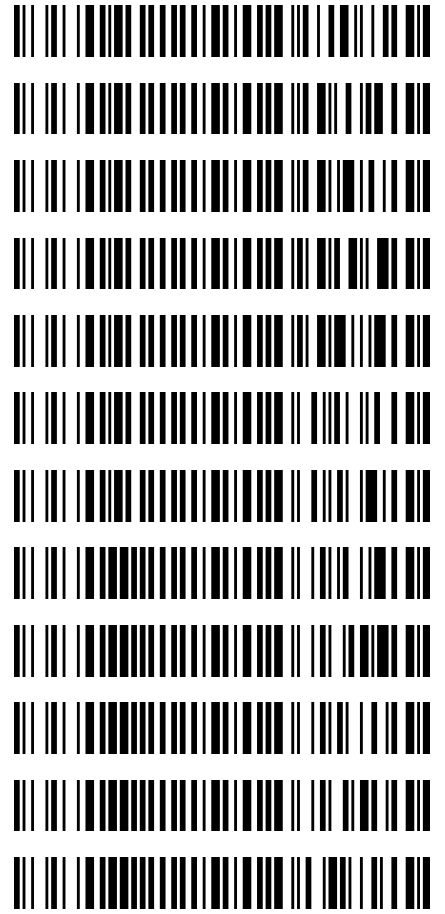


REVIEWED
By Tamara Salazar at 2:20 pm, Feb 24, 2020

§ 2/21/2020

Worklist: 4020

| <u>LAB CASE</u> | <u>ITEM</u> | <u>ITEM TYPE</u> | <u>DESCRIPTION</u> |
|-----------------|-------------|------------------|---------------------------------|
| M2020-0146 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0249 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0260 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0297 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0313 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0498 | 2 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| M2020-0525 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| P2020-0405 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| P2020-0406 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| P2020-0408 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| P2020-0409 | 1 | BCK | AM 27 Blood THC Quant by LC-QQQ |
| P2020-0459 | 1 | BCK | AM 27 Blood THC Quant 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# 27: Quantitation of THC and Metabolites in Blood by LC-MS/MS

Extraction Date: 2/19/20
 Plate lot#: IDP-108-190716

Analyst: Sarah Pickle
 Plate Expiration: 01/16/20

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: Hemostat 445283-3
LCMS-QQQ ID: 069901

Column: UCT Selectra DA 100 x 2.1mm 3um

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., 900rpm for 15 minutes. *Shaker ID: 067105*
- 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) 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. 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.
SPE Dry ID: 067103
- 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. 021920 AM 27 28 CS SP
 Worklist path: D:\MassHunter\Data\2020\AM 27-28\AM 27 28 CS SP ^{2/24/20} Batch Name: THCQ SP
- 2. Make any necessary integration changes, Curve weighting of Linear 1/x with r² 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: *Curves limited: THC 3-100, COOH 10-250, THC-OH 3-100*



Idaho State Police Forensic Services

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 | |

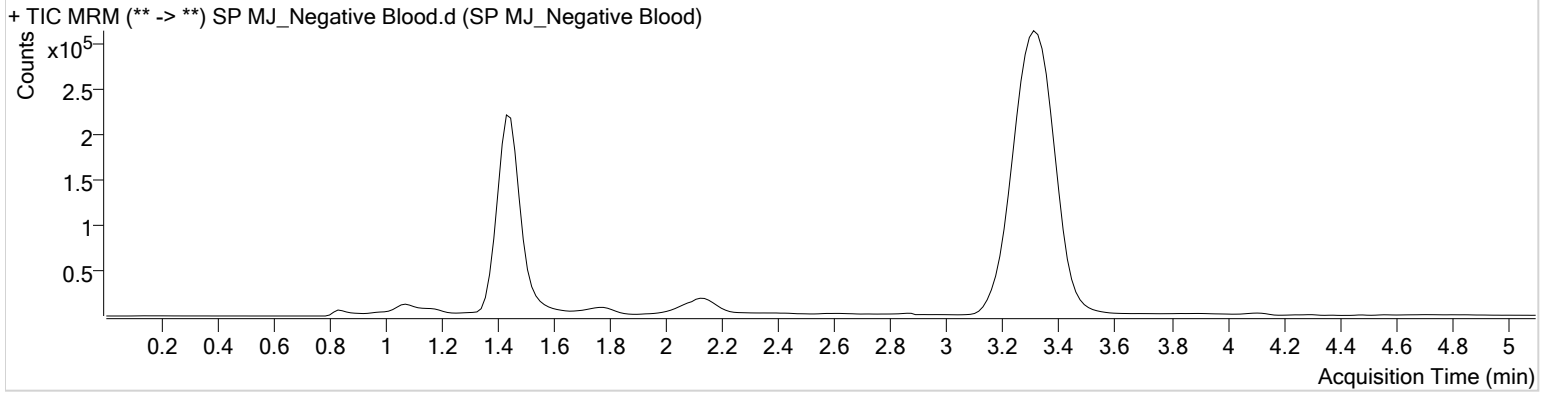
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|------------------------|
| Instrument | Falco | Data File | SP MJ_Negative Blood.d |
| Type | Sample | Sample | SP MJ_Negative Blood |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-A2 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 8:00:54 PM | | |
| Sample Info. | | | |

