Worklist: 6588

LAB_CASE	ITEM	ITEM_TYPE	DESCRIPTION	
C2023-2309	1	BCK	AM 27 Blood THC Quant by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖ ‖ ‖‖‖‖ ‖‖‖
C2023-2514	1	BCK	AM 27 Blood THC Quant by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖ ‖ ‖ ‖‖‖ ‖‖
C2023-2580	2	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖ ‖‖ ‖ ‖‖‖
C2023-2605	1	UCK	AM 27 Urine Cannabinoids Confirmation by LC-QQQ	‖‖‖ ‖‖ ‖‖‖‖‖‖‖‖‖‖‖‖‖‖ ‖‖ ‖‖ ‖‖‖

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

Extraction Date 11/30/23   Analyst: <u>Anne Nord</u>
Plate lot#: 230627   Plate re-test: 12/27/2023

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: 23J52629 **Urine Blank:** 8423   **Column**: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 69679

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.   Urine hydrolysis: <mark>add 1.5 ml urine to blank plate, add 250 ul 1N KOH mix and incubate at 40 degrees for 15 minutes.</mark>
     Pipette **1000μL (calibrated pipette) blood or 1000μL hydrolyzed urine Pipette ID: I41142J** in wells of analytical (standards) plate.
☒ 3.   Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 4.   Pipette **<mark style="background:red">500μL 0.1% formic acid in water blood sample</mark>**, **<mark>500 ul saturated phosphate buffer in urine</mark>** in wells 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) Manifold ID: 66792
☒ 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: 66819
☒ 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. SN > 10
☒ 4.   Case sample response for THC 1ng/ml LOD 3ng/ml LOQ, OH-THC 3ng/mL LOD and LOQ, Carboxy-THC: 5 ng/mL (qualitative only). 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 is it describe in comments section)
☒ 6.   Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: Mikel Buffaloe hands of the analyst

|	1	2	3	4	5	6
a	cal 1	Internal control urine				
b	cal 2	negative blood				
c	cal 3	2309-1				
d	cal 4	2514-1				
e	cal 5	negative urine				
f	cal 6	2580-2				
g	cal 7	2605-1				
h	Internal control (blood)					

Plate position 3

c2023-_____-_

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj internal control blood.d
Type	QC	**Sample**	mj internal control blood
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for
Injection Volume	10		interpretation purposes. Any drugs or values included in the notes but not included on the report
Acq. Date-Time	11/30/2023 5:52:35 PM		are used by laboratory personnel to make determinations/reach conclusions within the confines of
Sample Info.			the methods

Sample Chromatogram

+ TIC MRM (** -> **) mj internal control blood.d (mj internal control blood)
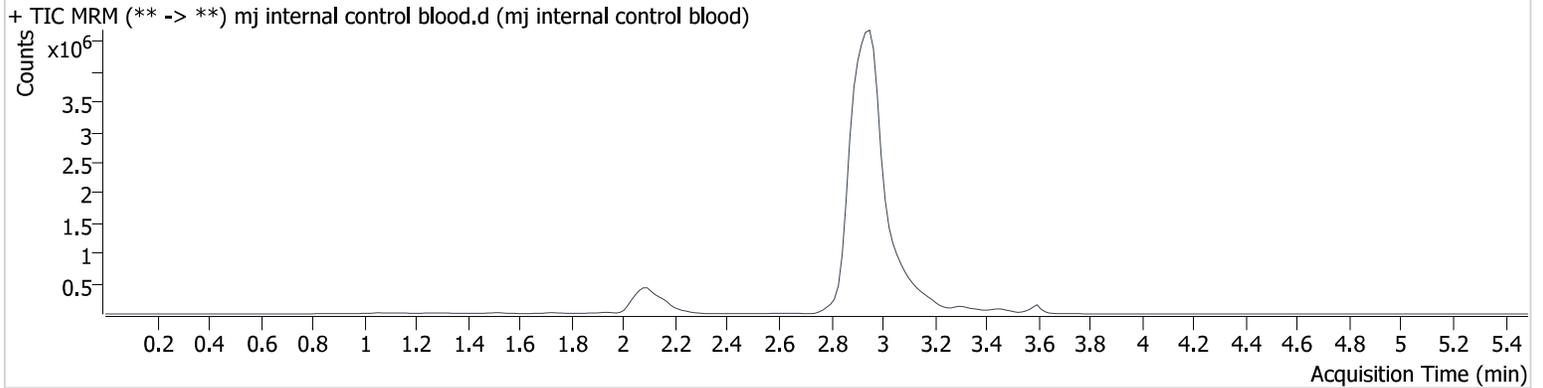
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	23467	465.6	881.59	∞	1748255	4.411 ng/ml
THC-COOH	2.152	48948	381.6	271.11	∞	1748255	17.297 ng/ml
THC	3.603	33409	1797.6	21.58	∞	281362	4.071 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj negative blood.d
Type	Sample	**Sample**	mj negative blood
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-B2	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for interpretation purposes. Any drugs or values included in the notes but not included on the report are used by laboratory personnel to make determinations/reach conclusions within the confines of the methods
Injection Volume	10		
Acq. Date-Time	11/30/2023 5:59:09 PM		
Sample Info.			

