SFL3

Method: DC-ISOMER1-30

Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope: Limited purpose (qualitative analysis and identification of phenethylamine isomers)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification in Microsolv Injection Solvent (3.75 mM Sodium Phosphate pH=2.6), dilute hydrochloric acid, or dilute phosphoric acid.

Method Parameters:

Instrument: Capillary Electorphoresis (Sciex P/ACE MDQ plus) equipped with an UVNis detector
Capillary Type and Dimensions: 40.2cm (30 cm LEF) X 50 µm ID bare fused silica capillary
Capillary Temperature: 20º C
Injection Parameters: Sample for 10.0 sec at .3 psi; Water for 5.0 sec at .1 psi
Buffer: DEA Custom Chiral (inlet); CELixir B pH= 2.5 (outlet)
Voltage: 0.17 minute ramp to +30kV
Detection Wavelength: 195 nm (Bandwidth=10nmm, Minimum Sampling Rate = 16HZ, Reference Wavelength=400nm, Reference Bandwidth=100nm)
Minimum Run Time: 4.0 minutes

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 within 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/or the individual relative retention times are within 1% of the values measured on week 1.

SFL3

Method: DC-PHEN-30

Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope: General purpose (qualitative analysis and identification of phenethylamine isomers)

Sample Preparation:

Samples will be prepared at a suitable concentration for identification in Microsolv Injection Solvent (3.75 mM Sodium Phosphate pH=2.6), dilute hydrochloric acid, or dilute phosphoric acid.

Method Parameters:

Instrument: Capillary Electorphoresis (Sciex P/ACE MDQ plus) equipped with an UVNis detector
Capillary Type and Dimensions: 40.2cm (30 cm LEF) X 50 µm ID bare fused silica capillary
Capillary Temperature: 15º C
Injection Parameters: Sample for 10.0 sec at .3 psi; Water for 5.0 sec at .1 psi
Buffer: CELixir B pH=2.5 (inlet); CELixir B pH= 2.5 (outlet)
Voltage: 0.17 minute ramp to +25kV
Detection Wavelength: 195 nm (Bandwidth=10nmm, Minimum Sampling Rate = 16HZ, Reference Wavelength=400nm, Reference Bandwidth=100nm)
Minimum Run Time: 3.5 minutes

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 migration times measured are within 0.3 minutes of the average of all injections, and/or the individual relative retention times are within 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/or the individual relative retention times are within 1% of the values measured on week 1.

SFL3

Method: 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 GC 7890 equipped with an FID
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 100ºC hold for 2 min, 11ºC/min to 280 ºC, hold for 3 min, 35 ºC/min to 310 ºC, hold for 4 min
Minimum Run Time: 26.221 min
Detector Temperature: 300ºC

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: COCSCRN

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 7890 equipped with an FID
Detector Temperature: 280 °C
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 250°C for 1 min, 30°C/min to 300 °C, hold for 2 min
Minimum Run Time: 4.67 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: G-SCRN-02

Instruments: DEA364408; DEA364315; DEA364316; DEA364407; DEA364311; DEA364353; DEA364381; DEA364409; DEA364410

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 7890 equipped with an FID
Column Type and Dimensions: DB-5 or HP-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- 135°C for 1 min, 20°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C, hold for 2 min
  - *DEA364408; DEA364316; DEA364407*
- 135°C for 1 min, 20°C/min to 280 °C, 30°C/min to 310 °C for 3 min
  - *DEA364381, DEA364409; DEA364410*
- 145°C for 1 min, 20°C/min to 280°C, hold for 1 min, 30°C/min to 310°C, hold for 2.3 min
  - *DEA364315*
- 125°C for 1.0 min, 20°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C for 2 min
  - *DEA364311*
- 125°C for 1.5 min, 20°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C for 1.75 min
  - *DEA364353*

Minimum Run Time:

- *DEA364408; DEA364316; DEA364407; DEA 364381; DEA364409; DEA364410***:** 12.25 min
- *DEA364315*: 12.05 min
- *DEA364311***:** 12.75 min
- *DEA364353***:** 13.00 min

Detector Temperature: 310 °C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364408; DEA364407; DEA364353; DEA364381; DEA364409; DEA364410***:** 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.
- *DEA364315; DEA364316; DEA364311:* Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections.

