100SLOWSCRN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

General purpose

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 100°C hold for 2 min, 11°C/min to 280°C, hold for 3 min, 35°C/min to 310°C, hold for 4 min
Minimum Run Time: 26.221 min
Detector Temperature: 300°C

Limitations:

See individual instrument validation reports.

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN1H2 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-CSI (or DB-5MS) 15 m × 0.25 mm × 0.25 µm LTM column, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 3.7 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program: 280°C
Minimum Run Time: 3.9667 min
LTM Program: 60°C hold for 0.5 min, ramp 120°C/min to 230°C, hold for 0.3 min, ramp 120°C/min to 320°C, hold for 1 min.
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GCGEN6b - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-1 15 m × 0.32 mm × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.8 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 80°C hold for 1 min, ramp 30°C/min to 300°C, ramp 20°C to 320°C, hold for 2 min
Minimum Run Time: 11.333 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN7 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-1 15 m × 0.32 mm × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.8 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 80°C hold for 1 min, ramp 15°C/min to 320°C, hold for 2 min
Minimum Run Time: 19 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN8 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 12 m × 0.2 mm × 0.33 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 80°C hold for 1 min, ramp 30°C/min to 320°C, hold for 2 min
Minimum Run Time: 11 min
Detector Temperature: 280°C or 300°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN12 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-17 20 m × 0.18 mm × 0.18 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 2.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 100°C hold for 1.0 min, ramp 15°C/min to 320°C, hold for 5 min
Minimum Run Time: 20.667 min
Detector Temperature: 280°C or 300°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCHIGH-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Cocaine, Heroin, Fentanyl, and Other Late Eluting Compounds)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Internal Standard Solution:

Tetracosane in selected solvent (optional)

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph with Flame Ionization Detector, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program: 210°C, hold 0.1 min, ramp 30°C/min to 300°C, hold 1.4 min
Total Run Time: 4.50 min
Detector Temperature: 280°C

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOW-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7-2.0 mL/min
Control Mode: Constant flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C, hold 1.0 min
Total Run Time: 4.00 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOWX-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7-2.0 mL/min, hold 3 min, ramp at 10 mL/min to 2.5 mL/min, hold for remainder of method runtime
Control Mode: Ramped flow
Oven Program: 75°C, hold 0.5 min, ramp at 40°C/min to 175°C, no hold, ramp at 30°C/min to 300°C, hold 1.9 min
Total Run Time: 9.00 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMIDC-H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.32 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.0 mL/min (0.5 min hold), ramped to 3 mL/min at 0.5 mL/min/min
Make-up Gas: Helium or Nitrogen
Control Mode: Ramped flow
Oven Program: 170°C initial temperature (0.5 min hold), ramp to 320°C at 35°C/min
Minimum Run Time: 4.78 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMIDE-H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.25 mm x 0.25µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.0 mL/min (0.5 min hold), ramped to 3 mL/min at 0.5 mL/min/min
Make-up Gas: Helium or Nitrogen
Control Mode: Ramped flow
Oven Program: 170°C initial (0.5 min hold), ramp to 320°C at 35°C /min
Minimum Run Time: 4.78 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMID-LTMA-H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.0 mL/min (hold 0.5 min), ramped to 3 mL/min at 0.5 mL/min/min
Make-up Gas: Helium or Nitrogen
Control Mode: Ramped flow
Oven Program: 170°C initial temperature (0.5 min hold), ramp to 320°C at 35°C /min
LTM Temperature: 170°C initial temperature (0.5 min hold), ramp to 320°C at 35 °C /min
Minimum Run Time: 4.78 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GENGC30 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent GC 7890A with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.320 mm x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min for 2.4 min; ramp at 45 mL/min/min to 2.5 mL/min for 1 min; ramp at 45 mL/min/min to 3.7 mL/min for 3.5 min and hold
Make-up Gas: Nitrogen
Control Mode: Ramped flow
Oven Program Set Points: 170°C for 0.5 min then 30°C/min to 300°C, 20°C/min to 320°C for 1.6 min
Minimum Run Time: 7.433 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

G-GEN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 8890 Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-5 12 m x 0.20 mm I.D. x 0.33 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 90°C (0.5 min hold); 40°C/min to 175°C; 20°C/min to 310°C (1.625 min hold)
Minimum Run Time: 11.0 min
Detector Temperature: 300°C

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GS1_361907; GS1F_361929; GS1F_361941 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: Pre-Column: DB-5 1 m x 0.180 mm ID x 0.18 µm; Column: DB-5MS LTM 15 m x 0.250 mm ID x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow Rate: Hydrogen, 3.25 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 300°C for 4.2 min
LTM Program Set Points: 100°C; ramp 80°C/min to 300°C hold for 1.7 min
Minimum Run Time: 4.2 min
Detector Temperatures:
GS1F_361929; GS1F_361941: 300 °C
GS1_361907: 280 °C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability:
Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:
Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GS1B_361942 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-5MS 15 m x 0.25 mm ID x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow Rate: Hydrogen, 3.25 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant Flow
Oven Program Set Points: 300°C for 4.2 min
LTM Program Set Points: 100°C; ramp 80°C/min to 300°C hold for 0.5 min
Minimum Run Time: 4.2 min
Detector Temperature: 300°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

GS5H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph equipped with Flame Ionization Detector, or equivalent
Column Type and Dimensions: 5% phenyl, 95% methylpolysiloxane; 15 m x 0.25 mm I.D. x 0.25 µm
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program: 100°C initial, ramp at 30 °C/min to 310°C, hold for 1 min
Minimum Run Time: 7.5 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GS50H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph equipped with Flame Ionization Detector, or equivalent
Column Type and Dimensions: 50% phenyl, 50% methylpolysiloxane; 30 m x 0.25 mm I.D. x 0.25 µm
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program: 100°C initial, ramp at 30°C/min to 300°C, hold for 15 min
Minimum Run Time: 21.667 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

HT30SCRN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890A Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-5 30 m x 0.320 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.5 mL/min (2.4 min); ramp at 45 mL/min/min to 2.5 mL/min (1 min); ramp at 45 mL/min/min to 3.7 mL/min (3.5 min)
Make-up Gas: Nitrogen
Control Mode: Constant Pressure
Oven Program Set Points: 240°C for 3.5 min then 45°C/min to 300°C for 2.2 min
Minimum Run Time: 7.033 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

ISOM01 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine Diastereoisomers)

Sample Preparation:

6-8 mg of sample is weighed into a test tube. 1 mL of 1 N sodium hydroxide is added and mixed thoroughly. 2 mL of hexane is added and mixed thoroughly to extract. Transfer the top (hexane) sample layer to a new test tube and discard the bottom basic layer. Three drops of α-methoxy-α trifluoromethylphenylacetic acid (MTPA) reagent is added to sample and vortexed for 5 seconds. Filter and transfer to an autosampler vial.

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.32 mm I.D. x 0.25 µm, or equivalent
Inlet Temperature: 260°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 35:1
Carrier Gas and Flow: Hydrogen, 1.75 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 220°C isothermal
Minimum Run Time: 5.5 min
Detector Temperature: 300°C

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

ISOM02 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Positional isomers of fluorofentanyl and chlorofentanyl)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.32 mm I.D. x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Pulsed Split; 10 psi until 0.75 min
Maximum Split Ratio: 25:1
Carrier Gas and Flow Rate: Hydrogen, 4 mL/min (2.25 min hold); 5 mL/min/min to 1.5 mL/min (1.25 min hold for fluorofentanyls; at least 2.75 min hold for chlorofentanyls
Make-up Gas: Nitrogen
Control Mode: Ramped flow
Oven Program Set Points: 250°C (0.5 min hold); 30°C/min to 265°C (2.5 min hold); 30°C/min to 280°C (no hold for fluorofentanyls; at least 1.5 min hold for chlorofentanyls)
Minimum Run Time: 4.0 min for fluorofentanyls; 5.5 min for chlorofentanyls
Detector Temperature: 280°C (Note: 300°C also validated during ruggedness testing)

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

LGS5H - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: 5% phenyl, 95% methylpolysiloxane; 15 m x 0.25 mm I.D. x 0.25 µm
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow Rate: Hydrogen, 2 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program: 280°C initial for 5.33 min, ramp at 30 °C/min to 310°C
LTM Program: 95°C initial hold for 0.25 min, ramp at 60°C/min to 280°C, hold for 2 min, ramp at 30°C/min to 310°C
Minimum Run Time: 6.2 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