Sample Chromatogram

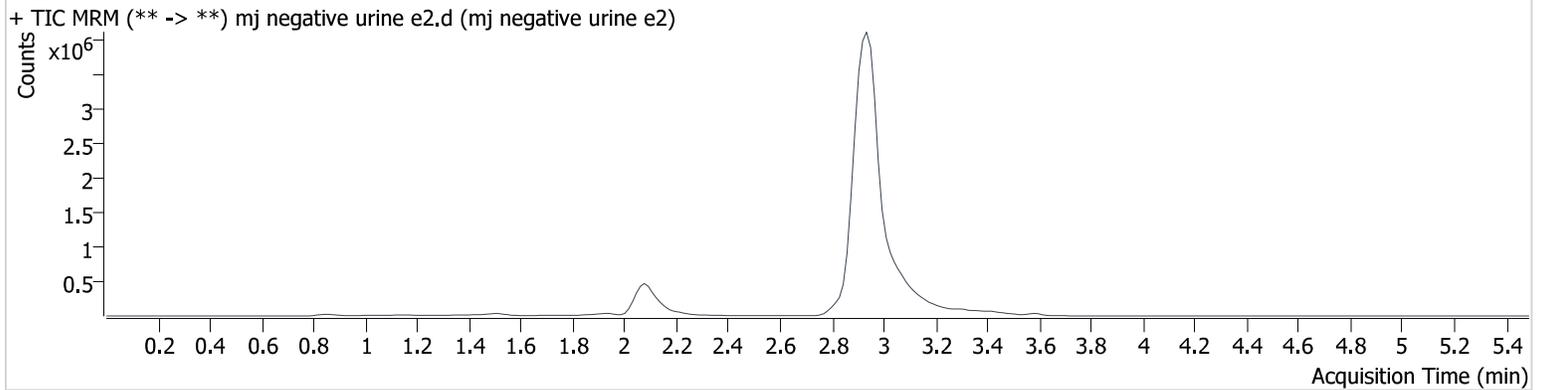+ TIC MRM (** -> **) mj negative blood.d (mj negative blood)

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update 12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj negative urine e2.d
Type	Sample	**Sample**	mj negative urine e2
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-E2	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for
Injection Volume	10		interpretation purposes. Any drugs or values included in the notes but not included on the report
Acq. Date-Time	11/30/2023 6:32:10 PM		are used by laboratory personnel to make determinations/reach conclusions within the confines of
Sample Info.			the methods

Sample Chromatogram

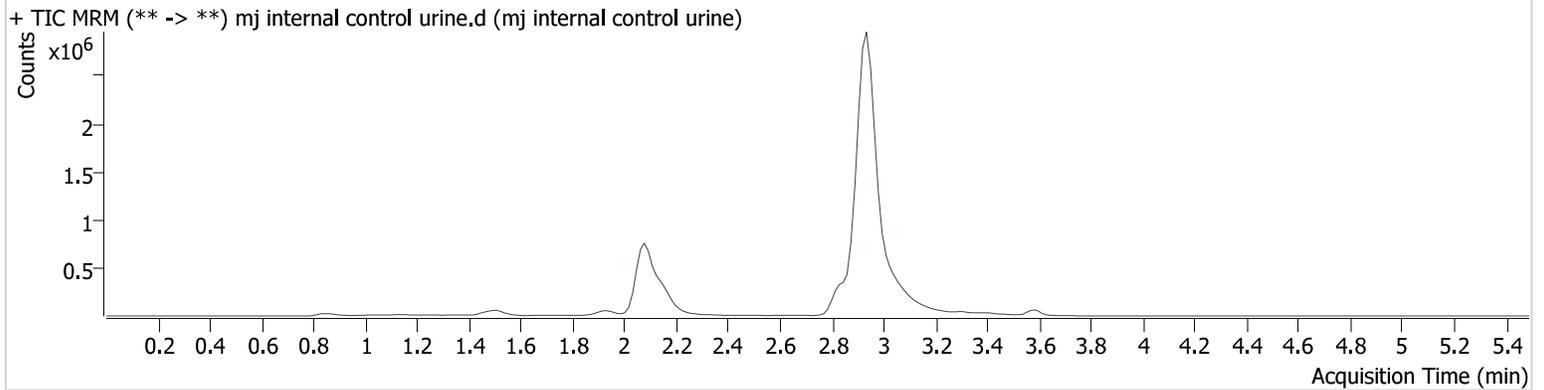+ TIC MRM (** -> **) mj negative urine e2.d (mj negative urine e2)

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj internal control urine.d
Type	Sample	**Sample**	mj internal control urine
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-A2	**Comment**	*Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for*
Injection Volume	10		*interpretation purposes. Any drugs or values included in the notes but not included on the report*
Acq. Date-Time	11/30/2023 7:24:55 PM		*are used by laboratory personnel to make determinations/reach conclusions within the confines of*
Sample Info.			*the methods*