Reproducibility:

- *DEA364408***:** 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 4 weeks are within 1% of the values measured on week 1.
- *DEA364315; DEA364316; DEA364353***:** Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364407; DEA364409:* 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.
- *DEA364311:* Individual retention times measured for representative compounds during 3 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364381*: 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 2 weeks are within 1% of the values measured on week 1.
- *DEA364410*: 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 1 week are within 1% of the values measured on week 1.

SFL3

Method: G-SCRN-02_30M

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 7890 equipped with an FID
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270ºC
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 140ºC, 25ºC/min to 290 ºC, hold for 5 min, 35 ºC/min to 310 ºC, hold for 1.5 min
Minimum Run Time: 13.071 min
Detector Temperature: 300ºC

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: ISOMER

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples will be based extracted into a suitable 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, and derivatized with MTPA.

Method Parameters:

Instrument: Agilent GC 7890 equipped with an FID
Detector Temperature: 310 °C
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 200 °C for 7 min, 30°C/min to 270°C
Minimum Run Time: 9.333 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 1 week are within 1% of the values measured on week 1.

SFL3

Method: LONGSCREEN

Instruments: DEA364381; DEA364409; DEA364410

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 7890 equipped with an FID
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 260°C for 5 min, 15°C/min to 310 °C, hold for 15 min
Minimum Run Time: 23.333 min
Detector Temperature: 280 °C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: LONGSCRN

Instruments: DEA364315; DEA364316; DEA364407

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364315*: 150°C for 1 min, 15°C/min to 280°C, hold for 1 min, 35°C/min to 310°C, hold for 6 min
- *DEA364316:* 150ºC for 1 min, 15ºC/min to 280 ºC, hold for 1 min, 35 ºC/min to 310 ºC, hold for 5.5 min
- *DEA364407:* 150ºC for 1 min, 15ºC/min to 280 ºC, hold for 1 min, 35 ºC/min to 310 ºC, hold for 7 min

Minimum Run Time:

- *DEA364315*: 17.524 min
- *DEA364316*: 17.024 min
- *DEA364407*: 18.524 min

Detector Temperature: 310°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364315:* 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 4 weeks are within 1% of the values measured on week 1.
- *DEA364316:* Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364407:* 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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: LONGSCRN_30M

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 7890 equipped with an FID
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270ºC
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 150ºC hold for 1 min, 15ºC/min to 280 ºC, hold for 1 min, 35 ºC/min to 310 ºC, hold for 8 min
Minimum Run Time: 19.524 min
Detector Temperature: 300ºC

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: LTM-FASTSCRN

Instruments: DEA364347; DEA364348; DEA364349; DEA364350; DEA364352

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 7890 equipped with an FID
Detector Temperature: 310 °C
Column Type and Dimensions:
Pre-Column: DB-5 1.0 m × 0.18mm I.D. × 0.18 µm film thickness;
LTM Column: DB-CSI LL-LTM 15m × 0.25mm I.D. × 0.25µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 ℃
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA364347; DEA364348; DEA364352:* Hydrogen, 2.0 mL/min
- *DEA364349; DEA364350:* Hydrogen, 1.0 mL/min

Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364347; DEA364348; DEA364352:* 280 °C
- *DEA364349; DEA364350:* 270 °C

LTM Program Set Points:

- 135°C for 1 min, 100°C/min to 280 °C, hold for 1 min, 250°C/min to 310 °C, hold for 1.9 min
  - *DEA364347; DEA364348*
- 135°C for 1 min, 100°C/min to 280 °C, hold for 1 min, 250°C/min to 310 °C, hold for 2.0 min
  - *DEA364349*
- 135°C for 1 min, 100°C/min to 280 °C, hold for 1 min, 250°C/min to 310 °C, hold for 1.7 min
  - *DEA364350*
- 135°C, 1.0 min, 100°C/min to 280°C for 1 min, 250°C/min to 310°C, hold for 1.5 min
  - *DEA364352*

