

## Laboratory Report

July 06, 2023

Shannon Shea - LFSB  
US EPA New England R1

Project Number: 23060033  
Project: MS4 Hartford, CT  
Analysis: HPLC/MS/MS Source Tracking Analysis  
EPA Chemist: Peter Philbrook

Date Samples Received by the Laboratory: 06/16/2023

### Analytical Procedure:

All samples were received and logged in by the laboratory according to the USEPA New England Laboratory SOP for Sample Log-in.

Sample preparation and analysis was done following the EPA Region I SOP, LSBSOP-LCMS\_STA3.

The SOP is based on an EPA Regional Analytical Method developed at the EPA New England Laboratory.

Data were reviewed in accordance with the internal verification procedures described in the EPA New England Quality Manual for NERL.

Results relate only to the items tested or to the samples as received by the Laboratory. This analytical report shall not be reproduced except in full, without written approval of the laboratory.

If you have any questions please call me at 617-918-8340 .

Sincerely,

DANIEL

BOUDREAU

Digitally signed by  
DANIEL BOUDREAU

Date: 2023.07.06  
16:09:43 -04'00'

23060033\$STA

**Qualifiers:**

**RL** = Reporting limit

**ND** = Not Detected above Reporting limit

**NA** = Not Applicable due to high sample dilutions or sample interferences

**NC** = Not calculated since analyte concentration is ND.

**J** = Estimated value

**J1** = Estimated value due to MS recovery outside acceptance criteria

**J2** = Estimated value due to LFB result outside acceptance criteria

**J3** = Estimated value due to RPD result outside acceptance criteria

**J4** = Estimated value due to LCS result outside acceptance criteria

**E** = Estimated value exceeds the calibration range

**L** = Estimated value is below the calibration range

**B** = Analyte is associated with the lab blank or trip blank contamination. Values are qualified when the observed concentration of the contamination in the sample extract is less than 10 times the concentration in the blank.

**R** = No recovery was calculated since the analyte concentration is greater than four times the spike level.

**P** = The confirmation value exceeded 35% difference and is less than 100%. The lower value is reported.

**C** = The identification has been confirmed by GC/MS.

**A** = Suspected Aldol condensation product.

**N** = Tentatively identified compound.

MS4 Hartford, CT

HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart SW-A	Lab Sample ID:	AC07104
Date of Collection:	6/14/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	2.6	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	4.0	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	16	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	0.47	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	88	37 - 113
Diphenhydramine-D3	70	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

MS4 Hartford, CT

HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart SW-B	Lab Sample ID:	AC07105
Date of Collection:	6/14/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	3.6	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	6.8	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	29	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	0.56	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	98	37 - 113
Diphenhydramine-D3	66	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart 09	Lab Sample ID:	AC07106
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	13	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	5.5	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	19	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	0.63	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	77	37 - 113
Diphenhydramine-D3	42	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart 01D	Lab Sample ID:	AC07107
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	0.71	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	ND	1	2.0	
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	ND	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	ND	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	84	37 - 113
Diphenhydramine-D3	69	8 - 114

Comments:

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart 01 AG	Lab Sample ID:	AC07108
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	1.1	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	8.4	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	44	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	1.7	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	97	37 - 113
Diphenhydramine-D3	75	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart 01C	Lab Sample ID:	AC07109
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	52	1	0.40	
103-90-2	Acetaminophen	23	1	2.0	
611-59-6	Paraxanthine	30	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	370	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	ND	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	65	37 - 113
Diphenhydramine-D3	35	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Hart 10A	Lab Sample ID:	AC07110
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	3.8	1	0.40	
103-90-2	Acetaminophen	24	1	2.0	
611-59-6	Paraxanthine	4.8	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	250	1	4.0	
56392-17-7	Metoprolol	4.9	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	ND	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	79	37 - 113
Diphenhydramine-D3	66	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Dup01	Lab Sample ID:	AC07111
Date of Collection:	6/15/2023	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	4.1	1	0.40	
103-90-2	Acetaminophen	25	1	2.0	
611-59-6	Paraxanthine	4.8	1	2.0	J
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	260	1	4.0	
56392-17-7	Metoprolol	5.1	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	ND	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	79	37 - 113
Diphenhydramine-D3	62	8 - 114

**Comments:** J = Estimated value - Paraxanthine continuing calibration verification was above QC criteria (145%), where 70=130% is required.