SCREEN - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: HP-5 30 m x 0.32 m x 0.25 µm, or equivalent
Inlet Temperature: 260°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 30:1
Carrier Gas and Flow Rate: Hydrogen, 3.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 100°C for 1.0 min, then 15°C/min ramp to 270°C, hold for 3.7 min
Minimum Run Time: 16.0 min
Detector Temperature: 300°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum $S/N_{pk\text{-}pk}$ =3 is observed, including a low-level (0.5%) marker compound. The earliest eluting compound was within the minimum acceptable retention time for the method ($k=2t_0$) and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during six weeks are within 0.1 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

SCREEN_LTM - Separation of Controlled and Non-Controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Flame Ionization Detector, or equivalent
Column Type and Dimensions: Guard Column: DB-5, 1.0 m x 0.18 mm x 0.18 µm; LTM Column: DB-5, 15 m x 0.25 m x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow Rate: Hydrogen, 3.25 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 300°C
LTM Program Set Points: 100°C, no hold, 80°C/min ramp to 300°C, hold 2 min
Minimum Run Time: 4.5 min
Detector Temperature: 300°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum $S/N_{pk\text{-}pk}$ =3 is observed, including a low-level (0.5%) marker compound. The earliest eluting compound was within the minimum acceptable retention time for the method ($k=2t_0$) and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during six weeks are within 0.1 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

SCRN30 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890A Gas Chromatograph with Flame Ionization Detector, or equivalent
Column Type and Dimensions: DB-5 30 m x 0.320 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen, 2.5 mL/min (hold 5 min); ramp at 45 mL/min/min to 3.5 mL/min (hold 2.2 min)
Make-up Gas: Nitrogen
Control Mode: Constant Pressure
Oven Program Set Points: 175°C for 1 min, then 15°C/min to 280°C for 3.5 min
Minimum Run Time: 11.5 min
Detector Temperature: 280°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

AUTO100/AUTO100-Split - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Analytical Solutions and Providers IRD3 Infrared Detector, or equivalent
Column Type and Dimensions: HP-5 25 m x 320 µm x 0.52 µm, or equivalent
Inlet Temperature: 250 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split or Splitless
Maximum Split Ratio: 10:1
Carrier Gas and Flow Rate: Helium, 2.0 mL/min
Control Mode: Constant flow
Oven Program Set Points: 100°C for 2.0 min, 25°C/min ramp to 270°C, hold for 12.2 min
Minimum Run Time: 21.0 min
Detector: See Vapor Phase Infrared Spectroscopy Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =$3_{pk=pk}$ is observed, including a low level (0.5%) marker compound. The earliest eluting compound was within the minimum acceptable retention time for the method ($k=2t_0$) and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during six weeks are within 0.1 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

AUTO180/AUTO180-Split - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Analytical Solutions and Providers IRD3 Infrared Detector, or equivalent
Column Type and Dimensions: HP-5 25 m x 320 µm x 0.52 µm, or equivalent
Inlet Temperature: 250 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split or Splitless
Maximum Split Ratio: 10:1
Carrier Gas and Flow Rate: Helium, 2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 180°C for 2 min, then 25 °C/min ramp to 270°C, hold 12.4 min
Minimum Run Time: 18 min
Detector: See Vapor Phase Infrared Spectroscopy Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN15 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Analytical Solutions and Providers IRD3 Infrared Detector, or equivalent
Column Type and Dimensions: HP-5 30 m × 0.32 mm × 0.25 µm, or equivalent
Inlet Temperature: 275°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split or Splitless
Maximum Split Ratio: 10:1
Carrier Gas and Flow Rate: Helium, 2.0 mL/min
Control Mode: Constant flow
Oven Program: 60°C hold for 2.0 min, ramp 20°C/min to 320°C, hold for 7 min
Minimum Run Time: 22 min
Detector: See Vapor Phase Infrared Spectroscopy Method Parameters.

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

IGS5A - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph with Analytical Solutions and Providers IRD3 Infrared Detector, or equivalent
Column Type and Dimensions: 5% phenyl, 95% methylpolysiloxane; 30 m x 0.32 mm I.D. x 0.25 µm film thickness
Inlet Temperature: 270°C
Minimum Injection Volume: 2 µL
Injection Mode: Splitless
Maximum Split Ratio: N/A
Carrier Gas and Flow Rate: Helium, 3.0 mL/min
Control Mode: Constant flow
Oven Program: 55°C initial, ramp 30°C/min to 295 °C, hold for 5.5 min
Minimum Run Time: 13.5 min
Detector: See Vapor Phase Infrared Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 4 weeks are within 0.1 minutes of the values measured on week 1.

AMINES – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
Limited purpose (amines)

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Agilent 5977A/B Mass Selective Detector, or equivalent
Column Type and Dimensions: RESTEK Rxi-1MS 30 m x 250 µm x 0.25 µm, or equivalent
Inlet Temperature: 265°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 75:1
Carrier Gas and Flow Rate: Helium, 2.75 mL/min
Control Mode: Constant flow
Oven Program Set Points: 170°C for 3.0 min, then 40°C/min to 300°C for 0.25 min
Minimum Run Time: 6.5 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Selected compounds are visually separated. All peaks are a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound if used. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 minutes of the average of all 30 injections, and/or the individual relative retention times are within 1% of the average of all injections.

Reproducibility: Individual retention times measured during at least 5 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during at least 5 weeks are within 1% of the values measured on week 1.

AUTO70 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Agilent 5977B Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-1 12 m x 200 µm x 0.33 µm, or equivalent
Inlet Temperature: 250°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 200:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min
Control Mode: Constant flow
Oven Program Set Points: 70°C for 1.2 min, then 25°C/min ramp to 280°C, hold for 2.5 min
Minimum Run Time: 12.1 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =$3_{pk=pk}$ is observed, including a low level (0.5%) marker compound. The earliest eluting compound was within the minimum acceptable retention time for the method ($k=2t_0$) and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during six weeks are within 0.1 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

DRUG 1 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: ZB-1MS 15 m x 0.25 mm I.D. x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.27 mL/min
Control Mode: Constant flow
Oven Program Set Points: 100°C for 0.5min, 30° C/min to 310°C, hold for 1.5 min
Minimum Run Time: 9.0 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility:
Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

DRUG2 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatography with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: ZB-1MS 15 m x 0.25 mm I.D. x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 3mL/min
Control Mode: Constant flow
Oven Program Set Points: 240°C for 0.6 min, 15°C/min to 320°C, hold for 0.66667 min
Minimum Run Time: 6.6 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

EARLY - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

General purpose

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph with Agilent Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-1 30 m x 250 µm x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 2 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow Rate: Helium, 1 mL/min
Make-up Gas: N/A
Control Mode: Constant flow
Oven Program: 90°C for 2 min, 14°C/min to 300°C, hold 10 min
Minimum Run Time: 27 min
Detector: See Mass Spectrometry Method Parameters

Limitations:

See individual instrument validation reports.

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Reproducibility testing was not conducted for this method. Positive controls must be analyzed contemporaneously with the sample.

GCGEN1He - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: 15 m DB-CSI (or DB-5MS) × 0.25 mm × 0.25 µm LTM column, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 3.7 mL/min
Control Mode: Constant flow
Oven Program: 280°C
LTM Program: 60°C hold for 0.5 min, ramp 120°C/min to 230°C, hold for 0.3 min, ramp 120°C/min to 320°C, hold for 1 min.
Minimum Run Time: 3.9667 min
Detector Temperature: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GCGEN2 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: 15 m DB-CSI (or DB-5MS) × 0.25 mm × 0.25 µm LTM column, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 2.0 mL/min
Control Mode: Constant flow
Oven Program: 280°C
Minimum Run Time: 11 min
LTM Program: 60°C hold for 2.0 min, ramp 40°C/min to 320°C, hold for 2.5 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GCGEN3 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5MS 20 m × 0.18 mm × 0.18 µm, or equivalent
Inlet Temperature: 275°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program: 80°C hold for 1 min, ramp 30°C/min to 320°C, hold for 2 min
Minimum Run Time: 11 min
Detector Temperature: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GCGEN4b - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-1MS 20 m × 0.18 mm × 0.18 µm, or equivalent
Inlet Temperature: 275°C
Minimum Injection Volume: 1.0 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 80°C hold for 1 min, ramp 30°C/min to 300°C, ramp 20°C to 320°C, hold for 2 min
Minimum Run Time: 11.33 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCGEN16 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-CSI 10 m × 0.15 mm × 0.3 µm LTM column, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program: 280°C
LTM Program: 80°C hold for 0.5 min, ramp 40°C/min to 320°C, hold for 4.0 min
Minimum Run Time: 10.5 min
Detector: See Mass Spectrometry Method Parameters.

