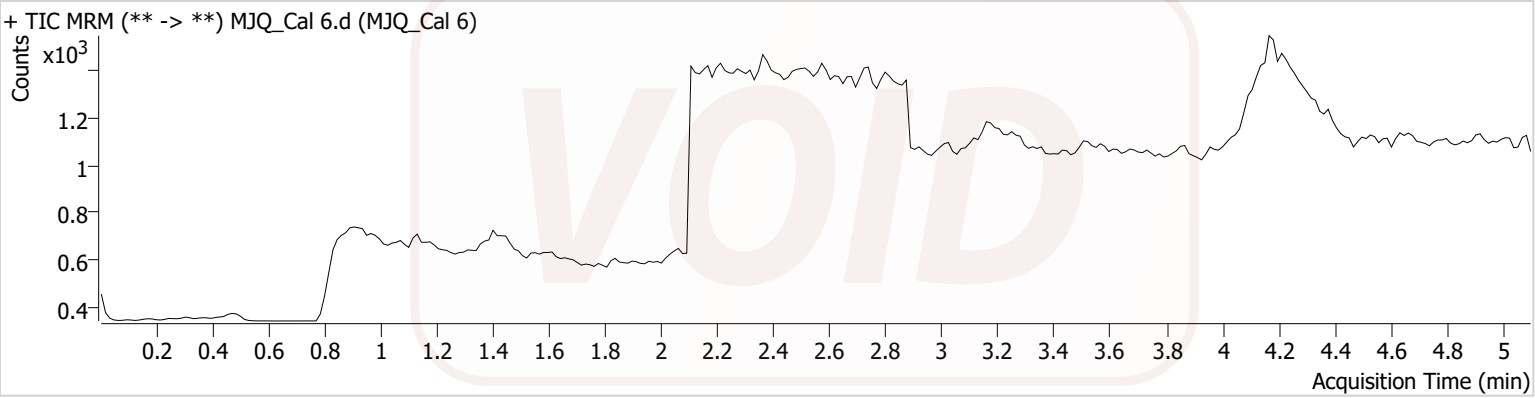
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	2/24/2021 7:48:33 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-F1	**Comment**	
Injection Volume	10		
Acq. Date-Time	2/23/2021 6:48:31 PM		
Sample Info.			

Sample Chromatogram



*Sample reconstituted and reinjected due to low internal standard response. See reinject data.

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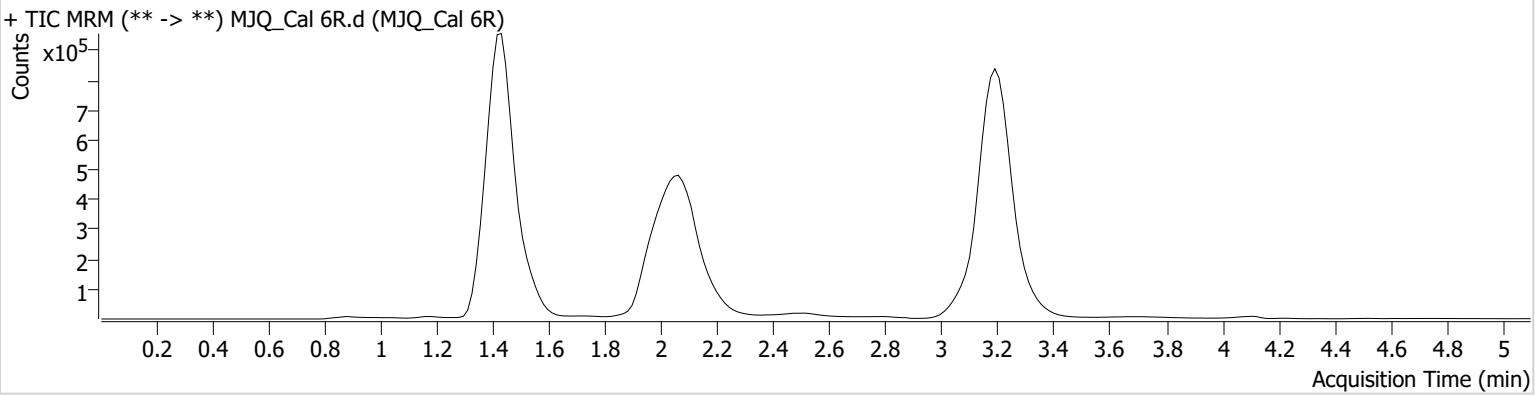
AM #27 Cannabinoid Quant. Results

Batch results	D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update	2/24/2021 7:48:33 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	2/23/2021 7:34:03 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	1468323	∞	11.6	∞	2253508	50.0825	ng/ml
THC-COOH	1.444	1390059	12793.43	58.7	∞	588539	100.0734	ng/ml
THC	3.194	2115972	∞	24.5	∞	4579575	50.3288	ng/ml

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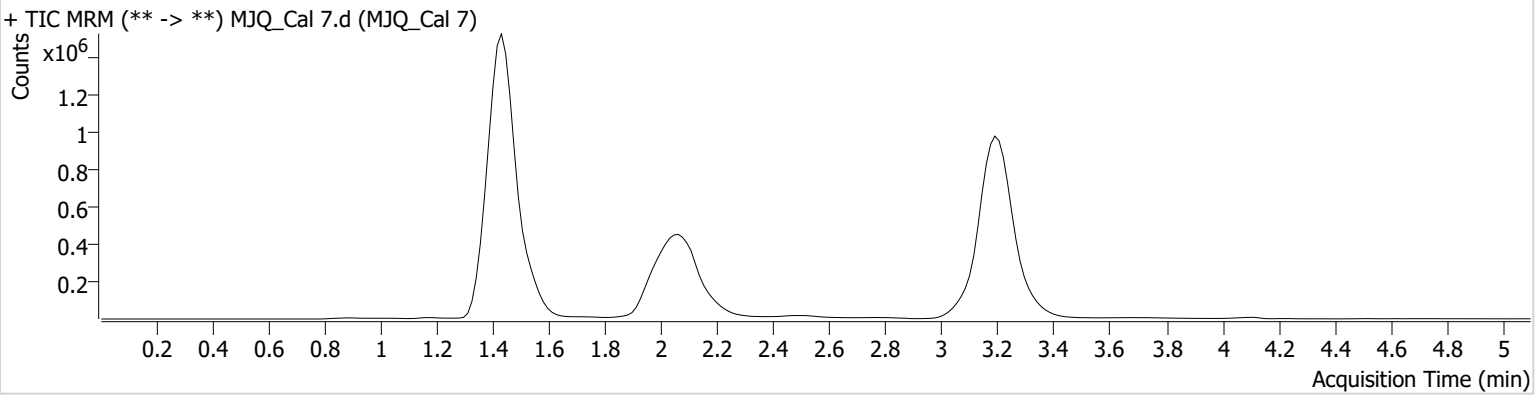
AM #27 Cannabinoid Quant. Results

Batch results D:\MassHunter\Data\2021\AM 25-26\022221 MDS THCS MDS Coroner Validatoin SAEC\QuantResults\AM 27.batch.bin
Calibration Last Update 2/24/2021 7:48:33 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	2/23/2021 6:56:06 PM		
Sample Info.			

Sample Chromatogram



Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	1.423	2683831	∞	11.7	∞	2064955	100.4862	ng/ml
THC-COOH	1.444	3037586	1731.79	58.0	9719.88	512324	251.8741	ng/ml
THC	3.194	3670633	∞	25.0	∞	3964534	100.5638	ng/ml