

Worklist: 4066

<u>LAB CASE</u>	<u>ITEM</u>	<u>ITEM TYPE</u>	<u>DESCRIPTION</u>
M2020-0260	1	BCK	AM 26 Blood THC Screen by LC-QQQ



**Idaho State Police
Forensic Services
Toxicology Discipline**

Request for Departure from an Analytical Method

Date of Request

01/13/2020

Forensic Scientist

Celena Shrum

Analytical Methods

Toxicology AM #25, Toxicology AM #26/27, and AM #28

Deviation

The expiration dates listed for the current batch of PinPoint ToxBox extraction plates are as follows:

- *MDS (batch IDP-107-190725)- Expiration is 1/25/2020
- *THC (batch IDP-108-190716)- Expiration is 1/16/2020
- *MDQ P1 (batch IDP-111-190729)- Expiration is 1/29/2020
- *MDQ P2 (batch IDP-112-190730)- Expiration is 1/30/2020

I am issuing a deviation to allow for the use of the remaining plates of these batches. The controls will be used to evaluate if the plate is working as intended. In addition, at least one external control must be included for each run.

Celena Shrum

Date: 01/13/2020

Celena Shrum

Toxicology Discipline Lead

AM# 26: THC and Metabolites Screen in Blood by LC-MS/MS

Extraction Date: 03/06/20

Analyst: Sarah Pickle

Plate lot#: IDP-108-190716

Plate Expiration: 01/16/20

Mobile phase A: 10mM Ammonium Formate
0.1% Formic Acid in Water

Mobile phase B: 0.1% Formic acid in MeOH
MTBE Hexane

Blank Blood Lot: Hemostat 445283-3

Column: Phenomenex Phenyl Hexyl (4.6x50mm: 2.6 um)

LCMS-QQ 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: 3382167**
- 3. Place on shaking incubator at ambient temp., 900 rpm for 15 minutes. *Shaker ID: 067105*
- 4. Pipette **500 µL 0.1% formic acid** in wells of analytical plate.
- 5. Place on shaking incubator at ambient temp., 900 rpm for 15 minutes.
- 6. Transfer **800 µL of blood+base** 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 **2.25 mL MTBE** (add in 3 increments of 750 µL).
- 10. Wait 5 minutes.
- 11. Apply positive pressure for approx. 10-15 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. 10-15 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.
SPE Dry ID: 067103
- 16. Reconstitute in **100 µL 100% LCMS MeOH** and heat seal plate with foil. Place in autosampler and run worklist.

Post-Analytic

- 1. Create batch and process data.

Worklist path: D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP

Batch Name: THCS SP

- 2. Calculated sample concentration of 3 ng/mL or greater for THC and THC-OH, a calculated sample concentration of 10 ng/mL or greater for Carboxy-THC.
- 3. Retention time within +/- 2% or +/-0.100 min whichever is greater of the average retention time of the calibrators.
- 4. Did all QCs pass for each analyte? Y / N
- 5. Central File Packet to include: LIMS Worklist, Method Checklist, Calibration and Control Reports

COMMENTS: *Due to differences in the AM 26 (worklist 3998) screen and AM 27 (worklist 4020) confirmation results, M2020-0260 was re-extracted and ran with this worklist.*



Idaho State Police Forensic Services

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AM #26 Blood THC and Metabolites Screen by LCMS-QQQ and AM #27 Quantitative Analysis of THC and Metabolites in Blood by LCMS-QQQ

Methanol External Control Solution (Lot: WS011620)

10 μL of 1mg/mL THC, 100 μL of 100 $\mu\text{g/mL}$ THC-OH, C-THC in 9790 μL MeOH
Approximate concentration 1 $\mu\text{g/mL}$.

Component	Source	Source Lot Number	Expiration Date
Methanol (LCMS)	Fisher	193941	
THC	Cerilliant	FE09101501	11/30/2020
C-THC	Cerilliant	FE07171501	09/30/2020
THC-OH	Cerilliant	FE07221601	07/31/2021
Prepared:	01/16/2020		
Prepared By:	Tamara Salazar		
Expires:	09/30/2020		

Blood External Control Solution (Lot: 021320)

200 μL of methanol external control solution was added to 9800 μL of blood.
Approximately 20 ng/mL of each compound.

Component	Source	Source Lot Number
Negative Blood	Hemostat	445283-3
Methanol External Control Solution	-	WS011620
Prepared:	02/13/2020	
Prepared by:	Celena Shrum	
Expires:	09/30/2020	