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GCHIGHB - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-35 15 m x 0.20 mm x 0.33 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min hold 0.5 min, ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Constant pressure, Ramped flow
Oven Program: 250°C initial, hold for 0.5 min, ramp to 320°C at 39°C /min, hold 1 min
Minimum Run Time: 2.00 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCHIGH-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Cocaine, Heroin, Fentanyl, and Other Late Eluting Compounds)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Internal Standard Solution:

Tetracosane in selected solvent (optional)

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7 mL/min
Control Mode: Constant flow
Oven Program: 210 °C, hold 0.1 min, ramp 30 °C/min to 300 °C, hold 0.4 min
Total Run Time: 3.50 min
Detector: See Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCHIGH-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Cocaine, Heroin, Fentanyl, and Other Late Eluting Compounds)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Internal Standard Solution:

Tetracosane in selected solvent (optional)

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Helium, 1.7 mL/min
Control Mode: Constant flow
Oven Program: 210°C, hold 0.1 min, ramp 30°C/min to 300°C, and hold 0.4 min
Total Run Time: 4.10 min
Detector: See Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCHIGH-LTMA - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Ramped flow
Oven Program: 250°C initial, hold initial for 0.5 min, ramp to 320°C at 39°C/min
LTM Temperature: 250°C initial, hold initial for 0.5 min, ramp to 320°C at 39°C/min
Minimum Run Time: 2.00 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOWB - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-35 15 m x 0.20 mm x 0.33 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5mL/min/min
Control Mode: Ramped flow
Oven Program: 100°C initial, ramp to 320°C at 35°C/min, hold 1 min
Minimum Run Time: 6.28 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOW-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7-2.0 mL/min
Control Mode: Constant flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C
Total Run Time: 3.00 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOW-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Helium, 1.7 mL/min
Control Mode: Constant flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C, hold 0.5 min
Total Run Time: 3.50 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOW-LTMA - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-5 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (hold 0.5 min) ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Ramped flow
Oven Program: 100°C initial, ramp to 320°C at 35°C/min
LTM Temperature: 100°C initial, ramp to 320°C at 35°C/min
Minimum Run Time: 6.28 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOWX-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Hydrogen, 1.7-2.0 mL/min, hold 3 min, ramp at 10 mL/min to 2.5 mL/min, hold for remainder of method runtime
Control Mode: Ramped flow
Oven Program: 75°C, hold 0.5 min, ramp at 40°C/min to 175°C, no hold, ramp at 30°C/min to 300°C, hold 1.9 min
Total Run Time: 9.0 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCLOWX-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General Purpose

Sample Preparation:
Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary. Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis. Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:
Dimethyl phthalate in selected solvent (optional)

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas and Flow Rate: Helium, 1.7 mL/min, hold 3.5 min, ramp at 10 mL/min to 2.5 mL/min, hold 2.34 min
Control Mode: Ramped flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C, hold 0.5 min, ramp 30°C/min to 300°C, hold 2.34 min
Total Run Time: 10.0 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMIDA - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Ramped flow
Oven Program: 170°C initial (0.5 min hold), ramp to 320°C at 35°C/min
Minimum Run Time: 4.78 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMIDB - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-35 15 m x 0.20 mm x 0.33 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Ramped flow
Oven Program: 170°C initial, hold for 0.5 min, ramp to 320°C at 35°C/min, hold 1 min
Minimum Run Time: 4.78 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMID-LTMA - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph equipped with Low Thermal Mass (LTM) module and Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 270 °C
Minimum Injection Volume: 0.5 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min (hold 0.5 min) ramped to 3 mL/min at 0.5 mL/min/min
Control Mode: Ramped flow
Oven Program: 170°C initial (0.5 min hold), ramp to 320°C at 35°C/min
LTM Temperature: 170°C initial (0.5 min hold), ramp to 320°C at 35°C /min
Minimum Run Time: 4.78 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GCMS_screen_H2 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

General Purpose

Sample Preparation:

Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph, or equivalent
Column: DB-5 20 m × 180 µm I.D. × 0.36 µm, or equivalent
Inlet Temperature: 270°C
Injection Volume: 0.5 µL
Mode: Split
Split Ratio: 50:1
Carrier Gas and Flow Rate: Hydrogen, 1.0 mL/min hold 0.5 min, ramp at 0.5 mL/min/min to 3.0 mL/min,
Control Mode: Ramped flow
Oven Program: 170°C initial, hold 0.5 min, ramp at 30°C/min to 300°C, no hold, ramp at 20°C/min to 320°C
Total Run Time: 5.8333 min
Detector: See Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GEN15 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph and 5977A/B Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5ms 15 m x 250 µm x 0.25 µm, or equivalent
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rate: Helium, 1.0 mL/min for 0.5 min, then 0.5 mL/min per min to 3.0 mL/min
Control Mode: Ramped flow
Oven Program Set Points: 170°C for 0.5 min, then 30°C/min to 300°C, then 20°C/min to 320°C
Minimum Run Time: 5.833 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Selected compounds are visually separated. All peaks are a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound if used. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 minutes of the average of all 30 injections, and/or the individual relative retention times are within 1% of the average of all injections.

Reproducibility: Individual retention times measured during at least 5 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during at least 5 weeks are within 1% of the values measured on week 1.

GENSCRN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977B Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-35 15 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 40:1
Carrier Gas and Flow Rate: Hydrogen, 2.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 120°C for 1.0 min, 35°C/min to 310°C, hold for 3.0 min
Minimum Run Time: 9.429 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated, and observed as a single peak with clear non-splitting apex. Peak fronting/tailing when observed, it does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1 and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GENSCRN-30 – SFL3 Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890 Gas Chromatograph with Agilent 5977 Mass Selective Detector, or equivalent
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow Rate: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 165°C for 1.5 min, 20°C/min to 280°C, hold for 4.5 min, 30°C/min to 295°C, hold for 6.25 min.
Minimum Run Time: 18.5 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated, and observed as a single peak with clear non-splitting apex. Peak fronting/tailing when observed, it does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1 and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

GS1_361917; GS1_361947; GS1_361952; GS1_361955; GS1_361997; GS1_361954; GS1_361949; GS1_361919; GS1_361953; GS1_361948; GS1_361936 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph with Agilent 5977A Mass Selective Detector, or equivalent
Column Type and Dimensions: HP-5MS 15 m x 250 µm ID x 0.25 µm, or equivalent
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow Rates:
GS1_361917; GS1_361947; GS1_361952; GS1_361955; GS1_361954; GS1_361949; GS1_361953; GS1_361948; GS1_361936: Helium, 1.5061 mL/min*:*
GS1_361997; GS1_361919: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 110°C for 0.3 min; 65°C/min to 175°C; 45°C/min to 200°C; 35°C/min to 310°C for 1 min
Minimum Run Time: 5.99 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:
GS1_361917; GS1_361947; GS1_361955; GS1_361953; GS1_361936: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1. *GS1_361952; GS1_361997; GS_361954; GS_361949; GS1_361919; GS1_361948; GS1_361935*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

GS50 - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph with Agilent Mass Selective Detector, or equivalent
Column Type and Dimensions: 50% phenyl, 50% methylpolysiloxane; 30 m x 0.25 mm I.D. x 0.25 µm film thickness
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow Rate: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program: 100°C initial, ramp at 30°C/min to 300°C, hold for 18 min
Minimum Run Time: 24.667 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

GSRXI1a - Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
General purpose

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:
Instrument: Agilent Gas Chromatograph (220V fast heating oven) with Agilent Mass Selective Detector, or equivalent
Column Type and Dimensions: 100% dimethylpolysiloxane; 30 m x 0.25 mm I.D. x 0.25 µm film thickness
Inlet Temperature: 265 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow Rate: Helium, 2 mL/min
Control Mode: Constant flow
Oven Program: 180°C initial, hold for 2 min, ramp at 50°C/min to 310°C, hold for 4 min
Minimum Run Time: 8.6 min
Detector: See Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

THCSCRN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:
Limited Purpose (Cannabinoids)

Sample Preparation:
Weigh approximately 50 mg of plant material (excluding seeds, stems, stalks and roots) into a test tube or beaker. Add 5 mL of internal standard solution and extract at room temperature for at least 10 minutes. Vortex at least twice for 10-15 seconds during the extraction period. Filter into autosampler vial prior to analysis.