Sample Chromatogram

+ TIC MRM (** -> **) mj internal control urine.d (mj internal control urine)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	37950	∞	865.48	∞	2746455	4.529 ng/ml
THC-COOH	2.152	57403	1032.6	282.46	∞	2746455	13.371 ng/ml
THC	3.588	19462	∞	26.65	179.8	179124	3.760 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update 12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	end of run blood control blood h1.d
Type	QC	**Sample**	end of run blood control blood h1
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-H1	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for interpretation purposes. Any drugs or values included in the notes but not included on the report are used by laboratory personnel to make determinations/reach conclusions within the confines of the methods
Injection Volume	10		
Acq. Date-Time	11/30/2023 7:31:32 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) end of run blood control blood h1.d (end of run blood control blood h1)
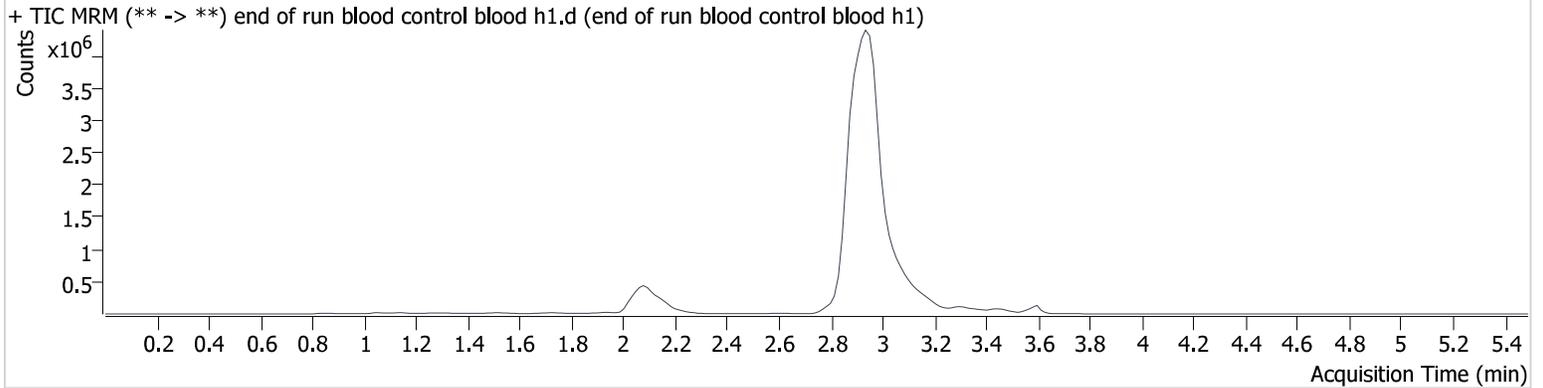
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	24019	1420.4	892.04	∞	1868940	4.241 ng/ml
THC-COOH	2.152	43645	409.5	287.25	∞	1868940	14.727 ng/ml
THC	3.603	30476	∞	22.81	∞	251255	4.150 ng/ml

Compound Calibration Report

Agilent | Trusted Answers

Batch results	D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Last Cal. Update	12/1/2023 12:03 PM
Analyst Name	ISP\datastor
Analyte	THC

Internal Standard   THC-d3

THC - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 2 QCs

$y = 0.032392 * x - 0.013129$
$R^2 = 0.99846282$
Type:Linear, Origin:Ignore, Weight:1/x

Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	1.0	1.2	120.2
mj cal 2	2	✔	3.0	2.8	94.8
mj cal 3	3	✔	5.0	4.7	93.1
mj cal 4	4	✔	10.0	9.0	89.7
mj cal 5	5	✔	25.0	25.6	102.5
mj cal 6	6	✔	50.0	48.8	97.7
mj cal 7	7	✔	100.0	101.9	101.9

Compound Calibration Report

Agilent | Trusted Answers

Batch results	D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Last Cal. Update	12/1/2023 12:03 PM
Analyst Name	ISP\datastor
Analyte	THC-COOH

Internal Standard THC-OH-d3

THC-COOH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 2 QCs

$y = 0.001808 * x - 0.003274$
$R^2 = 0.99126446$
Type:Linear, Origin:Ignore, Weight:1/x
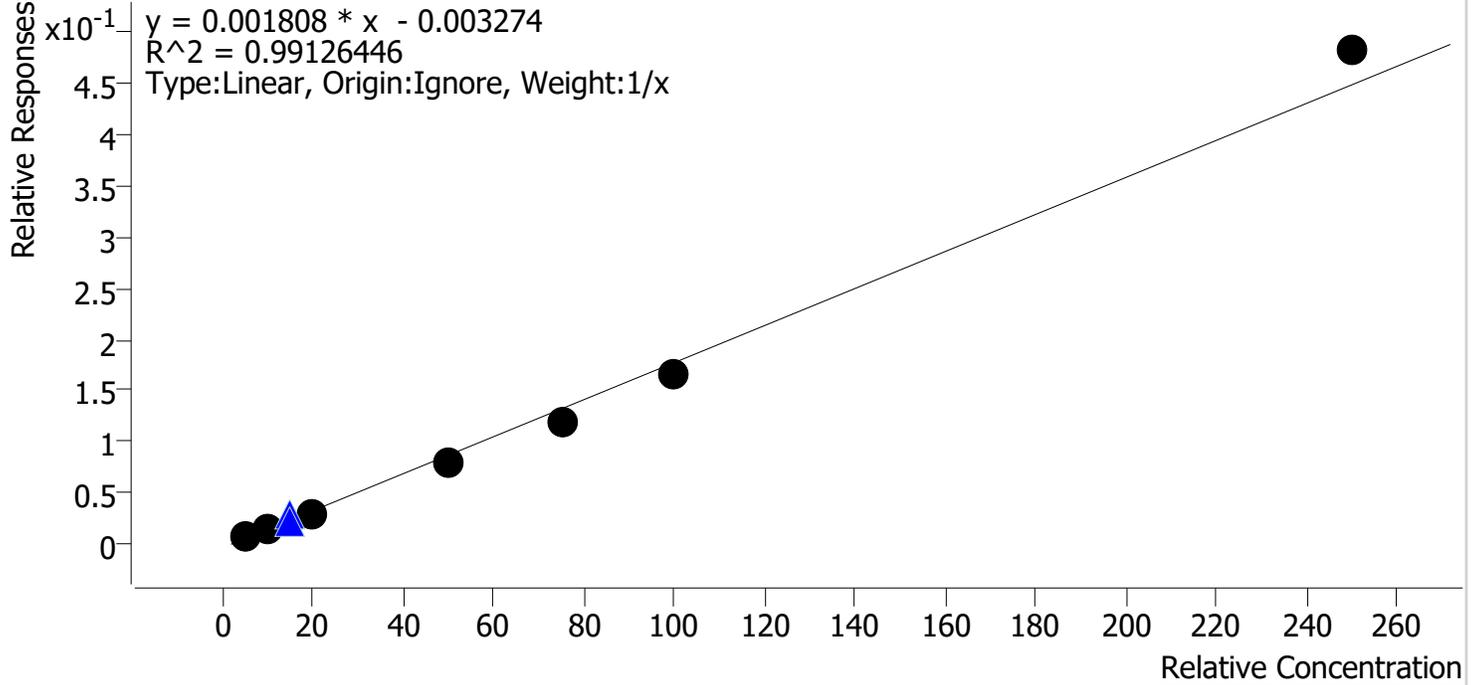
Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	5.0	6.4	128.2
mj cal 2	2	✔	10.0	9.5	94.5
mj cal 3	3	✔	20.0	18.8	94.2
mj cal 4	4	✔	50.0	46.6	93.1
mj cal 5	5	✔	75.0	67.0	89.4
mj cal 6	6	✔	100.0	93.2	93.2
mj cal 7	7	✔	250.0	268.5	107.4

