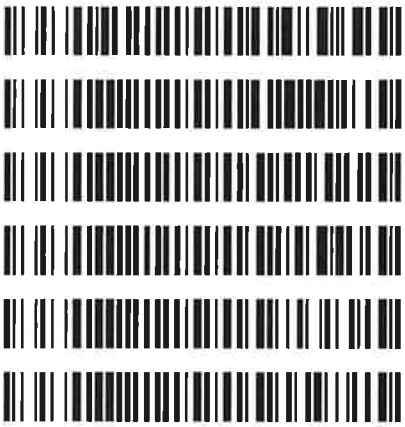
REVIEWED
By Celena Shrum at 11:44 am, Apr 23, 2019

4/22/2019

Worklist: 3318

LAB_CASE	ITEM	TASK_ID	DESCRIPTION
M2019-1432	2	149340	AM 27 Blood THC Quant by LC-QQQ
P2019-1046	1	149341	AM 27 Blood THC Quant by LC-QQQ
P2019-1055	1	149342	AM 27 Blood THC Quant by LC-QQQ
P2019-1096	1	149343	AM 27 Blood THC Quant by LC-QQQ
P2019-1167	1	149344	AM 27 Blood THC Quant by LC-QQQ
P2019-1170	1	149345	AM 27 Blood THC Quant by LC-QQQ



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

Extraction Date: 4/17/19 Analyst: Sarah Pickle
Plate lot#: 0539904 Plate Expiration: 09/10/19

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: 445283-1 **Column**: UCT Selectra DA 100 x 2.1mm 3um
LCMS-QQQ ID: 59740

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.
☒ 3. Create worklist: Data Path: _____

Analytic:

☒ 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
☒ 2. Pipette **1000µL blood (calibrated pipette) Pipette ID: #3** in wells of analytical (standards) plate.
☒ 3. Place on shaking incubator at ambient temp., 900rpm for 15 minutes. *Shaker ID: 067105*
☒ 4. Pipette **500µL 0.1% formic acid in water** in wells of analytical plate.
☒ 5. Place on shaking incubator at ambient temp., 900rpm for 15 minutes.
☒ 6. Transfer **800µL 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 85-100 PSI- Selector to the right)*** Manifold ID: 067104
☒ 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: 067103*
☒ 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. C:\MassHunter\Data\2019\Am27\041719 THCQ SP
Worklist path: ~~G:\TOX\Pocatello\Abby\2019\AM 27\041719 THCQ SP~~ Batch Name: THCQ
☒ 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 and OH-THC 3ng/mL (quantitative), Carboxy-THC: 10ng/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: Carboxy-THC 10-100*

Idaho State Police
Forensic Services

AM #27 Quantitative Analysis of THC and Metabolites in Blood by LCMS-QQQ

Methanol External Control Solution (Lot: WS041619)

10 ul of 1mg/mL THC, 100 ul of 100 ug/mL THC-OH, C-THC in 9790 ul MeOH

Approximate concentration 1ug/mL.

Component	*Source*	*Source Lot Number*	*Expiration Date*
Methanol (LCMS)	Fisher	184782	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2019
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	04/16/2019		
Prepared By:	Tamara Salazar		
Expires:	01/31/2020		

Blood External Control Solution (Lot: 041619)

100 ul of methanol external control solution was added to 9900 ul of blood.

Approximately 10ng/mL of each compound.

Component	*Source*	*Source Lot Number*
Negative Blood	Hemostat	445283-1
Methanol External Control Solution	-	WS041619
Prepared:	04/16/19	
Prepared by:	Tamara Salazar	
Expires:	01/31/2020	

