

B

Worklist: 2528

<u>LAB CASE</u>	<u>ITEM</u>	<u>TASK ID</u>	<u>DESCRIPTION</u>
M2018-2829	1	119889	AM 27 Blood THC Quant by LC-QQQ
M2018-2959	1	119890	AM 27 Blood THC Quant by LC-QQQ
P2018-1639	1	119895	AM 27 Blood THC Quant by LC-QQQ
P2018-1744	1	119892	AM 27 Blood THC Quant by LC-QQQ
P2018-1757	1	119893	AM 27 Blood THC Quant by LC-QQQ
P2018-1835	1	119894	AM 27 Blood THC Quant by LC-QQQ



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

TS

Extraction Date: 06/28/18
Plate lot#: 0515037

Analyst: Tamara Salazar
Plate Expiration: 09/28/18

Mobile phase A: 0.1% Formic Acid in LCMS Water
MTBE LCMS Methanol

Mobile phase B: 0.1% Formic acid in Acetonitrile
Hexane

Blank Blood Lot: 361331-1

Column: UCT Selectra DA 100 x 2.1mm 3um

Blank Urine: POC062718

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: _____

Urine Hydrolysis:

- 1. Pipette 1.5 mL urine into empty 48 well plate.
- 2. Add 250ul 1N KOH to urine samples.
- 2. Place on shaking incubator at 40 degrees for 15 mins.

Analytic:

- 1. Remove standards, plate, controls, and samples from cold storage. Allow to reach room temperature.
- 2. Pipette **1000µL blood/urine (calibrated pipette) Pipette ID: 27** 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 for blood samples. Pipette **500ul saturated phosphate buffer** for urine samples.
- 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.
Worklist path: C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528
Batch Name: 062818 THCQ TS Worklist 2528
- 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.

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- 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:

Curves limited: THC-COOH 5-250 -15



Idaho State Police Forensic Services

TS

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

Analyst: Tamara Salazar
Extraction Date: 06/28/18
Worklist Number: 2528

Reagent	Lot Number	Expiration Date	Date in Service	Date Out of Service	Initials
ToxBox THC/THC Metabolite Plate	0515037	09/28/18			
Negative Blood	361331-1		05/25/18		
Negative Urine	POC062718		06/27/18		
Methanol External Control Solution	WS020718	02/07/19	02/07/18		
Blood External Control Solution	061718	06/17/19	06/17/18		
Urine External Controls	062818	06/28/19	06/28/18		
Methyl Tert-Butyl Ether (MTBE) 99.9%	A0375555		06/26/17		
Hexanes (ACS)	101642		10/26/17		
Methanol (LCMS Grade)	177145		04/11/18		
1 N KOH	091817		09/18/18		
Saturated Phosphate Buffer pH 1.8	020118		02/01/18		
0.1% Formic Acid in Water (Mobile Phase A)	166541		06/26/17		
0.1% Formic Acid in Acetonitrile (Mobile Phase B)	176190		02/06/18		
Needle Rinse--75% LCMS MeOH in LCMS Water	052918		05/29/18		

Methanol External Control Solution (Lot: WS020718)
10 ul of 1mg/mL THC, 100 ul of 100 ug/mL THC-OH, C-THC in 9790 ul MeOH

Component	Source	Source Lot Number	Expiration Date
Methanol (LCMS)	Fisher	172516	
THC	Cerilliant	FE04231406	04/30/2019
C-THC	Cayman	0497429	02/08/2019
THC-OH	Cerilliant	FE01121503	01/31/2020
Prepared:	02/07/18		
Prepared By:	Tamara Salazar		
Expires:	02/07/19		

Blood External Control Solution (Lot: ~~0261718~~ ⁰⁶¹⁷¹⁸ TS)
100 ul of methanol external control solution was added to 9900 ul of blood.

Component	Source	Source Lot Number
Negative Blood	Hemostat	361331-1
Methanol External Control Solution		WS020718

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

Prepared:	06/17/18
Prepared by:	Tamara Salazar
Expires:	06/17/19

Urine External Control Solutions (Lot: 062818)

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

Three controls were made by adding 50ul, 100ul, and 200ul of methanol external control to 4.5mL of negative urine.

Component	Source	Source Lot Number
Negative Blood Urine 15		POC062718
Methanol External Control Solution		WS020718
Prepared:	06/28/18	
Prepared by:	Sarah Pickle	
Expires:	06/28/19	

1 N KOH (Lot: 091817)

Component	Source	Source Lot Number
Potassium Hydroxide	Fisher	034727
DI Water	-	-
Prepared:	09/18/17	
Prepared By:	Celena Shrum	

Saturated Phosphate Buffer pH 1.8 (Lot: 020118)

Component	Source	Source Lot Number
Potassium Phosphate monobasic	Fisher	L10-021-61
DI Water	-	-
Prepared:	02/01/18	
Prepared By:	Celena Shrum	

Needle Rinse (75% LCMS MeOH in LCMS Water) (Lot: 052918)

Component	Source	Source Lot Number
MeOH (LCMS Grade)	Fisher	177145
Water (LCMS Grade)	Fisher	177528
Prepared:	05/29/18	
Prepared By:	Tamara Salazar	

