

TBI-FORENSIC SERVICES DIVISION PERFORMANCE VERIFICATION FORM

Instrument/Method to Verify:

Piney - Agilent 1260 Liquid Chromatograph iQ Single Quadrapole Mass Spectrometer

Check the appropriate box: New Validation ☐ Modification ☐ Performance Check ☒

Laboratory: NCL ☒ KCL ☐ JCL ☐ Unit: Forensic Chemistry

Based on training and competency, the following analyst(s) are authorized by the Technical Leader to participate in this study:

Name Andrea King Title FS III Initials AK

Completed: Andrea King
Andrea King (Aug 29, 2024 11:33 CDT)

Name _____ Title _____ Initials _____

Completed:

Name _____ Title _____ Initials _____

Completed:

Verification of final review:

Unit Supervisor: Rebecca Hernandez

Section Manager: Glenn B. Everett

Unit Supervisor: Brett Trotter

Section Manager:

Unit Supervisor:

Section Manager:

Regional Administrator: Janifer Hall

IT Manager: Matthew Buck

Regional Administrator:

Regional Administrator:

Quality Assurance Manager: chad johnson

By signing below, I have verified this study meets all requirements and I deem this procedure/instrument fit for the intended use:

Technical Leader: Brett Trotter

All information must be submitted to the Quality Assurance Manager prior to the implementation of the procedure/instrument. If applicable, include training plan and competency testing schedule.

Piney

1. Introduction

1.1 Purpose

The purpose of this validation report is to document the performance and accuracy of the LCMS (Liquid Chromatography-Mass Spectrometry) instrument, following the analysis of 15 standards. This validation aims to ensure that the LCMS is operating correctly and producing reliable and accurate results.

1.2 Instrument Details

- **Instrument Model:** Agilent 1260 Infinity Lab LC/MSD Single Quadrupole
- **Agilent 1260 Vialsampler**
 - **Serial number:** DEAEQ69876
 - **Product number:** G7129A
- **Agilent 1260 Binary Pump**
 - **Serial number:** DEAE902046
 - **Product number:** G7112B
- **Agilent Single Quadrupole Mass Spectrometer**
 - **Serial number:** DEAEQ69876
 - **Product number:** G7129A
- **Software Version:** OpenLab CDS v. 2.6

2. Methodology

- **Method Name:** TBI Drug Screen
 - **Mobile Phases**
 - Mobile A: 0.1% Formic Acid in H₂O
 - Mobile B: 0.1% Formic Acid in MeOH

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○ **Binary Pump**

- Pressure limits
 - Min 0.00 bar Max 600.00 bar
- Gas Flow 12.0 L/min
- Nebulizer 15.0 psi
- Gas Temperature 300 C
- Oven (Column) Temperature 50.0 C
- Injection Volume 5.00 uL

Time(min)	A(%)	B(%)	Flow rate(mL/min)
0.00	99.0	1.0	0.350
2.00	99.0	1.0	-
8.00	20.0	80.0	-
8.50	2.0	98.0	-
10.00	2.0	98.0	-

2.1 Standards

• **Standards Analyzed:**

▪ **Buprenorphine**

- Cerilliant lot#FE03191903 exp June 25,2025 MeOH

▪ **Clonazepam**

- Cerilliant lot#FN05252144 exp 2/26 Acetonitrile

▪ **Etizolam**

- Cerilliant lot#FN06202103 exp 11/30/26 MeOH

▪ **GCMS Standard (Hydrocodone, Pethidine, Cocaine)**

- Made: Dec 15,2023 Exp: Dec 15,2024 MeOH

▪ **GCIR Standard**

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- Made: March 6, 2024 Exp: March 6, 2025 MeOH
- **Alprazolam**
 - Cerilliant lot# FE050032221 exp Jan 31, 2028 MeOH
- **Psilocyn**
 - Cerilliant lot# FE08092301 exp Aug 31, 2028 MeOH
- **Psilocybin**
 - Cerilliant lot# FE7232102 exp Aug 31, 2025 MeOH
- **Fentanyl**
 - Cerilliant lot# FE03252103 exp April 2026 MeOH
- **Oxycodone**
 - Cerilliant lot# FE10161901 exp Jan, 2025 MeOH
- **Xylazine**
 - Sigma lot# BCBL9775V exp Nov 7, 2024 MeOH
- **LSD**
 - Cerilliant lot# FE10161901 exp Oct, 2024 MeOH
- **Testosterone**
 - Sigma lot# 50H0345 exp April 10, 2025 MeOH
- **Tianeptine**
 - Cayman Chemical lot# 05755807-12 exp July 19, 2025 MeOH+HCl
- **Isotonitazene**
 - Cayman Chemical lot# 0577984-1 exp Feb 14, 2025 MeOH
- **Preparation:** Standards were prepared and diluted according to established procedures.

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2.2 Procedure

1. **Instrument Setup:** The LCMS was set up and a weekly tune was run before performing the validation with the TBI drug Screen method.
2. **Running Standards:** Each of the 15 standards was injected into the LCMS under standardized conditions.
3. **Data Collection:** Data was collected for each standard, including retention times, peak areas, and mass spectra.

3. Results

3.1 System Suitability

- **Resolution:** The resolution between peaks was adequate for all standards for all standards except Testosterone which is expected, meeting the specifications for this method.
- **Sensitivity:** The sensitivity was assessed for each compound, with acceptable data for all standards except Testosterone. The TBI drug screen method is not necessarily the appropriate method for analysis of Testosterone. If Testosterone is suspected in case work, it might be necessary to use an alternative method such as one of the isocratic methods.

3.2 Method Performance

- **Specificity:** The LCMS method was able to distinguish the analytes from other substances.

3.3 Issue with Testosterone

- **Testosterone Performance:** Testosterone did not lead to a chromatographic peak and no mass spectroscopy data was collect for this standard. This issue is known to occur with the TBI drug screen method and is considered acceptable in this context of this validation.

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4. Conclusion

- **Instrument Performance:** The LCMS instrument demonstrated overall satisfactory performance based on the analysis of 15 standards. All acceptance criteria for system suitability, and method performance were met, except for the noted issue with Testosterone that is expected for this method.
- **Validation Status:** The LCMS system is considered validated for the intended use, with the exception of the known issue with Testosterone.
- **Routine Maintenance:** Continue with the scheduled maintenance as per policy.

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Acquisition Method: TBI Drug Screen

Path: C:\CDSPProjects\TBI - Piney\Methods

1 Method Information

Last Saved As:	C:\CDSPProjects\TBI - Piney\Methods\TBI Drug Screen.amx	Modified:	2024-08-21 13:13:10-05:00	Modifier:	SYSTEM (SYSTEM)
Created:	2024-06-20 13:32:21-05:00	Creator:	SYSTEM (SYSTEM)	Description:	TBI general purpose method using full scan (45 - 600 amu). Starts 99% water and 1% methanol and ramps to 2% water and 98% methanol.
Version:	2024-0821-1813-10351	Method Status:	Generic		

2 Binary Pump Method

Flow:	0.350 mL/min	Use Solvent Types:	Yes	Low Pressure Limit:	0.00 bar
High Pressure Limit:	600.00 bar	Maximum Flow Gradient:	100.000 mL/min ²	Module Display Name:	Binary Pump
Module Type:	G7112B	Order:	1		

Solvent Composition

Empty columns are hidden.

Channel	Solvent 1	Name 1	Solvent 2	Selected	Used	Percent
A	H2O	0.1 % FA	H2O	Ch. 1	Yes	99.0 %
B	MeOH	0.1 % FA	H2O	Ch. 1	Yes	1.0 %

Timetable

Time	A	B	Flow	Pressure
Start. Cond. min	99.0 %	1.0 %	0.350 mL/min	600.00 bar
2.00 min	99.0 %	1.0 %	--- mL/min	--- bar
8.00 min	20.0 %	80.0 %	--- mL/min	--- bar
8.50 min	2.0 %	98.0 %	--- mL/min	--- bar
10.00 min	2.0 %	98.0 %	--- mL/min	--- bar

2.1 Stroke A

Automatic Stroke Calculation A: Yes

2.2 Stroke B

Automatic Stroke Calculation B: Yes

2.3 Stoptime

Stoptime Mode: Time set Stoptime: 10.00 min

2.4 Posttime

Posttime Mode: Time set Posttime: 2.00 min

3 Sampler Method

Module Display Name: Sampler Module Type: G7129A Order: 1

3.1 Auxiliary

Draw Speed:	100 µL/min	Eject Speed:	100 µL/min	Wait Time After Draw:	1.2 s
Needle Height Offset:	0.2 mm				

3.2 Injection

Injection Mode: Injection with needle wash Injection Volume: 5.00 µL

3.2.1 Needle Wash

Needle Wash Location: Flush Port Wash Time: 3 s

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3.3 High throughput

Sample Flush-Out Factor: 6.0 | Injection Valve to Bypass for Delay Volume Reduction: Yes

3.3.1 Overlapped Injection

Overlapped Injection Mode: Off

3.4 Stoptime

Stoptime Mode: As Pump/No Limit

3.5 Posttime

Posttime Mode: Off

4 Column Oven Method

Module Display Name: Column Oven | Module Type: G7130A | Order: 1

4.1 Temperature Control

Temperature Control Mode: Temperature Set | Temperature: 50.0 °C

4.1.1 Temperature Not Ready Limit

Temperature Not Ready Limit On: Yes | Temperature Not Ready Limit Value: 0.8 °C | Temperature Equilibration Time: 0.0 min

4.2 Enforce column for run

Enforce column for run is enabled: No

4.3 Stoptime

Stoptime Mode: As Pump/Injector

4.4 Posttime

Posttime Mode: Off

5 Method

Module Display Name: SQ Mass Spectrometer | Module Type: LC/MSD iQ | Order: 1

5.1 General

Acquire Mode: Advanced | Tune File: atunes.tune | Ion Source: ESI
Optimized Method: Off | Last Compound Optimized Time: | Last Source Optimized Time:
Time Filter Enabled: On | Time filter window: 0.03 min

5.2 Stop Time

Stop Mode: By Pump Time

5.3 Time Segment

Time Segment

Start Time	Scan Type
0 min	Scan

5.3.1

Targeted Points Per Second Enabled: Off | Targeted Points Per Second: 0.93 Hz

5.3.2 Time Segment 1

5.3.2.1 Scan Type: Scan

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Start mass	End mass	Scan time	Detector Gain Factor	Fragmentor	Polarity	Threshold	Data Storage	Ion Mode
45 m/z	600 m/z	500 ms	1	135 V	+	0	Centroid	ESI

5.3.2.2 Fragmentor Ramp

N/A:

5.3.2.3 Source Parameter

Source Parameter

Parameter Id	Positive Value	Negative Value
Gas Temperature (°C)	300	300
Gas Flow (L/min)	12	12
Nebulizer (psi)	15	15
Capillary Voltage (V)	4000	4000