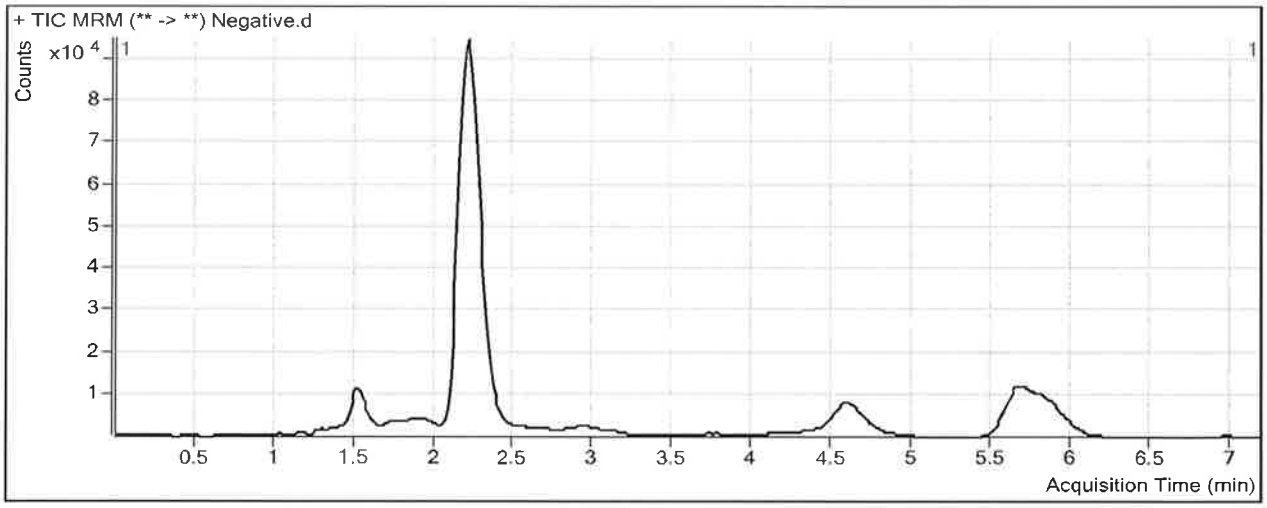
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:14 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 11:30	**Data File**	Negative.d
Sample Type	Sample	**Sample Name**	Negative
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-H5	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



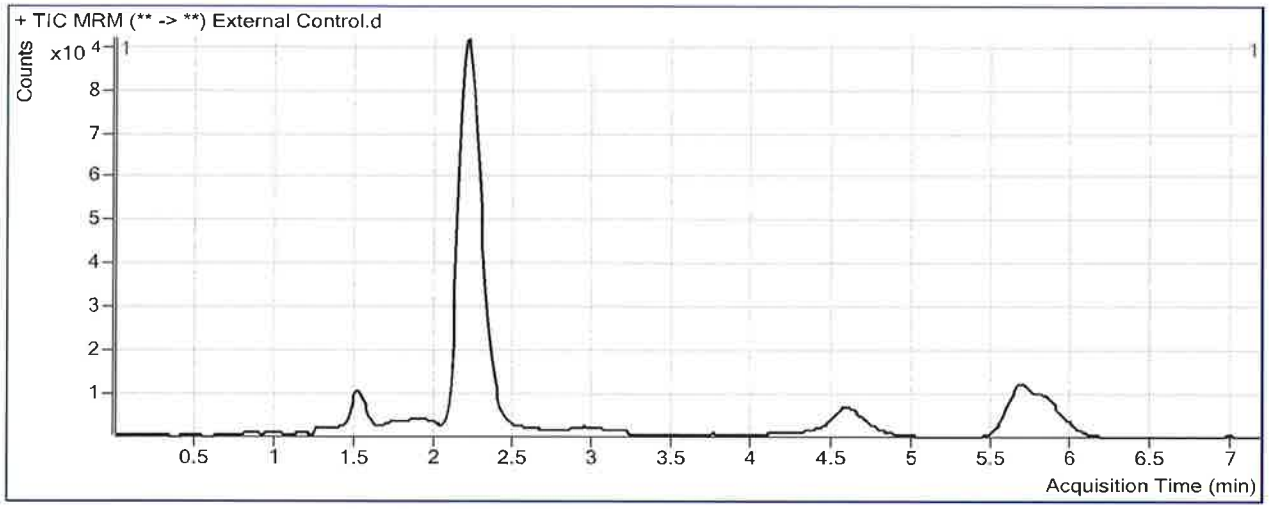
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:14 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 11:54	**Data File**	External Control.d
Sample Type	Sample	**Sample Name**	External Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-G5	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.212	54425	594340	0.0916	8.0137
THC-COOH	THC-COOH-D9	2.299	39533	210250	0.1880	7.4009
THC	THC-D3	5.732	15930	224879	0.0708	8.5237