Sample Chromatogram



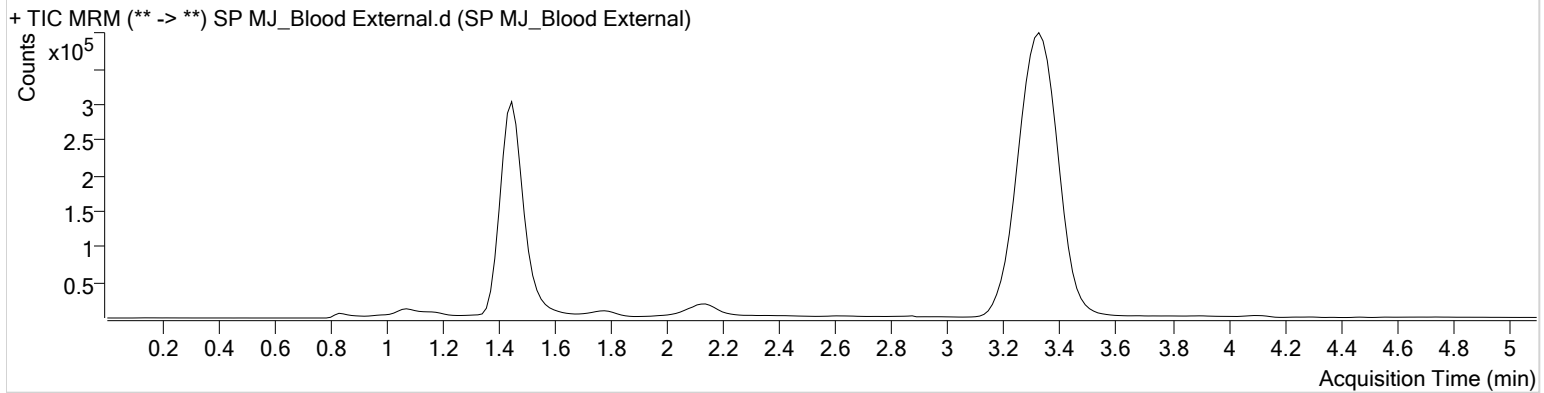
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|------------------------|
| Instrument | Falco | Data File | SP MJ_Blood External.d |
| Type | Sample | Sample | SP MJ_Blood External |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-B2 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 8:16:07 PM | | |
| Sample Info. | | | |

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|--------|---------|-------|--------|------------|---------------|
| THC-OH | 1.453 | 324576 | ∞ | 11.1 | 335.95 | 1043752 | 19.8626 ng/ml |
| THC-COOH | 1.489 | 94120 | 281.45 | 54.4 | 696.41 | 215933 | 18.0983 ng/ml |
| THC | 3.345 | 460519 | 1451.41 | 25.8 | 188.38 | 3411547 | 16.9311 ng/ml |

AM #27 Cannabinoid Quant. Results

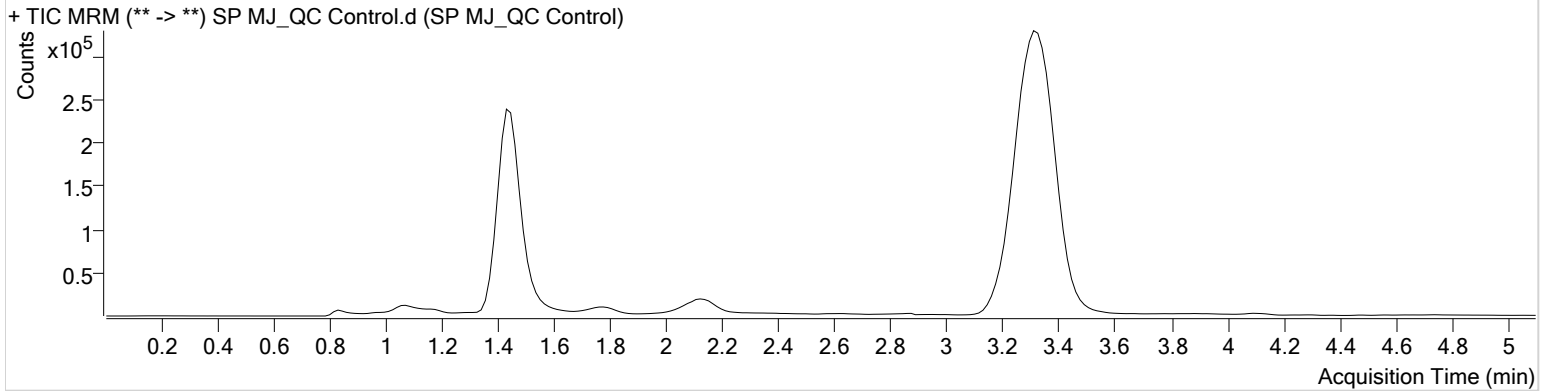


Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|--------------------|
| Instrument | Falco | Data File | SP MJ_QC Control.d |
| Type | Sample | Sample | SP MJ_QC Control |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-H1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 7:45:43 PM | | |

Sample Info.