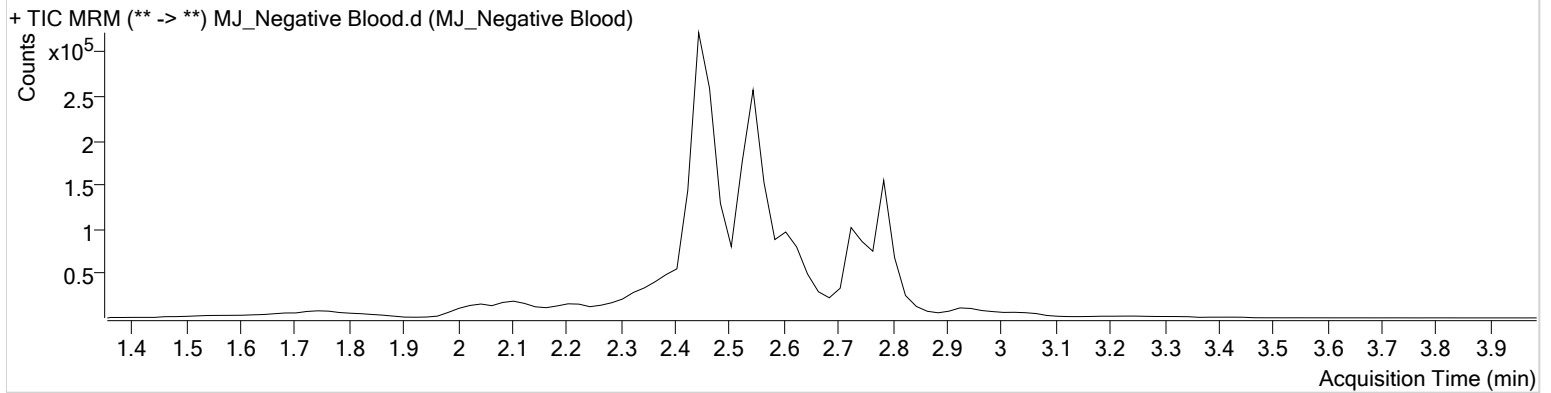
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Negative Blood.d
Type	Sample	Sample	MJ_Negative Blood
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-H5	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:52:46 AM		
Sample Info.			

Sample Chromatogram



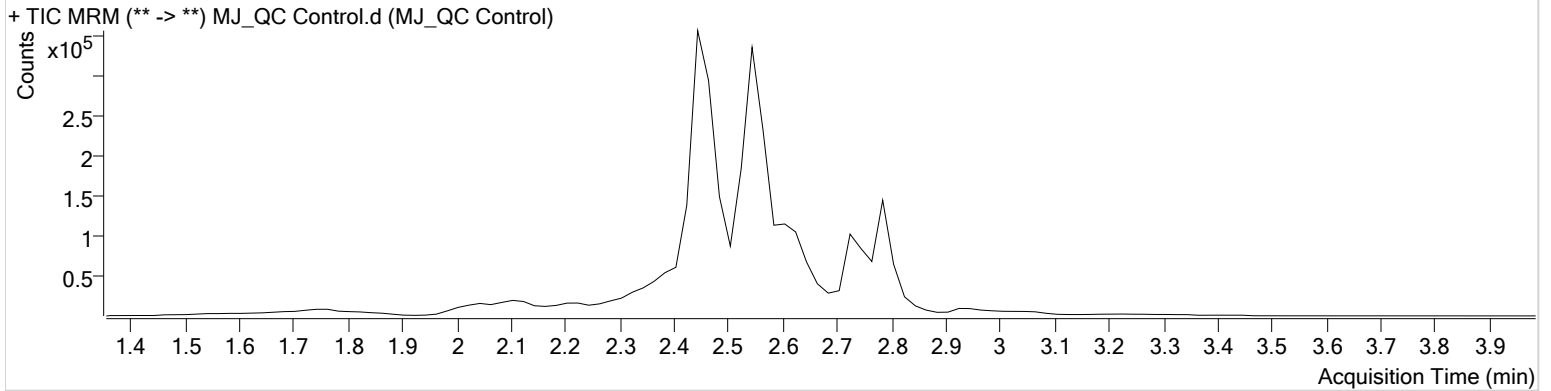
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_QC Control.d
Type	Sample	Sample	MJ_QC Control
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-A6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:39:42 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.799	5709	199765	3.2819 ng/ml
THC-COOH	2.545	92452	444742	12.4735 ng/ml
THC-OH	2.451	47678	964219	4.2862 ng/ml