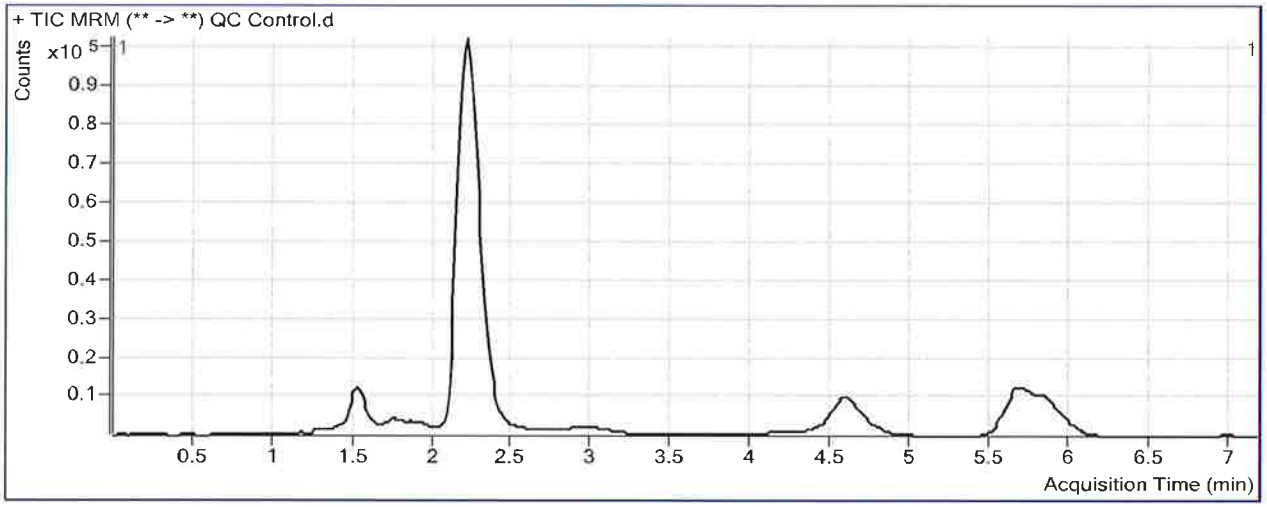
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:14 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 11:06	**Data File**	QC Control.d
Sample Type	QC	**Sample Name**	QC Control
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-A6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

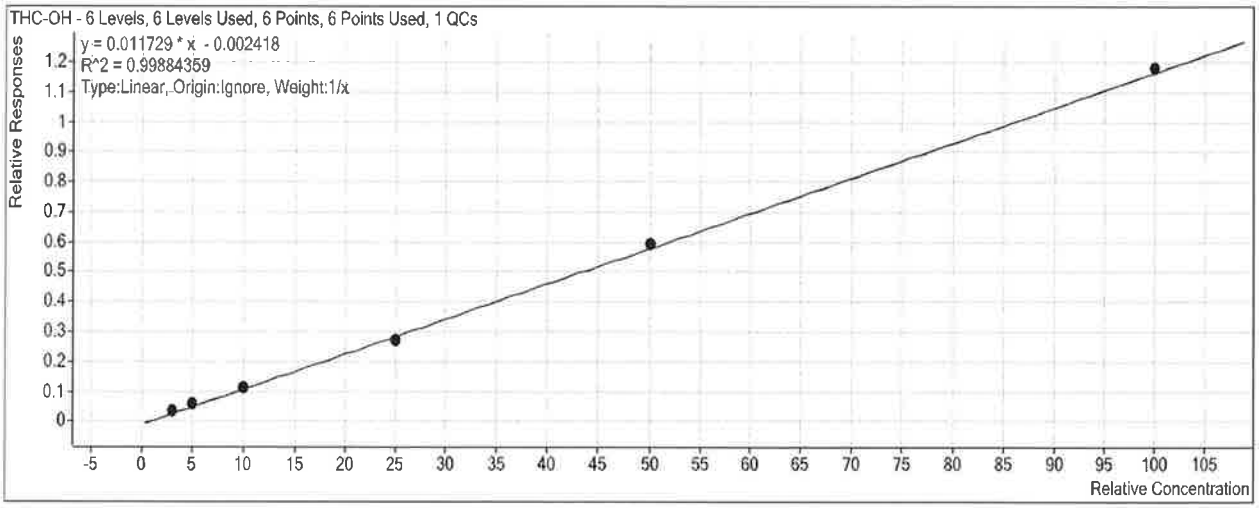
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.212	37151	677073	0.0549	4.8844
THC-COOH	THC-COOH-D9	2.299	56117	237017	0.2368	10.5664
THC	THC-D3	5.759	10274	257740	0.0399	4.6692