Agilent | Trusted Answers

Batch results	D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Last Cal. Update	12/1/2023 12:03 PM
Analyst Name	ISP\datastor
Analyte	THC-OH	**Internal Standard** THC-OH-d3

THC-OH - 7 Levels, 7 Levels Used, 7 Points, 7 Points Used, 2 QCs
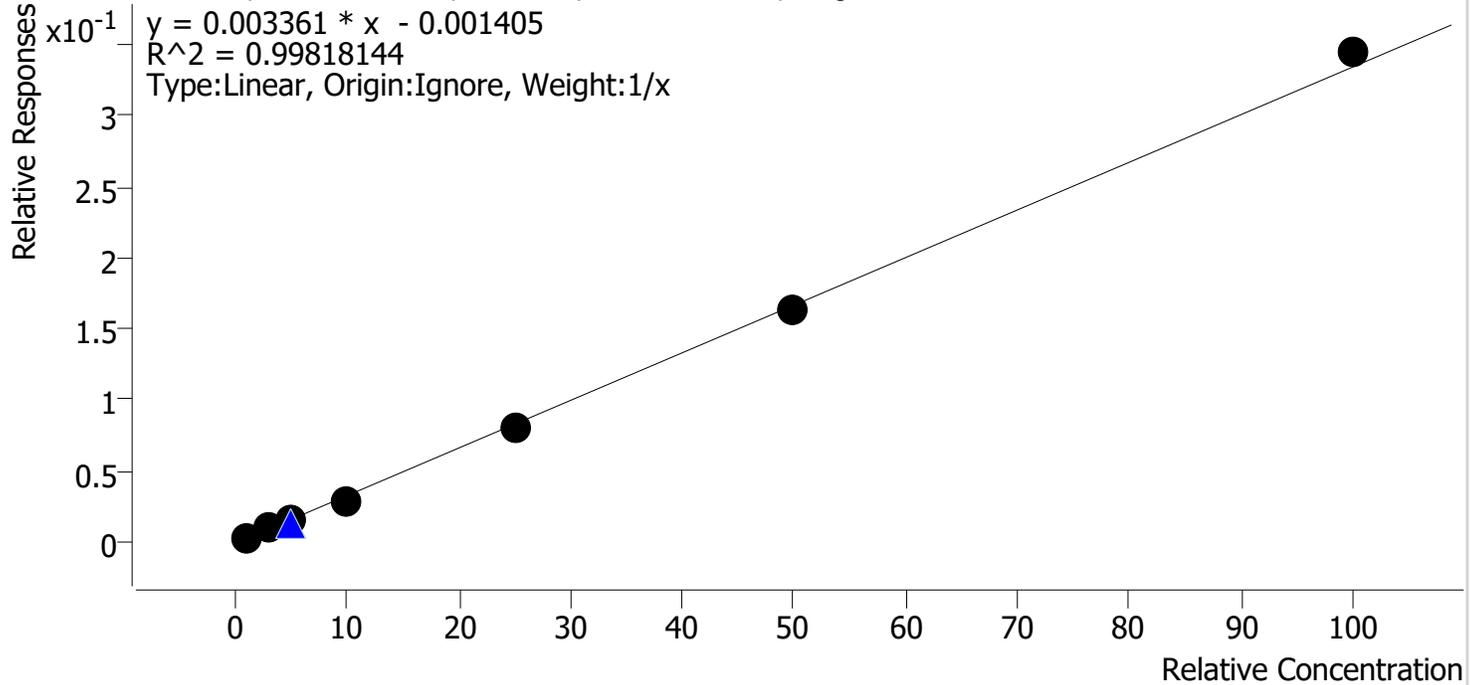
$y = 0.003361 * x - 0.001405$
$R^2 = 0.99818144$
Type:Linear, Origin:Ignore, Weight:1/x