AM #26 Cannabinoids Screen Results

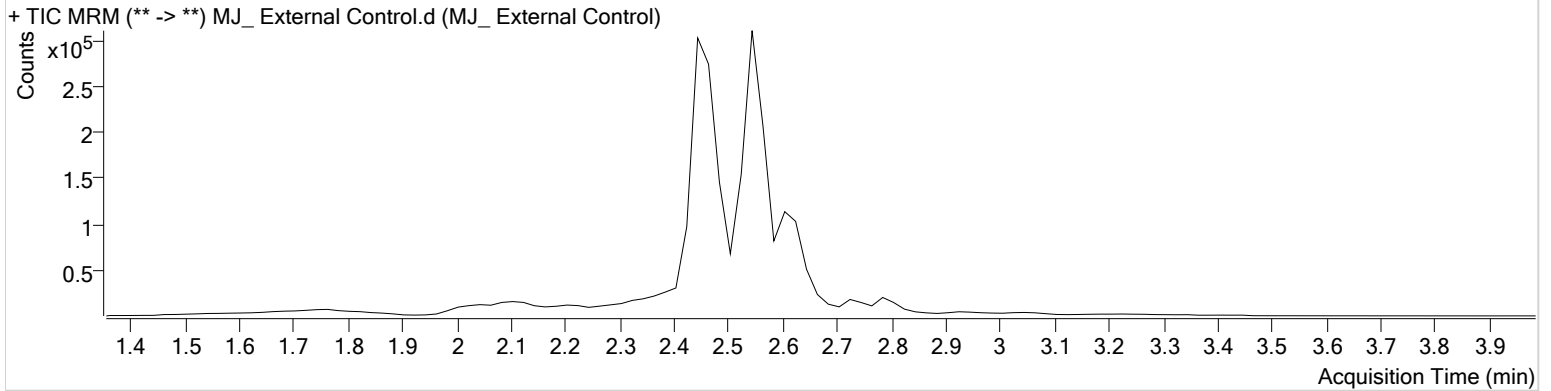


Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_ External Control.d
Type	Sample	Sample	MJ_ External Control
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-G5	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:59:18 AM		

Sample Info.

Sample Chromatogram



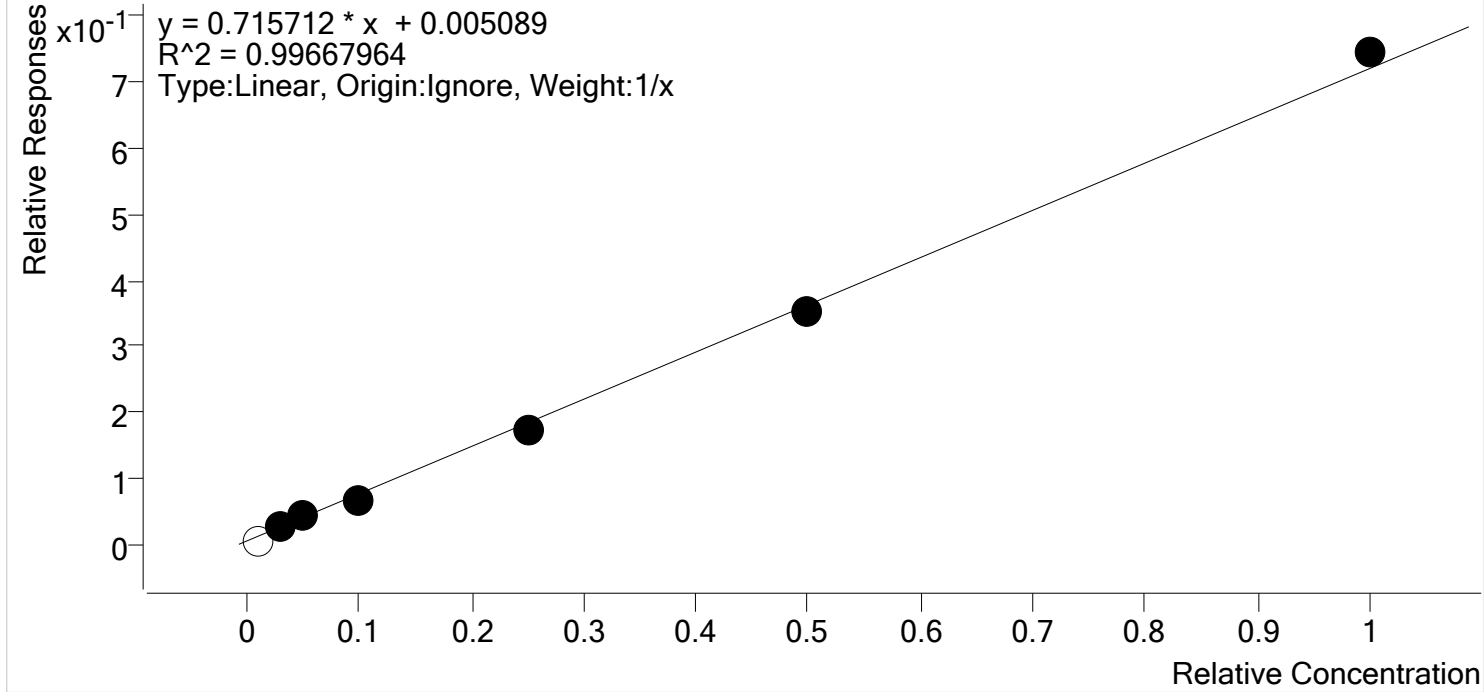
Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.799	3276	29446	14.8320 ng/ml
THC-COOH	2.545	119742	379821	18.4947 ng/ml
THC-OH	2.471	173765	718338	19.3576 ng/ml



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Last Cal. Update 3/6/2020 12:19 PM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-d3