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin

Last Calib Update 4/23/2019 11:35 AM **Analyst Name** ISP TOX

Target Compound *THC-OH*
Internal Standard *THC-OH-D3*



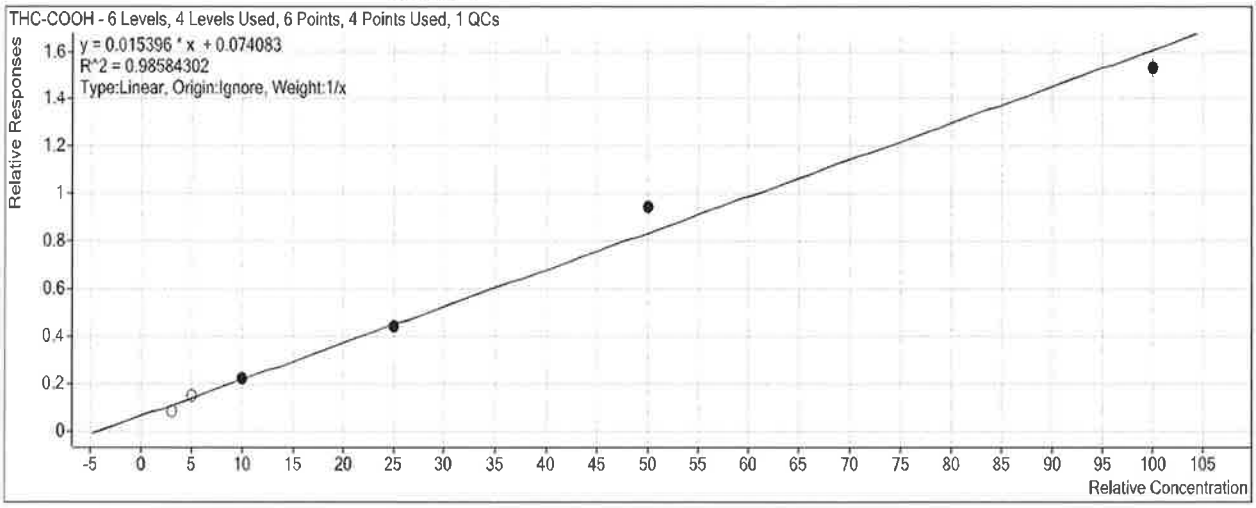
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☑	3	3.2	105.1
Cal 2-5ng	2	☑	5	5.1	101.6
Cal 3-10ng	3	☑	10	9.8	97.9
Cal 4-25ng	4	☑	25	23.2	92.8
Cal 5-50ng	5	☑	50	50.8	101.6
Cal 6-100ng	6	☑	100	101.0	101.0
QC Control	8	☑	5	4.9	97.7

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin

Last Calib Update 4/23/2019 11:35 AM **Analyst Name** ISP TOX

Target Compound *THC-COOH*
Internal Standard *THC-COOH-D9*



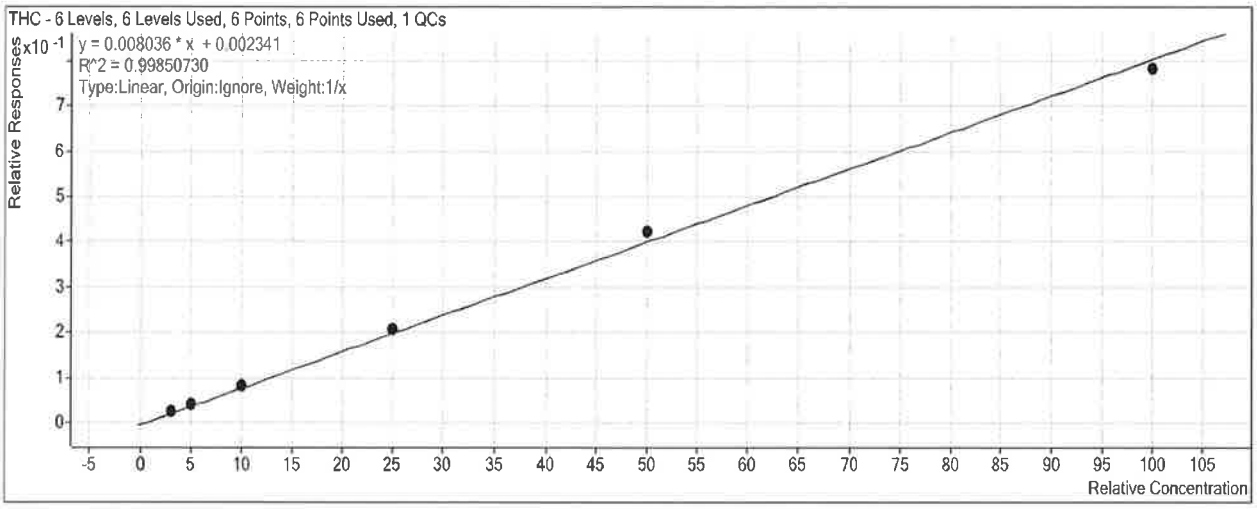
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☐	3	0.6	21.0
Cal 2-5ng	2	☐	5	5.1	101.4
Cal 3-10ng	3	☑	10	9.6	96.3
Cal 4-25ng	4	☑	25	23.8	95.3
Cal 5-50ng	5	☑	50	56.9	113.8
Cal 6-100ng	6	☑	100	94.7	94.7
QC Control	8	☐	10	10.6	105.7