ISP FORENSICS - Pocatello Instrument # 59740

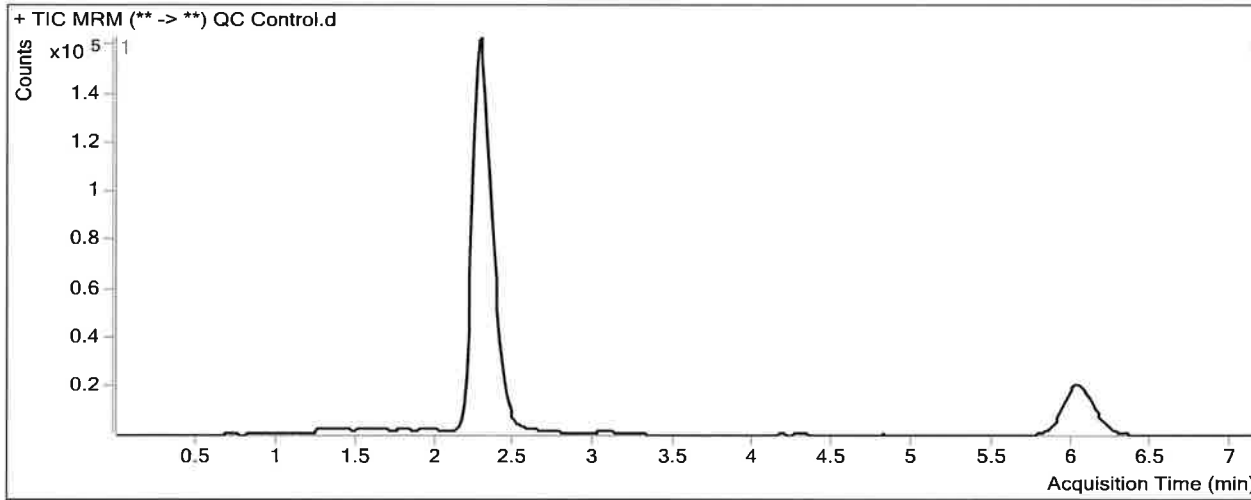
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W		
Analysis Time	7/8/2018 8:26 AM	Analyst Name	ISPUser
Report Time	7/8/2018 8:28 AM	Reporter Name	ISPUser
Last Calib Update	7/8/2018 8:26 AM	Batch State	Processed

Analysis Info

Acq Time	2018-06-28 13:55	Data File	QC Control.d
Sample Type	Sample	Sample Name	QC Control
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-H1	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	2.292	106155	934193	0.1136	9.6357
THC-COOH	THC-COOH-D9	2.392	72669	256820	0.2830	10.2043
THC	THC-D3	6.079	29751	241626	0.1231	9.8262

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ISP FORENSICS - Pocatello Instrument # 59740

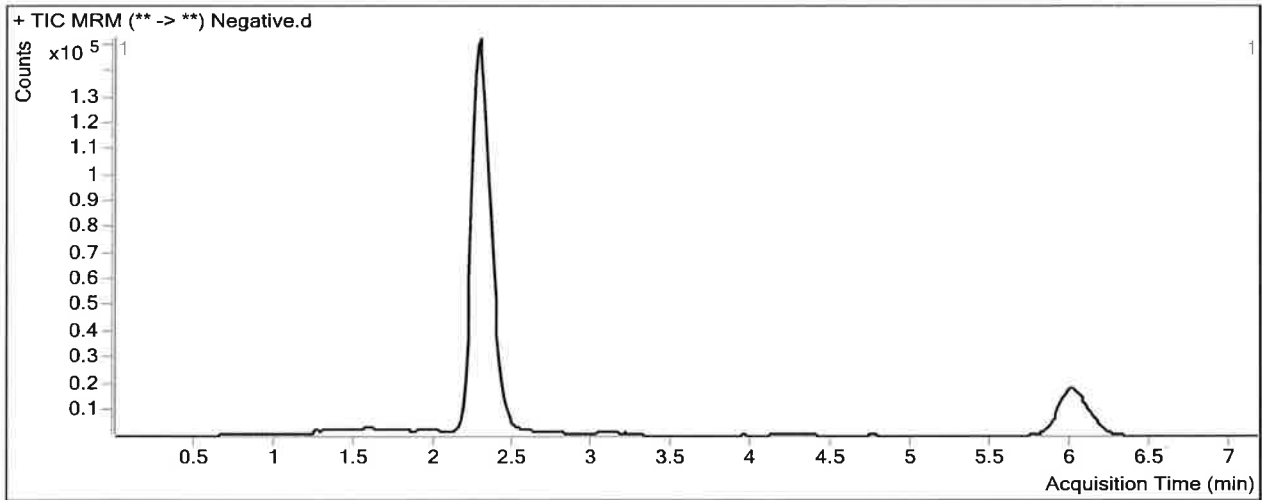
Cannabinoids Analysis Report

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Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISUser
Report Time 7/8/2018 8:28 AM **Reporter Name** ISUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 14:19 **Data File** Negative.d
Sample Type Sample **Sample Name** Negative
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-A2 **Sample Info**
Inj Vol -1 **Comment** Hemostat 361331-1