Internal Standard Solution:
0.05 mg/mL 4-androsten-3,17-dione in 9:1 methanol/chloroform, or 0.05 mg/mL testosterone in 9:1 methanol/chloroform

Method Parameters:
Instrument: Agilent 7890B Gas Chromatograph System, or equivalent
Column Type and Dimensions: DB-5MS 15 m x 0.25 mm x 0.25 µm, or equivalent
Inlet Temperature: 250°C
Injection Volume: 1 µL
Mode: Split
Split Ratio: 50:1
Carrier Gas: Helium
Carrier Gas Flow Rate: 1.2 mL/min, hold 4.3 min, ramp at 2 mL/min to 1.5 mL/min
Control Mode: Ramped flow
Oven Program: 210°C, ramp at 30°C/min to 235°C, hold 3 min, ramp at 30°C/min to 280°C
Minimum Run Time: 5.3 min
Detector: See Mass Spectrometer Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.
Repeatability: Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.
Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

MS01 – Identification of Controlled and Non-controlled Substances by Mass Spectrometry (EI)

Scope:

General Purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent Technologies 5977 Mass Spectrometer, or equivalent
Mass Analyzer: Quadrupole
Ionization Mode: Positive Electron Ionization
Scan Range: 40-500 m/z
Scan Rate: N = 2
Source Temperature: 230°C
MS Temperature: 150°C
Transfer Line Temperature: 280°C
Tune Type: Standard Tune

Limitations:

See individual instrument validation reports.

Acceptance Criteria:

Selectivity: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum. The molecular ion is present in the sample spectrum if it is present in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods are established via evaluation of system-wide historical spectral data.

LTMMS1 - Identification of Controlled and Non-controlled Substances by Mass Spectrometry (EI)

Scope:
General purpose

Sample Preparation:
GC effluent

Method Parameters:
Instrument: Agilent 5977B Mass Selective Detector [coupled to Gas Chromatograph equipped with Low Thermal Mass (LTM) module], or equivalent
Mass Analyzer: Quadrupole
Ionization Mode: Positive Electron Ionization, 70eV
Scan Range: 34-550 amu
Scan Rate: N=1
Quad Temperature: 150 °C
Source Temperature: 230 °C
Transfer Line Temperature: 280 °C
Tune Type: stune.u

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum. The molecular ion is present in the sample spectrum if it is present in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

MS2 - Identification of Controlled and Non-controlled Substances by Mass Spectrometry (EI)

Scope:
General purpose

Sample Preparation:
GC effluent

Method Parameters:
Instrument: Agilent Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole
Ionization Mode: Positive Electron Ionization
Scan Range: m/z 34-550
Scan Rate: N=1
Source Temperature: 280 °C
MS Temperature: 150 °C
Transfer Line Temperature: 280 °C
Tune Type: stune.u

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum. The molecular ion is present in the sample spectrum if it is present in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

SFL1_EARLY - Identification of Controlled and Non-controlled Substances by Mass Spectrometry (EI)

Scope:
General purpose

Sample Preparation:
GC effluent

Method Parameters:
Instrument: Agilent 5975C Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 34-550 m/z
Scan Rate: N = 2
Source Temperature: 230°C
MS Temperature: 150°C
Transfer Line Temperature: 280°C
Tune Type: stune.u

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum are of the same nominal mass as those in the reference spectrum. The molecular ion is present in the sample spectrum if it is present in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

LCMUSH – Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (Psilocybin and Psilocin in mushrooms)

Sample Preparation:
Cut plant material into pieces and grind using a mortar and pestle. Extract a portion of the ground sample into methanol. Remove 3-4 drops of the methanol extract and dilute to approximately 1.5 mL with 85mM sodium phosphate buffer for a final target analyte concentration of approximately 0.05-0.10 mg/mL. Filter through a minimum 0.2 µm filter.

Method Parameters:
Instrument: Waters Acuity (I-Class) Ultra Performance Liquid Chromatograph, equipped with a photo diode array detector, or equivalent
Column: Waters BEH C18 10 cm x 2.1 mm id x 1.7 µm, or equivalent
Column Temperature: 30°C
Buffer/Mobile Phase: 85mM sodium phosphate, pH ~1.8. Stock: Add 44 mL H_3PO_4 (85%) and 5.4 g sodium hydroxide (pellets) to 4 L deionized water. Working: Dilute 530 mL of stock to 1 L deionized water. Filter working buffer through a 0.2 µm filter.
Minimum Injection Volume: 1.0 µL, using a flow-through needle
Gradient Set Points: 97% Buffer: 3% acetonitrile for 0.5 min, linear gradient to 82% Buffer: 18% acetonitrile for 2.5 min, hold for 3.0 min, linear gradient to 97% Buffer: 3% acetonitrile for 0.5 min, hold for 2 min.
Flow Rate: 0.45 mL/min
Minimum Run Time: 8.5 minutes
Detection Wavelength: 266 nm

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

LCOPIOID1 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (opiods and related compounds)

Sample Preparation:
Samples dissolved or extracted into solvents miscible with water. Insoluble material removed from sample prior to injection; 0.22 µm filter recommended. Oxycodone was used as the fixed compound for validation purposes.

Method Parameters:
Instrument: Waters Acquity I-class Ultra Performance Liquid Chromatograph, or equivalent
Column: BEH C18 2.1 × 100 mm x 1.7 µm, or equivalent
Column Temperature: 25°C ± 5°C
Buffer: 10 mM Ammonium Formate with TFA in water, pH 3.6
Mobile Phase: 90% Buffer/10% acetonitrile, ramped to 50:50 over 2 min. Ramped back to 90:10 over 0.6 min, hold 90:10 for 0.4 min.
Minimum Injection Volume: 1.0 µL
Flow Rate: 0.35 mL/min
Minimum Run Time: 3 min
Detection Wavelength: 235 nm

Limitations:
See individual instrument validation packets.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual absolute retention times measured are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during 6 weeks were within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

LCPHEN1 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (analysis of phenethylamines and related compounds)

Sample Preparation:
Samples dissolved or extracted into solvents miscible with water. Insoluble material removed from sample prior to injection; 0.45 µm filter recommended. Methamphetamine was used as the fixed compound for validation purposes.

Method Parameters:
Instrument: Waters Acquity I-class Ultra Performance Liquid Chromatograph, or equivalent
Column: BEH C18 2.1 × 100 mm 1.7 µm, or equivalent
Column Temperature: 30°C ± 2°C
Buffer/Mobile Phase: (A) 85 mM sodium phosphate in water, pH 1.8; (B) acetonitrile
Minimum Injection Volume: 1.0 µL
Gradient Set Points: Isocratic (90% A: 10% B)
Flow Rate: 0.45 mL/min
Minimum Run Time: 5 min
Detection Wavelength: 210 nm

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria.

Repeatability: Individual absolute retention times measured are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during 6 weeks were within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

LSDSCRN - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (LSD, iso-LSD, LAMPA)

Sample Preparation:
Samples will be prepared at a suitable concentration for identification and in any appropriate solvent, including and in any appropriate solvent, including but not limited to methanol, water, and phosphate buffer, or any combination of these. Internal standards may be used. Perform filtration and centrifugation, as necessary.

Method Parameters:
Instrument: Agilent 1200 series Liquid Chromatograph, or equivalent
Column: XDB C18 150 mm x 4.6 mm x 5 µm, or equivalent
Column Temperature: 50°C
Buffer/Mobile Phase: 85Mm Sodium Phosphate buffer w/ Hexylamine and sodium azide (pH ~2.5)
Minimum Injection Volume: 5 µL
Gradient Set Points: Isocratic 80% buffer: 20% acetonitrile
Flow Rate: 1.0 mL/min
Minimum Run Time: 7.5 min
Detection Wavelength: 210 nm

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 minutes of the average of 30 injections, and the individual relative retention times are with 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during 6 weeks are within 0.1 minutes of the values measured on week 1, and the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

Amines_XDB - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (analysis of amines)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1200 Infinity Liquid Chromatograph, or equivalent
Column: Eclipse XDB C18 4.6 x 50 mm, 1. 8µm, or equivalent
Column Temperature: 40°C
Injection Volume: 0.1µL
Buffer/Mobile Phase: 2.5224g ammonium formate (≥99.995% trace metals basis) in 4L Millipore H20, pH to 3.7 using formic acid (Optima LC/MS grade)
Minimum Injection Volume: 0.1µL
Gradient: Isocratic **88% buffer:12% acetonitrile (0.1% formic acid)**
Flow Rate: 0.6 mL/min
Minimum Run Time: 4.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Selected compounds are visually separated. All peaks are a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 or 0.3 (for LCMS) minutes of the average of all injections, and/or the individual relative retention times are with 1% of the average of all injections.