ISP Forensics
Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin

Last Calib Update 4/23/2019 11:35 AM **Analyst Name** ISP TOX

Target Compound *THC*
Internal Standard *THC-D3*



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1-3ng	1	☑	3	2.9	97.9
Cal 2-5ng	2	☑	5	4.8	95.7
Cal 3-10ng	3	☑	10	10.3	102.5
Cal 4-25ng	4	☑	25	25.5	102.1
Cal 5-50ng	5	☑	50	52.3	104.6
Cal 6-100ng	6	☑	100	97.2	97.2
QC Control	8	☑	5	4.7	93.4

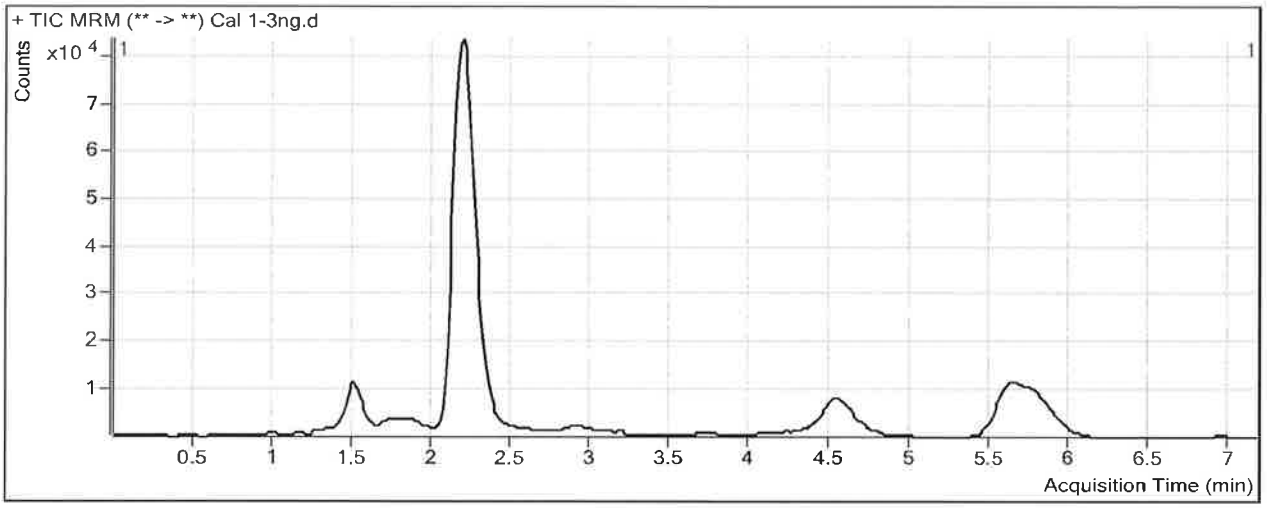
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 09:43	**Data File**	Cal 1-3ng.d
Sample Type	Calibration	**Sample Name**	Cal 1-3ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-G6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.199	19460	562982	0.0346	3.1532
THC-COOH	THC-COOH-D9	2.285	16711	199447	0.0838	0.6302
THC	THC-D3	5.772	6310	243291	0.0259	2.9361