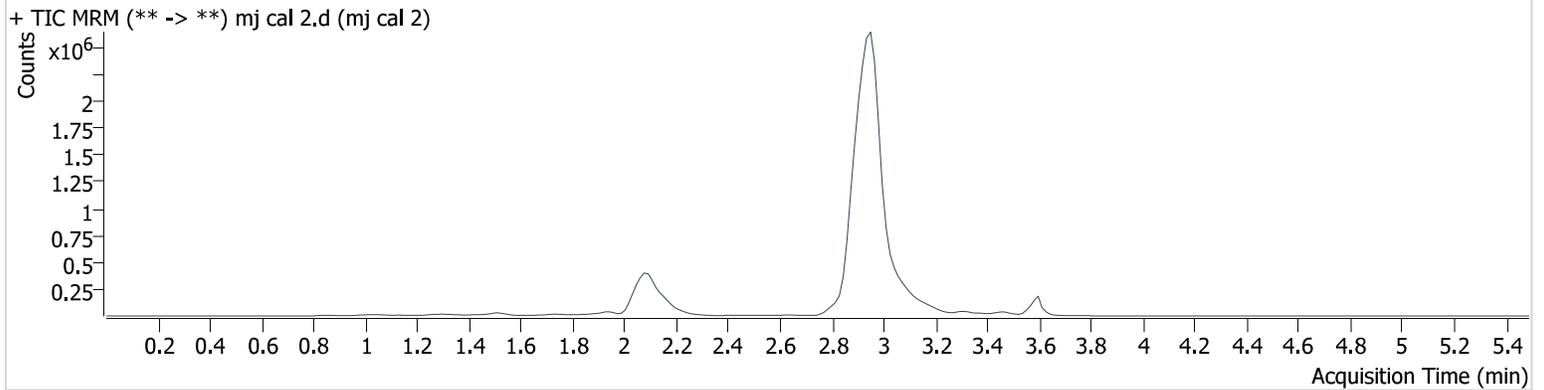| Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
mj cal 1	1	✔	1.0	1.2	115.7
mj cal 2	2	✔	3.0	3.1	103.0
mj cal 3	3	✔	5.0	4.7	94.6
mj cal 4	4	✔	10.0	9.0	89.8
mj cal 5	5	✔	25.0	23.9	95.6
mj cal 6	6	✔	50.0	49.1	98.2
mj cal 7	7	✔	100.0	103.0	103.0

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 1.d
Type	Cal	**Sample**	mj cal 1
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-A1	**Comment**	*Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for*
Injection Volume	10		*interpretation purposes. Any drugs or values included in the notes but not included on the report*
Acq. Date-Time	11/30/2023 5:06:17 PM		*are used by laboratory personnel to make determinations/reach conclusions within the confines of*
Sample Info.			*the methods*

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 1.d (mj cal 1)
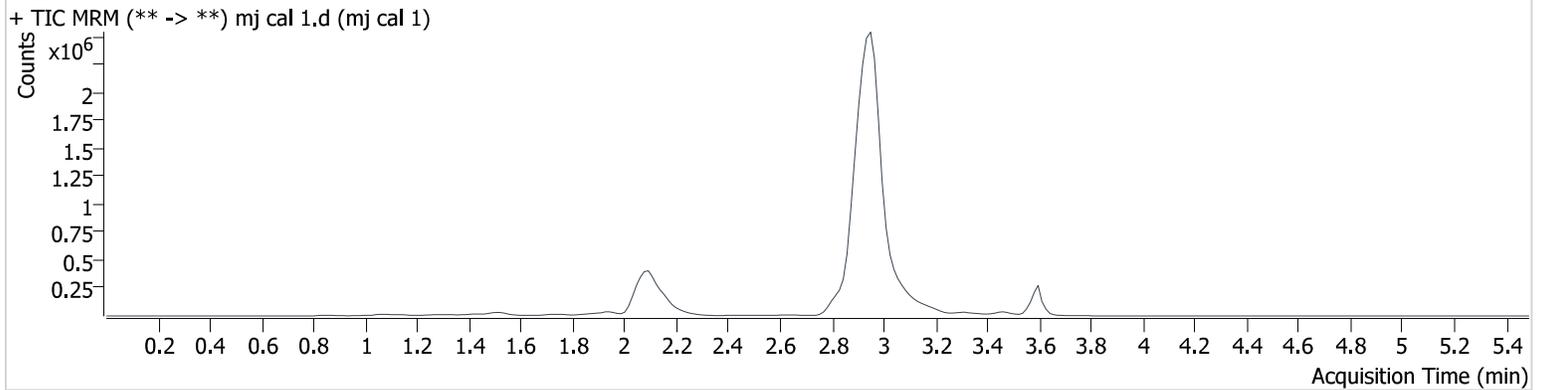
Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.	
THC-OH	2.096	4466	∞	808.83	∞	1797378	1.157 ng/ml	**Low**
THC-COOH	2.167	14947	∞	271.72	∞	1797378	6.410 ng/ml	
THC	3.603	12955	∞	21.47	∞	501671	1.202 ng/ml	