THC - 7 Levels, 6 Levels Used, 7 Points, 6 Points Used, 0 QCs

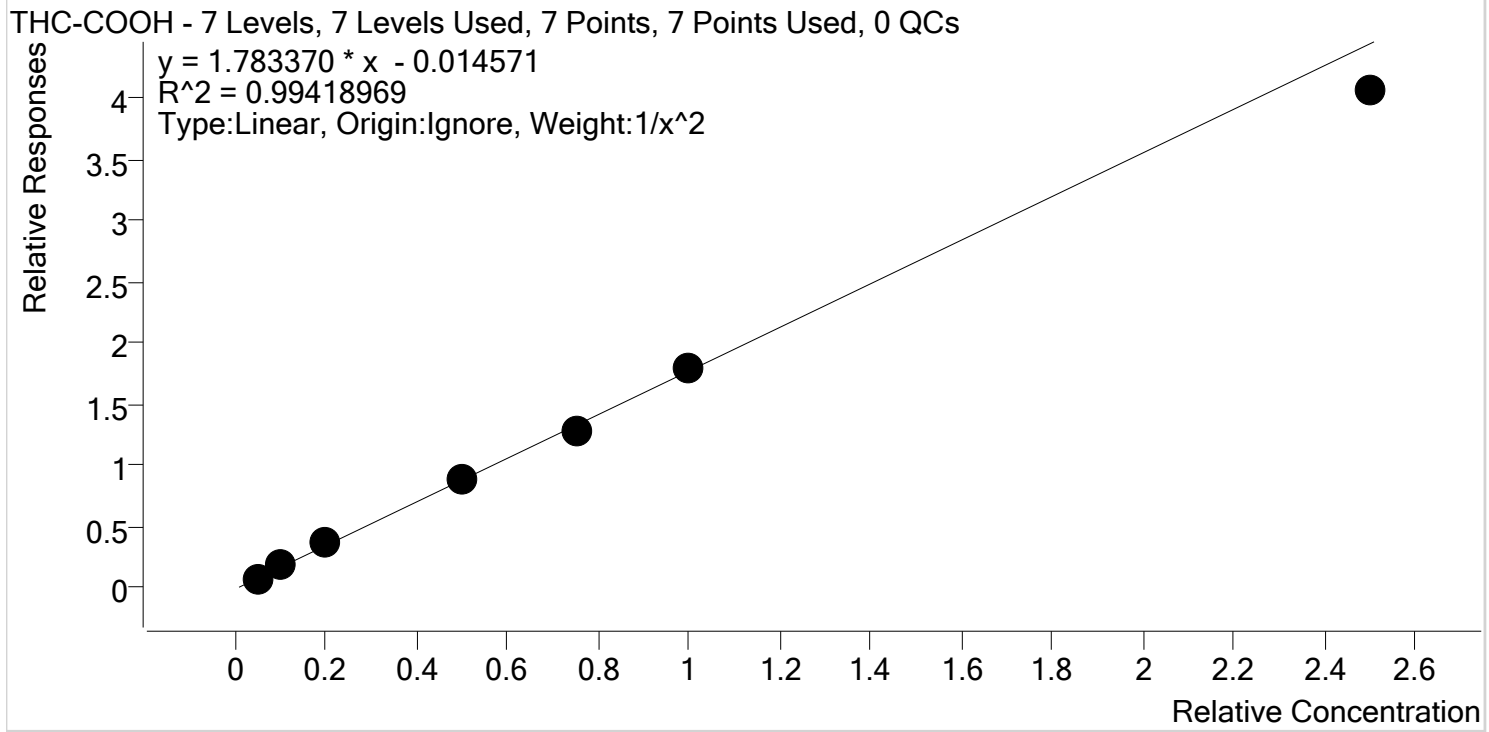


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	x	1.0	0.1	5.8
MJ_Cal 2	2	✓	3.0	3.0	100.5
MJ_Cal 3	3	✓	5.0	5.8	115.5
MJ_Cal 4	4	✓	10.0	8.9	89.3
MJ_Cal 5	5	✓	25.0	23.7	94.9
MJ_Cal 6	6	✓	50.0	48.2	96.4
MJ_Cal 7	7	✓	100.0	103.3	103.3



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Last Cal. Update 3/6/2020 12:19 PM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-d9