Sample Chromatogram



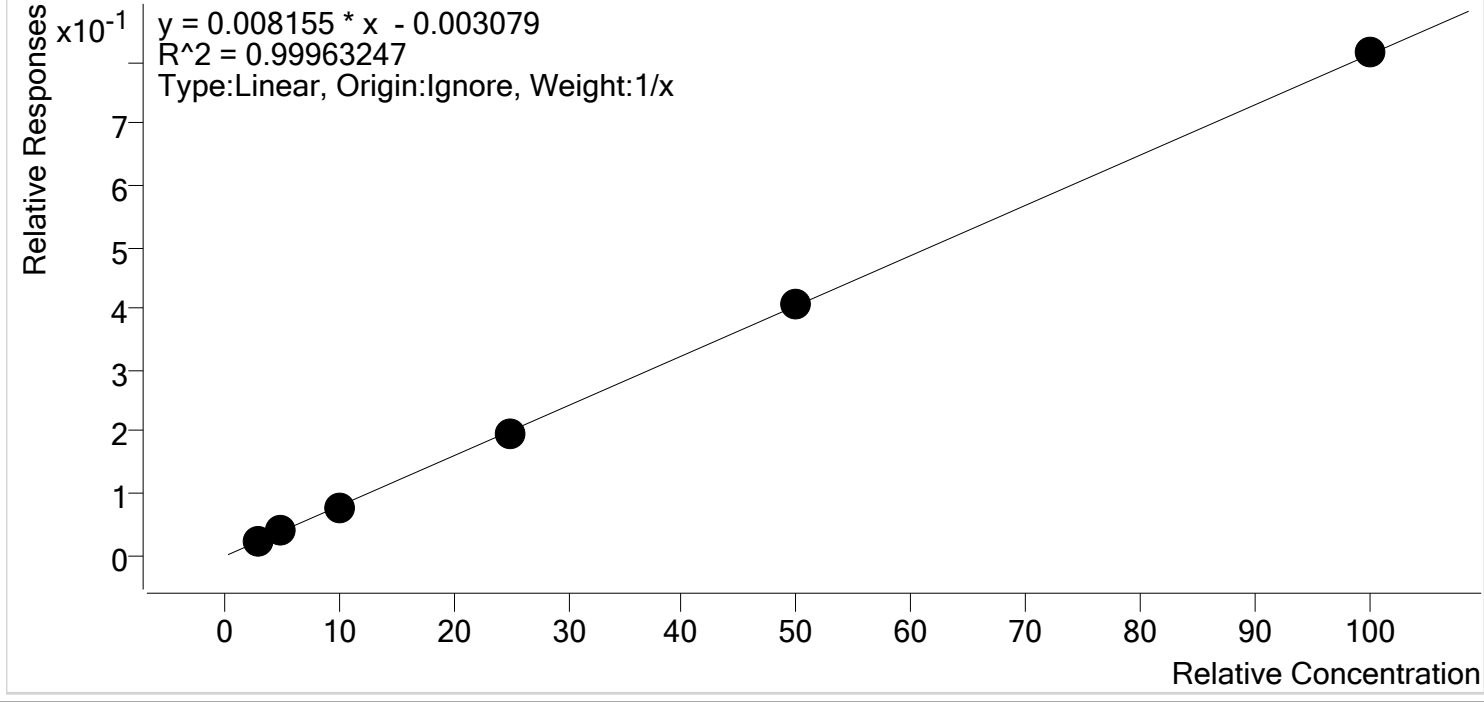
| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|--------|--------|-------|--------|------------|---------------|
| THC-OH | 1.453 | 100947 | ∞ | 9.2 | 55.21 | 979030 | 4.2642 ng/ml |
| THC-COOH | 1.474 | 62199 | ∞ | 60.1 | 348.76 | 201282 | 12.7294 ng/ml |
| THC | 3.330 | 106953 | 322.01 | 28.7 | 81.63 | 3153138 | 4.5370 ng/ml |



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Last Cal. Update 2/20/2020 8:02 AM
Analyst Name ISP\datastor
Analyte THC **Internal Standard** THC-D3