5.4 Chromatogram

Chromatogram

Chromatogram Type	Label
TIC	TIC

5.5 Timetable

Timetable

Start Time	Timetable Type	Timetable Value
0 min	Diverter Valve	To MS

6 Method Properties

Instrument Technique: Liquid Chromatography

7 Schema version

Schema version: 2.3

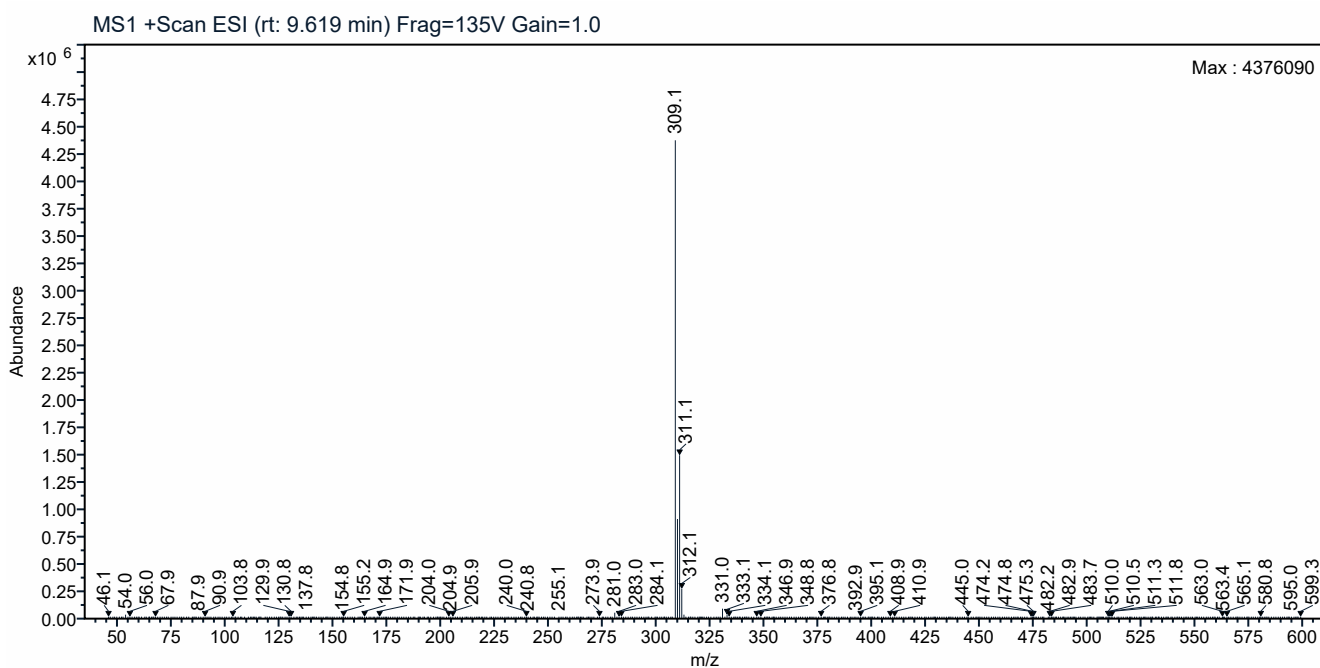
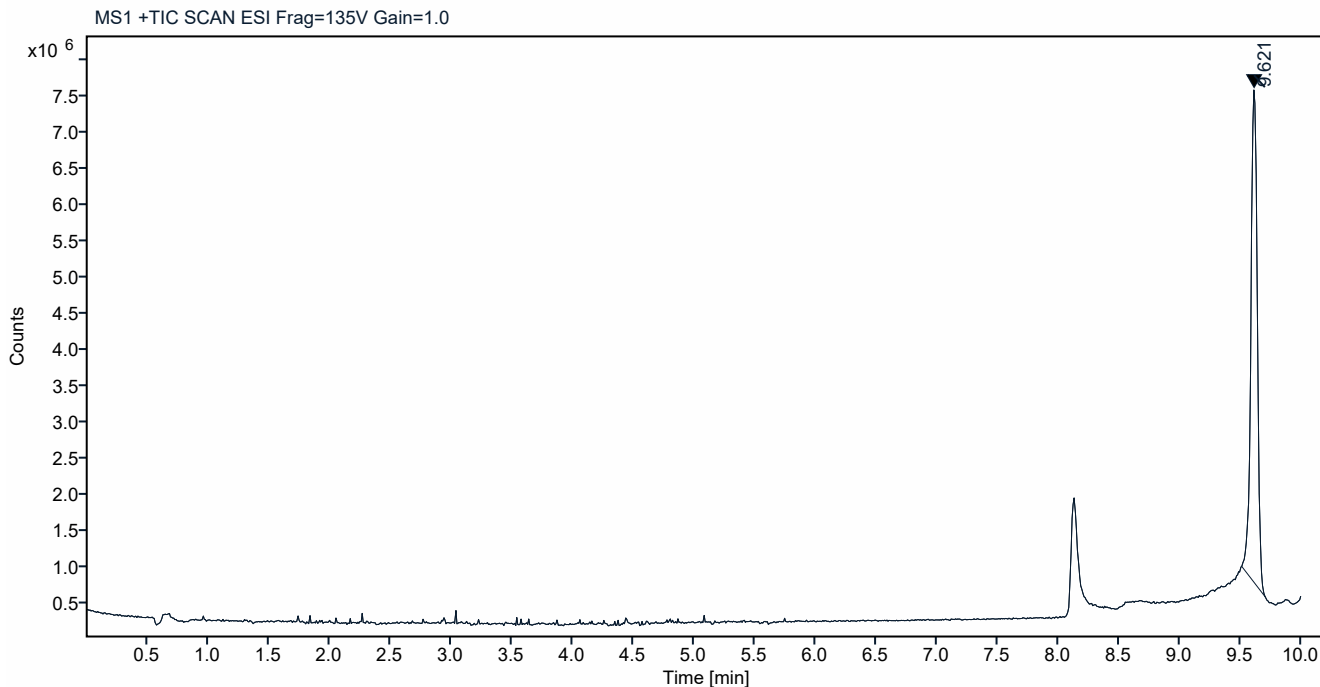
Method Audit Trail

Method audit trail is not printed

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Analysis Report

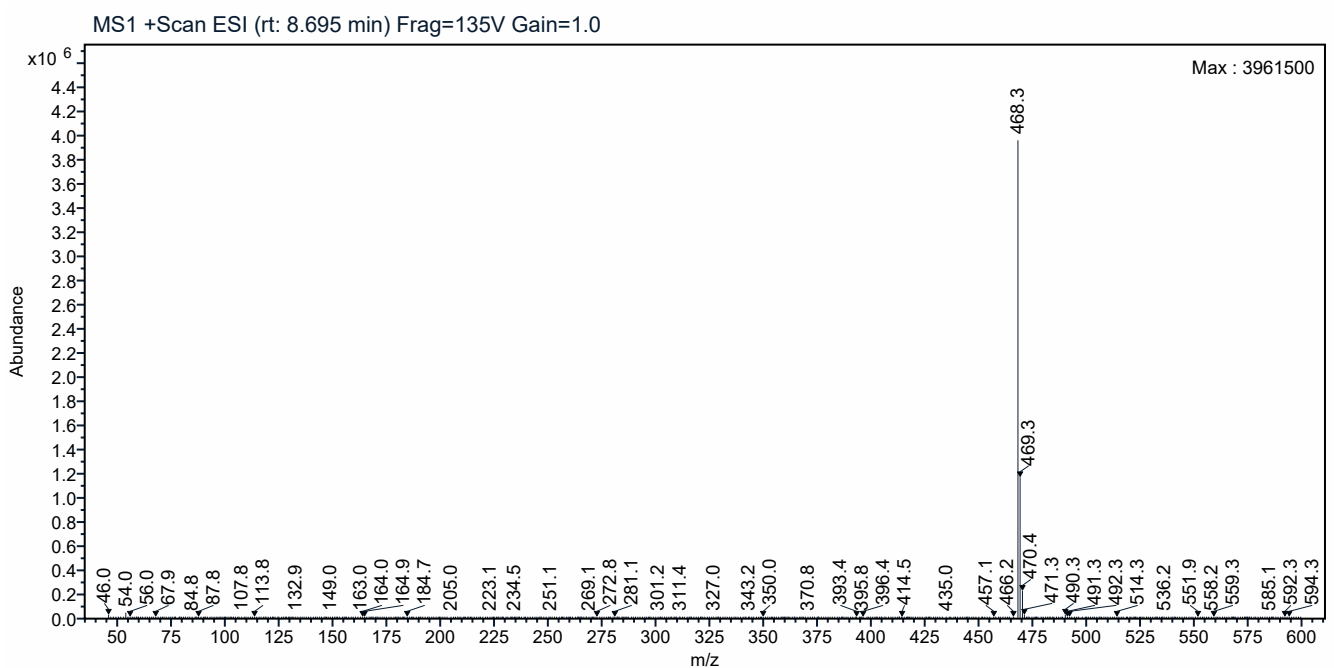
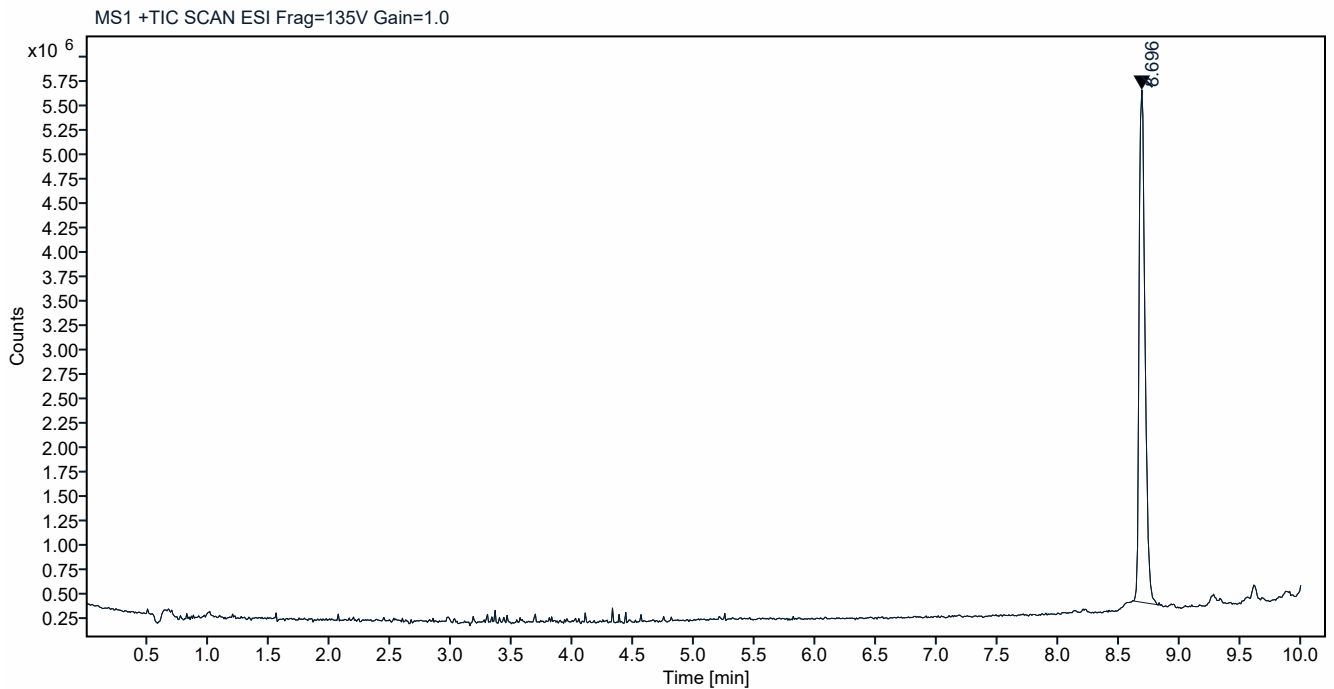
Sample Name	Alprazolam lot#FE050032221 exp 1-31-28 cerilliant MeOH	Injection Data File Name	Alprazolam-20240821 132723.dx
Instrument Name	Piney	Injection Acq Method Name	TBI Drug Screen.amx
Sample Vial Number	P1-A2	Injection Acquired Date	2024-08-21 13:28:00-05:00
Injection Volume	5.000	Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21-24\Alprazolam-20240821 132723.sirsl



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Analysis Report

Sample Name	Buprenorphine lot# FE03191903 exp 6-25-25 cerilliant MeOH	Injection Data File Name	Buprenorphine-20240821 135241.dx
Instrument Name	Piney	Injection Acq Method Name	TBI Drug Screen.amx
Sample Vial Number	P1-A3	Injection Acquired Date	2024-08-21 13:53:18-05:00
Injection Volume	5.000	Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21-24\Buprenorphine-20240821 135241.sirsl