Reproducibility: Individual retention times measured during 6 weeks are within 0.1 or 0.3 (for LCMS) minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

Benzo_XDB - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited Purpose (Benzodiazepines)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1200 Infinity Liquid Chromatograph, or equivalent
Column: Eclipse XDB C18 4.6 x 50 mm, 1.8 µm, or equivalent
Column Temperature: 40°C
Buffer/Mobile Phase: (A) 10mM ammonium formate buffer; (B) acetonitrile (0.1% formic acid)
Minimum Injection Volume: 0.1 µL
Gradient Set Points: Isocratic (55% A: 45% B)
Flow rate: 0.9 mL/min
Minimum Run Time: 5.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

Cyclopropyl_Crotonyl_Fentanyl - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (cyclopropyl fentanyl, crotonyl fentanyl, 2-furanyl fentanyl, 3-furanyl fentanyl)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, acetonitrile, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Agilent Poroshell 120 EC-C18 2.1 mm x 100 mm x 2.7 µm, or equivalent
Column Temperature: 55°C
Buffer/Mobile Phase: (A): water with 0.1% formic acid and 0.1% ammonium formate; (B): 95:5 acetonitrile:water with 0.1% formic acid
Minimum Injection Volume: 1.0 µL
Gradient Set Points: 77:23 A:B for 8.0 min, 5:95 A:B for 1.0 min, 1.0 min post-time
Flow Rate: 0.5 mL/min
Minimum Run Time: 10.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. The earliest eluting compound was within the minimum acceptable retention time of the method and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during six weeks are within 0.3 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

GEN1 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
General purpose

Sample Preparation:
Stock sample solutions should be prepared using methanol or deionized water at a target analyte concentration of 0.5 mg/mL. For analysis, the stock solution should be diluted 1:10 using the LCMS injection solvent (0.1% formic acid in deionized water), then filtered to 0.2 µm.

Method Parameters:
Instrument: ThermoScientific Ultimate 3000 Ultra-High Performance Liquid Chromatograph, or equivalent
Column: Hypersil GOLD C18 50 x 2.1 mm x 1.9 µm, or equivalent
Column Temperature: 20°C
Buffer/Mobile Phase: (A): deionized water with 0.1% formic acid; (B): acetonitrile with 0.1% formic acid
Minimum Injection Volume: 1 µL
Gradient Set Points: 0-1.0 min: 95:5 A/B; 1.0-4.0 min: 95:5 to 5:95 A/B (Re-equilibration: 4.0-4.5 min: 5:95 to 95:5 A/B; 4.5-6.0 min: 95:5 A/B)
Flow Rate: 0.400 mL/min
Minimum Run Time: 4.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections.

Reproducibility: Individual retention times measured during weeks 2-6 are within 0.3 minutes of the values measured on week 1.

LCFENT1 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (heroin and fentanyl related compounds)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Synergi Hydro-RP 80Å 3.0 × 150 mm 4 µm, or equivalent
Column Temperature: Not controlled
Buffer: 0.1% Formic acid in water
Mobile Phase: 70% Buffer:30% ACN
Minimum Injection Volume: 1 µL
Flow Rate: 0.4 mL/min
Minimum Run Time: 13 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N = 3 is observed. Earliest eluting compound met minimum retention factor criteria.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections.

Reproducibility: Individual retention times measured during 2 weeks are within 0.3 minutes of the values measured on week 1.

LCLSD2 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (LSD)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Synergi Hydro-RP 80Å 3.0 × 150 mm 4 µm, or equivalent
Column Temperature: Not controlled
Buffer: 0.1% Formic acid in water
Mobile Phase: 80% Buffer:20% ACN
Minimum Injection Volume: 1 µL
Flow Rate: 0.4 mL/min
Minimum Run Time: 18 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N = 3 is observed. Earliest eluting compound met minimum retention factor criteria.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections and the individual relative retention times are with 1% of the average of 30 injections. However, multiple repeatability studies revealed that a positive control should be run within 15 sequential injections of the unknown sample.

Reproducibility: Individual retention times measured during 2 weeks were not within 0.3 minutes of the values measured on week 1. Based on the repeatability testing, positive controls shall be analyzed within a maximum of 15 sequential injections of the unknown sample, within the same sequence on the same day.

LCMUSHROOM2 - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (psilocybe mushrooms)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Synergi Hydro-RP 80Å 3.0 × 150 mm 4 µm, or equivalent
Column Temperature: Not controlled
Buffer: 0.1% Formic acid in water
Mobile Phase: 85% Buffer:15% ACN
Minimum Injection Volume: 1 µL
Flow Rate: 0.4 mL/min
Minimum Run Time: 5 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N = 3 is observed. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual absolute retention times measured are within 0.3 minutes of the average of 30 injections.

Reproducibility: Individual retention times measured during 4 weeks were within 0.3 minutes of the values measured on week 1. Based on this testing, positive controls shall be analyzed within 21 days of the unknown sample.

LC-SCRN - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
General Purpose

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: ThermoFisher Scientific Vanquick Ultra-High Performance Liquid Chromatograph, or equivalent
Column: Accucore aQ 100 mm x 2.1 mm, 2.6 µm, or equivalent
Column Temperature: 30°C
Buffer/Mobile Phase: (A) Millipore water (0.1% formic acid); (B) acetonitrile (0.1% formic acid)
Minimum Injection Volume: 0.2 µL
Gradient Set Points: 98% A: 2% B for 5.0 min, then 10% A: 90% B for 1.0 min
Flow rate: 0.5 mL/min
Minimum Run Time: 6.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

LSD - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited Purpose (LSD and related compounds)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: ThermoFisher Scientific Vanquick Ulta-High Performance Liquid Chromatograph, or equivalent
Column: Accucore aQ 100 mm x 2.1 mm, 2.6 µm, or equivalent
Column Temperature: 30°C
Buffer/Mobile Phase: (A) Millipore water (0.1% formic acid); (B) acetonitrile (0.1% formic acid)
Minimum Injection Volume: 0.2 µL
Gradient Set Points: 85% A: 15% B for 2.0 min, then 70% A: 30% B for 1.5 min, then 10% A: 90% B
Flow rate: 0.5 mL/min
Minimum Run Time: 5.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

LSD_XDB - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (LSD, iso-LSD, and LAMPA)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1200 Infinity Liquid Chromatograph, or equivalent
Column: Eclipse XDB C18 4.6 x 50mm, 1.8 µm, or equivalent
Column Temperature: 30°C
Injection Volume: 0.2 µL
Buffer/Mobile Phase: 2.5224 g ammonium formate (≥99.995% trace metals basis) in 4L Millipore H20, pH to 3.7 using formic acid (Optima LC/MS grade)
Minimum Injection Volume: 0.1 µL
Gradient: Isocratic **75% buffer:25% acetonitrile (0.1% formic acid)**
Flow Rate: 0.5 mL/min
Minimum Run Time: 5.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Selected compounds are visually separated. All peaks are a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.1 or 0.3 (for LCMS) minutes of the average of all injections, and/or the individual relative retention times are with 1% of the average of all injections.

Reproducibility: Individual retention times measured during 6 weeks are within 0.1 or 0.3 (for LCMS) minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

LTQXL-SCRN - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
General Purpose

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, acetonitrile, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: ThermoFisher Scientific Vanquick Ultra-High Performance Liquid Chromatograph, or equivalent
Column: Hypersil GOLD C18 50 mm x 2.1 mm, 1.9 µm, or equivalent
Column Temperature: 30 °C
Buffer/Mobile Phase: (A) Millipore water (0.1% formic acid); (B) acetonitrile (0.1% formic acid); (C) Millipore water; (D) water (0.1% formic acid)
Minimum Injection Volume: 0.5 µL
Gradient Set Points: 98% A, 1% B, 1% D for 7.0 min then 5% A, 70% B, 25% D for 1.4 min
Flow rate: 0.5 mL/min
Minimum Run Time: 8.4 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1.