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



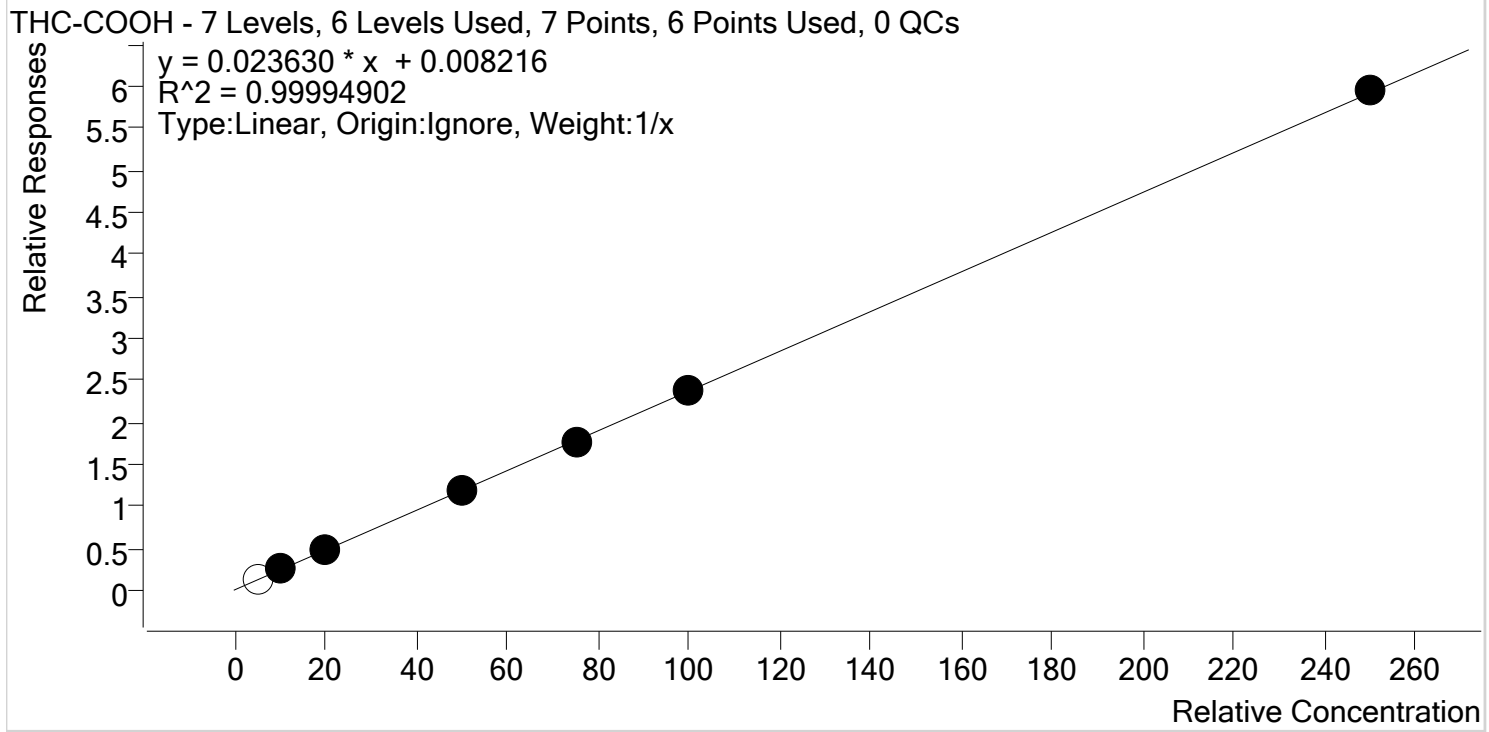
| Sample | Level | Enabled | Expected Concentration | Final Concentration | Accuracy |
|-------------|-------|---------|------------------------|---------------------|----------|
| SP MJ_Cal 2 | 2 | ✓ | 3.0 | 3.2 | 105.5 |
| SP MJ_Cal 3 | 3 | ✓ | 5.0 | 4.9 | 98.7 |
| SP MJ_Cal 4 | 4 | ✓ | 10.0 | 9.8 | 97.6 |
| SP MJ_Cal 5 | 5 | ✓ | 25.0 | 24.2 | 96.7 |
| SP MJ_Cal 6 | 6 | ✓ | 50.0 | 50.6 | 101.2 |
| SP MJ_Cal 7 | 7 | ✓ | 100.0 | 100.4 | 100.4 |

S



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Last Cal. Update 2/20/2020 8:02 AM
Analyst Name ISP\datastor
Analyte THC-COOH **Internal Standard** THC-COOH-D9