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Analysis Report

Sample Name Clonazepam lot# FN05252144 exp 2-2026 cerilliant Acetonitrile

Instrument Piney
Name

Sample Vial P1-A4
Number

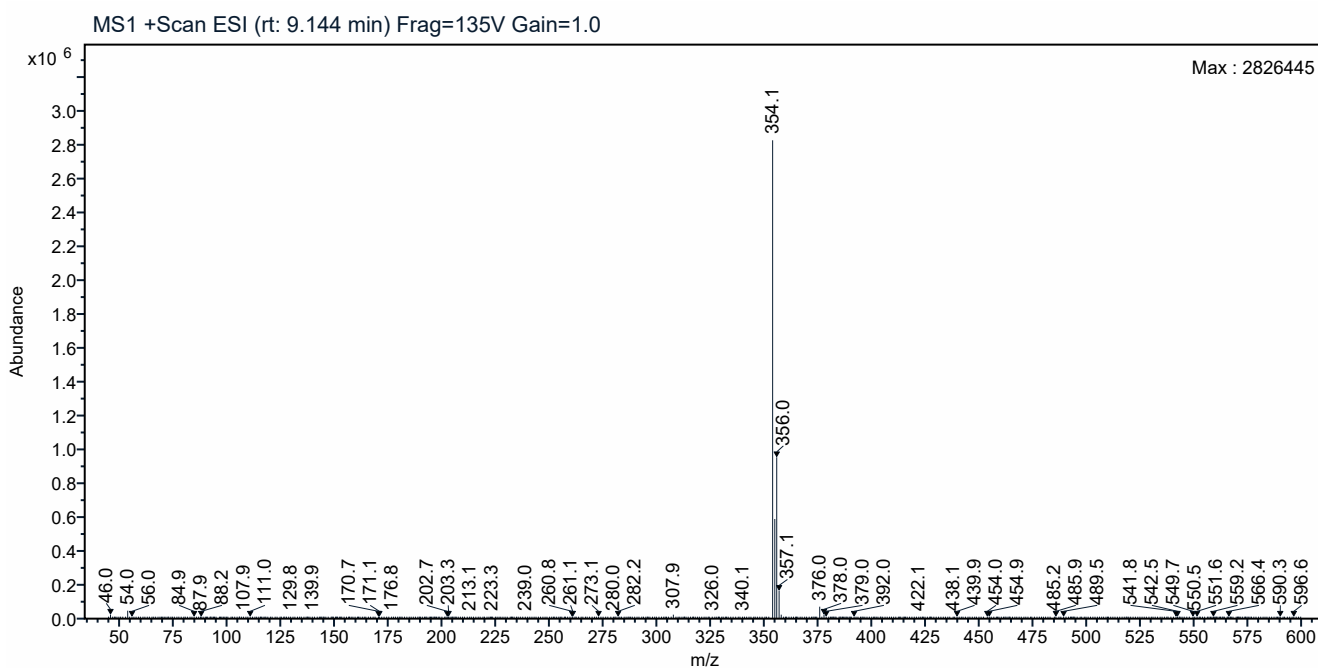
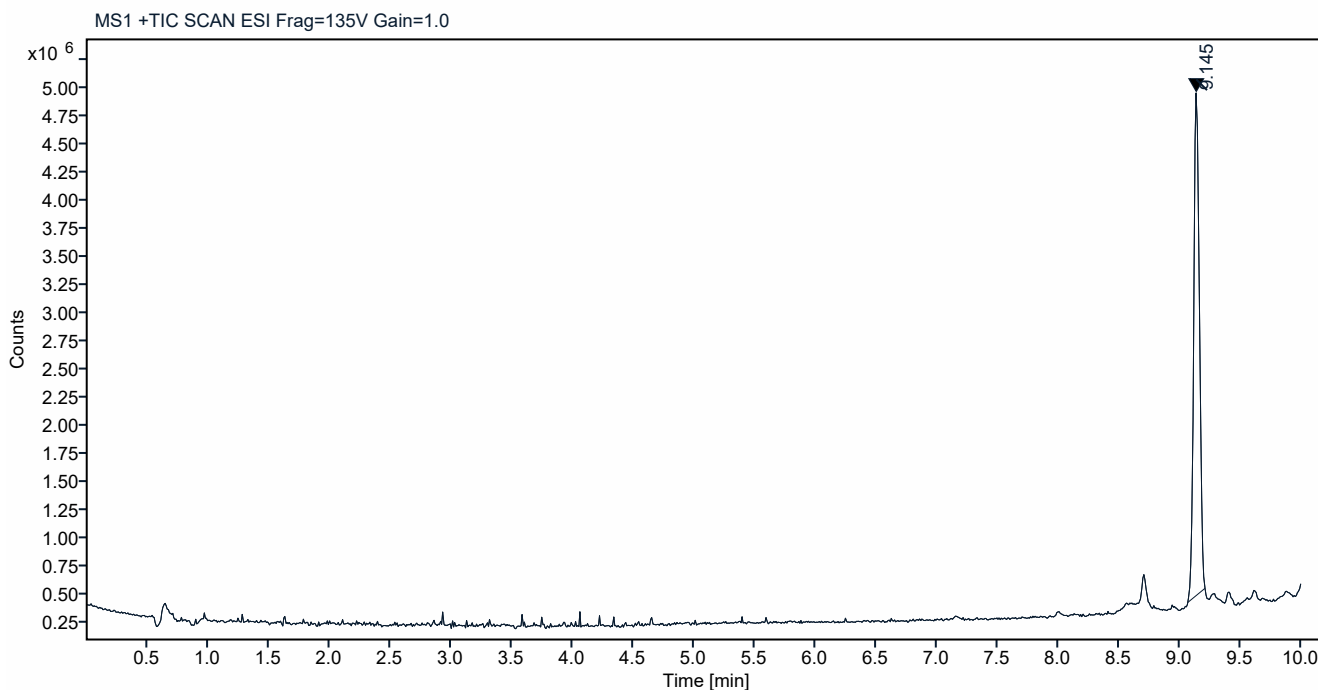
Injection Volume 5.000

Injection Data File Clonazepam-20240821 141758.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 14:18:34-05:00
Date

Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Clonazepam-20240821 141758.sirsl



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Analysis Report

Sample Name Etizolam lot# FN06202103 exp 11-30-26 cerilliant MeOH

Instrument Piney
Name

Sample Vial P1-A5
Number

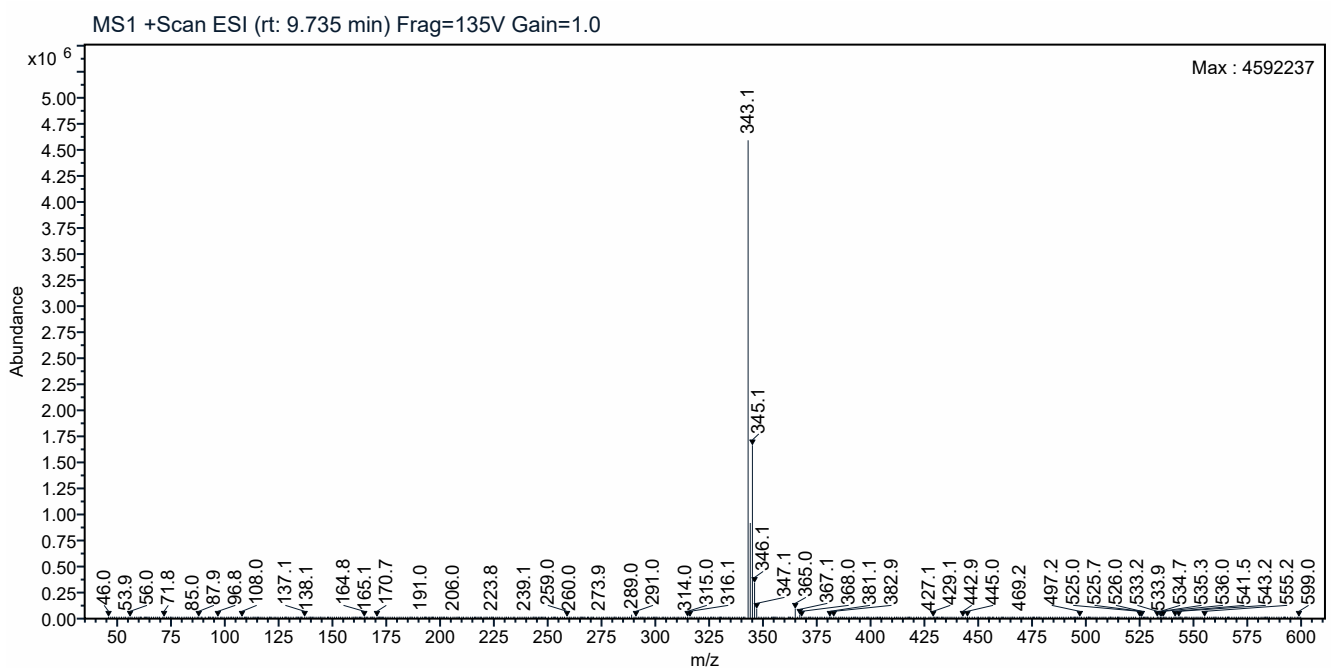
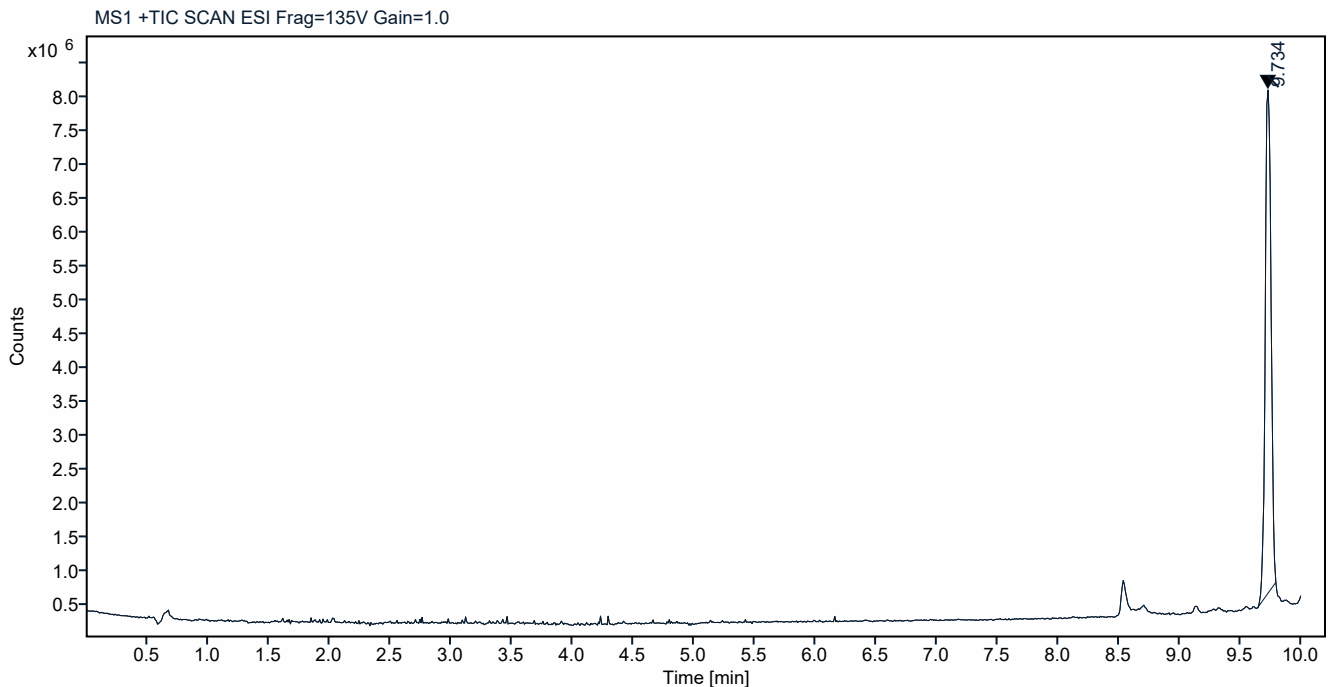
Injection Volume 5.000

Injection Data File Etizolam.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 14:43:48-05:00
Date

Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Etizolam.sirslt



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Analysis Report

Sample Name Fentanyl lot#FE03252103 cerilliant exp
4-2024

Instrument Piney
Name

Sample Vial P1-A6
Number

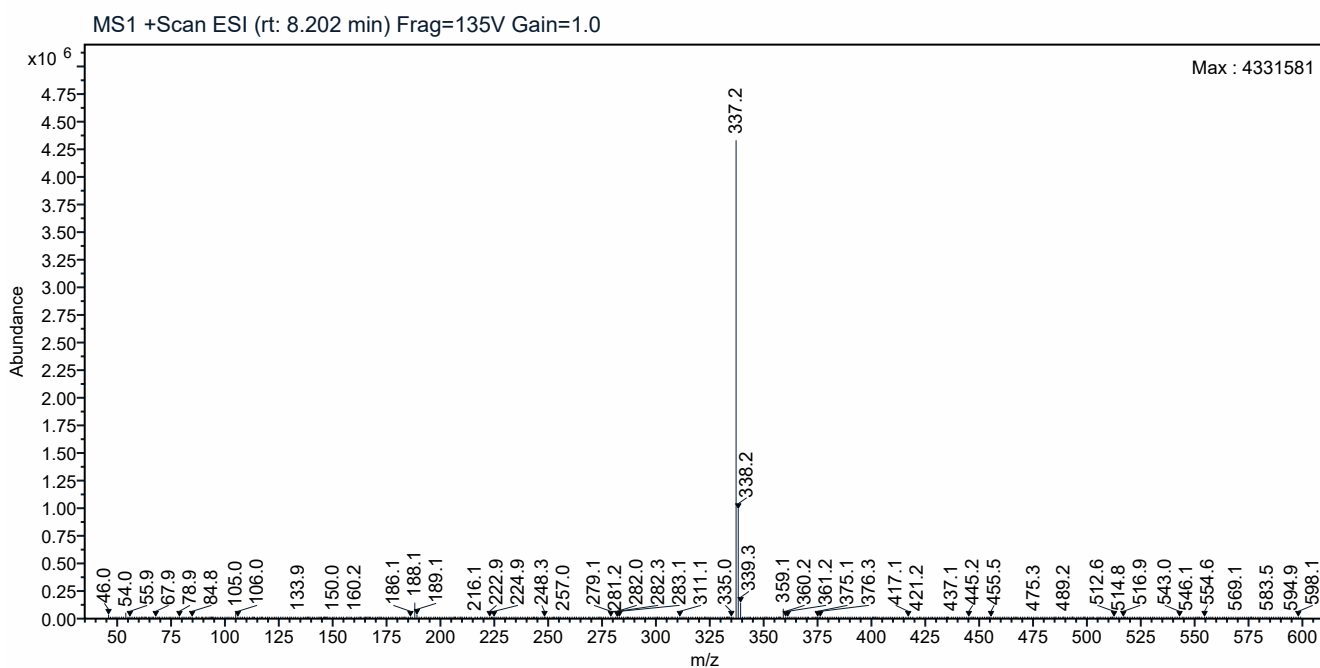
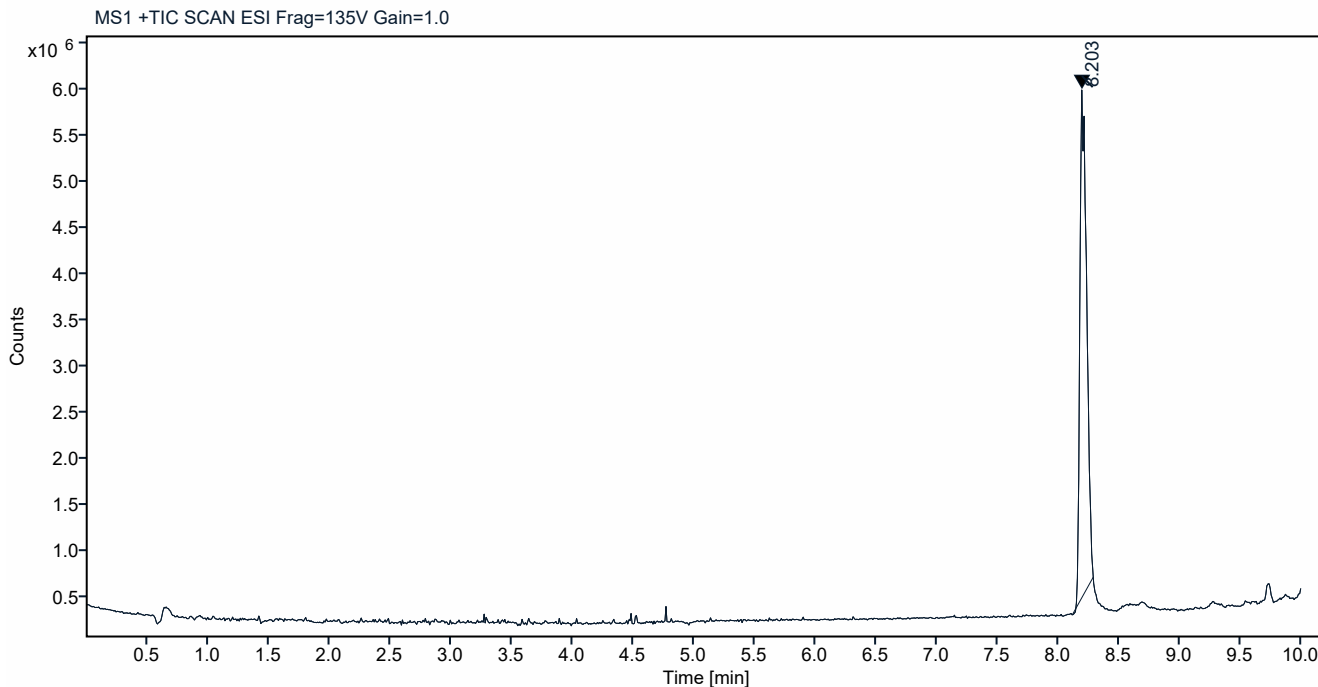
Injection Volume 5.000