Oxy_XDB - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited Purpose (oxycodone, hydrocodone, hydromorphone and related substances)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1200 Infinity Series Liquid Chromatograph, or equivalent
Column: Eclipse XDB C18 4.6 x 50 mm, 1.8 µm, or equivalent
Column Temperature: 40°C
Buffer/Mobile Phase: (A) 10mM ammonium formate buffer in Millipore water (adjusted to pH 3.7 using formic acid); (B) acetonitrile (0.1% formic acid)
Minimum Injection Volume: 0.1 µL
Gradient Set Points: Isocratic (85% A: 15% B)
Flow rate: 0.5 mL/min
Minimum Run Time: 3.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed, including a low level (0.5%) marker compound. Earliest eluting compound fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.
Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

Psilocybin_XDB - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited Purpose (Psilocybin and related compounds)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, 0.01 N hydrochloric acid, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1200 Infinity Series Liquid Chromatograph, or equivalent
Column: Eclipse XDB C18 4.6 x 50 mm, 1.8 µm, or equivalent
Column Temperature: 30°C
Buffer/Mobile Phase: (A) 10mM ammonium formate buffer in Millipore water (adjusted to pH 3.7 using formic acid); (B) methanol (0.1% formic acid)
Minimum Injection Volume: 0.1 µL
Gradient Set Points: Isocratic (95% A: 5% B)
Flow rate: 0.9 mL/min
Minimum Run Time: 6.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured for representative compounds are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured for representative compounds during 6 weeks are within 0.3 minutes of the values measured on week 1, and/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

SCREEN_FENTANYL - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
Limited purpose (fentanyl and related compounds)

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, acetonitrile, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Agilent Poroshell 120 EC-C18 2.1 mm x 100 mm x 2.7 µm, or equivalent
Column Temperature: 55°C
Buffer/Mobile Phase: (A): water with 0.1% formic acid and 0.1% ammonium formate; (B): 95:5 acetonitrile/water with 0.1% formic acid
Minimum Injection Volume: 0.50 µL
Gradient Set Points: 75:25 A:B for 3.2 min, 30:70 A:B for 0.01 min, 5:95 A:B for 0.79 min, 1.0 min post-time
Flow Rate: 0.5 mL/min
Minimum Run Time: 5.0 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. The earliest eluting compound was within the minimum acceptable retention time of the method and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during six weeks are within 0.3 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

SCREEN - Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope:
General purpose

Sample Preparation:
Samples must be prepared in an appropriate LC/MS grade solvent such as, but not limited to, methanol, acetonitrile, or Millipore filtered water. All samples should be filtered using 0.2 µm syringe filters or similar.

Method Parameters:
Instrument: Agilent 1290 Liquid Chromatograph, or equivalent
Column: Agilent Poroshell 120 EC-C18 2.1 mm x 100 mm x 2.7 µm, or equivalent
Column Temperature: 55°C
Buffer/Mobile Phase: (A): water with 0.1% formic acid and 0.1% ammonium formate; (B): 95:5 acetonitrile/water with 0.1% formic acid
Minimum Injection Volume: 0.50 µL
Gradient Set Points: 95:5 A:B for 1.0 min, 90:10 A:B for 5.0 min, 50:50 A:B for 2.0 min, 5:95 A:B 1.5 min
Flow Rate: 0.5 mL/min
Minimum Run Time: 9.5 min
Detector: See ESI Mass Spectrometry Method Parameters

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. The earliest eluting compound was within the minimum acceptable retention time of the method and/or fulfilled the repeatability and reproducibility acceptance criteria.

Repeatability: Individual retention times measured are within 0.3 minutes of the average of 30 injections, and/or the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility: Individual retention times measured during six weeks are within 0.3 minutes of the values measured on week one, and/or the individual relative retention times during six weeks are within 1% of the values measured on week one.

Amines_MS - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
Limited Purpose (Amines)

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6130 Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50-400 **m/z**
MS Scan Rate: 0.77 sec/cycle (peak width 0.080 min)
Collision Gas: Nitrogen
Collision Energy: 4 kV
Tune File: atunes.tun
Activation Type: Source-induced dissociation (SID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

Benzos_MS - Identification of Controlled and Non-controlled Substances Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
Limited purpose (benzodiazepines)

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6100 Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50 - 400 m/z
MS Scan Rate: 0.77 sec/cycle (peak width 0.080 min)
Collision Gas: Nitrogen
Collision Energy: 4 kV
Tune File: atunes.tun
Activation Type: Source-induced dissociation (SID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

LSD_MS - Identification of Controlled and Non-controlled Substances by Electrospray Ionization Mass Spectrometry (ESI-MS)

Scope:
Limited purpose (LSD)

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6100 Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50 - 400 m/z
MS Scan Rate: 0.77 sec/cycle (peak width 0.080 min)
Collision Gas: Nitrogen
Collision Energy: 3 kV
Tune File: atunes.tun
Activation Type: Source-induced dissociation (SID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass (or within 5 ppm for high-resolution system) as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

MSMS - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General Purpose

Sample Preparation:
LC or DART effluent

Method Parameters:
Instrument: ThermoFisher Scientific LTQ or LXQ
Mass Analyzer: Linear Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50 – 700 m/z
MSMS Range: Precursor-ion dependent
MS Scan Rate: Normal (16,666 Da/ second)
MSMS Scan Rate: Normal (16,666 Da/ second)
Collision Gas: Nitrogen
Collision Energy: 45 V
Tune File: Monthly.LTQTune
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

MSMS1 - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6545 Q-TOF Mass Spectrometer with dual AJS ESI source, or equivalent
Mass Analyzer: Quadrupole Time-Of-Flight
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
MS Scan Range: 50 – 1000 m/z
MSMS Range: 50 – 500 m/z
MS Scan Rate: 1.5 spectra/s
MSMS Scan Rate: 1 spectra/s
Collision Gas: Nitrogen
Collision Energy: 10 – 50 eV
Tune File: TOFMassCalibration-1700mzRange
Reference Masses: 121.050873, 922.009798
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample MS spectrum were all within 5 ppm of those in the reference spectrum. The measured m/z values for prominent ions in the sample MSMS spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

MSMSNEG - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
Direct analysis or LC effluent

Method Parameters:
Instrument: Agilent 6545 Q-TOF Mass Spectrometer with dual AJS ESI source, or equivalent
Mass Analyzer: Quadrupole Time-Of-Flight
Ionization Mode: Negative mode electrospray ionization
Drying Gas: Nitrogen
MS Scan Range: 50 – 1000 m/z
MSMS Range: 50 – 500 m/z
MS Scan Rate: 1.5 spectra/s
MSMS Scan Rate: 1 spectra/s
Collision Gas: Nitrogen
Collision Energy: 0 eV (MS); 10 – 50 eV (MSMS)
Tune File: TOFMassCalibration-1700mzRange
Reference Masses: 112.985587 (enabled)
Activation Type: Collision-Induced Dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample MS spectrum were all within 5 ppm of those in the reference spectrum. The measured m/z values for prominent ions in the sample MSMS spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

NESI-MS2 - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
Direct analysis or LC effluent

Method Parameters:
Instrument: ThermoScientific LCQ Fleet mass spectrometer, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Negative mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 275°C
MS Scan Range: 50-500 m/z
MSMS Range: 50-500 m/z (variable based on precursor ion)
MS Scan Rate: 60 µs/u (normal scan)
MSMS Scan Rate: 60 µs/u (normal scan)
Collision Gas: Helium
Collision Energy: 30 eV
Tune File: LCMS-NEG.LTQTune
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

Oxy_MS - Identification of Controlled and Non-controlled Substances by Electrospray Ionization Mass Spectrometry (ESI-MS)

Scope:
Limited Purpose (oxycodone, hydrocodone, and hydromorphone)

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6100 Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50 - 400 **m/z**
MS Scan Rate: 2500 u/s (standard mode)
Collision Gas: Nitrogen
Collision Energy: 4 kV
Tune File: atunes.tun
Activation Type: Source-induced dissociation (SID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

PESI-MS2 - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
Direct analysis or LC effluent

Method Parameters:
Instrument: ThermoScientific LCQ Fleet mass spectrometer, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 275°C
MS Scan Range: 50-500 m/z
MSMS Range: 50-500 m/z (variable based on precursor ion)
MS Scan Rate: 60 µs/u (normal scan)
MSMS Scan Rate: 60 µs/u (normal scan)
Collision Gas: Helium
Collision Energy: 30 eV
Tune File: LCMS.LTQTune
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

Psilocybin_MS - Identification of Controlled and Non-controlled Substances by Electrospray Ionization Mass Spectrometry (ESI-MS)

Scope:
Limited Purpose (Psilocybin and related compounds)

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6100 Mass Selective Detector, or equivalent
Mass Analyzer: Quadrupole Ion Trap
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
Capillary Temperature: 350°C
MS Scan Range: 50 - 400 m/z
MS Scan Rate: 2500 u/s (standard mode)
Collision Gas: Nitrogen
Collision Energy: 4 kV
Tune File: atunes.tun
Activation Type: Source-induced dissociation (SID)

Limitation:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

QMS1 - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6545 Q-TOF Mass Spectrometer with dual AJS ESI source, or equivalent
Mass Analyzer: Quadrupole Time-Of-Flight
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
MS Scan Range: 50-1050 m/z
MS Scan Rate: 3.00 spectra/s
Collision Gas: Nitrogen
Tune File: TOFMassCalibration-1700mzRange
Reference Masses: 121.050873, 922.009798
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample MS spectrum were all within 5 ppm of those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