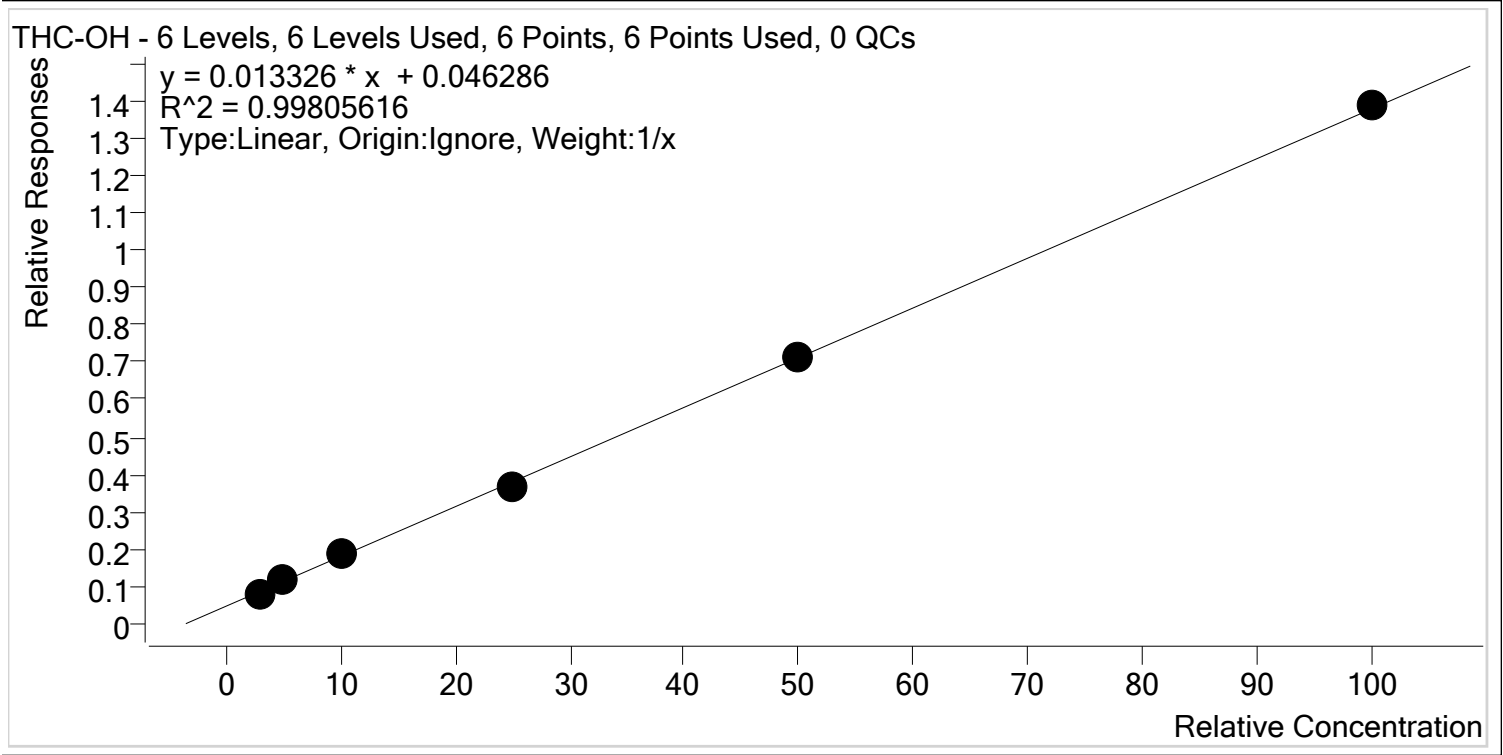


| Sample | Level | Enabled | Expected Concentration | Final Concentration | Accuracy |
|-------------|-------|---------|------------------------|---------------------|----------|
| SP MJ_Cal 1 | 1 | x | 5.0 | 5.5 | 109.3 |
| SP MJ_Cal 2 | 2 | ✓ | 10.0 | 10.2 | 101.6 |
| SP MJ_Cal 3 | 3 | ✓ | 20.0 | 19.9 | 99.4 |
| SP MJ_Cal 4 | 4 | ✓ | 50.0 | 49.9 | 99.8 |
| SP MJ_Cal 5 | 5 | ✓ | 75.0 | 74.5 | 99.3 |
| SP MJ_Cal 6 | 6 | ✓ | 100.0 | 99.4 | 99.4 |
| SP MJ_Cal 7 | 7 | ✓ | 250.0 | 251.2 | 100.5 |



AM #27 Cannabinoids Quant. Calibration Curve Report

Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Last Cal. Update 2/20/2020 8:02 AM
Analyst Name ISP\datastor
Analyte THC-OH **Internal Standard** THC-OH-D3



| Sample | Level | Enabled | Expected Concentration | Final Concentration | Accuracy |
|-------------|-------|---------|------------------------|---------------------|----------|
| SP MJ_Cal 2 | 2 | ✓ | 3.0 | 2.6 | 85.0 |
| SP MJ_Cal 3 | 3 | ✓ | 5.0 | 5.6 | 112.3 |
| SP MJ_Cal 4 | 4 | ✓ | 10.0 | 10.8 | 107.8 |
| SP MJ_Cal 5 | 5 | ✓ | 25.0 | 23.7 | 94.8 |
| SP MJ_Cal 6 | 6 | ✓ | 50.0 | 49.7 | 99.5 |
| SP MJ_Cal 7 | 7 | ✓ | 100.0 | 100.6 | 100.6 |

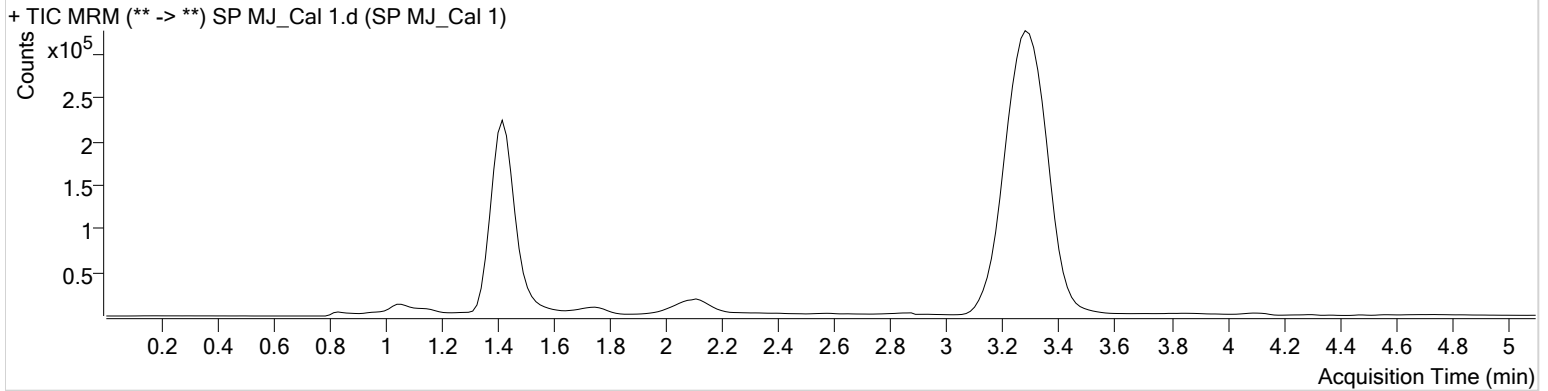
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 1.d |
| Type | Cal | Sample | SP MJ_Cal 1 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-A1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 6:44:48 PM | | |

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|-------|-----|-----------------|--------|------------|-------------------------|
| THC-COOH | 1.459 | 29836 | ∞ | 43.5 Low | 632.74 | 217280 | 5.4633 ng/ml Low |