Sample Chromatogram



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ISP FORENSICS - Pocatello Instrument # 59740

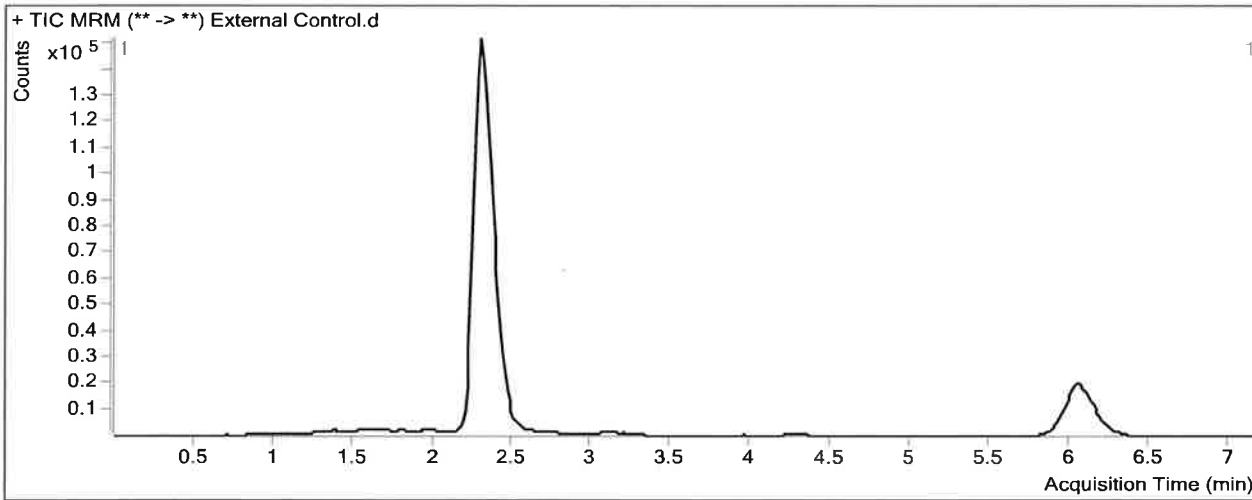
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W
Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISPUser
Report Time 7/8/2018 8:28 AM **Reporter Name** ISPUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 14:42 **Data File** External Control.d
Sample Type Sample **Sample Name** External Control
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-B2 **Sample Info**
Inj Vol -1 **Comment** Hemostat 361331-1 + WS 020718

Sample Chromatogram



Results

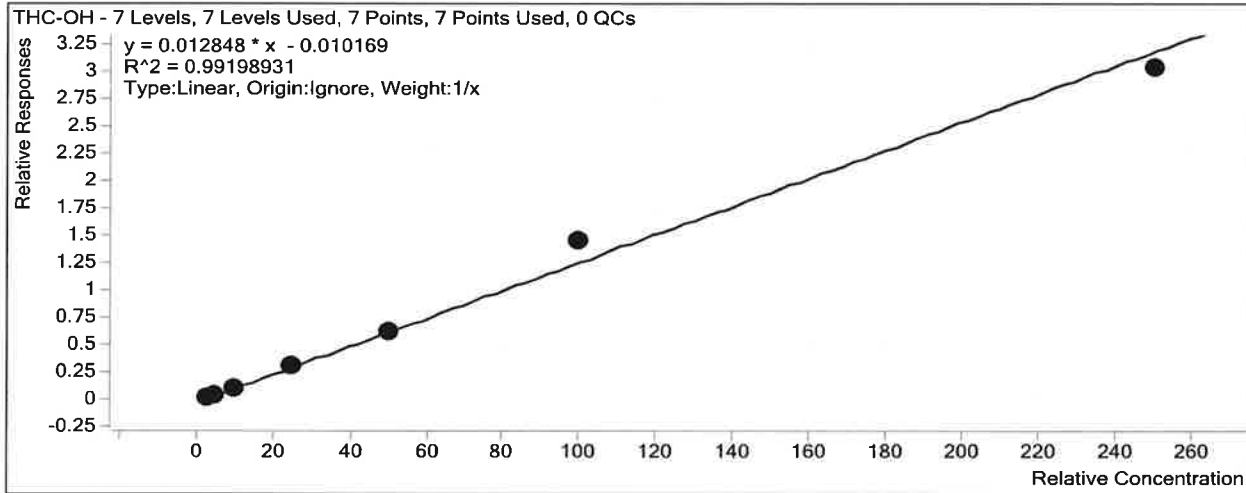
Compound	ISTD Compound	RT	Response	ISTD Resp	Resp Ratio	Final Conc
THC-OH	THC-OH-D3	2.305	85798	894768	0.0959	8.2546
THC-COOH	THC-COOH-D9	2.406	60450	255811	0.2363	8.4189
THC	THC-D3	6.106	23777	227118	0.1047	8.4158