AM #27 Cannabinoids

Instrument	69679	**Data File**	mj cal 2.d
Type	Cal	**Sample**	mj cal 2
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-B1	**Comment**	*Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for*
Injection Volume	10		*interpretation purposes. Any drugs or values included in the notes but not included on the report*
Acq. Date-Time	11/30/2023 5:13:01 PM		*are used by laboratory personnel to make determinations/reach conclusions within the confines of*
Sample Info.			*the methods*

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 2.d (mj cal 2)

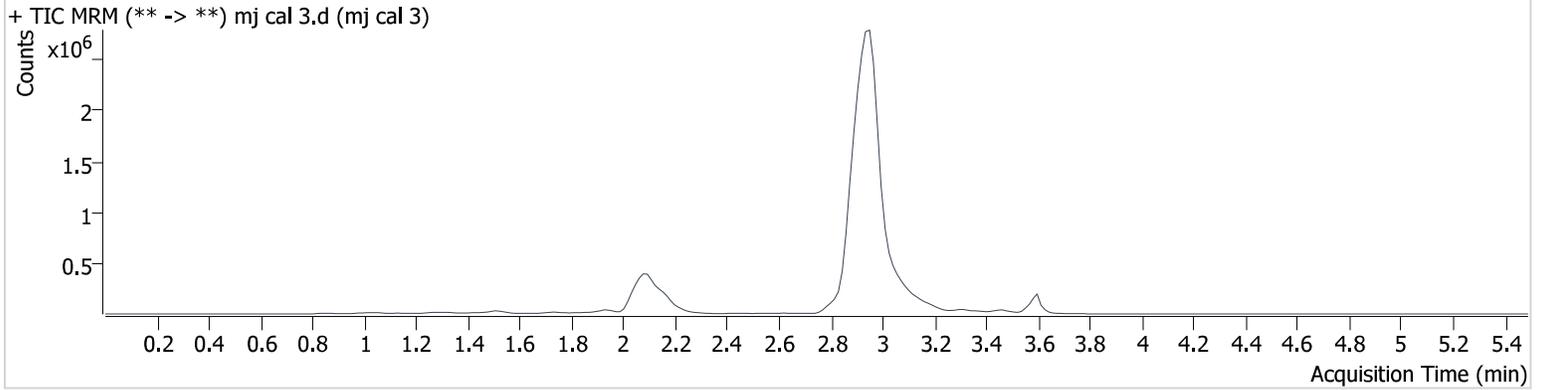| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.096	15608	∞	795.40	∞	1737212	3.091 ng/ml
THC-COOH	2.167	24001	∞	276.06	∞	1737212	9.452 ng/ml
THC	3.603	25489	∞	23.32	∞	322500	2.845 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update 12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 3.d
Type	Cal	**Sample**	mj cal 3
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-C1	**Comment**	*Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for*
Injection Volume	10		*interpretation purposes. Any drugs or values included in the notes but not included on the report*
Acq. Date-Time	11/30/2023 5:19:37 PM		*are used by laboratory personnel to make determinations/reach conclusions within the confines of*
Sample Info.			*the methods*

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 3.d (mj cal 3)

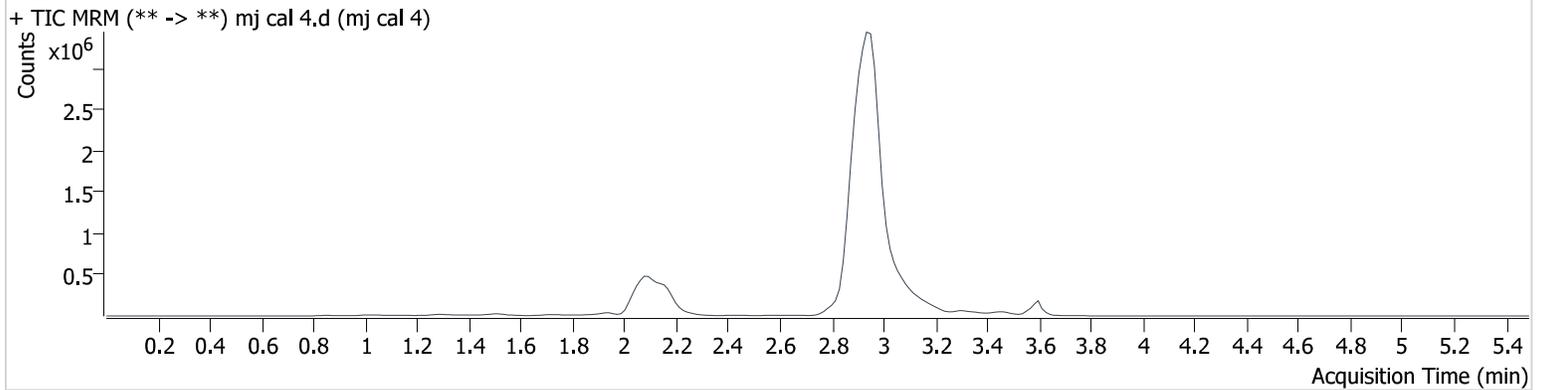| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	23621	263.0	870.75	∞	1629649	4.730 ng/ml
THC-COOH	2.152	50160	∞	271.43	∞	1629649	18.835 ng/ml
THC	3.603	44269	378.9	23.15	∞	321413	4.657 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 4.d
Type	Cal	**Sample**	mj cal 4
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-D1	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for interpretation purposes. Any drugs or values included in the notes but not included on the report are used by laboratory personnel to make determinations/reach conclusions within the confines of the methods
Injection Volume	10		
Acq. Date-Time	11/30/2023 5:26:11 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 4.d (mj cal 4)