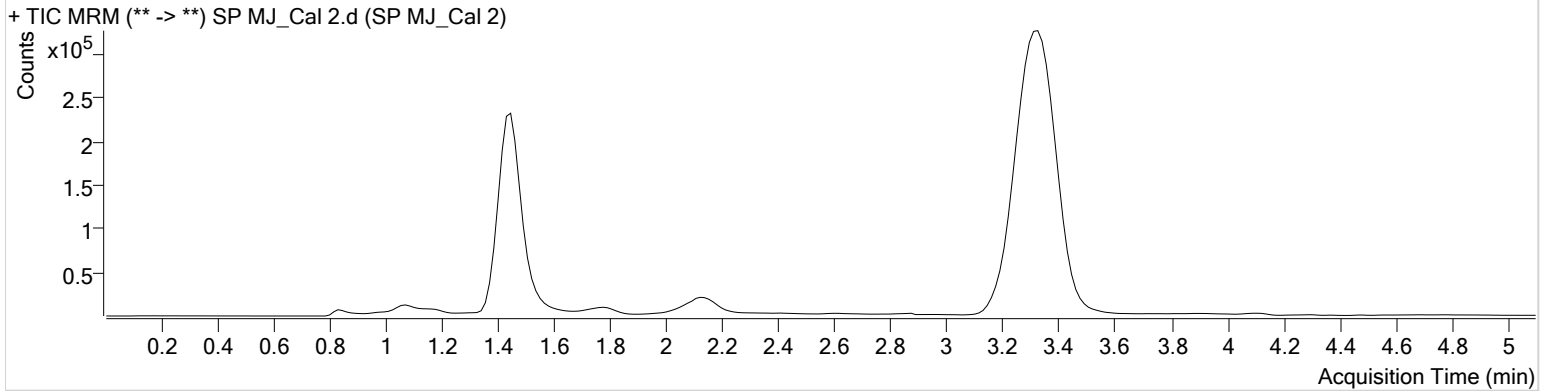
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 2.d |
| Type | Cal | Sample | SP MJ_Cal 2 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-B1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 6:52:40 PM | | |

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|-------|--------|-------|-------|------------|-------------------------|
| THC-OH | 1.483 | 80387 | ∞ | 8.9 | 34.08 | 1001255 | 2.5515 ng/ml Low |
| THC-COOH | 1.474 | 51318 | 109.99 | 50.8 | ∞ | 206629 | 10.1626 ng/ml |
| THC | 3.345 | 73158 | ∞ | 28.1 | 27.67 | 3219033 | 3.1645 ng/ml |

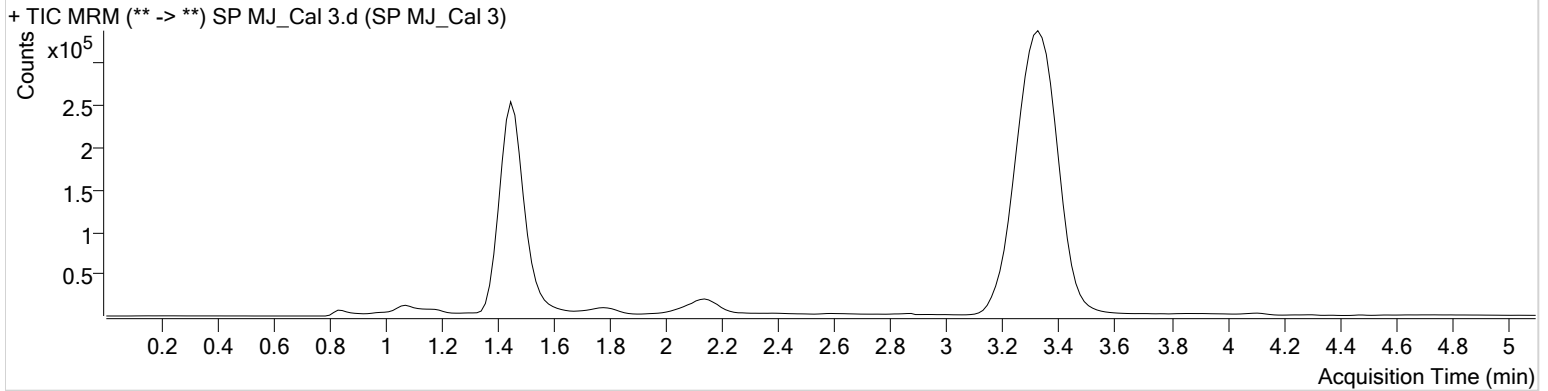
AM #27 Cannabinoid Quant. Results



Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 3.d |
| Type | Cal | Sample | SP MJ_Cal 3 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-C1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 7:00:14 PM | | |
| Sample Info. | | | |

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|--------|--------|-------|--------|------------|---------------|
| THC-OH | 1.468 | 126611 | ∞ | 8.9 | ∞ | 1045543 | 5.6139 ng/ml |
| THC-COOH | 1.489 | 102708 | 708.02 | 55.5 | 411.79 | 214917 | 19.8764 ng/ml |
| THC | 3.345 | 124886 | 195.90 | 28.9 | 105.22 | 3361124 | 4.9339 ng/ml |