QMSMS1 - Identification of Controlled and Non-controlled Substances by Electrospray Ionization-Mass Spectrometry (ESI-MS)

Scope:
General purpose

Sample Preparation:
LC effluent

Method Parameters:
Instrument: Agilent 6545 Q-TOF Mass Spectrometer with dual AJS ESI source, or equivalent
Mass Analyzer: Quadrupole Time-Of-Flight
Ionization Mode: Positive mode electrospray ionization
Drying Gas: Nitrogen
MS Scan Range: 50 - 1050 m/z
MSMS Range: 40 – 700 m/z
MS Scan Rate: 3.00 spectra/s
MSMS Scan Rate: 3.00 spectra/s
Collision Gas: Nitrogen
Collison Energy: 10 – 40 eV
Tune File: TOFMassCalibration-1700mzRange
Reference Masses: 121.050873, 922.009798
Activation Type: Collision-induced dissociation (CID)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample MS spectrum were all within 5 ppm of those in the reference spectrum. The measured m/z values for prominent ions in the sample MSMS spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

DART-MS(QTOF)-(QS/M)-Identification of Controlled and Non-controlled Substances by Direct Analysis in Real Time (DART) Mass Spectrometry

Scope:
General purpose

Sample Preparation:

For DART-MS(QTOF)-QS (use of QuickStrip sample cards), testing solutions can be prepared using organic volatile solvents (e.g., MeOH, $CHCl_3$, acetonitrile, acetone, hexane, etc.), or deionized water. Optimum target analyte concentrations for testing are dependent on the analyte, its ionization efficiency, the presence of other (suppressing or enhancing) compounds, the mass analyzer's sensitivity, etc. During this validation, analyte concentrations of 100-200 µg/mL were used. Solutions are deposited (1-5 µL depending on concentration) on the designated card spots and allowed to evaporate to dryness at room temperature. For DART-MS(QTOF)-M (manual sampling), items such as tablets, blotter paper, etc. can be analyzed directly and require no sample preparation. Solutions can be prepared as described above.

Method Parameters:

Instrument: IonSense DART JumpShot ionization source, or equivalent
Mass Analyzer: Agilent Q-TOF (quadrupole time-of-flight) mass spectrometer, or equivalent
DART Ceramic Cap: Small orifice (QS) or Large orifice (M)
Ionization Gas: Helium
Standby Gas: Nitrogen
Contact Closure: Yes (QS) or No (M)
DART Parameters:
- **DART Method:** DART-MSMS(QTOF)-QS-CC (QS) or None/Free Run (M)
- **Sampling Technique:** Pulsing (QS) or Continuous (M)
- **Pulse Duration:** 5 s (QS) or N/A (M)
- **Temperature Profile:** Fixed (QS) or N/A (M)
- **Run Temperature:** 250°C
- **Contact Closure:** On (22 s delay) (QS) or Off (M)
- **MS Ready Signal:** Off (QS) or N/A (M)
- **Ion Mode:** Positive
- **Heater Wait Time:** 30 s (QS) or N/A (M)
- **Post-Run State:** Standby (QS) or N/A (M)
- **Post-Run Heater Temperature:** 250°C
- **Starting Sample:** Variable (QS) or Manual (M)
- **Sample Count:** Variable, up to 12 (QS) or Manual (M)

MS Parameters:
- **MS Tune:** TOF Transmission Tune (1700 m/z range)
- **Scan Range:** m/z 40-600
- **Ionization Mode:** Positive
- **Data:** Centroid
- **MS Scan Rate:** 5 spectra/s
- **MS/MS Scan Rate:** 5 spectra/s
- **Fragmentor Voltage:** 100 V
- **Collision Gas:** Nitrogen
- **Collision Energy:** Variable
- **Total Analysis Time:** 0.3 min (QS) or Variable (M)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:

Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MSMS fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

DART-MS(QQQ)-(QS/M)-Identification of Controlled and Non-controlled Substances by Direct Analysis in Real Time (DART) Mass Spectrometry

Scope:
General purpose

Sample Preparation:

For DART-MS(QQQ)-QS (use of QuickStrip sample cards), testing solutions can be prepared using organic volatile solvents (e.g., MeOH, CHCl3, acetonitrile, acetone, hexane, etc.), or deionized water. Optimum target analyte concentrations for testing are dependent on the analyte, its ionization efficiency, the presence of other (suppressing or enhancing) compounds, the mass analyzer's sensitivity, etc. During this validation, analyte concentrations of 100-200 µg/mL were used. Solutions are deposited (1-5 µL depending on concentration) on the designated card spots and allowed to evaporate to dryness at room temperature. For DART-MS(QQQ)-M (manual sampling), items such as tablets, blotter paper, etc. can be analyzed directly and require no sample preparation. Solutions can be prepared as described above.

Method Parameters:

Instrument: IonSense DART JumpShot ionization source, or equivalent
Mass Analyzer: Waters Xevo TQ-S (triple quadrupole), or equivalent
DART Ceramic Cap: Small orifice (QS) or Large orifice (M)
Ionization Gas: Helium
Standby Gas: Nitrogen
Contact Closure: Yes (QS) or No (M)
DART Parameters:
- **DART Method:** QuickStrip-Pulsed-CCon-Pos (QS) or None/Free Run (M)
- **Sampling Technique:** Pulsing (QS) or Continuous (M)
- **Pulse Duration:** 1 s (QS) or N/A (M)
- **Temperature Profile:** Fixed (QS) or N/A (M)
- **Run Temperature:** 250°C
- **Contact Closure:** On (12 s delay) (QS) or Off (M)
- **MS Ready Signal:** Off (QS) or N/A (M)
- **Ion Mode:** Positive
- **Heater Wait Time:** 30 s (QS) or N/A (M)
- **Post-Run State:** Standby (QS) or N/A (M)
- **Post-Run Heater Temperature:** 250°C
- **Starting Sample:** 1 (QS) or Manual (M)
- **Sample Count:** 12 (QS) or Manual (M)

MS Parameters:
- **MS Tune:** DART_TUNE_POS
- **Scan Range:** m/z 50-500
- **Ionization Mode:** Positive
- **Data:** Centroid
- **Scan Time:** 0.2 s
- **Cone Voltage:** 0 V
- **APCI Probe**: N/A
- **Collision Gas:** Argon
- **Collision Energy:** Variable
- **Total Analysis Time:** 6 s (QS) or Variable (M)

Limitations:
See individual instrument validation reports.

Acceptance Criteria:

Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MSMS fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

DART-MS2 - Identification of Controlled and Non-controlled Substances by Direct Analysis in Real Time (DART) Mass Spectrometry

Scope:
General purpose

Sample Preparation:
Samples can be prepared using organic volatile solvents (methanol, acetonitrile, acetone, hexane, etc.), deionized water, or the LCMS injection solvent. Powder samples can be directly analyzed (manually) and require no sample preparation (use DIP-it tips or disposable glass capillaries or needles). Items such as tablets, blotter paper, etc. can also be analyzed without any sample preparation.

Method Parameters:
Instrument: IonSense DART ionization source and ThermoScientific LCQ Fleet mass spectrometer, or equivalent
Mass Analyzer: Quadrupole ion trap (QIT)
Ionization Mode: DART
Drying Gas: N/A
Ionization gas: Helium
Capillary Temperature: 200°C
MS Scan Range: 50-500 m/z
MSMS Range: 50-500 m/z (variable based on precursor ion)
MS Scan Rate: 60 µs/u (normal scan)
MSMS Scan Rate: 60 µs/u (normal scan)
Collision Gas: Helium
Collision Energy: 30 V
Tune File: DART Tune.LTQTune
Activation Type: Collision-induced dissociation (CID)

Limitations:
Analysis of mixtures may result in the production of multiple protonated molecular ions during DART ionization. Also, some fragmentation of molecular ions may be observed due to the high temperature of the source.

Under DART-MS2 conditions, analysis of dipyrone does not produce an intact protonated molecular ion. Instead, the degradation product 4-methylaminoantipyrine is observed.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS fragmentation spectra correspond to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

DART-SCRN-OrbiTrapMS – Identification of Controlled and Non-controlled Substances by Direct in Real Time (DART) Mass Spectrometry

Scope:
General purpose

Sample Preparation:
Samples can be analyzed directly as powders or as solutions in a suitable solvent.