Injection Data File Fentanyl.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 15:09:05-05:00
Date

Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Fentanyl.sirsl



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Analysis Report

Sample Name Methamphetamine, Amphetamine,
Phentermine made: 3-6-24 exp: 3-6-25

Instrument Piney

Sample Vial P1-B5

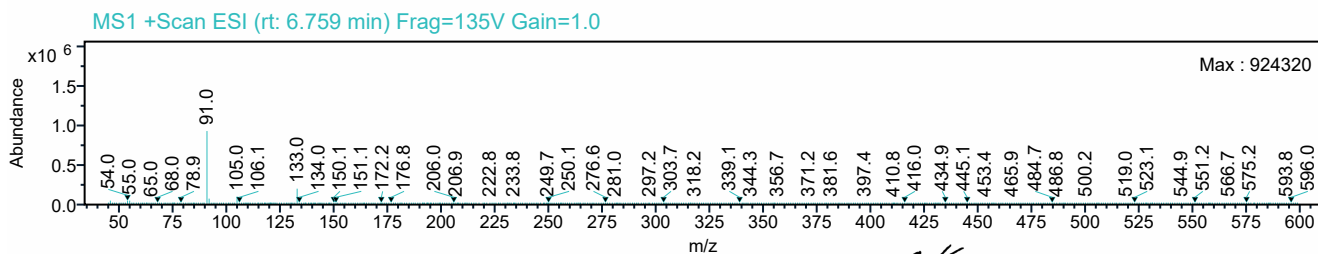
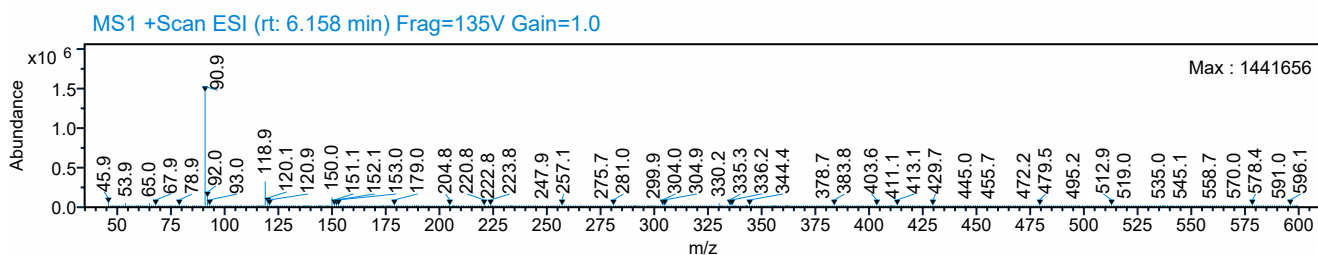
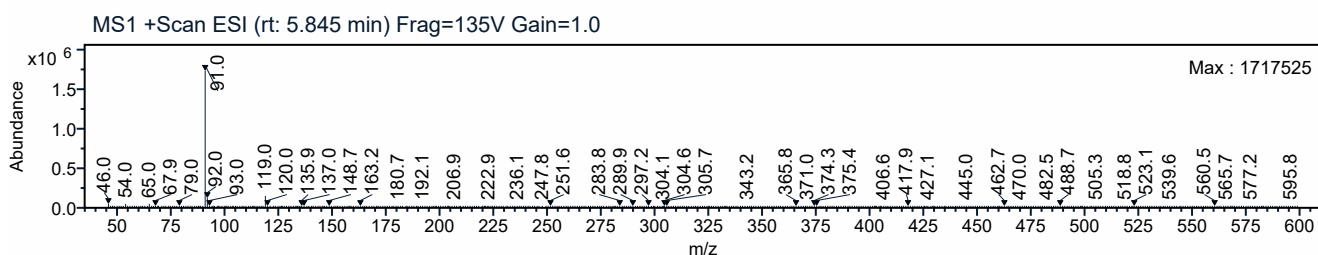
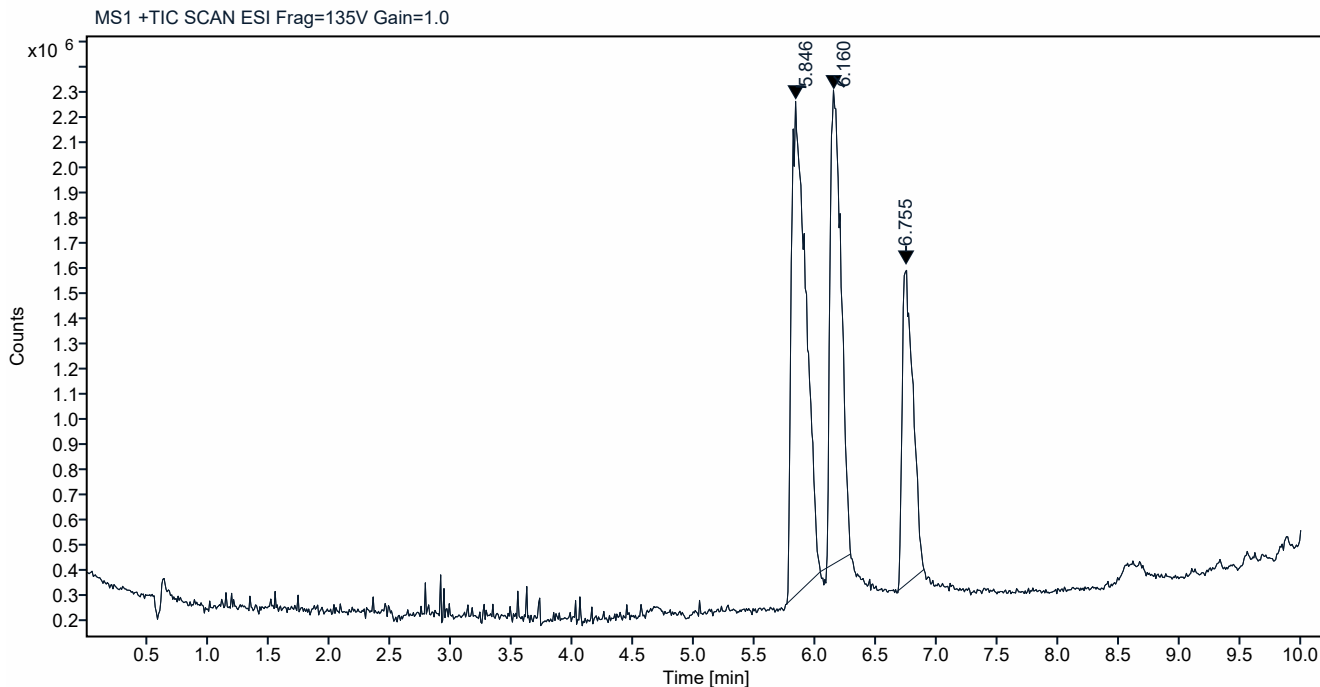
Injection Volume 5.000

Injection Data File GCIR test solution.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 19:21:58-05:00
Date

Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-24\GCIR
test solution.sirslt



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Analysis Report

Sample Name Hydrocodone, Cocaine, Pethidine
made:12-15-23 exp 12-15-24

Instrument Piney
Name

Sample Vial P1-B4
Number

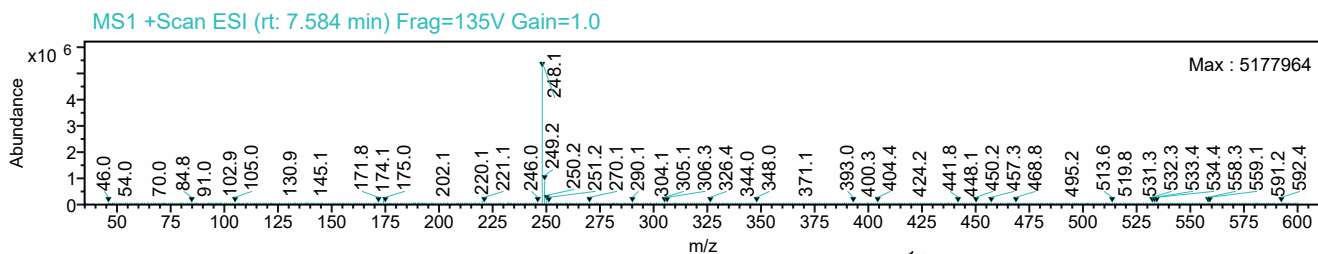
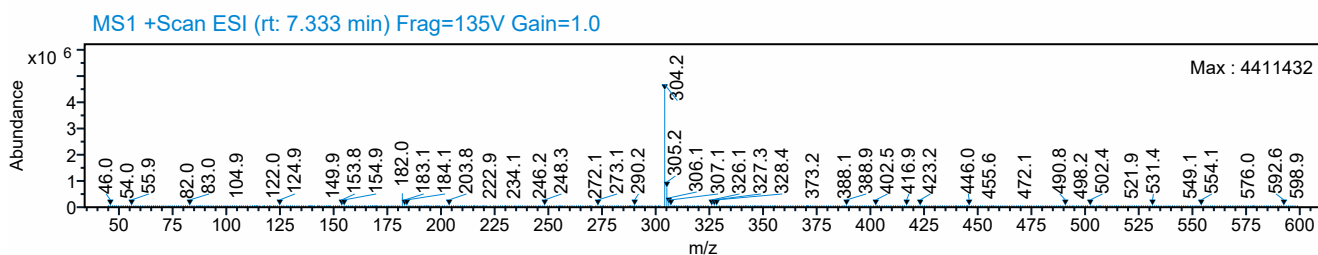
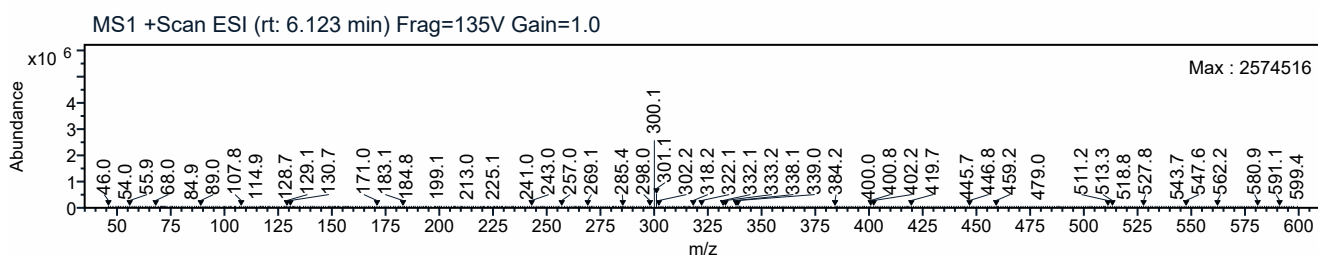
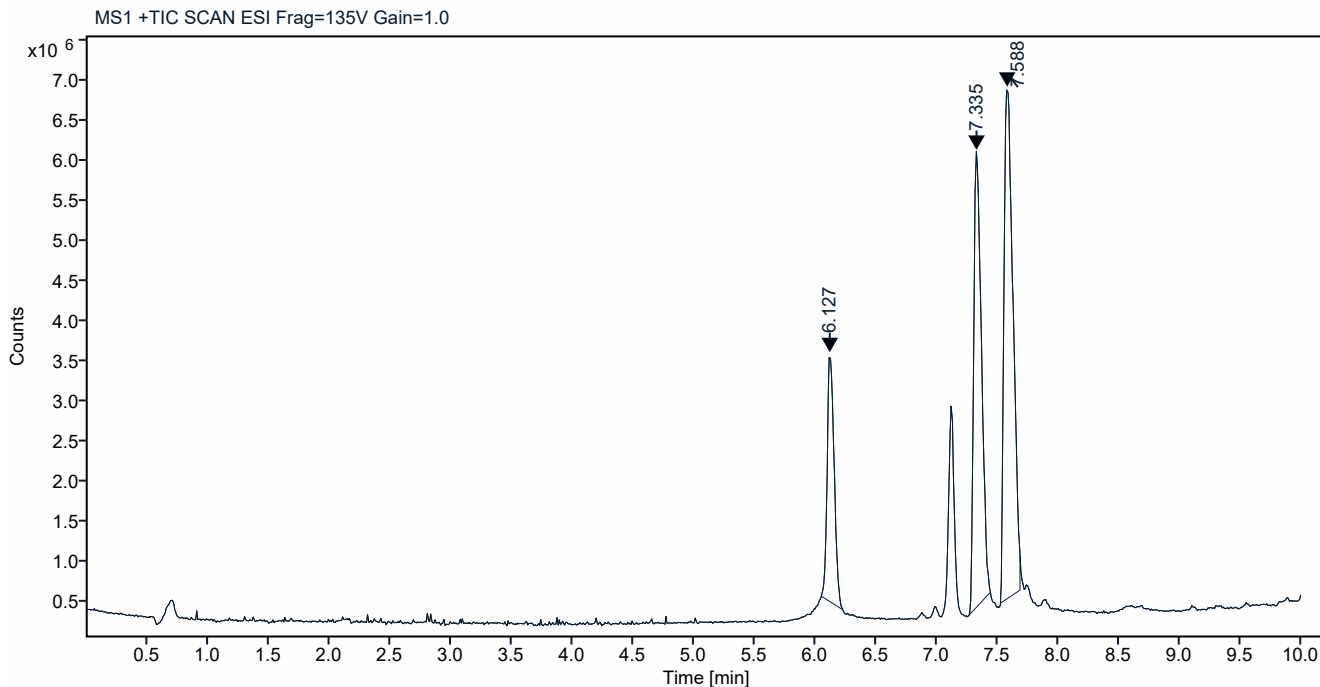
Injection Volume 5.000