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**Laboratory Blank**

Client Sample ID:	N/A	Lab Sample ID:	N/A
Date of Collection:	N/A	Matrix:	Stormwater
Date of Preparation:	6/20/2023	Amount Prepared:	500 mL
Date of Analysis:	6/21/2023	Percent Solids:	N/A
Dry Weight Prepared:	N/A	Extract Dilution:	1
Wet Weight Prepared:	N/A	pH:	N/A

CAS Number	Compound	Concentration ng/L	Dilution	RL ng/L	Qualifier
486-56-6	Cotinine	ND	1	0.40	
103-90-2	Acetaminophen	ND	1	2.0	
611-59-6	Paraxanthine	ND	1	2.0	
29122-68-7	Atenolol	ND	1	2.0	
58-08-2	Caffeine	ND	1	4.0	
56392-17-7	Metoprolol	ND	1	2.0	
58-73-1	Diphenhydramine	ND	1	0.40	
298-46-4	Carbamazepine	ND	1	0.40	

Surrogate Compounds	Recoveries (%)	QC Ranges
Phentermine-D5	94	37 - 113
Diphenhydramine-D3	79	8 - 114

**Comments:**

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MATRIX SPIKE (MS) RECOVERY

Sample ID: AC07108

PARAMETER	SPIKE ADDED ng/L	SAMPLE CONCENTRATION ng/L	MS CONCENTRATION ng/L	MS % REC	QC LIMITS (% REC)
Acetaminophen	120	ND	96.1	80	55 - 99
Atenolol	120	ND	111	93	24 - 160
Caffeine	240	44.0	251	86	34 - 131
Carbamazepine	24.0	1.7	22.1	85	61 - 107
Cotinine	24.0	1.1	22.1	87	48 - 112
Diphenhydramine	24.0	ND	19.2	80	17 - 112
Metoprolol	120	ND	120	100	31 - 137
Paraxanthine	120	8.4	139	109	41 - 132

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Laboratory Duplicate Results

Sample ID: AC07107

PARAMETER	SAMPLE RESULT ng/L	SAMPLE DUPLICATE RESULT ng/L	PRECISION RPD %	QC LIMITS
Acetaminophen	ND	ND	NC	50
Atenolol	ND	ND	NC	50
Caffeine	ND	ND	NC	50
Carbamazepine	ND	ND	NC	50
Cotinine	0.70562	0.735	4.1	50
Diphenhydramine	ND	ND	NC	50
Metoprolol	ND	ND	NC	50
Paraxanthine	ND	ND	NC	50

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14laboratory Fortified Blank (LFB) Results

PARAMETER	LFB AMOUNT SPIKED ng/L	LFB RESULT ng/L	LFB RECOVERY %	QC LIMITS %
Acetaminophen	120	93.1	78	45 - 106
Atenolol	120	99.1	83	33 - 129
Caffeine	240	185	77	41 - 120
Carbamazepine	24	20.9	87	63 - 108
Cotinine	24	19.4	81	55 - 106
Diphenhydramine	24	19.9	83	15 - 113
Metoprolol	120	99.7	83	41 - 122
Paraxanthine	120	128	107	52 - 115

Comments:

Samples in Batch: AC07104, AC07105, AC07106, AC07107, AC07108, AC07109, AC07110, AC07111

## CHAIN OF CUSTODY RECORD

[illegible]

# **US EPA REGION 1 SAMPLE RECEIPT CHECKLIST**

PROJ#: 23060033	RECEIPT DATE: 6-16-23
SURVEY NAME: MS4 LOCATION: HARTFORD, CT	REC'D BY: Daniel Casey (ESAT)
OSC/PO: SHANNON SHEA (LFSB)	SITE ID: SUPERFUND: N <input checked="" type="checkbox"/>

WERE SAMPLES SHIPPED? N <input checked="" type="checkbox"/> <input checked="" type="checkbox"/> TRACKING #: _____ DATE SENT: _____ NO. Hand Delivered <input checked="" type="checkbox"/>	COMMENTS: STORMWATER SAMPLES  8 #SSTA
COOLER TEMPERATURE UPON ARRIVAL <u>NA</u> °C / NA	
CHAIN OF CUSTODY PRESENT? Y <input checked="" type="checkbox"/> COMPLETE? Y <input checked="" type="checkbox"/>	
CUSTODY SEALS PRESENT ON COOLER? N <input checked="" type="checkbox"/> SAMPLES? N <input checked="" type="checkbox"/>	
WERE SAMPLE CONTAINERS INTACT? Y <input checked="" type="checkbox"/>	
WAS SAMPLE PRESERVATION DOCUMENTED? Y <input checked="" type="checkbox"/> COC Sample Container ✓	
APPROPRIATE SAMPLES VOLUME FOR REQUESTED ANALYSIS? Y <input checked="" type="checkbox"/>	
SAMPLES AND COC MATCH? Y <input checked="" type="checkbox"/>	
IF ANY PROBLEMS WAS PROJECT MANAGER NOTIFIED? BY WHOM? _____	
APPROPRIATE SAMPLE CONTAINERS? Y <input checked="" type="checkbox"/>	
SAMPLES WITHIN HOLDING TIMES? Y <input checked="" type="checkbox"/>	
ALL ANALYSIS SPECIFIED ON COC? Y <input checked="" type="checkbox"/>	
DATE/TIME OF COLLECTION ON COC Y <input checked="" type="checkbox"/>	
TURN-AROUND TIME: 4 weeks	