AM #27 Cannabinoid Quant. Results

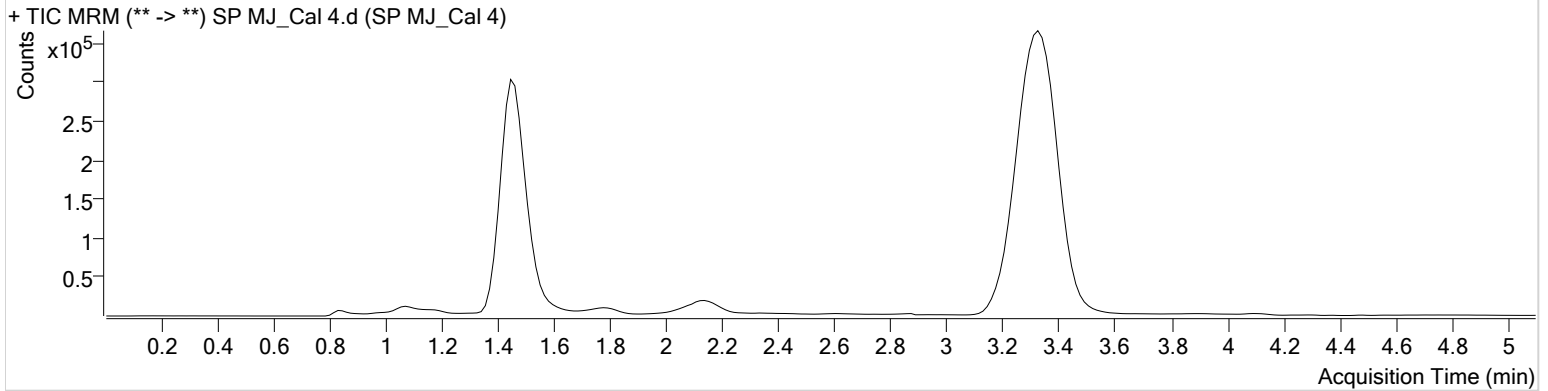


Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 4.d |
| Type | Cal | Sample | SP MJ_Cal 4 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-D1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 7:07:48 PM | | |

Sample Info.

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|--------|--------|-------|--------|------------|---------------|
| THC-OH | 1.453 | 199820 | ∞ | 9.8 | 764.22 | 1052132 | 10.7786 ng/ml |
| THC-COOH | 1.489 | 256480 | ∞ | 57.3 | 663.52 | 215951 | 49.9140 ng/ml |
| THC | 3.345 | 262795 | 465.23 | 26.8 | ∞ | 3436389 | 9.7555 ng/ml |

AM #27 Cannabinoid Quant. Results

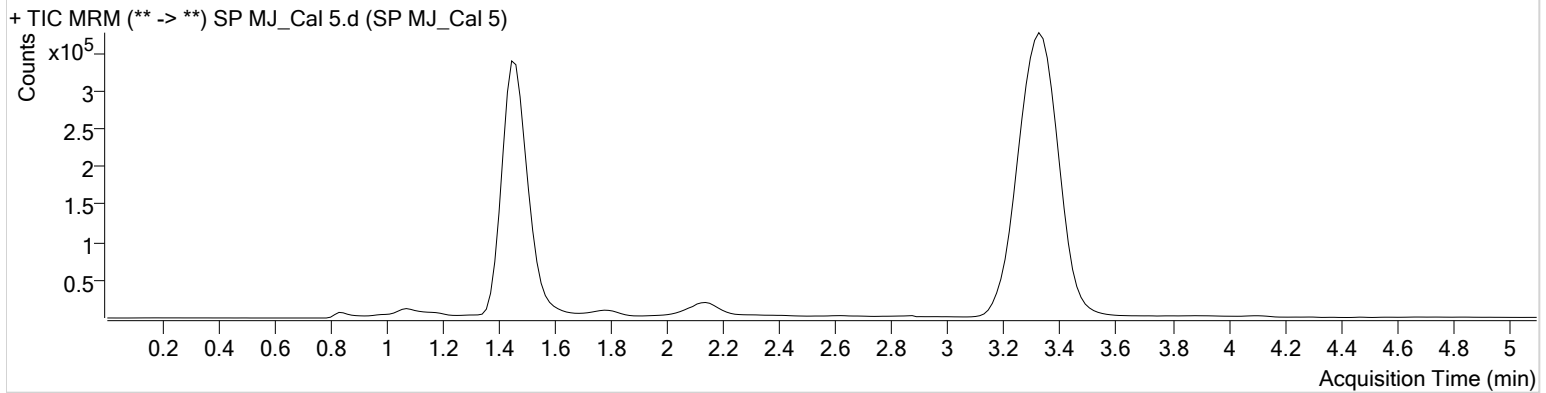


Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 5.d |
| Type | Cal | Sample | SP MJ_Cal 5 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-E1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 7:15:23 PM | | |

Sample Info.

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|--------|---------|-------|---------|------------|---------------|
| THC-OH | 1.453 | 345529 | ∞ | 12.6 | 294.23 | 954357 | 23.6961 ng/ml |
| THC-COOH | 1.489 | 333811 | ∞ | 59.1 | 3940.03 | 188817 | 74.4687 ng/ml |
| THC | 3.345 | 586591 | 1085.46 | 26.8 | 488.20 | 3023003 | 24.1729 ng/ml |