Injection Data File GCMS standard mix.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 18:56:42-05:00
Date

Data File Path C:\CDSProjects\TBI -
Piney\Results\Validation\8-21-
24\GCMS standard mix.sirslt



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Analysis Report

Sample Name Isonitazene lot#0577984-1 cayman
chemical exp 2-14-24 MeOH

Instrument Piney
Name

Sample Vial P1-A7
Number

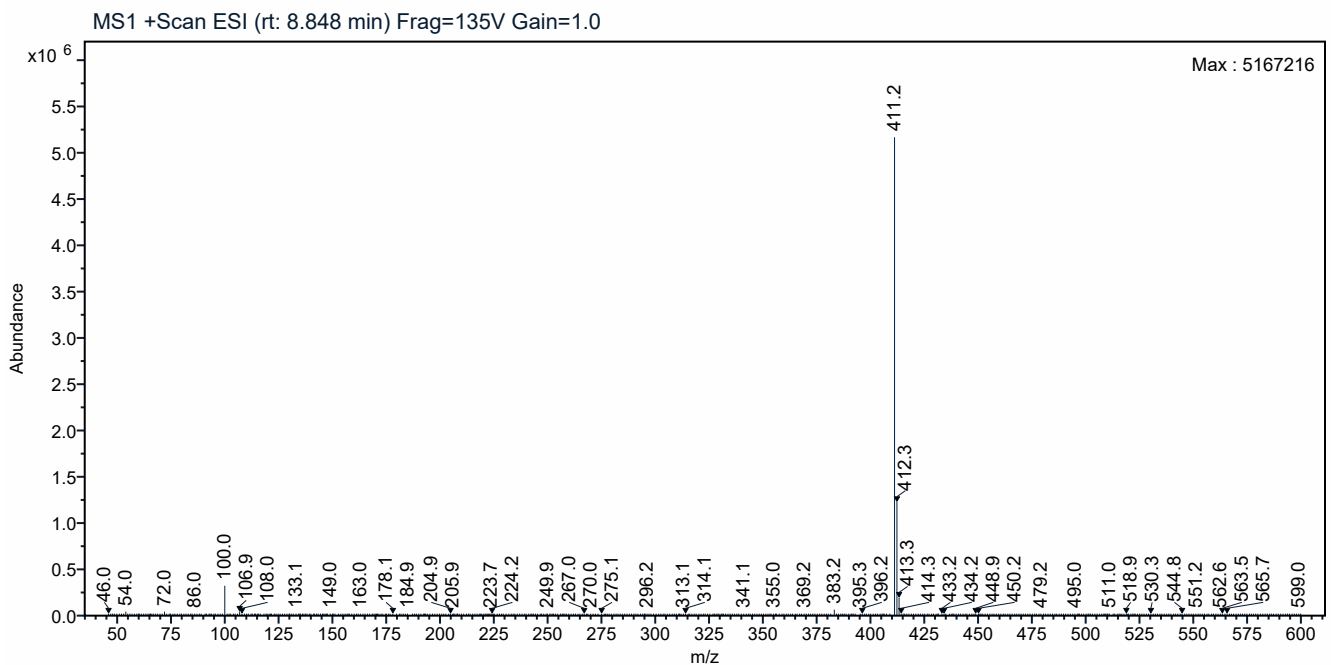
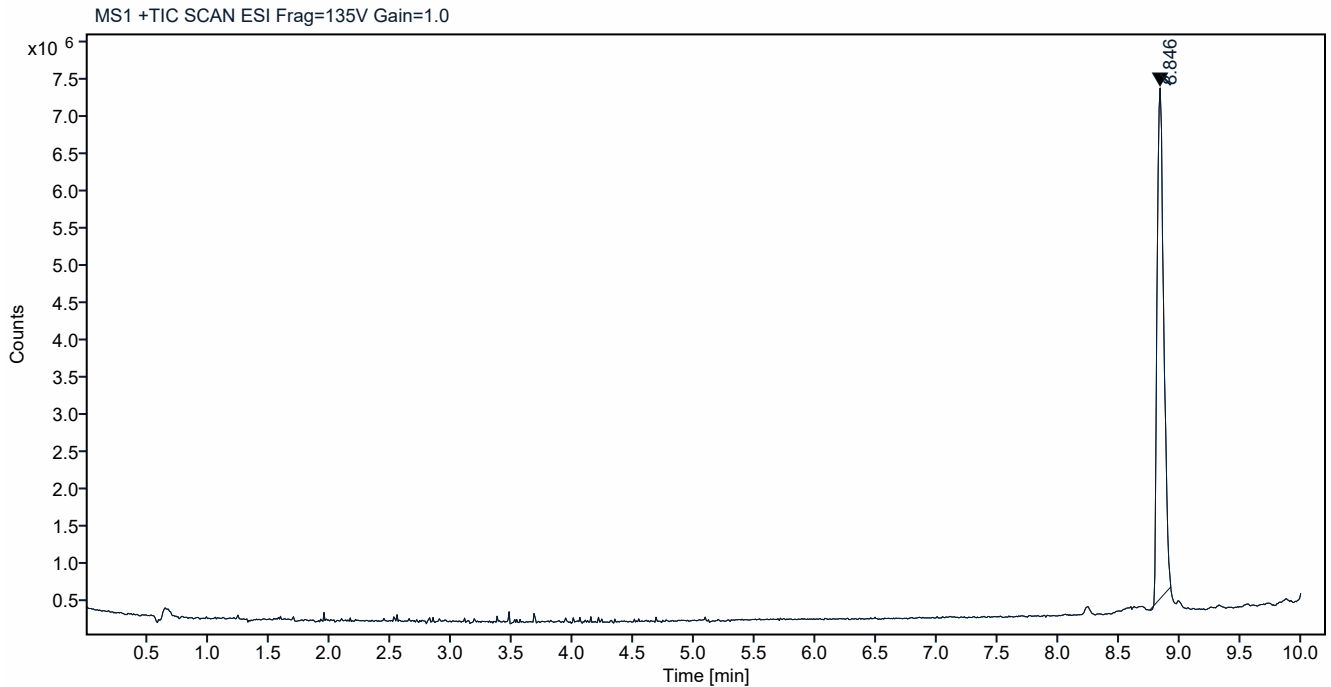
Injection Volume 5.000

Injection Data File Isonitazene.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 15:34:21-05:00
Date

Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Isonitazene.sirslt



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Analysis Report

Sample Name LSD lot# FE10161901 cerilliant exp 10-2024 MeOH

Injection Data File LSD.dx
Name

Instrument Piney
Name

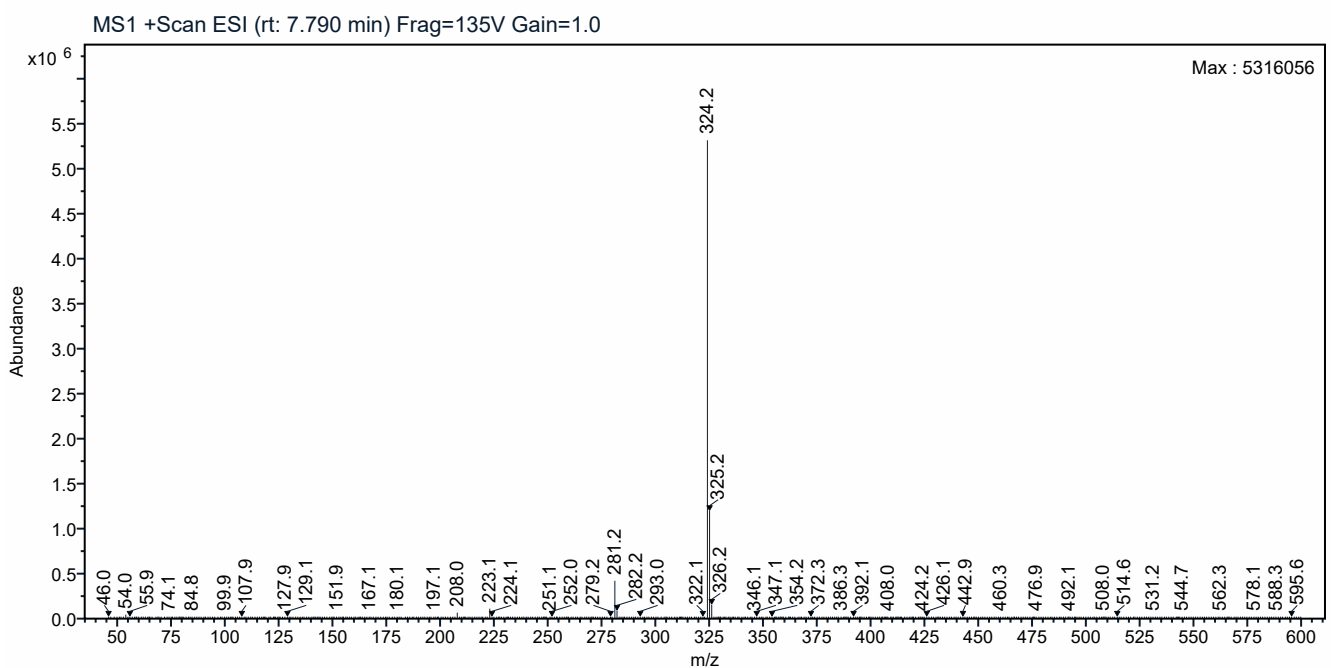
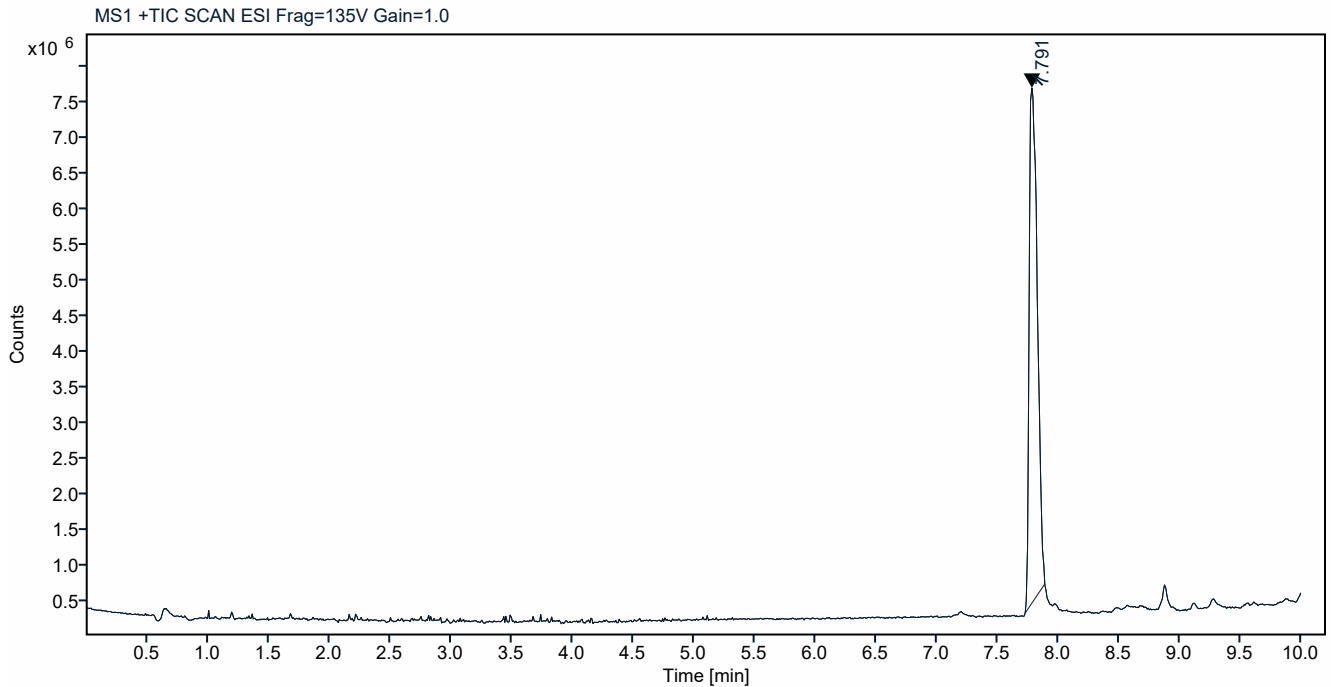
Injection Acq TBI Drug Screen.amx
Method Name