Method Parameters:
Instrument: ThermoFisher Scientific Exactive Plus HRAM DART-MS, or equivalent
Mass Analyzer: Orbitrap
Ionization Mode: DART
Drying Gas: Nitrogen
Ionization gas: Nitrogen
Capillary Temperature: 250°C
MS Scan Range: 50-750 m/z
MS Scan Rate: 7.2 scans/s
Collision Gas: Nitrogen
Collision Energy: 1V, 30V, 60V
Tune File: Exactive Plus calibration
Reference Masses (high resolution only): 195.087, 524.264, 1221.990, 1421.977, 1621.965
Activation Type: Source-induced dissociation (SID)

Limitations summary:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall MS and MSMS (if used) fragmentation spectra corresponds to those of the reference spectrum. The measured m/z values for prominent ions in the sample spectrum were all of the same nominal mass (or within 5 ppm for high-resolution system) as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

IRD or IRD1 – Identification of Controlled and Non-controlled Substances by Vapor Phase Infrared Spectroscopy

Scope:
General purpose

Sample Preparation:
GC effluent

Method Parameters:

Instrument: Analytical Solution and Providers, Model IRD3, or equivalent
Scan Range: 550-4000 cm^{-1}
Transfer Line Temperature: 280°C or 295°C
Resolution: 4, 8, or 16 cm^{-1}
Light Pipe Temperature: 280°C or 295°C

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum and the prominent, well-defined, measured signals in the sample spectra were all within 4 cm^{-1} of those in the reference spectrum. No prominent extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

IR01/IR01X – Identification of Controlled and Non-controlled Substances by Solid Phase Infrared Spectroscopy

Scope:

General Purpose

Sample Preparation:

Powder and liquid samples are analyzed by direct analysis. If needed to separate mixtures, samples may be extracted using solid-or liquid based procedures, or by sublimation.

Method Parameters:

Instrument: Thermo Scientific Nicolet iS10 Infrared Spectrometer, or equivalent
Number of Background Scans: 8 scans (IR01); 32 scans (IR01X)
Minimum Number of Sample Scans: 8 scans (IR01); 32 scans (IR01X)
Scan Range: 650-4000 cm^{-1} (IR01); 400-4000 cm^{-1} (IR01X)
Sample Gain: Autogain
Resolution: 4.000 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 150 (Open)
Accessory: Smart Golden Gate ATR

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: Analyte spectra are compared to a verified reference database, commercial library, published spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum and the prominent, well-defined, measured signals in the sample spectra were all within 4 cm^{-1} of those in the reference spectrum. No prominent extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods are established via evaluation of system-wide historical spectral data.

SFL1_32Scans - Identification of Controlled and Non-controlled Substances by Solid Phase Infrared Spectroscopy

Scope:
General Purpose

Sample Preparation:
Powder and liquid samples are analyzed by direct analysis. If needed to separate mixtures, samples may be extracted using solid-or liquid based procedures, or by sublimation.

Method Parameters:
Instrument: Thermo Scientific Nicolet iS10 Infrared Spectrometer, or equivalent
Number of Background Scans: 32
Minimum Number of Sample Scans: 32
Scan Range: 400-4000 cm^{-1}
Sample Gain: 8.0
Resolution: 4 cm^{-1}
Optical Velocity: 0.4747
Aperture: 150.00
Accessory: Smart Golden Gate ATR

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum and the prominent, well-defined, measured signals in the sample spectrum were all within 4 cm^{-1} of those in the reference spectrum. No prominent extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

IRMicroARO1 - Identification of Controlled and Non-controlled Substances by Solid Phase Infrared Spectroscopy

Scope:
General Purpose

Sample Preparation:
Sample directly analyzed.

Method Parameters:
Instrument: Smiths Detection IlluminatIR FTIR with Olympus microscope attachment
Number of Background Scans: 8
Minimum Number of Sample Scans: 8
Scan Range: 650 – 4000 cm^{-1}
Resolution: 4 cm^{-1}
Detector: MCT
Accessory: ARO IR Microscope 15x objective

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum and the prominent, well-defined, measured signals in the sample spectrum between 2000 – 650 cm^{-1} were all within 8 cm^{-1} of those in the reference spectrum. No prominent extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

IRMicroATR1 - Identification of Controlled and Non-controlled Substances by Solid Phase Infrared Spectroscopy

Scope:
General Purpose

Sample Preparation:
Sample directly analyzed.

Method Parameters:
Instrument: Smiths Detection IlluminatIR FTIR with Olympus microscope attachment
Number of Background Scans: 16
Minimum Number of Sample Scans: 16
Scan Range: 650 – 4000 cm^{-1}
Resolution: 4 cm^{-1}
Detector: MCT
Accessory: ATR IR Microscope 36x objective

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published spectra or spectra from another ISO/IEC 17025 accredited laboratory. The overall sample spectral pattern corresponds to that of the reference spectrum and the prominent, well-defined, measured signals in the sample spectrum between 2000 – 650 cm^{-1} were all within 8 cm^{-1} of those in the reference spectrum. No prominent extraneous signals are observed in the sample spectrum.

Repeatability and Reproducibility: The repeatability and reproducibility of confirmatory methods is established via evaluation of system-wide historical spectral data.

CEPHENISOMER1 - Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope:

Limited purpose (select controlled and non-controlled substances and optical isomers)

Sample Preparation:

Samples in aqueous solution. Insoluble material filtered or removed from sample. Nicotinamide and/or noscapine may be used as optional internal standards (IS). Nicotinamide was used as the fixed compound for validation purposes.

Method Parameters:

Instrument: Agilent 7100 Capillary Electrophoresis system, or equivalent
Capillary Type and Dimensions: 48.5 cm × 50 mm ID (40 cm effective length)
Capillary Temperature: 15°C
Injection Parameters: 150 mbar*s (50 mbar for 3s or equivalent). Optional buffer, water, or IS injections permissible
Buffer: Microsolv custom buffer (PN 05375-M2-X) or equivalent (2-Hydroxypropyl-β-cyclodextrin in Microsolv Celixer pH Accelerator B (PN 06125-CE))
Voltage: 30 kV after 0.2 minutes
Detection Wavelength: 195 nm
Spectral Acquisition Range: 190-400 nm
Spectrum Step: 2 nm
Minimum Run Time: 6 min

Limitations:

See individual instrument validation reports.

Additional reproducibility testing conducted using differing batches of run buffer resulted in migration times outside of the acceptance range. As such, all samples and positive controls must be analyzed using the same batch of run buffer.

Acceptance Criteria:

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed.

Repeatability: Individual migration times measured are within 0.3 minutes of the average of all injections, and the individual relative retention times are with 1% of the average of all injections.

Reproducibility: Individual absolute and migration times measured during 6 weeks. Absolute migration times are within 0.3 minutes of the values measured on week 1, and the individual relative retention times are with 1% of the values measured on week 1.

CHIRAL 2B_FAST - Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope:
Limited purpose (optical isomers of methamphetamine, amphetamine, pseudoephedrine, and ephedrine)

Sample Preparation:
Samples in Injection Solvent solution (Add 45.00 g NaH_2PO_4 + 250 mg (± 1 mg) NaH_2PO_4 in 5 L volumetric flask. Add 4.9 L deionized water, mix to dissolve. Titrate with approximately 10 mL of 4.4M phosphoric acid to pH of 2.5 ± 0.1. Dilute to 5 L volume.) Insoluble material filtered or removed from sample.

Internal Standard Solution:
0.05 mg/mL procaine HCl in Injection Solvent Solution

Method Parameters:
Instrument: Agilent 7100 Capillary Electrophoresis system, or equivalent
Capillary Type and Dimensions: 50 cm × 50 µm ID (41.5 cm effective length)
Capillary Temperature: 9°C
Buffer: Custom Chiral2 Buffer: (7.000 g 2-hydroxylpropyl)-β-Cyclodextrin into 50 mL volumetric flask, dilute to volume with Microsolv CElixer pH Accelerator B (PN 06125-CE) to final concentration of approximately 0.07000 g/mL
Preconditioning Parameters: Flush, 0.1N sodium hydroxide for 60 s; flush, deionized water for 60 s; flush, Microsolve CElixir pH Initiator A for 60 s; flush, Custom Chiral2 Buffer for 120 s
Injection Parameters: Sample at 50mbar for 8 sec; water at 35 mbar for 1 s;
Voltage: 27.5 kV at 0.5 min
Detection Wavelength: Signal A: 195nm, 10nm bandwidth; Signal B: 205nm, 20nm bandwidth; No Signal C; Signal D: 250nm, 4nm bandwidth; Signal E: 280nm, 4nm bandwidth
Minimum Run Time: 10.5 min

Limitations:
See individual instrument validation reports.

Acceptance Criteria:
Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed.

Repeatability: Individual migration times measured are within 0.3 minutes of the average of all injections, and/or the individual relative retention times are with 1% of the average of all injections.

Reproducibility: Reproducibility testing was as not conducted for this method. Positive controls must be analyzed concurrently with samples.