Minimum Run Time:

- *DEA364347; DEA364348:* 5.47 min

- *DEA364349:* 5.57 min
- *DEA364350:* 5.27 min
- *DEA364352:* 5.07 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364347:* Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections.
- *DEA364348; DEA364349; DEA364350; DEA364352:* 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:

- *DEA364347:* Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364348; DEA364350:* 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 2 weeks are within 1% of the values measured on week 1.
- *DEA364349:* 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 3 weeks are within 1% of the values measured on week 1.
- *DEA364352:* Individual retention times measured for representative compounds during 4 weeks are within 0.1 minutes of the values measured on week 1.

SFL3

Method: LTM-G-SCRN-02

Instruments: DEA364347; DEA364348; DEA364349; DEA364350

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 7890 equipped with an FID
Detector Temperature: 310 °C
Column Type and Dimensions:
Pre-Column: DB-5 1.0 m × 0.18mm I.D. × 0.18 µm film thickness;
LTM Column: DB-CSI LL-LTM 15m × 0.25mm I.D. × 0.25µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364347; DEA364348:* 280 °C
- *DEA364349; DEA364350:* 270 °C

LTM Program Set Points:

- 135°C for 1 min, 15°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C, hold for 2.6 min
  - *DEA364347; DEA364348*
- 135°C for 1 min, 15°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C, hold for 3 min
  - *DEA364349*
- 135°C for 1 min, 15°C/min to 280 °C, hold for 1 min, 30°C/min to 310 °C, hold for 2.3 min
  - *DEA364350*

Minimum Run Time:

- *DEA364347; DEA364348:* 15.267 min
- *DEA364349:* 15.667 min
- *DEA364350:* 14.967 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364347; DEA364348; DEA364349:* 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.
- *DEA364350:* Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections.

Reproducibility:

- *DEA364347; DEA364349; DEA364350:* Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364348:* 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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: LTM-ISOMER

Instrument: DEA364349; DEA364350; DEA364348

Separation of Controlled and Non-controlled Substances by Gas

Chromatography **Scope: Limited purpose for isomers of methamphetamine**

Sample Preparation:

Samples will be based extracted into a suitable 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, and derivatized with MTPA.

Method Parameters:

Instrument: Agilent GC 7890 equipped with an FID
Detector Temperature: 310 °C
Column Type and Dimensions:
Pre-Column: DB-5 1.0 m × 0.18mm I.D. × 0.18 µm film thickness;
LTM Column: DB-CSI LL-LTM 15m × 0.25mm I.D. × 0.25µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 280 °C
LTM Program Set Points: 200°C for 9 min
Minimum Run Time: 9 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364349*: Individual retention times measured for representative compounds during 3 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.
- *DEA364350*: Individual retention times measured for representative compounds during 4 weeks are within 0.1 minutes of the values measured on week 1, and/or the individual relative retention times during 2 weeks are within 1% of the values measured on week 1.
- *DEA364348*: 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.

SFL3

Method: LTM-LONGSCRN

Instruments: DEA364348; DEA364350; DEA364349

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 7890 equipped with an FID
Detector Temperature: 310 °C
Column Type and Dimensions:
Pre-Column: DB-5 1.0 m × 0.18mm I.D. × 0.18 µm film thickness;
LTM Column: DB-CSI LL-LTM 15m × 0.25mm I.D. × 0.25µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 280 °C
LTM Program Set Points: 135°C for 1 min, 20°C/min to 320 °C, hold for 5 min
Minimum Run Time: 15.25 min

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364348:* Individual retention times measured for representative compounds are within 0.1 minutes of the average of 30 injections.

- *DEA364349; DEA364350:* 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:

- *DEA364348:* Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364349; DEA364350:* 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 4 weeks are within 1% of the values measured on week 1.