Sample Vial P1-A8
Number

Injection Acquired 2024-08-21 15:59:38-05:00
Date

Injection Volume 5.000

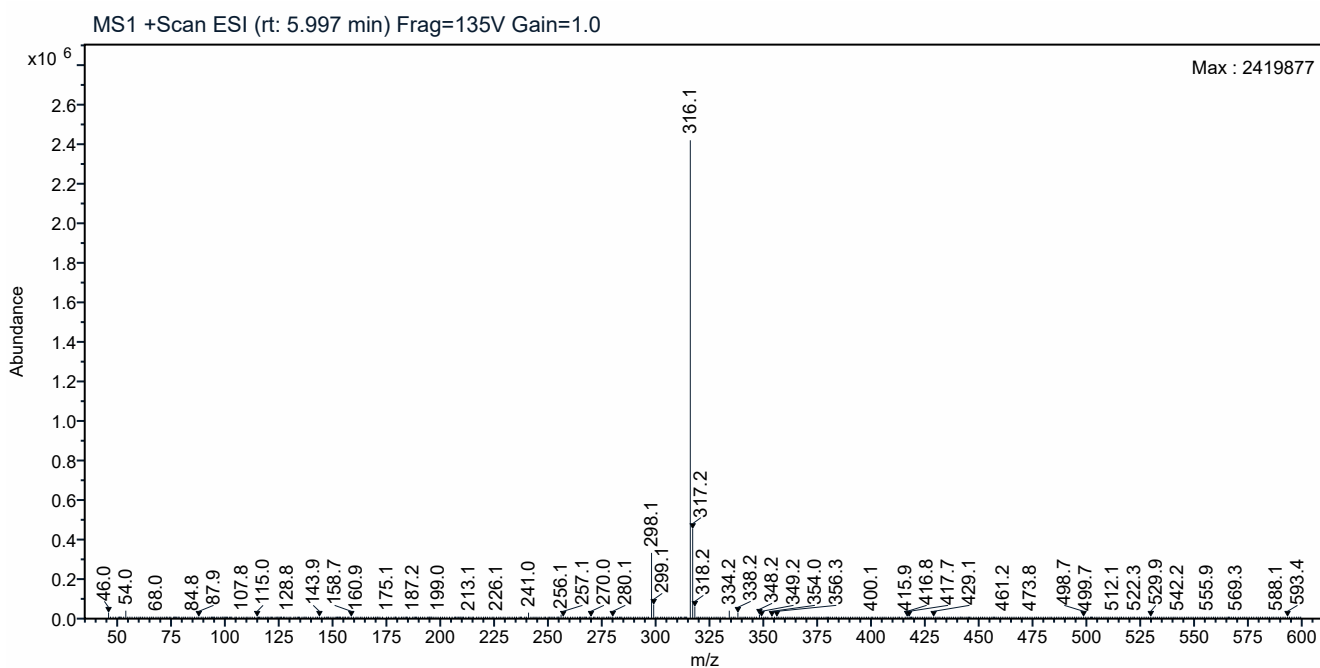
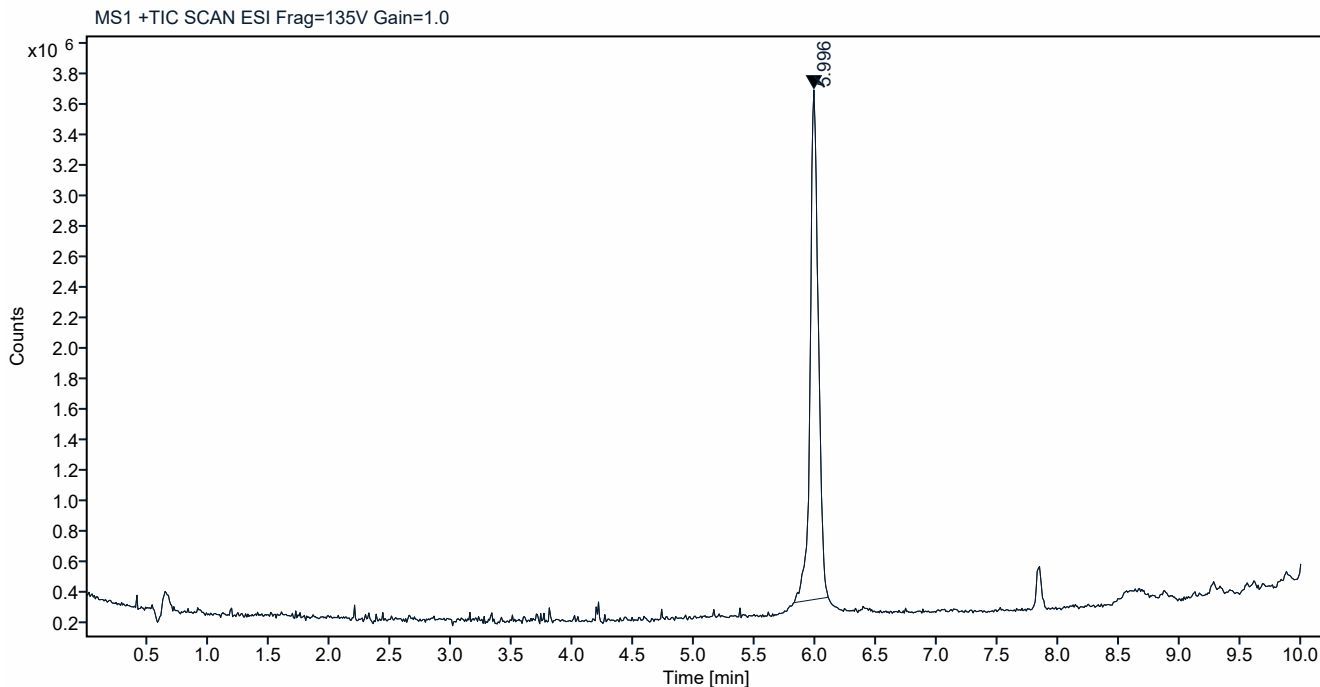
Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\LSD.sirsl



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Analysis Report

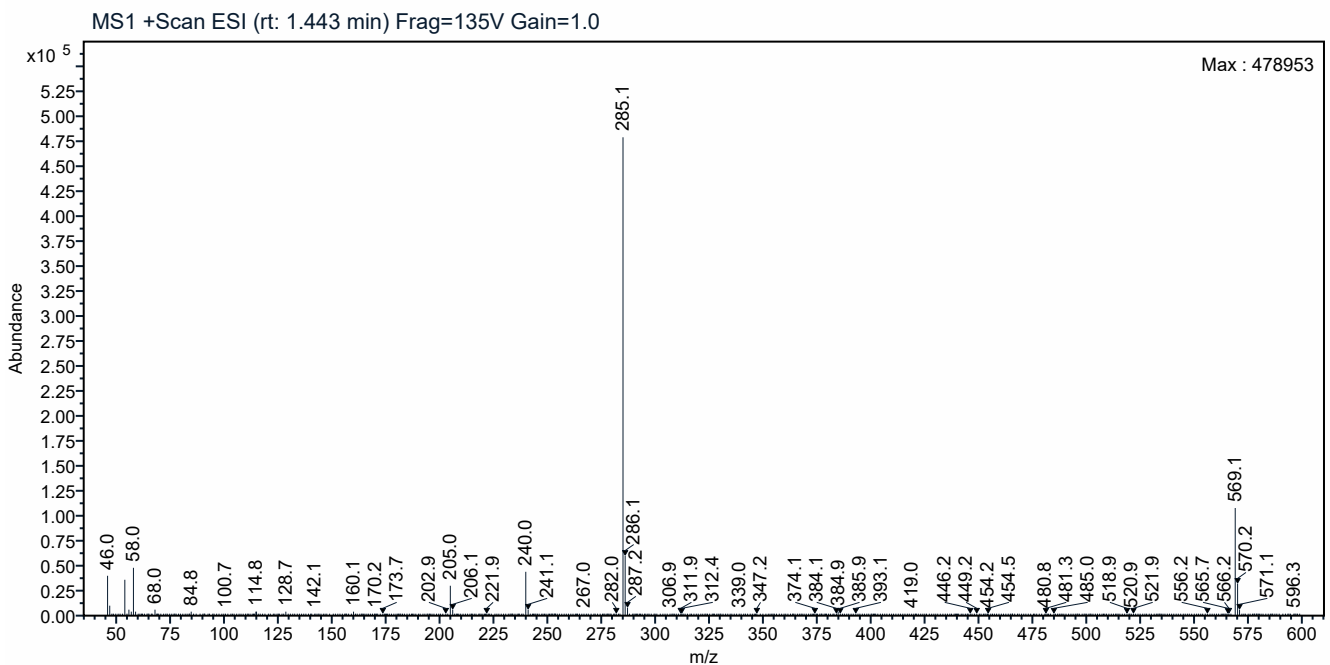
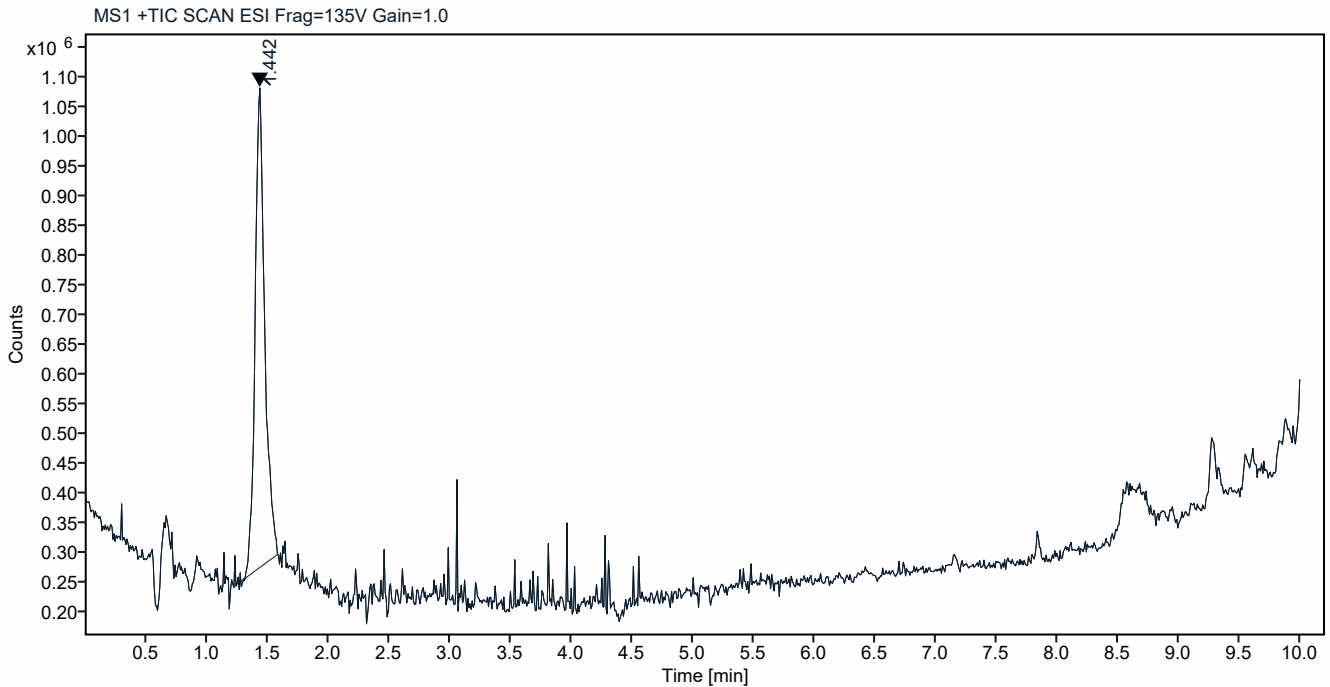
Sample Name	Oxycodone lot#FE10161901 cerillant 01-2025 MeOH	Injection Data File	Oxycodone.dx
Instrument	Piney	Name	
Injection Name		Injection Acq	TBI Drug Screen.amx
Sample Vial	P1-A9	Method Name	
Number		Injection Acquired	2024-08-21 16:24:55-05:00
Injection Volume	5.000	Date	
		Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21- 24\Oxycodone.sirsl