ISP Forensics Calibration Curve Report

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Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist
2528\QuantResults\062818 THCQ TS Worklist 2528.batch.bin
Last Calib Update 7/8/2018 8:26 AM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input checked="" type="checkbox"/>	3	3.1	104.0
Cal 2	2	<input checked="" type="checkbox"/>	5	4.8	96.5
Cal 3	3	<input checked="" type="checkbox"/>	10	9.3	92.9
Cal 4	4	<input checked="" type="checkbox"/>	25	24.5	97.9
Cal 5	5	<input checked="" type="checkbox"/>	50	49.5	98.9
Cal 6	6	<input checked="" type="checkbox"/>	100	115.0	115.0
Cal 7	7	<input checked="" type="checkbox"/>	250	236.8	94.7

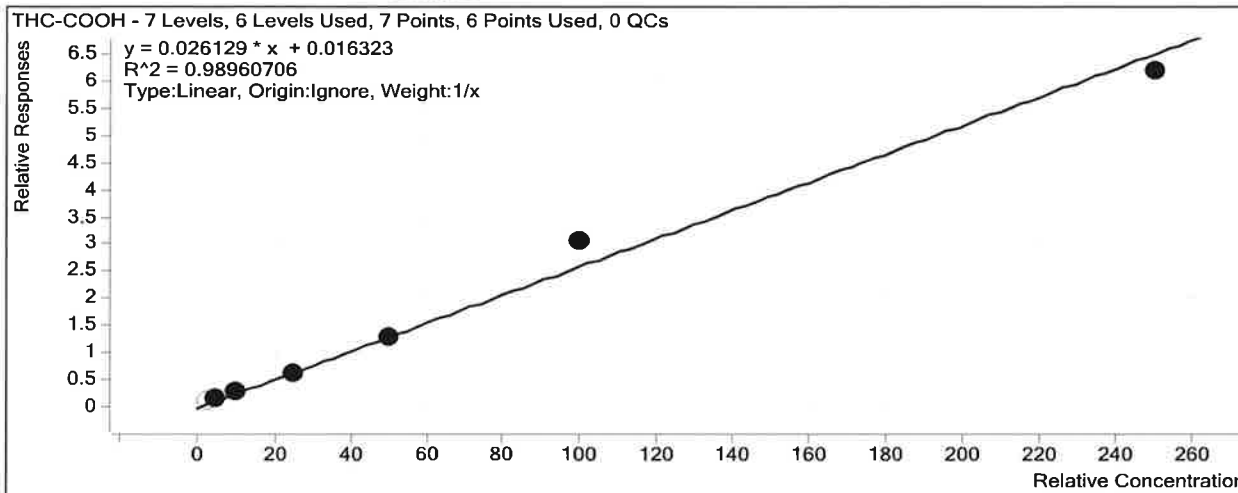
ISP Forensics Calibration Curve Report

TS

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist
2528\QuantResults\062818 THCQ TS Worklist 2528.batch.bin

Last Calib Update 7/8/2018 8:26 AM **Analyst Name** ISP TOX

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



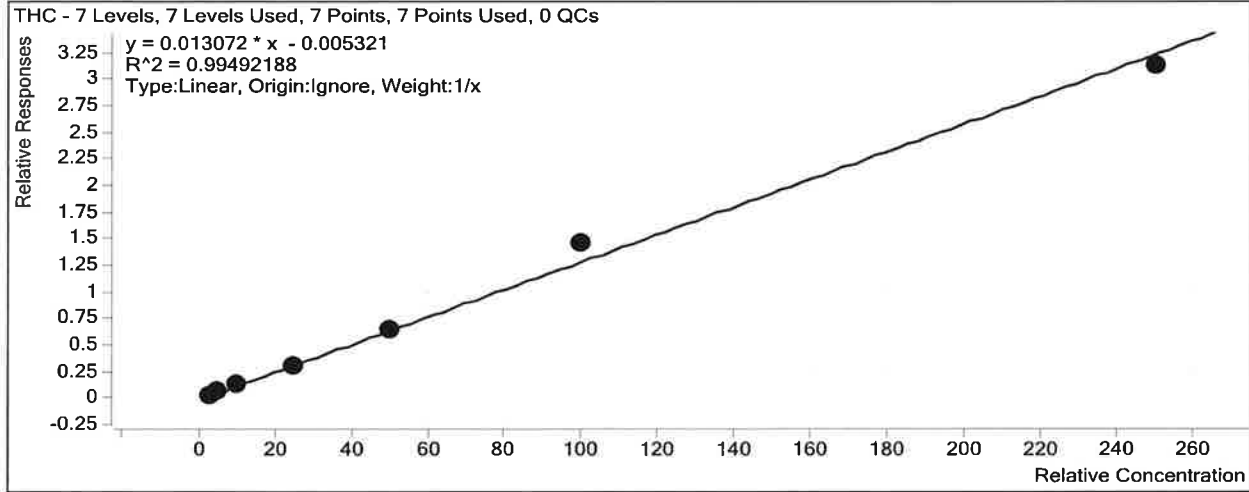
Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input type="checkbox"/>	3	4.5	150.9
Cal 2	2	<input checked="" type="checkbox"/>	5	5.0	99.6
Cal 3	3	<input checked="" type="checkbox"/>	10	9.7	96.8
Cal 4	4	<input checked="" type="checkbox"/>	25	23.9	95.8
Cal 5	5	<input checked="" type="checkbox"/>	50	48.3	96.7
Cal 6	6	<input checked="" type="checkbox"/>	100	116.6	116.6
Cal 7	7	<input checked="" type="checkbox"/>	250	236.4	94.6