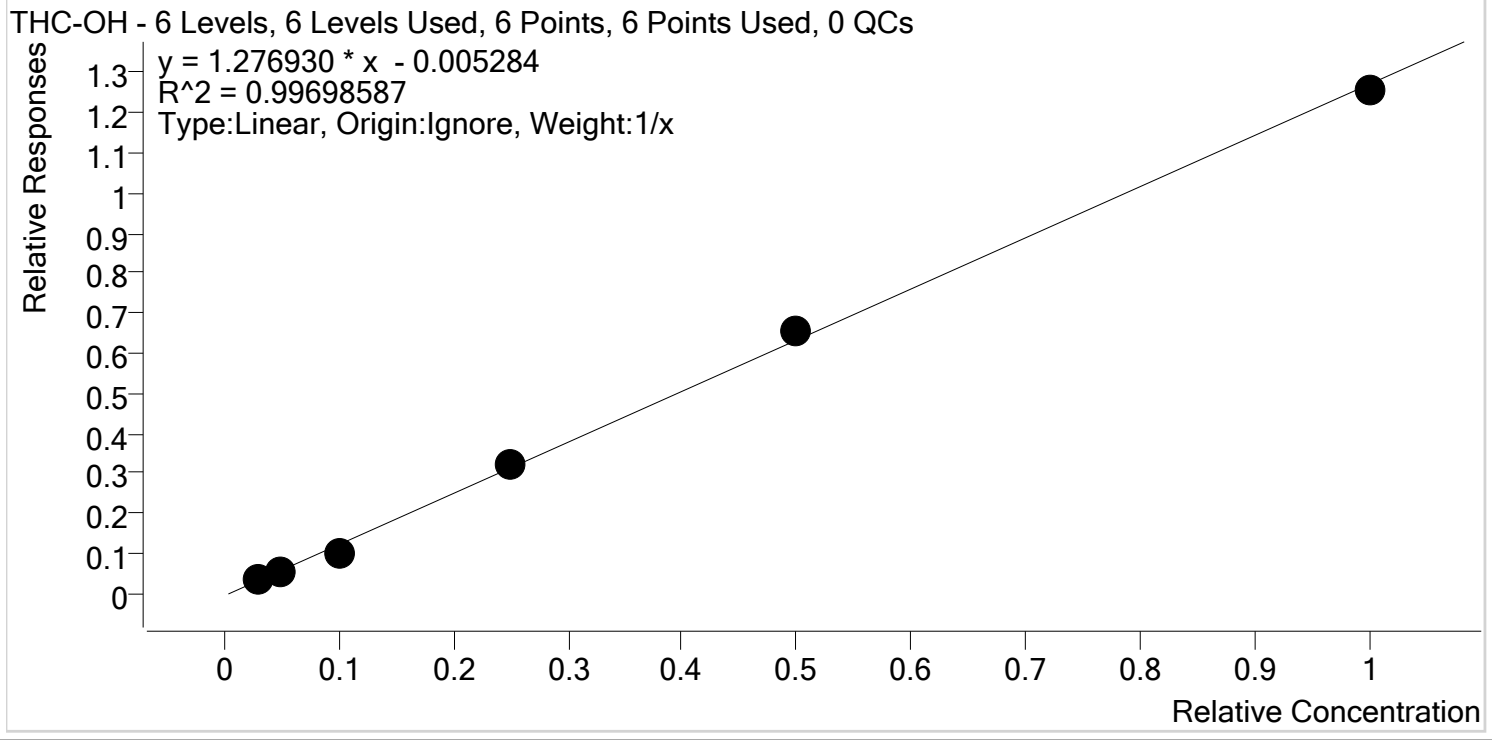


Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 1	1	✓	5.0	4.7	94.7
MJ_Cal 2	2	✓	10.0	10.7	106.8
MJ_Cal 3	3	✓	20.0	21.7	108.7
MJ_Cal 4	4	✓	50.0	49.9	99.8
MJ_Cal 5	5	✓	75.0	72.8	97.0
MJ_Cal 6	6	✓	100.0	101.5	101.5
MJ_Cal 7	7	✓	250.0	228.5	91.4



AM #26 Cannabinoids Screen Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Last Cal. Update 3/6/2020 12:19 PM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-d3



Sample	Level	Enabled	Expected Concentration	Final Concentration	Accuracy
MJ_Cal 2	2	✓	3.0	3.4	113.4
MJ_Cal 3	3	✓	5.0	4.8	96.8
MJ_Cal 4	4	✓	10.0	8.4	84.0
MJ_Cal 5	5	✓	25.0	25.9	103.5
MJ_Cal 6	6	✓	50.0	51.8	103.6
MJ_Cal 7	7	✓	100.0	98.7	98.7

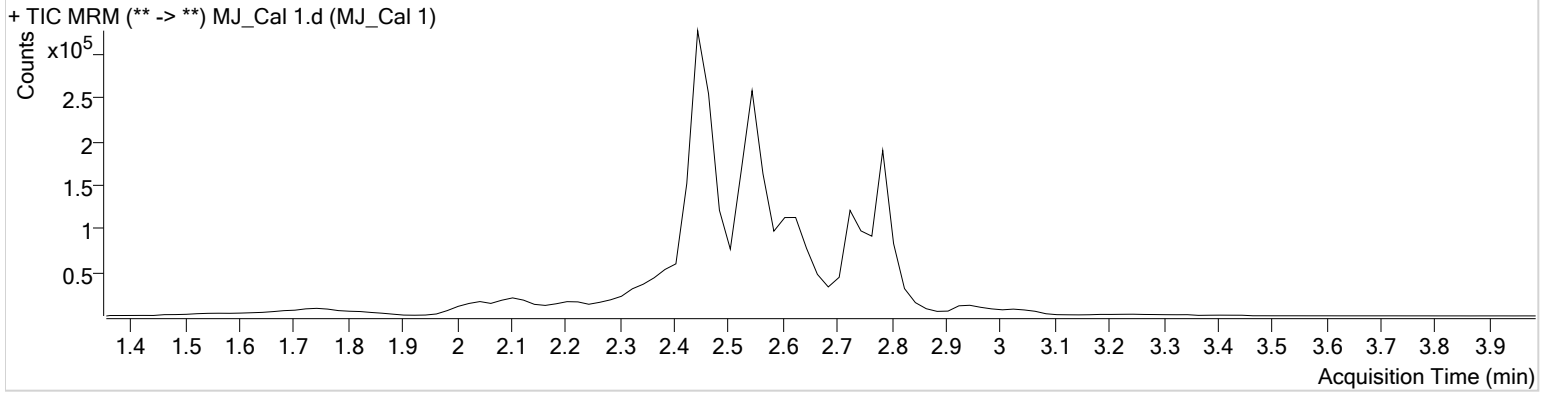
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 1.d
Type	Cal	Sample	MJ_Cal 1
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-B6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 10:47:15 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC-COOH	2.545	27000	386287	4.7364 ng/ml Low