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Analysis Report

Sample Name	Psilocybin lot# FE7232102 cerilliant exp 4-2026 MeOH	Injection Data File Name	Psilocybin.dx
Instrument Name	Piney	Injection Acq Method Name	TBI Drug Screen.amx
Sample Vial Number	P1-A11	Injection Acquired Date	2024-08-21 17:15:30-05:00
Injection Volume	5.000	Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21-24\Psilocybin.sirsl



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Analysis Report

Sample Name Psilocyn lot#FE08092301 cerilliant exp
8-31-25 MeOH

Injection Data File Psilocyn.dx
Name

Instrument Piney
Name

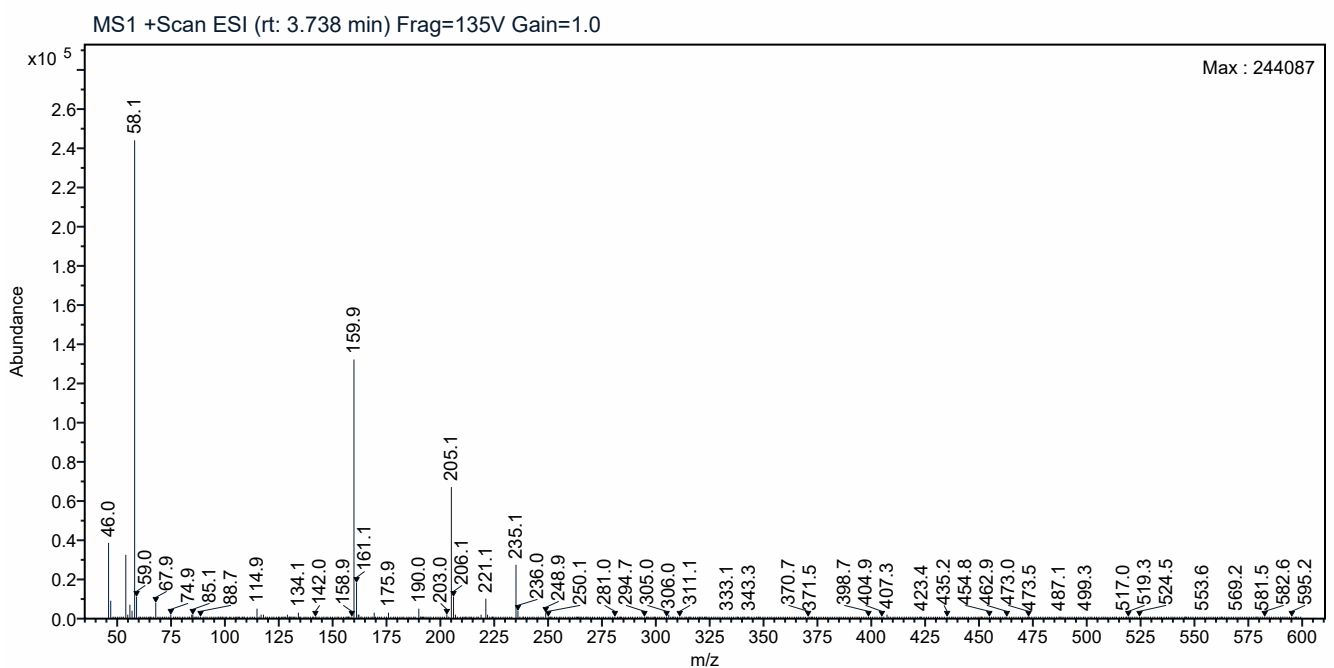
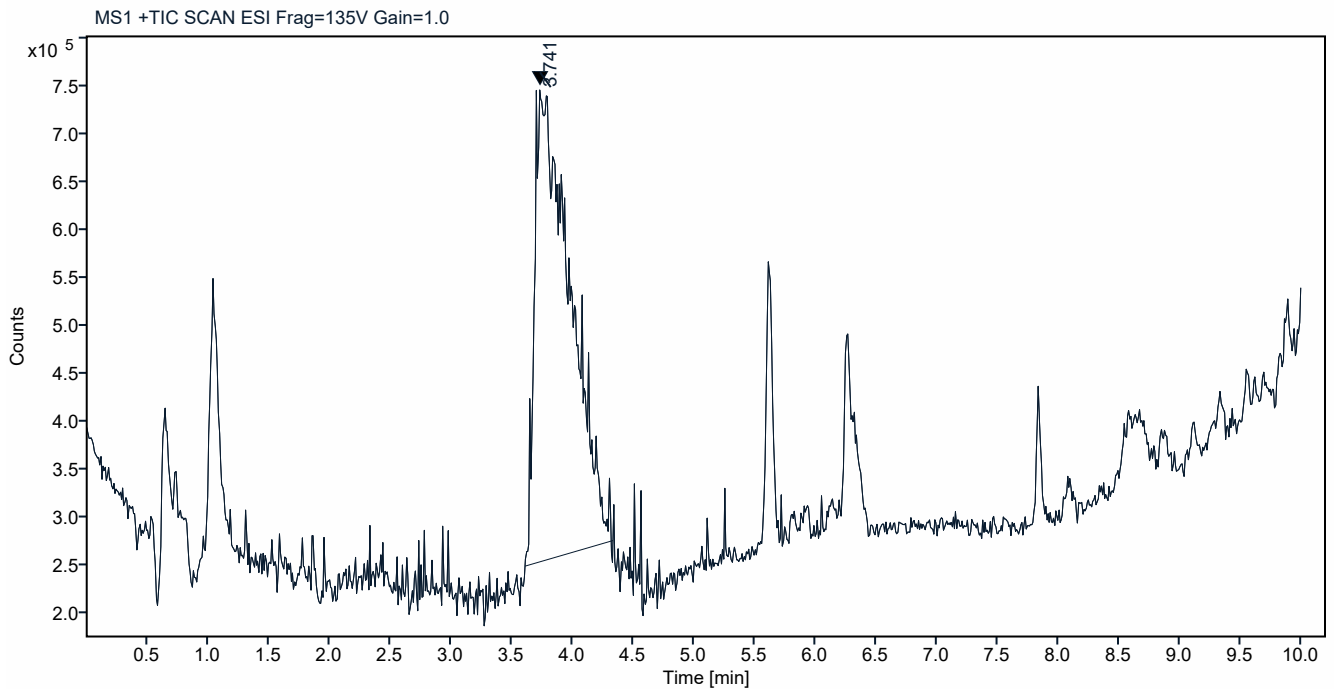
Injection Acq TBI Drug Screen.amx
Method Name

Sample Vial P1-A10
Number

Injection Acquired 2024-08-21 16:50:13-05:00
Date

Injection Volume 5.000

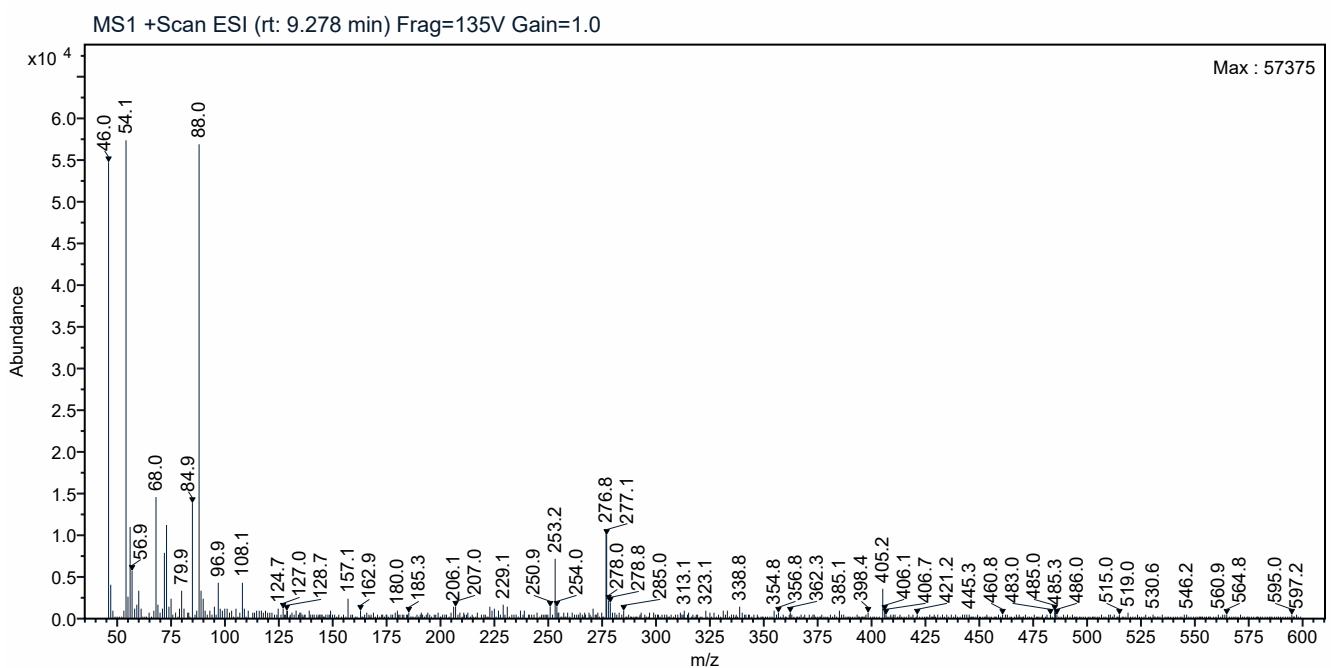
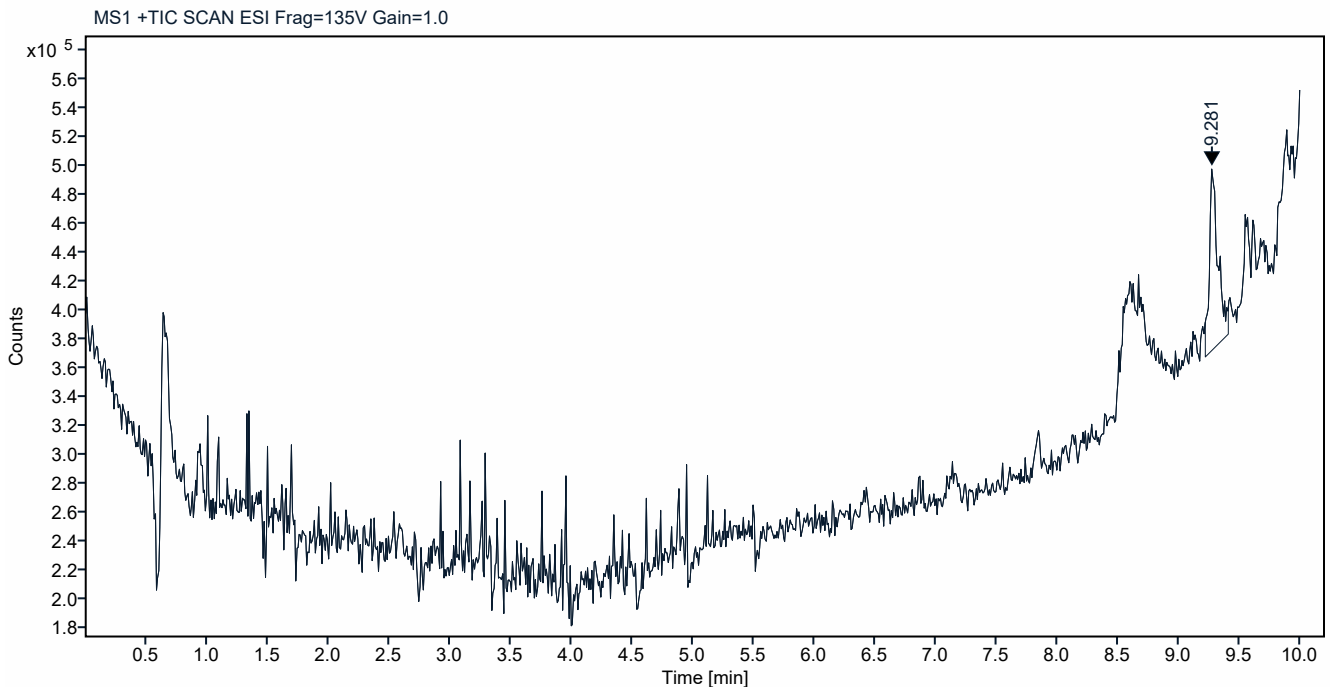
Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Psilocyn.sirsl