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ISP Forensics Calibration Curve Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist
2528\QuantResults\062818 THCQ TS Worklist 2528.batch.bin
Last Calib Update 7/8/2018 8:26 AM **Analyst Name** ISP TOX

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



Sample	Level	Enabled	Exp Conc	Final Conc	Accuracy
Cal 1	1	<input checked="" type="checkbox"/>	3	3.0	100.0
Cal 2	2	<input checked="" type="checkbox"/>	5	5.0	100.7
Cal 3	3	<input checked="" type="checkbox"/>	10	9.7	97.2
Cal 4	4	<input checked="" type="checkbox"/>	25	23.6	94.6
Cal 5	5	<input checked="" type="checkbox"/>	50	49.8	99.7
Cal 6	6	<input checked="" type="checkbox"/>	100	111.9	111.9
Cal 7	7	<input checked="" type="checkbox"/>	250	239.9	95.9

ISP FORENSICS - Pocatello Instrument # 59740

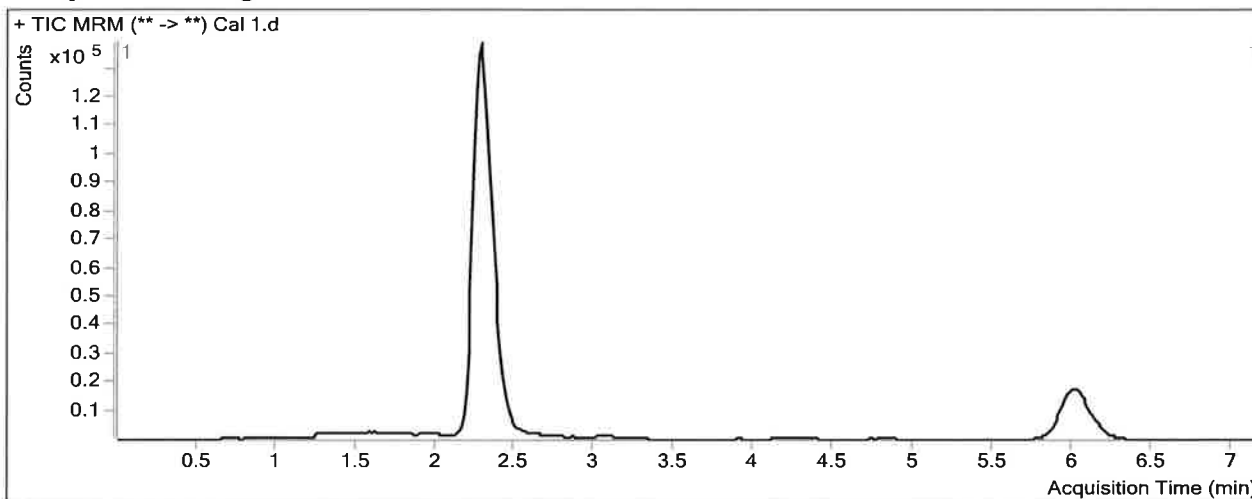
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W		
Analysis Time	7/8/2018 8:26 AM	Analyst Name	ISPUser
Report Time	7/8/2018 8:27 AM	Reporter Name	ISPUser
Last Calib Update	7/8/2018 8:26 AM	Batch State	Processed

Analysis Info

Acq Time	2018-06-28 12:20	Data File	Cal 1.d
Sample Type	Calibration	Sample Name	Cal 1
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-A1	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	2.292	26028	869888	0.0299	3.1202
THC-COOH	THC-COOH-D9	2.379	33086	245771	0.1346	4.5274
THC	THC-D3	6.025	7720	227679	0.0339	3.0008