SFL3

Method: MTPA

Instruments: DEA364315; DEA364316; DEA364407

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (l-Methamphetamine and d-Methamphetamine)

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364315:* 165°C for 1 min, 15°C/min to 270°C for 1 min
- *DEA364316; DEA364407:* 200°C for 7 min, 30°C/min to 270°C

Minimum Run Time:

- *DEA364315:* 9.0 min
- *DEA364316; DEA364407*: 9.3 min

Detector Temperature: 310°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:

- *DEA364315; DEA364316*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364407*: 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.

SFL3

Method: MTPA_30M

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (for the qualitative analysis and identification of methamphetamine optical isomers)

Sample Preparation:

Samples will be base extracted into a suitable 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, and derivatized with MTPA.

Method Parameters:

Instrument: Agilent GC 7890 equipped with an FID
Column Type and Dimensions: DB-5 30 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase
Inlet Temperature: 270ºC
Minimum Injection Volume: 1 μL
Injection Mode): Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.5 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant Flow
Oven Program Set Points: 200°C for 7 min, 30°C/min to 270°C
Minimum Run Time: 9.333 min
Detector Temperature: 310 ºC

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 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.

SFL3

Method: MTPA-ISOMER

Instruments: DEA364381; DEA364409; DEA364410; DEA364311; DEA364353

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (l-Methamphetamine and d-Methamphetamine)

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA364381; DEA364409; DEA364410; DEA364311*: Hydrogen at 1.1 mL/min
- *DEA364353*: Hydrogen at 1.0 mL/min

Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- 225°C, 15°C/min to 240°C, hold 0.5min, 15°C/min to 280°C
  - *DEA364381; DEA364409; DEA364410*
- 200°C, 15°C/min to 280°C
  - *DEA364311; DEA364353*

Minimum Run Time:

- *DEA364381; DEA364409; DEA364410*: 4.17 min
- *DEA364311; DEA364353*: 5.33 min

Detector Temperature: 300°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 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.

SFL3

Method: QUICKSCRN

Instruments: DEA364316; DEA364407

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364316:* 165ºC for 1 min, 20ºC/min to 255 ºC
- *DEA364407*: 165ºC for 1 min, 20ºC/min to 255 ºC, hold for 0.5 min

Minimum Run Time:

- *DEA364316:* 5.5 min
- *DEA364407*: 6.0 min

Detector Temperature: 310°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:

- *DEA364316*: 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 1 week are within 1% of the values measured on week 1.
- *DEA364407*: 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.

SFL3

Method: SHORTSCREEN

Instruments: DEA364311; DEA364353; DEA364381; DEA364409; DEA364410

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364311; DEA364381; DEA364409; DEA364410:* 250°C for 1 min, 30°C/min to 280°C, hold for 3 min
- *DEA364353:* 250°C for 1 min, 35°C/min to 300°C, hold for 2.5 min

Minimum Run Time:

- *DEA364311; DEA364381; DEA364409; DEA364410:* 5.0 min
- *DEA364353:* 4.9 min

Detector Temperature: 310°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:

- *DEA364311*: 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 3 weeks are within 1% of the values measured on week 1.
- *DEA364353*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *DEA364381; DEA364409; DEA364410*: 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.

SFL3

Method: SHORTSCRN

Instruments: DEA364315; DEA364316; DEA364407

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 7890 equipped with an FID detector
Column Type and Dimensions: DB-5 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen at 1.0 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364315; DEA364316:* 250°C for 1 min, 30°C/min to 300°C, hold for 2 min
- *DEA364407*: 250ºC for 1 min, 30ºC/min to 300 ºC, hold for 2.5 min

Minimum Run Time:

- *DEA364315; DEA364316:* 4.667 min
- *DEA364407*: 5.1667 min

Detector Temperature:

- *DEA364315*: 310°C
- *DEA364316; DEA364407*: 280℃

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 the individual relative retention times are within 1% of the average of 30 injections.