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Analysis Report

Sample Name	Testosterone lot#50H0345 sigma exp 11-7-24 MeOH	Injection Data File	Testosterone.dx
Instrument Name	Piney	Injection Acq Method Name	TBI Drug Screen.amx
Sample Vial Number	P1-B1	Injection Acquired Date	2024-08-21 17:40:49-05:00
Injection Volume	5.000	Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21-24\Testosterone.sirsl



AK

Analysis Report

Sample Name Tianeptine lot#05755807-12 cayman
cehmical exp7-19-25 MeOH+HCl

Instrument Piney
Name

Sample Vial P1-B2
Number

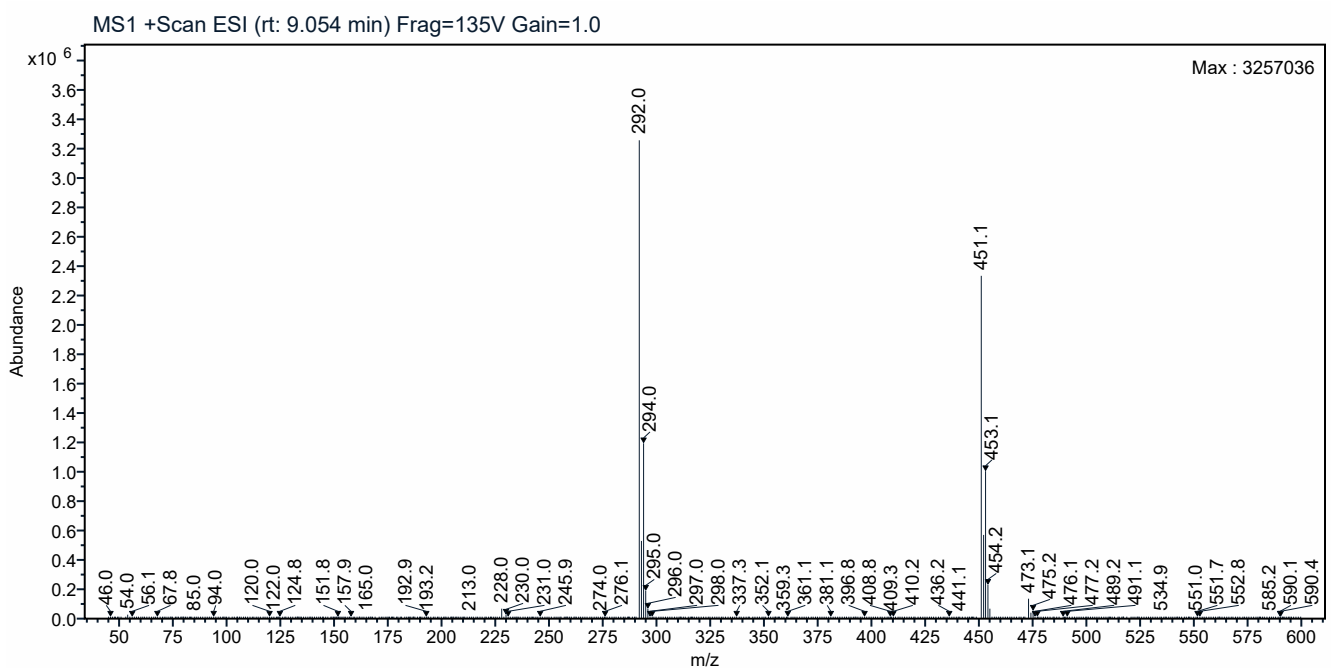
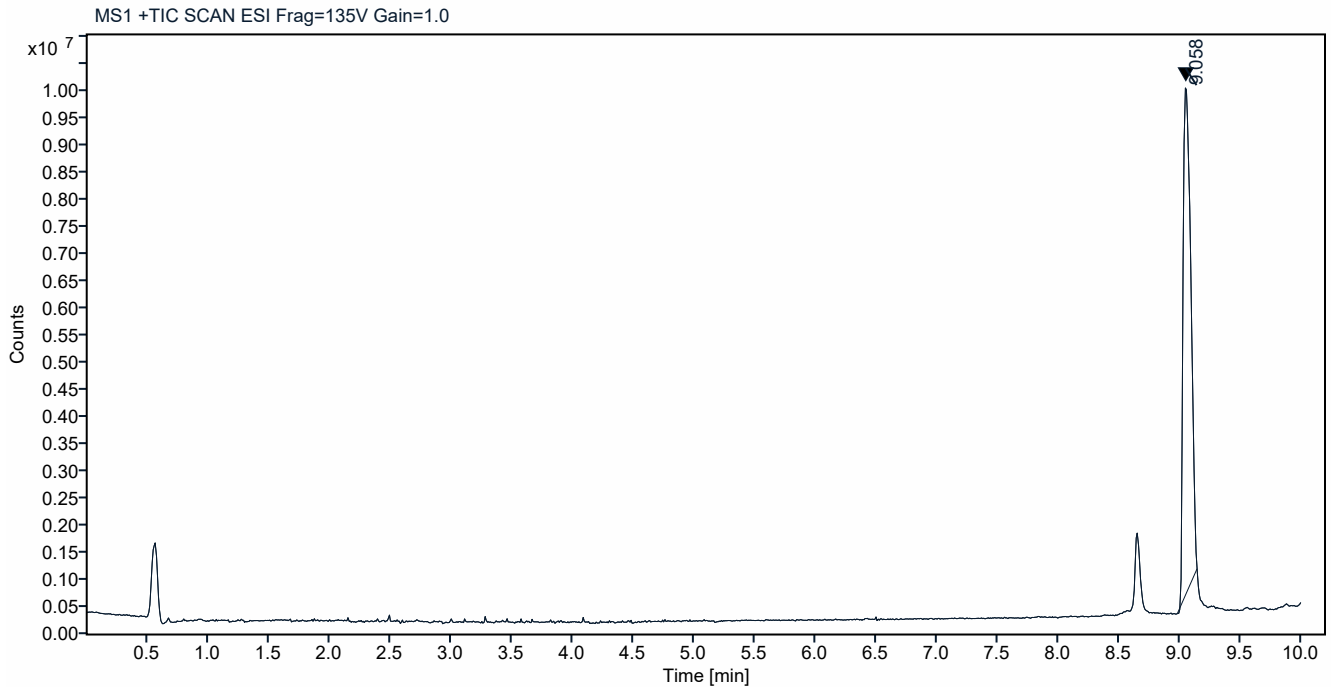
Injection Volume 5.000

Injection Data File Tianeptine.dx
Name

Injection Acq TBI Drug Screen.amx
Method Name

Injection Acquired 2024-08-21 18:06:07-05:00
Date

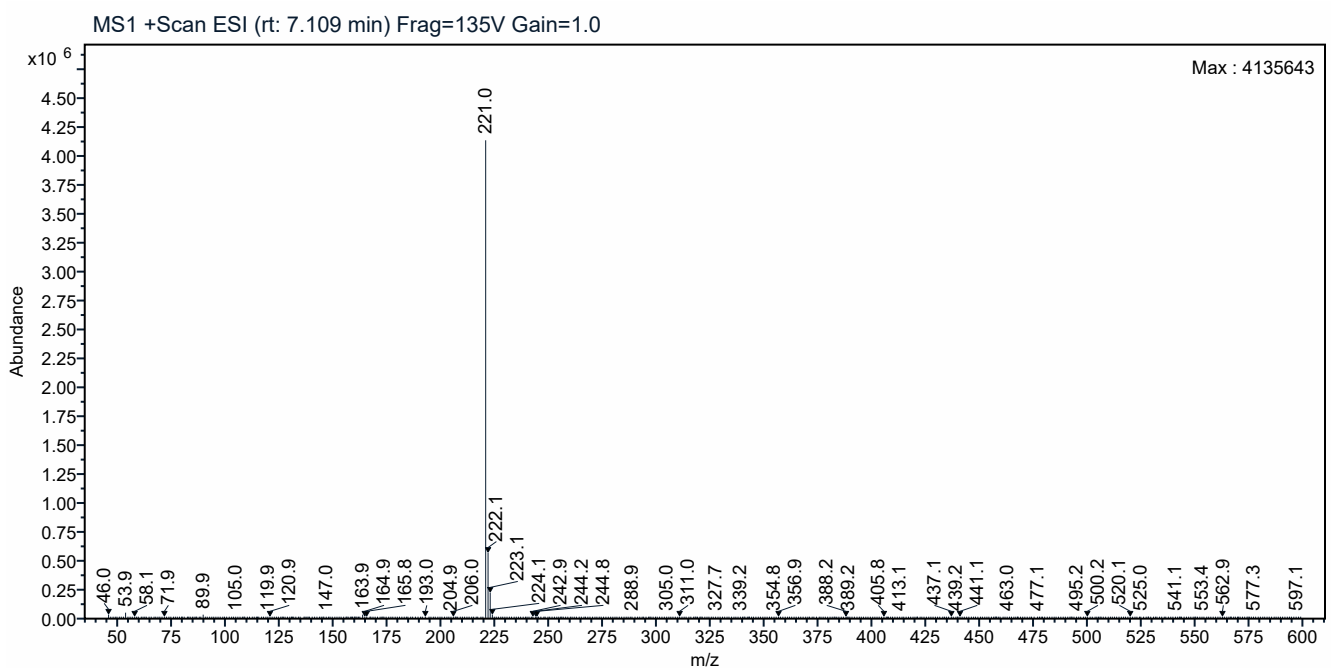
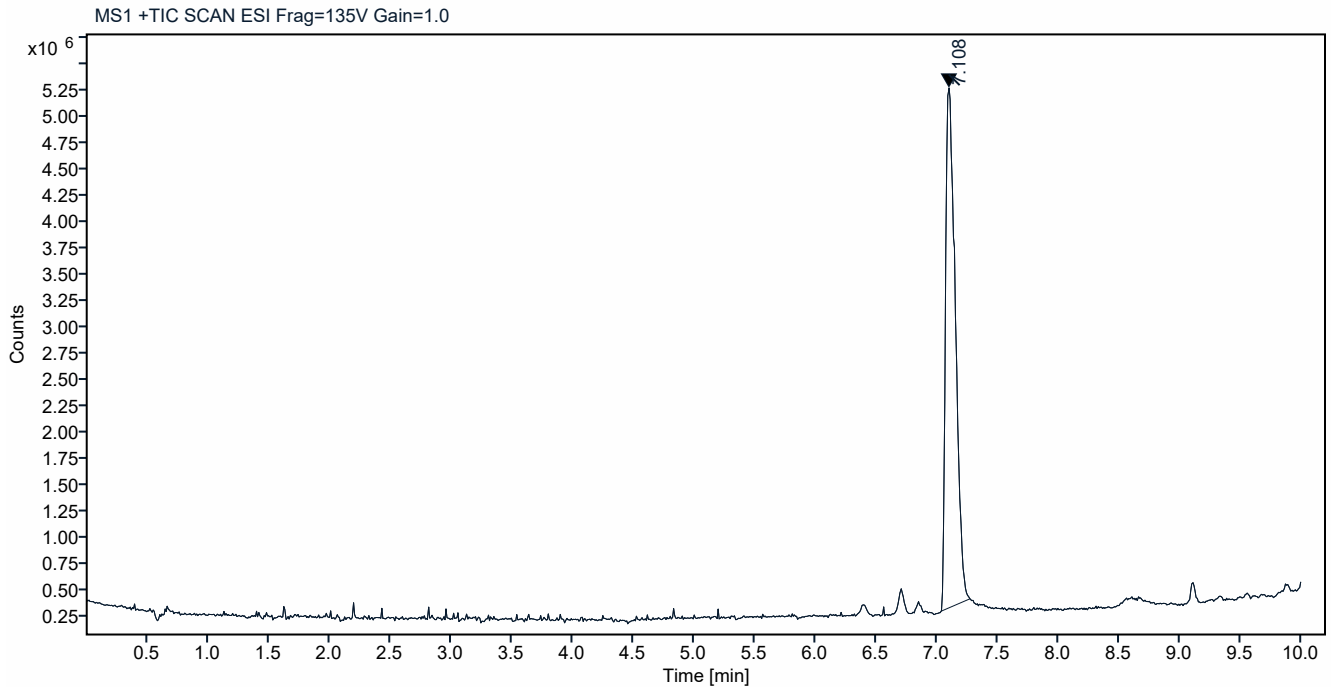
Data File Path C:\CDSPProjects\TBI -
Piney\Results\Validation\8-21-
24\Tianeptine.sirsl



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Analysis Report

Sample Name	Xylazine lot# BCBL9775V sigma 4-10-25 MeOH	Injection Data File Name	Xylazine.dx
Instrument Name	Piney	Injection Acq Method Name	TBI Drug Screen.amx
Sample Vial Number	P1-B3	Injection Acquired Date	2024-08-21 18:31:24-05:00
Injection Volume	5.000	Data File Path	C:\CDSPProjects\TBI - Piney\Results\Validation\8-21-24\Xylazine.sirsl



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