AM #27 Cannabinoid Quant. Results

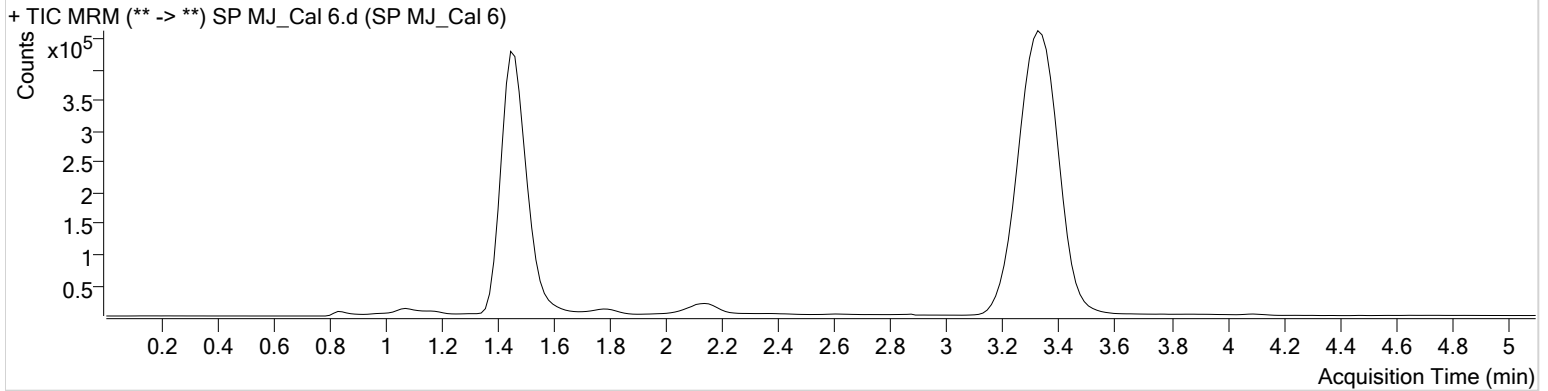


Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

| | | | |
|-------------------------|----------------------|------------------|---------------|
| Instrument | Falco | Data File | SP MJ_Cal 6.d |
| Type | Cal | Sample | SP MJ_Cal 6 |
| Acq. Method | AM 27 THC quant.m | Operator | Sarah Pickle |
| Sample Position | P4-F1 | Comment | |
| Injection Volume | 10 | | |
| Acq. Date-Time | 2/19/2020 7:22:57 PM | | |

Sample Info.

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|---------|---------|-------|---------|------------|---------------|
| THC-OH | 1.453 | 666838 | ∞ | 12.7 | 1100.35 | 940350 | 49.7423 ng/ml |
| THC-COOH | 1.489 | 439071 | 514.47 | 59.4 | 3065.98 | 186279 | 99.4013 ng/ml |
| THC | 3.345 | 1244059 | 5852.07 | 25.5 | 377.82 | 3036543 | 50.6184 ng/ml |

AM #27 Cannabinoid Quant. Results

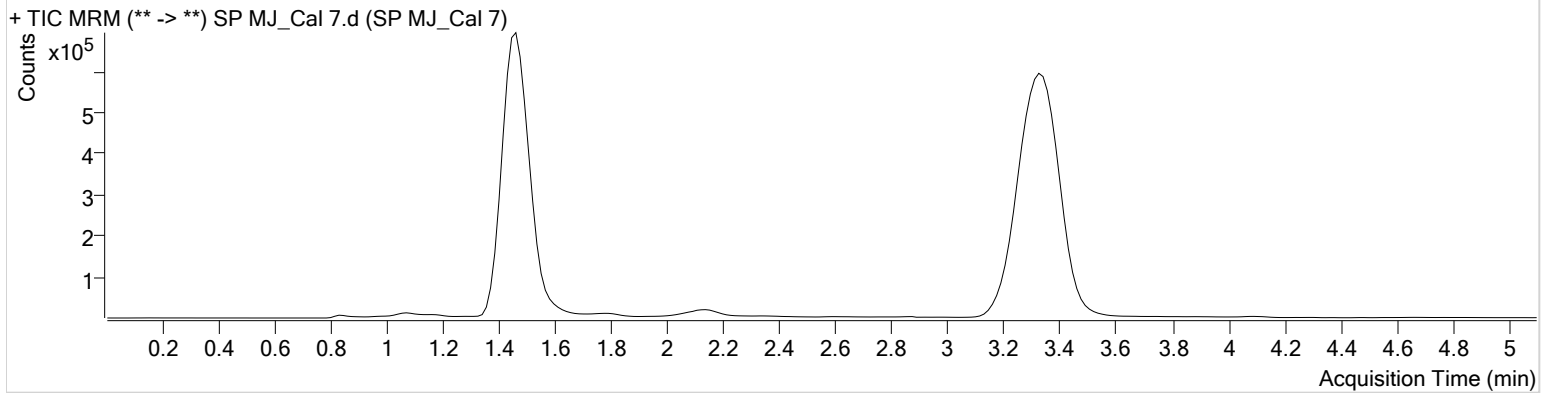


Batch results D:\MassHunter\Data\2020\AM 27-28\021920 AM 27 28 CS SP\QuantResults\THCQ SP.batch.bin
Calibration Last Update 2/20/2020 8:02:54 AM

Instrument Falco
Type Cal
Acq. Method AM 27 THC quant.m
Sample Position P4-G1
Injection Volume 10
Acq. Date-Time 2/19/2020 7:30:32 PM
Sample Info.

Data File SP MJ_Cal 7.d
Sample SP MJ_Cal 7
Operator Sarah Pickle
Comment

Sample Chromatogram



| Name | RT | Resp. | S/N | Ratio | S/N | ISTD Resp. | Final Conc. |
|----------|-------|---------|-----|-------|---------|------------|----------------|
| THC-OH | 1.453 | 1328833 | ∞ | 12.5 | 2659.78 | 958000 | 100.6177 ng/ml |
| THC-COOH | 1.489 | 1100303 | ∞ | 58.4 | 7183.05 | 185127 | 251.1769 ng/ml |
| THC | 3.345 | 2482138 | ∞ | 25.7 | 2223.05 | 3044526 | 100.3548 ng/ml |