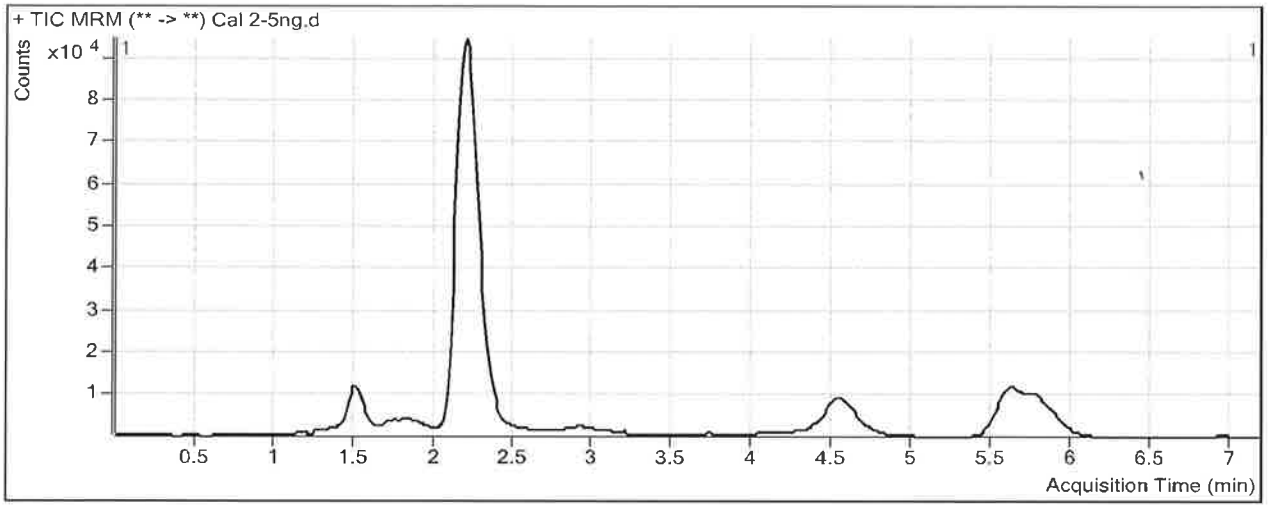
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 09:55	**Data File**	Cal 2-5ng.d
Sample Type	Calibration	**Sample Name**	Cal 2-5ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-F6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.199	35873	627552	0.0572	5.0800
THC-COOH	THC-COOH-D9	2.272	33241	218481	0.1521	5.0704
THC	THC-D3	5.692	9841	241230	0.0408	4.7850

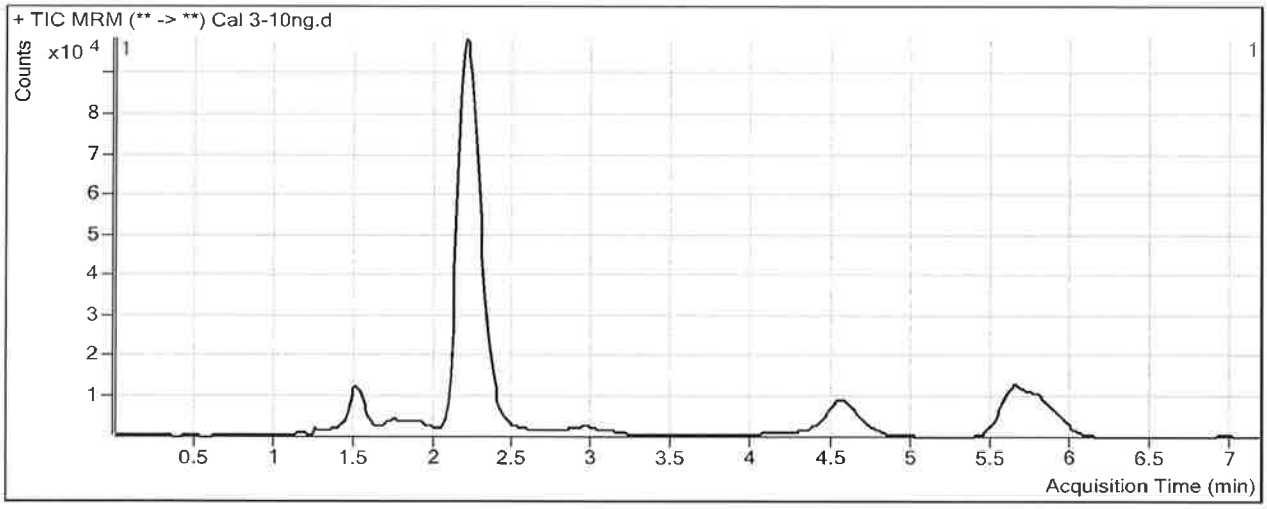
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 10:07	**Data File**	Cal 3-10ng.d
Sample Type	Calibration	**Sample Name**	Cal 3-10ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-E6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.199	70936	630886	0.1124	9.7928
THC-COOH	THC-COOH-D9	2.285	48412	217739	0.2223	9.6294
THC	THC-D3	5.772	20413	240882	0.0847	10.2539