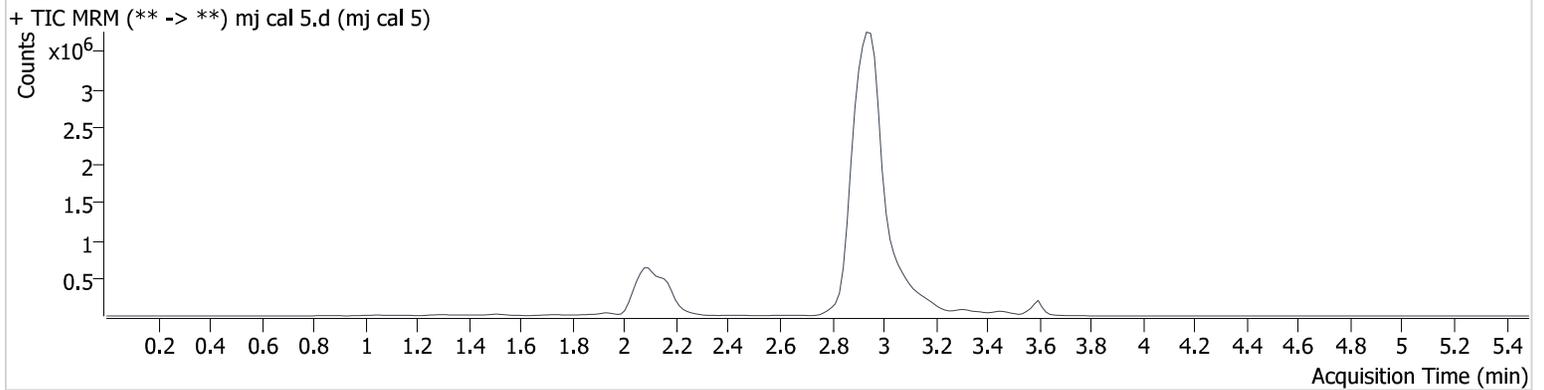| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	50376	7335.3	932.92	∞	1750246	8.981 ng/ml
THC-COOH	2.152	141581	∞	267.75	∞	1750246	46.552 ng/ml
THC	3.603	80394	∞	24.80	∞	289699	8.972 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 5.d
Type	Cal	**Sample**	mj cal 5
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-E1	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for interpretation purposes. Any drugs or values included in the notes but not included on the report are used by laboratory personnel to make determinations/reach conclusions within the confines of the methods
Injection Volume	10		
Acq. Date-Time	11/30/2023 5:32:48 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 5.d (mj cal 5)

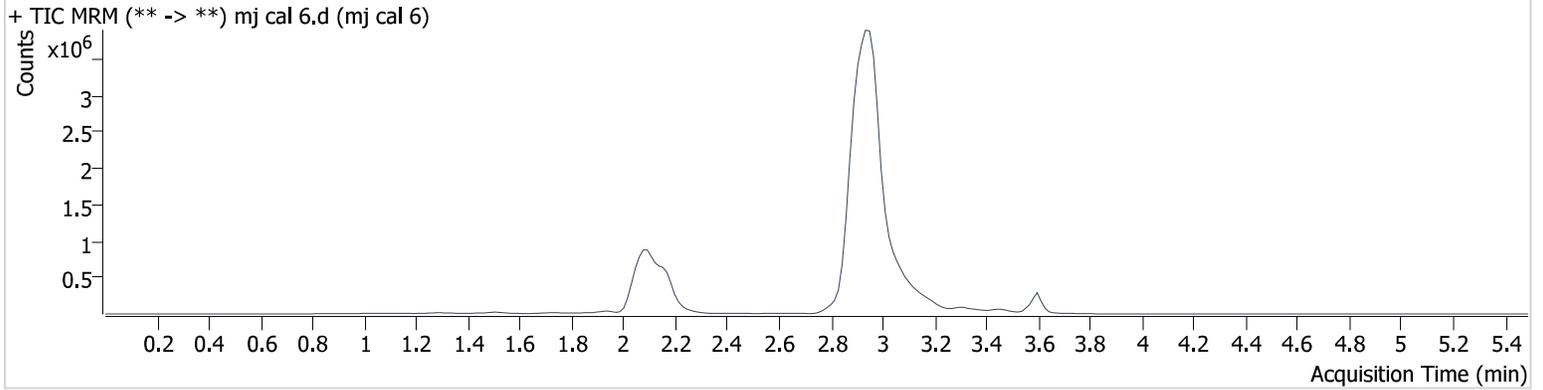| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	136539	∞	892.66	∞	1729392	23.906 ng/ml
THC-COOH	2.152	203926	5496.3	278.82	∞	1729392	67.031 ng/ml
THC	3.603	200308	657213.7	20.48	∞	245224	25.622 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 6.d
Type	Cal	**Sample**	mj cal 6
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-F1	**Comment**	Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for
Injection Volume	10		interpretation purposes. Any drugs or values included in the notes but not included on the report
Acq. Date-Time	11/30/2023 5:39:24 PM		are used by laboratory personnel to make determinations/reach conclusions within the confines of
Sample Info.			the methods

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 6.d (mj cal 6)