TS

ISP FORENSICS - Pocatello Instrument # 59740

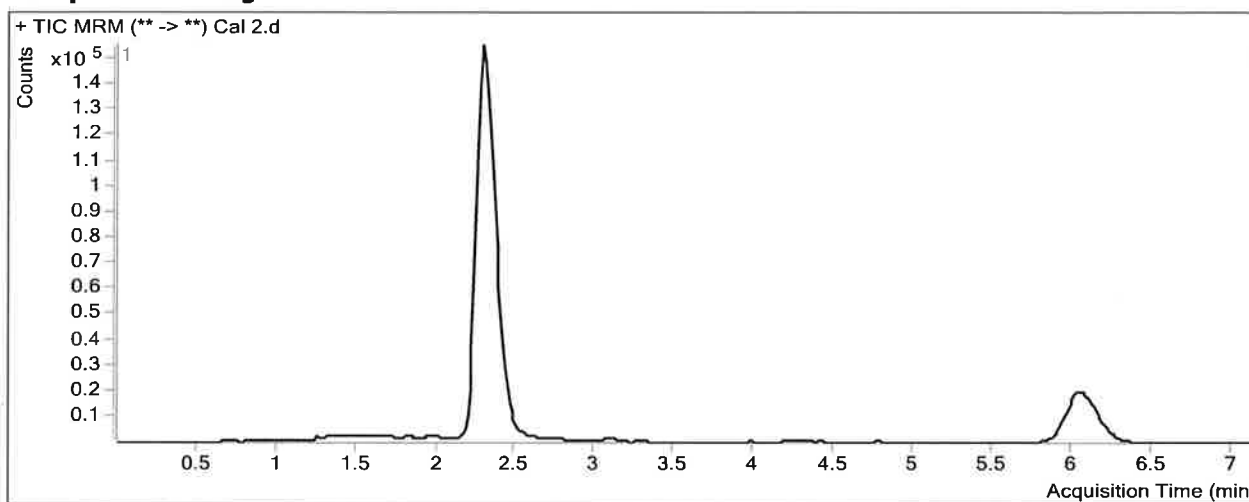
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W
Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISPUser
Report Time 7/8/2018 8:27 AM **Reporter Name** ISPUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 12:32 **Data File** Cal 2.d
Sample Type Calibration **Sample Name** Cal 2
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-B1 **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	2.305	50135	967135	0.0518	4.8262
THC-COOH	THC-COOH-D9	2.406	39326	268564	0.1464	4.9792
THC	THC-D3	6.079	15330	253340	0.0605	5.0362

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ISP FORENSICS - Pocatello Instrument # 59740

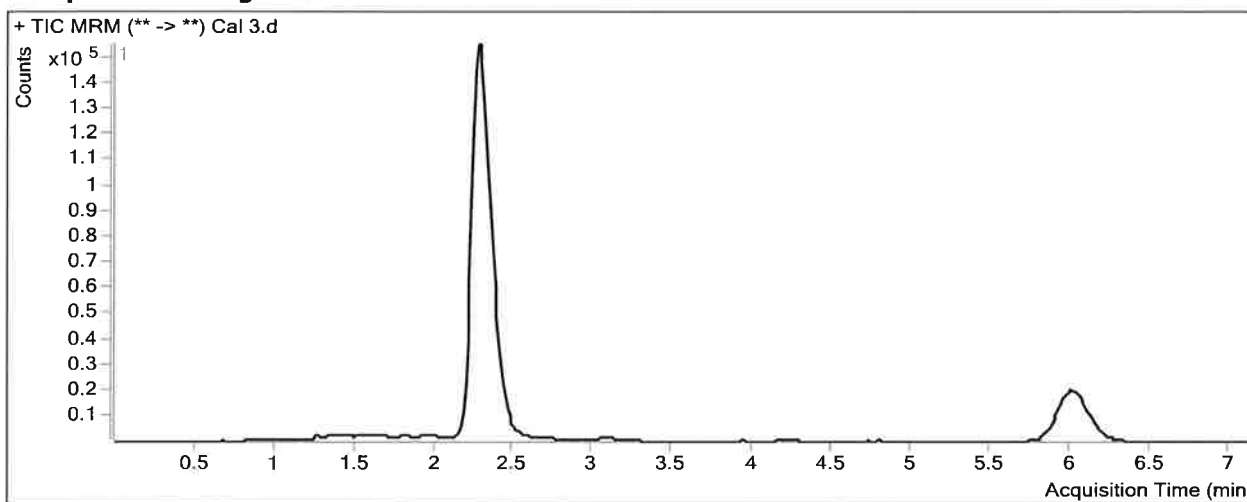
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W
Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISPUser
Report Time 7/8/2018 8:28 AM **Reporter Name** ISPUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 12:44 **Data File** Cal 3.d
Sample Type Calibration **Sample Name** Cal 3
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-C1 **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	2.292	96721	885417	0.1092	9.2936
THC-COOH	THC-COOH-D9	2.392	66719	247836	0.2692	9.6781
THC	THC-D3	6.052	28303	232587	0.1217	9.7161