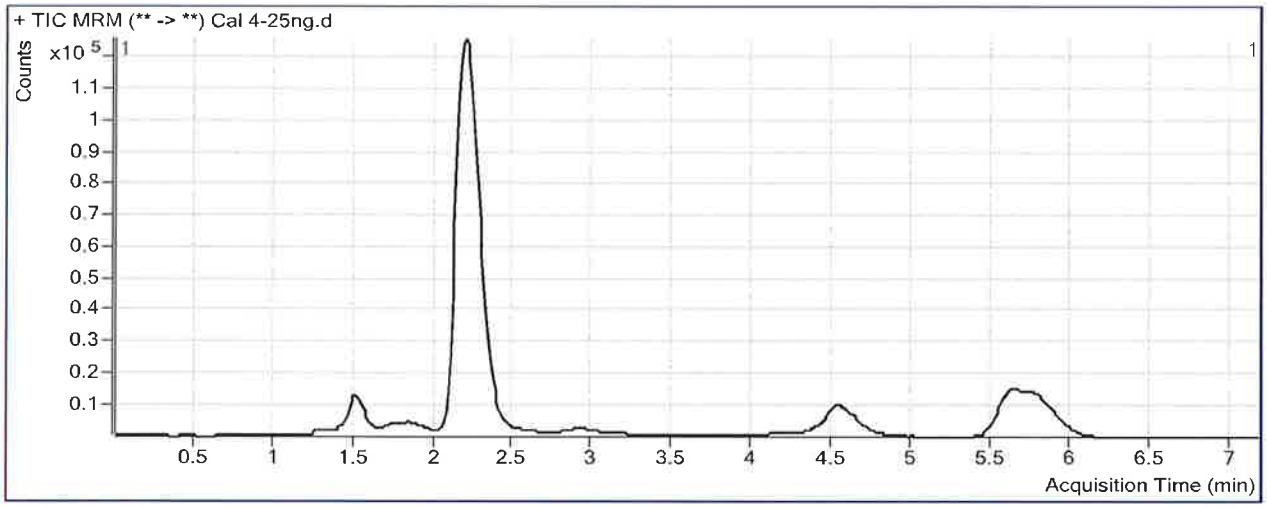
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 10:19	**Data File**	Cal 4-25ng.d
Sample Type	Calibration	**Sample Name**	Cal 4-25ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-D6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.185	188340	698209	0.2697	23.2050
THC-COOH	THC-COOH-D9	2.285	105021	238292	0.4407	23.8140
THC	THC-D3	5.759	54525	262848	0.2074	25.5223

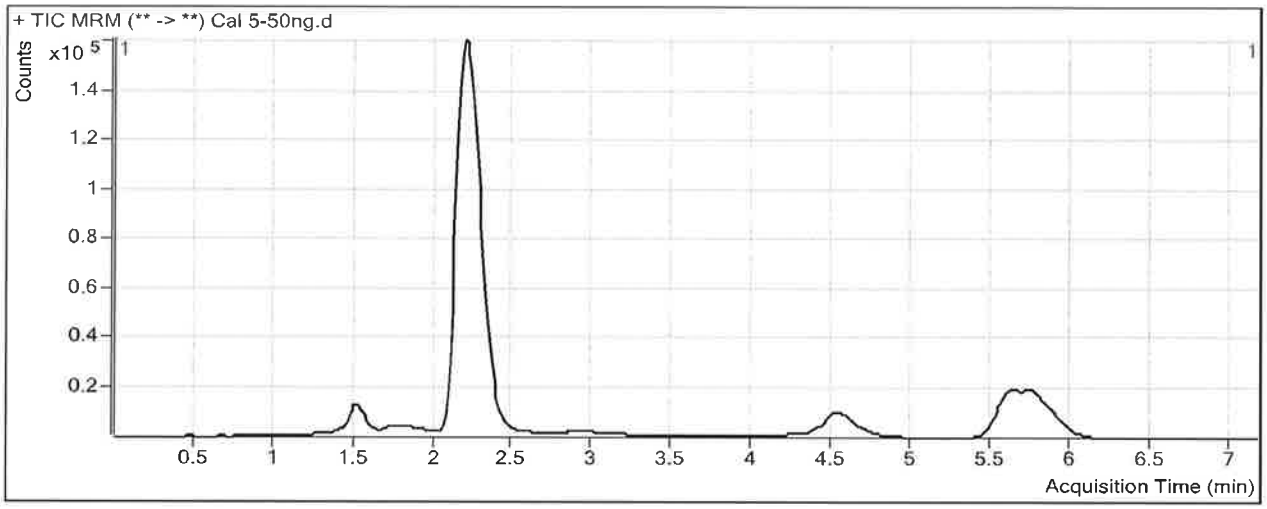
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 10:31	**Data File**	Cal 5-50ng.d
Sample Type	Calibration	**Sample Name**	Cal 5-50ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-C6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.199	415638	700757	0.5931	50.7766
THC-COOH	THC-COOH-D9	2.285	226915	238855	0.9500	56.8931
THC	THC-D3	5.745	116272	275122	0.4226	52.3000