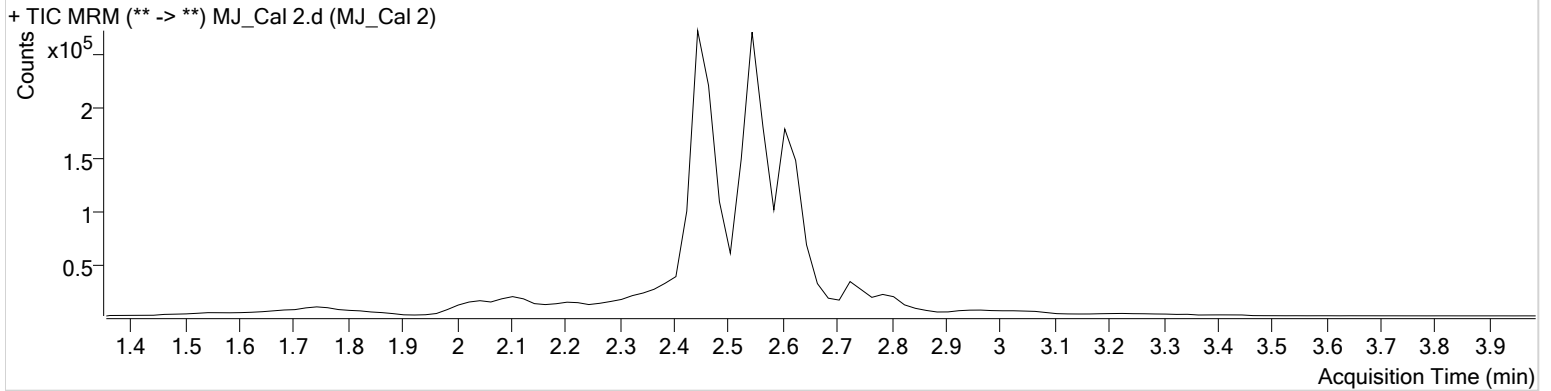
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 2.d
Type	Cal	Sample	MJ_Cal 2
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-C6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 10:53:58 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.819	777	29147	3.0141 ng/ml
THC-COOH	2.545	66599	378625	10.6803 ng/ml
THC-OH	2.451	26702	699742	3.4023 ng/ml

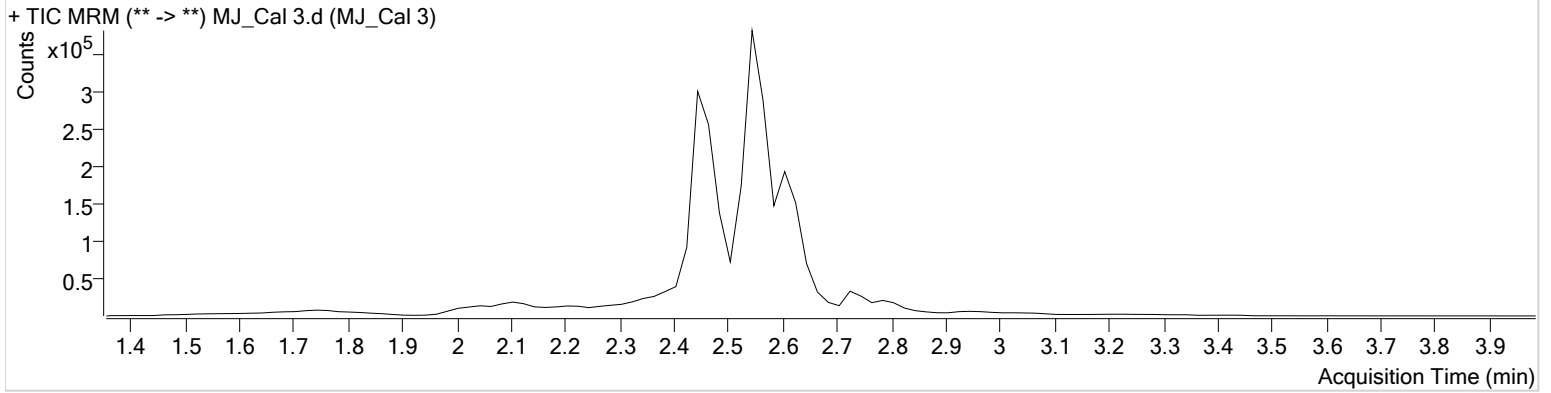
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 3.d
Type	Cal	Sample	MJ_Cal 3
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-D6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:00:30 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.819	1203	25906	5.7747 ng/ml
THC-COOH	2.545	155931	417697	21.7499 ng/ml
THC-OH	2.451	44214	781875	4.8423 ng/ml