TS

ISP FORENSICS - Pocatello Instrument # 59740

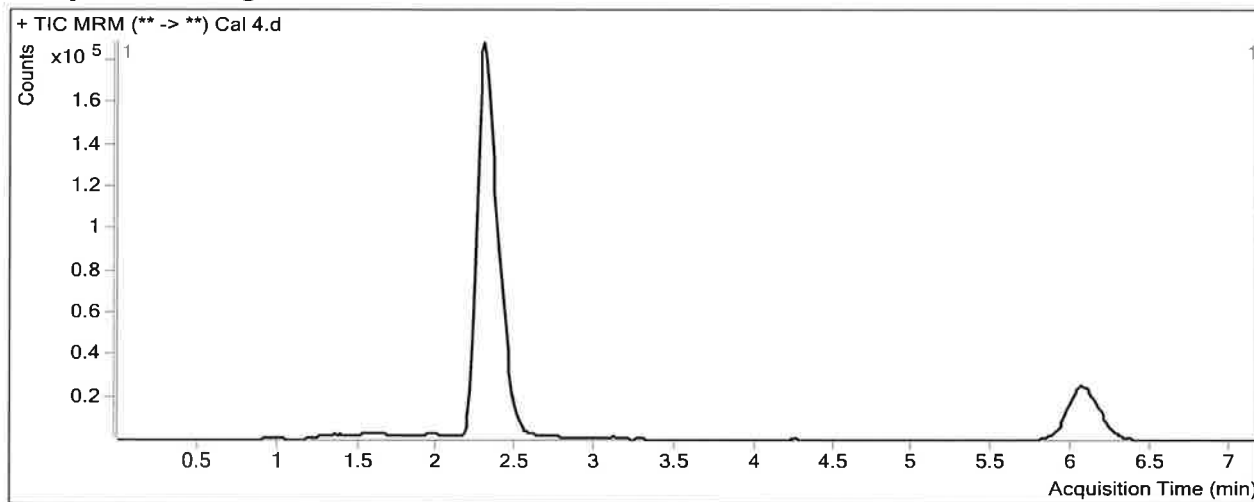
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W		
Analysis Time	7/8/2018 8:26 AM	Analyst Name	ISPUser
Report Time	7/8/2018 8:28 AM	Reporter Name	ISPUser
Last Calib Update	7/8/2018 8:26 AM	Batch State	Processed

Analysis Info

Acq Time	2018-06-28 12:56	Data File	Cal 4.d
Sample Type	Calibration	Sample Name	Cal 4
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-D1	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	2.305	282231	927526	0.3043	24.4743
THC-COOH	THC-COOH-D9	2.406	158563	247007	0.6419	23.9428
THC	THC-D3	6.079	73127	240753	0.3037	23.6433

TS

ISP FORENSICS - Pocatello Instrument # 59740

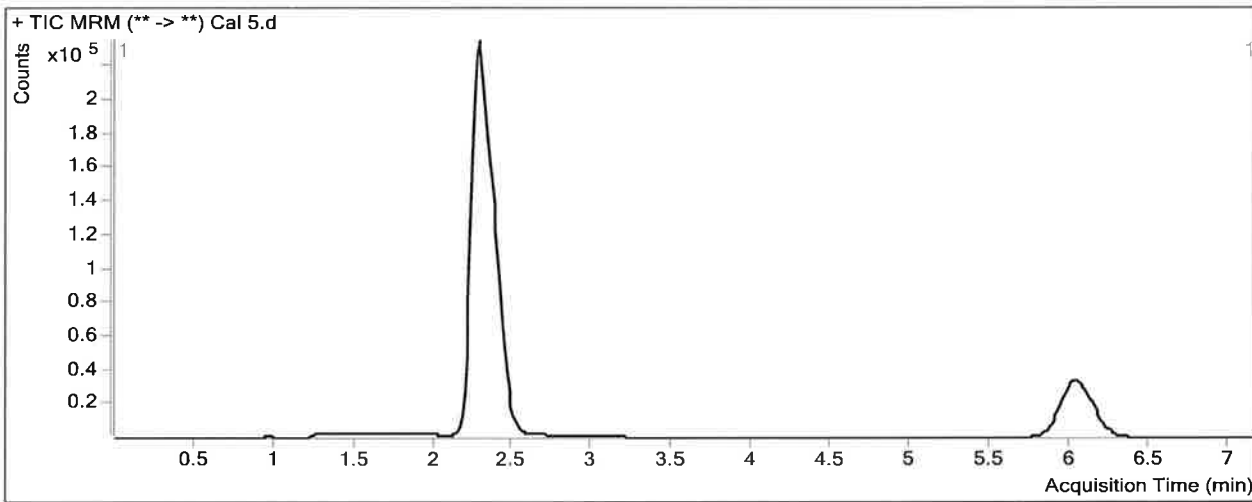
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W
Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISPUser
Report Time 7/8/2018 8:28 AM **Reporter Name** ISPUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 13:08 **Data File** Cal 5.d
Sample Type Calibration **Sample Name** Cal 5
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-E1 **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	2.292	575608	920633	0.6252	49.4540
THC-COOH	THC-COOH-D9	2.392	320184	250309	1.2792	48.3297
THC	THC-D3	6.052	156658	242453	0.6461	49.8361