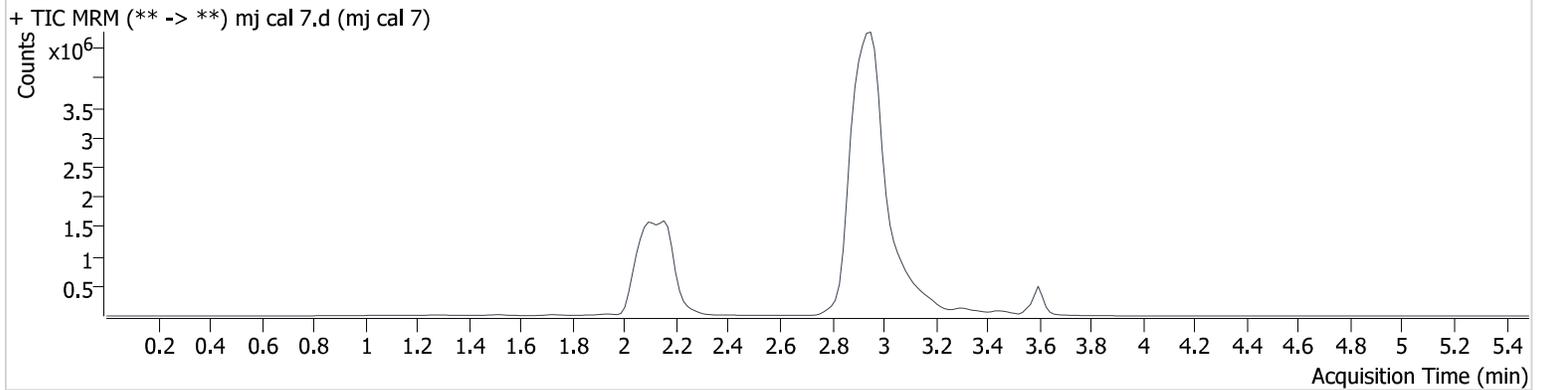| Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	277081	∞	874.26	∞	1692839	49.112 ng/ml
THC-COOH	2.152	279782	∞	272.48	∞	1692839	93.224 ng/ml
THC	3.603	385797	∞	23.31	∞	245862	48.848 ng/ml

AM #27 Cannabinoids

Batch results   D:\MassHunter\Data\2023\am 27-28\113023\QuantResults\thc.batch.bin
Calibration Last Update   12/1/2023 12:03:10 PM

Instrument	69679	**Data File**	mj cal 7.d
Type	Cal	**Sample**	mj cal 7
Acq. Method	thc quant 50 50.m	**Operator**	Anne Nord
Sample Position	P3-G1	**Comment**	*Only drugs and concentrations listed on the laboratory report itself are appropriate to be used for interpretation purposes. Any drugs or values included in the notes but not included on the report are used by laboratory personnel to make determinations/reach conclusions within the confines of the methods*
Injection Volume	10		
Acq. Date-Time	11/30/2023 5:46:00 PM		
Sample Info.			

Sample Chromatogram

+ TIC MRM (** -> **) mj cal 7.d (mj cal 7)

Name	RT	Resp.	S/N	Ratio	S/N	ISTD Resp.	Final Conc.
THC-OH	2.081	616798	13595.2	853.54	∞	1788348	103.024 ng/ml
THC-COOH	2.152	862276	3471.1	263.90	∞	1788348	268.496 ng/ml
THC	3.603	865634	15415.6	24.12	10573.0	263424	101.852 ng/ml

Idaho State Police
Forensic Services

Request for Departure from an Analytical Method or Quality Standard

Deviation Number (assigned by QM): ISP Dev QUAL-23-02

Date of Request: **8/8/23**

Requestor/Discipline: Anne Nord / Quality manual

Analytical Method/Quality Standard, Revision #: Quality manual Revision 9
16.2.5c.2.6 Training in the use and understanding of analytical methods shall include the analysis of training samples. The trainee may, under the direct observation of a competent analyst, handle case samples, but the trainer will make all conclusions and must be present and observe all aspects of the work (the trainee works as the hands of the trainer). All evidence in the "hands of the trainer" process will be checked out by the trainer and the chain of custody shall be maintained in the name of the trainer/trained analyst. Probative samples may be independently handled by the trainee if the evidence can be analyzed without changing it (e.g. comparison of latent prints or bullets). Examination reports shall be based solely on examinations performed by or directly observed by approved analysts. The report will be issued by the trainer/trained analyst. The analytical notes will clearly indicate the samples handled by the trainee. In the case of controlled substances, if an additional training sample is taken it will be stored in a secure locked location (either a drug locker or the controlled substance cabinet)

Temporary or Permanent Deviation: Permanent until the next version of the quality
     manual is released and analytical notes can be defined.

Scope of Deviation (record specific information, e.g. affected programs, evidence types, expected end date; etc): Documentation for hands of the trainer, in case records.

Deviation Request (Describe detailed instructions of the changes being made; include reference to specific section number(s) in the method manual):

The quality manual currently requires that documentation that the trainee handled samples will be in the "analytical notes" the manual does not further define what is meant by analytical notes. I am requesting to document this in the case notes that are attached in ILIMS or if there is batch data associated with the analysis it may be documented in the central batch data.

Technical Justification for Analytical Method Deviations: The batch data and the notes packet are both part of the case record. They both provide a path to clear documentation of what the trainee handled or did.

Technical Review

☐ Departure approved
 Comments:

☐ Departure Not Approved
 Comments:

Approver: Date:
Title:

Quality Review

Quality Approver: Corinna Owsley
Title: Acting Quality Manager
Date: 8-8-23