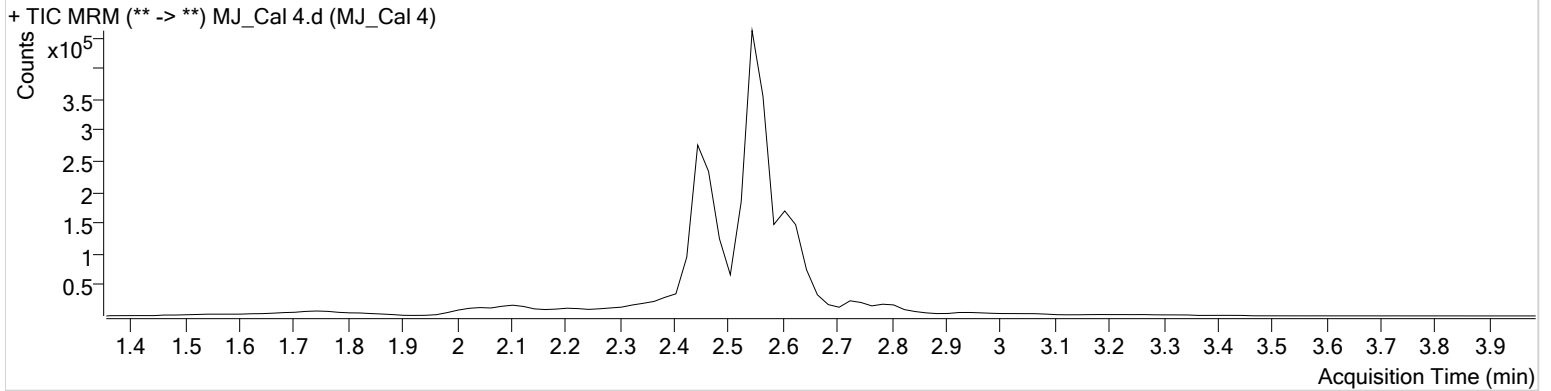
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 4.d
Type	Cal	Sample	MJ_Cal 4
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-E6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:07:02 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.819	1769	25633	8.9342 ng/ml
THC-COOH	2.545	294025	335965	49.8908 ng/ml
THC-OH	2.451	75888	744507	8.3963 ng/ml

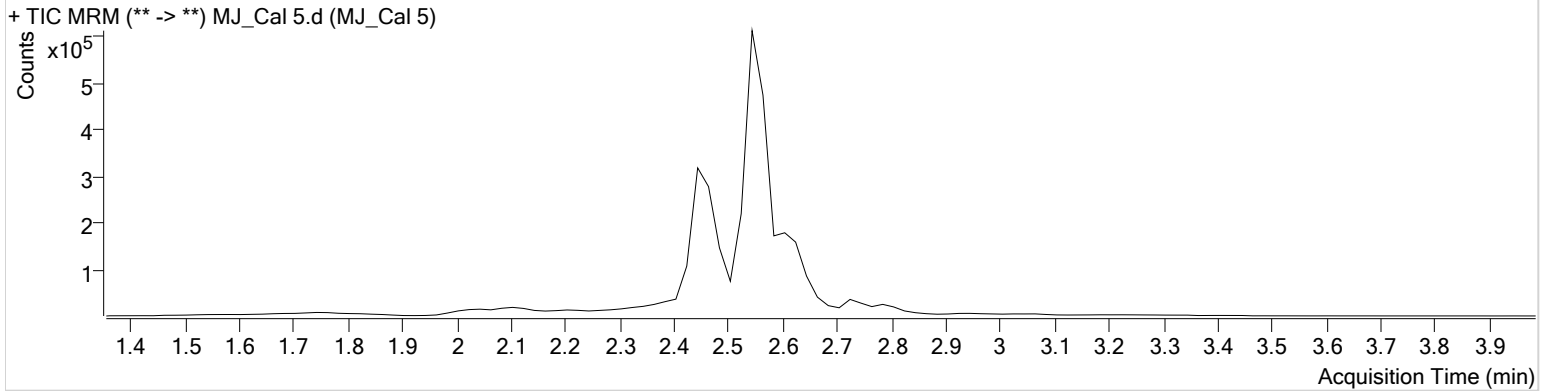
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 5.d
Type	Cal	Sample	MJ_Cal 5
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-F6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:13:34 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.799	5176	29586	23.7346 ng/ml
THC-COOH	2.545	449893	350557	72.7802 ng/ml
THC-OH	2.471	222386	684045	25.8737 ng/ml