TS

ISP FORENSICS - Pocatello Instrument # 59740

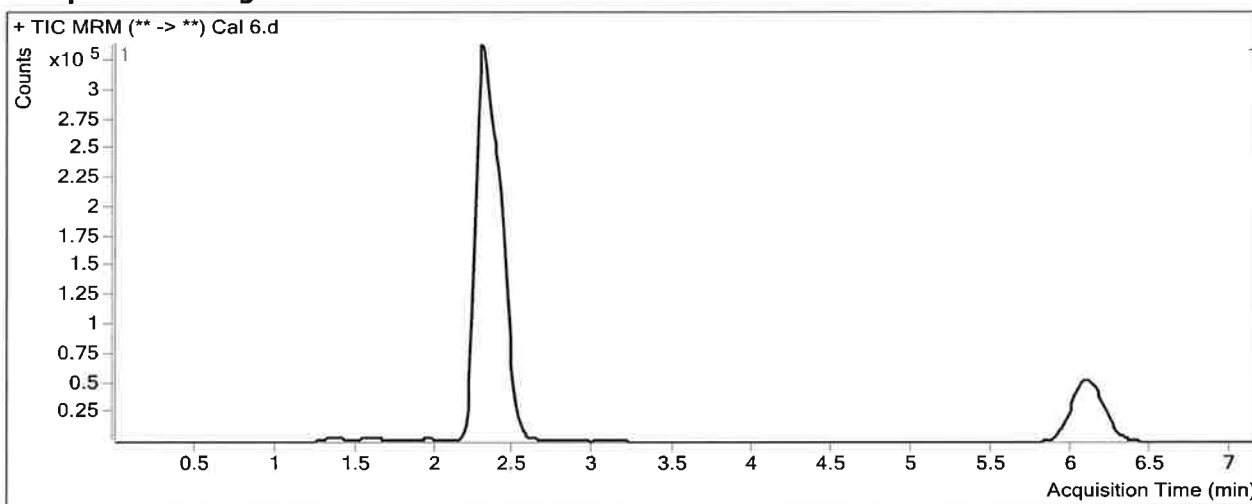
Cannabinoids Analysis Report

Batch Data Path	C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W		
Analysis Time	7/8/2018 8:26 AM	Analyst Name	ISPUser
Report Time	7/8/2018 8:28 AM	Reporter Name	ISPUser
Last Calib Update	7/8/2018 8:26 AM	Batch State	Processed

Analysis Info

Acq Time	2018-06-28 13:19	Data File	Cal 6.d
Sample Type	Calibration	Sample Name	Cal 6
Dilution	1	Acq Method	THC Quant 051517 workingmm.m
Position	P1-F1	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	2.305	1291897	880473	1.4673	114.9916
THC-COOH	THC-COOH-D9	2.419	699493	228317	3.0637	116.6255
THC	THC-D3	6.106	337708	231725	1.4574	111.8945

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ISP FORENSICS - Pocatello Instrument # 59740

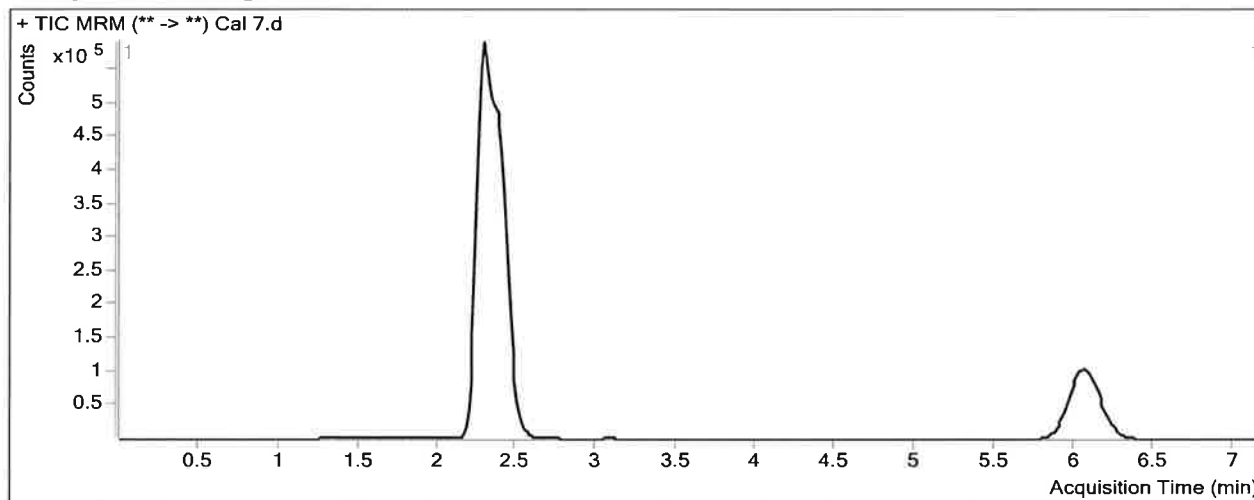
Cannabinoids Analysis Report

Batch Data Path C:\MassHunter\Data\2018\THC Quant\062818 THC Quant TS Worklist 2528\QuantResults\062818 THCQ TS W
Analysis Time 7/8/2018 8:26 AM **Analyst Name** ISPUser
Report Time 7/8/2018 8:28 AM **Reporter Name** ISPUser
Last Calib Update 7/8/2018 8:26 AM **Batch State** Processed

Analysis Info

Acq Time 2018-06-28 13:31 **Data File** Cal 7.d
Sample Type Calibration **Sample Name** Cal 7
Dilution 1 **Acq Method** THC Quant 051517 workingmm.m
Position P1-G1 **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	2.292	2829701	933025	3.0328	236.8402
THC-COOH	THC-COOH-D9	2.392	1494768	241306	6.1945	236.4446
THC	THC-D3	6.052	774086	247288	3.1303	239.8731