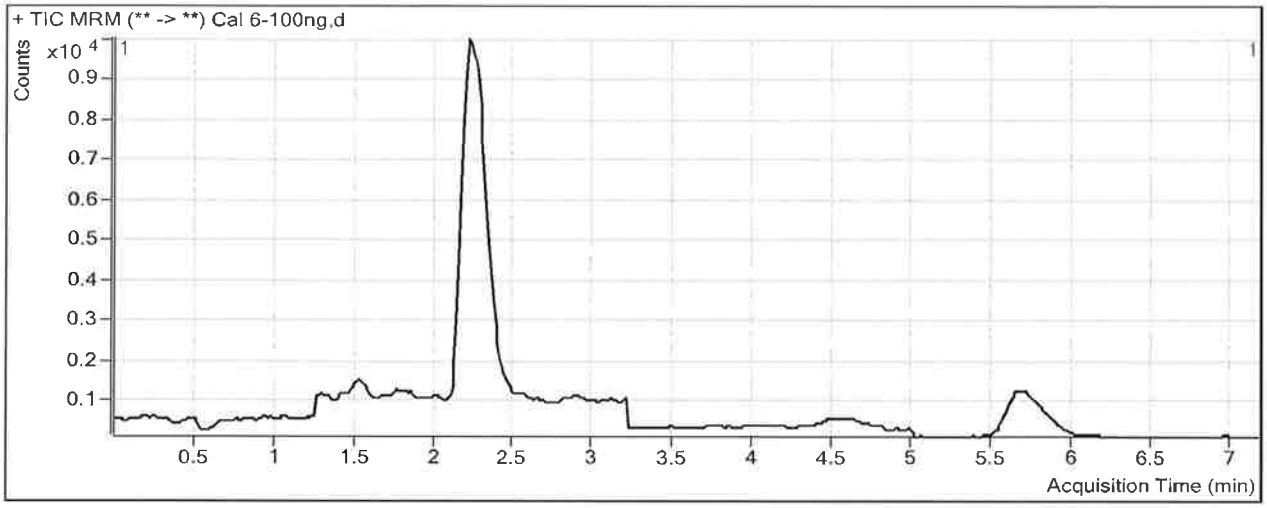
ISP FORENSICS
Pocatello
Benzodiazepine Analysis Report

Batch Data Path	C:\MassHunter\Data\2019\AM 27\041719 THCQ SP\QuantResults\THCQ.batch.bin		
Analysis Time	4/22/2019 3:11 PM	**Analyst Name**	datastor
Report Time	4/22/2019 3:13 PM	**Reporter Name**	datastor
Last Calib Update	4/22/2019 3:11 PM	**Batch State**	Processed

Analysis Info

Acq Time	2019-04-18 10:43	**Data File**	Cal 6-100ng.d
Sample Type	Calibration	**Sample Name**	Cal 6-100ng
Dilution	1	**Acq Method**	THC Quant 051517 workingmm.m
Position	P2-B6	**Sample Info**	
Inj Vol	-1	**Comment**	

Sample Chromatogram



Results

Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.212	32896	27829	1.1821	100.9924
THC-COOH	THC-COOH-D9	2.312	14212	9280	1.5315	94.6635
THC	THC-D3	5.652	6720	8577	0.7835	97.2028