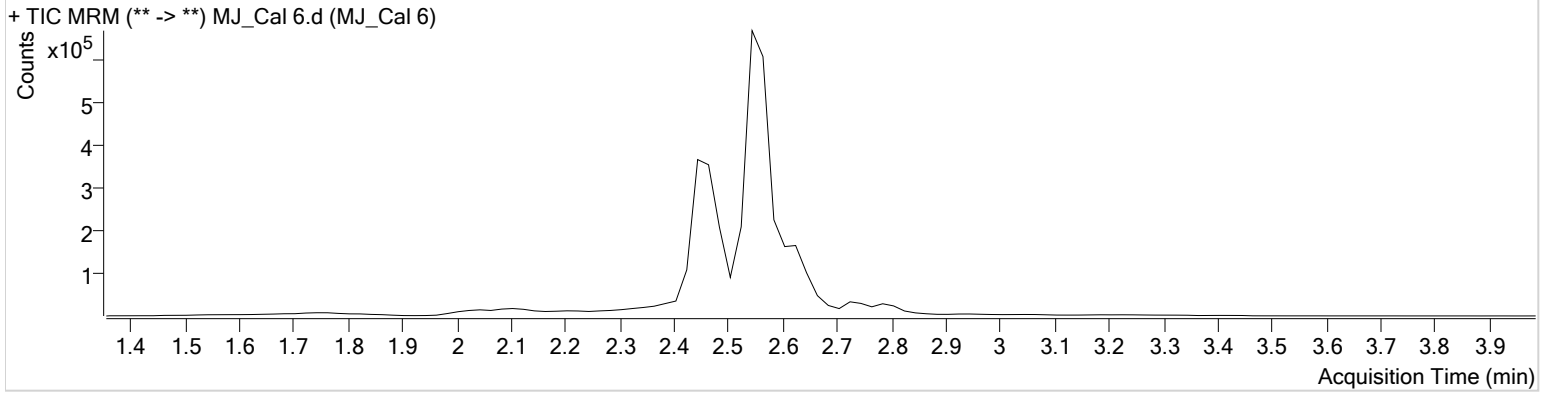
AM #26 Cannabinoids Screen Results



Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 6.d
Type	Cal	Sample	MJ_Cal 6
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-G6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:20:06 AM		

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.799	11429	32639	48.2130 ng/ml
THC-COOH	2.545	603455	336090	101.4982 ng/ml
THC-OH	2.471	476259	725816	51.8004 ng/ml

AM #26 Cannabinoids Screen Results

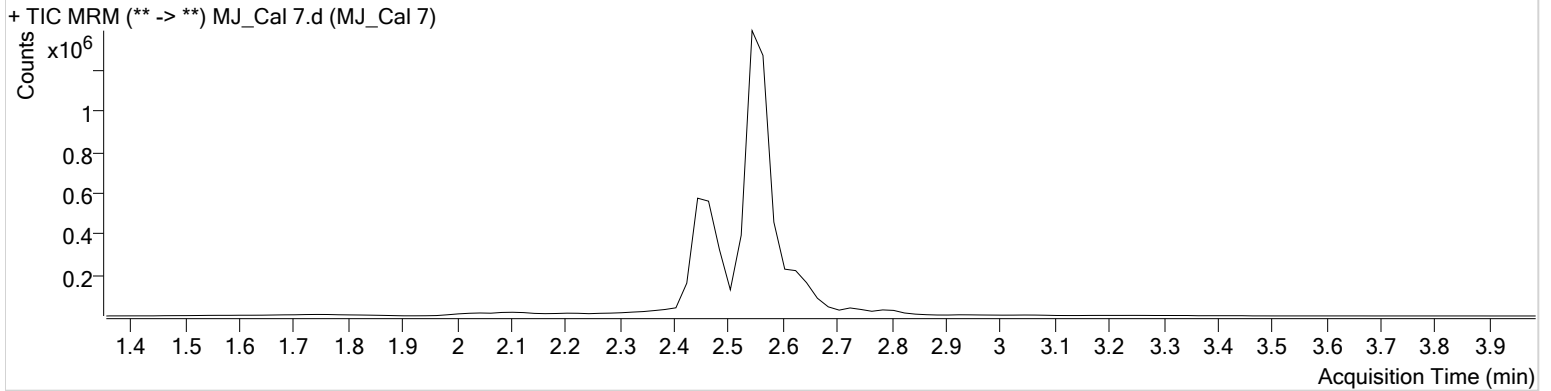


Batch results D:\MassHunter\Data\2020\AM 25-26\030620 AM 26 SP\QuantResults\THCS SP.batch.bin
Calibration Last Update 3/6/2020 12:19:17 PM

Instrument	Falco	Data File	MJ_Cal 7.d
Type	Cal	Sample	MJ_Cal 7
Acq. Method	am 26 test.m	Operator	Sarah Pickle
Sample Position	P3-H6	Comment	
Injection Volume	10		
Acq. Date-Time	3/6/2020 11:26:38 AM		

Sample Info.

Sample Chromatogram



Name	RT	Resp.	ISTD Resp.	Final Conc.
THC	2.799	19119	25676	103.3293 ng/ml
THC-COOH	2.545	1314459	323724	228.5002 ng/ml
THC-OH	2.471	1013385	807573	98.6849 ng/ml