Reproducibility:

- *DEA364315; DEA364407*: 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.
- *DEA364316*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

SFL3

Method: ISOMERSCRN

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 ether, hexane or chloroform. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B equipped with Analytical Solutions and Providers, Model IRD3
Column Type and Dimensions: HP-5 30 m × 0.32 mm I.D. × 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280ºC
Minimum Injection Volume: 2 µL
Injection Mode: Split
Maximum Split Ratio: 5:1
Carrier Gas and Flow: Helium at 2.0 mL/min
Control Mode: Constant flow
Oven Program Set Points: 70ºC, hold for 2 min, 10ºC/min to 280ºC, hold for 4 min, 35ºC/min to 310ºC, hold for 3 min
Minimum Run Time: 30.857 min
Detector Temperature: see IRD1 method for DEA364380

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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. This method is not intended for the use of identifying substances at low concentrations. Substances at concentrations of 0.5% and lower are not within the scope of this method. 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 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.

SFL3 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
Scan Range: 4000 – 550 cm^{-1}
Transfer Line Temperature: 280ºC
Resolution: 8 cm^{-1}
Light Pipe Temperature: 280ºC

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: 30M_LONGSCRN

Instrument: DEA364308

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/5977A GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature: 270 ºC
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 30:1
Carrier Gas and Flow: Helium, 1 mL/min
Control Mode: Constant flow
Oven Program Set Points: 130°C for 1 min, 15°C/min to 265 °C, 10°C/min to 310 °C hold for 16 min
Minimum Run Time: 30.5 min
Detector: See *DEA364308* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 2 weeks are within 0.1 minutes of the values measured on week 1 and the individual relative retention times during 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: DRUG1_30M

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/5977B GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature: 280 ºC
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow: Helium, 1 mL/min for 3 min, ramp 1 mL/min per min to 1.6 mL/min
Control Mode: Ramped flow
Oven Program Set Points: 110 °C for 2 min, 30 °C/min to 250 °C, hold for 0.5 min
Minimum Run Time: 7.167 min
Detector: See *DEA364310* MSD1 Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: DRUG2; DRUG2-15m

Instruments: DEA364354; DEA364355; DEA364376; DEA364360; DEA364359

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/5977A GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. x 0.25 µm film thickness (or equivalent)
Inlet Temperature:

- *DEA364354; DEA364355; DEA364376*: 270 ºC
- *DEA364359; DEA364360*: 280°C

Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio:

- *DEA364354; DEA364355; DEA364376; DEA364360*: 60:1
- *DEA364359*: 50:1

Carrier Gas and Flow: Helium, 1 mL/min
Control Mode: Constant flow
Oven Program Set Points:

- *DEA364354; DEA364355; DEA364376*: 200°C for 1 min, ramp temperature 30°C/min to 290°C, hold for 2 min
- *DEA364359*: 180°C for 0.5 min, ramp temperature 25°C/min to 280°C, hold for 1 min
- *DEA364360*: 180 °C for 0.5 min, ramp temperature 25°C/min to 290 °C, hold for 0.55 min

Minimum Run Time:

- *DEA364354; DEA364355; DEA364376*: 6 min
- *DEA364359*: 5.5 min
- *DEA364360*: 5.45 min

Detector: See *DEA364354; DEA364355; DEA364376; DEA364359; DEA364360* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364354; DEA364355; DEA364376; DEA364359:* 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.
- *DEA364360*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

SFL3

Method: DRUG2_30M; 30M_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 7890A/5977B GC/MSD

Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)

Inlet Temperature:

- *DEA364310:* 280ºC
- *DEA364308:* 270ºC

Minimum Injection Volume: 1 μL

Injection Mode: Split

Maximum Split Ratio:

- *DEA364310:* 50:1
- *DEA364308:* 60:1

Carrier Gas and Flow:

- *DEA364310:* Helium, 1 mL/min for 1.5 min, ramp 1 mL/min per min to 1.6 mL/min
- *DEA364308:* Helium, 1 mL/min

Control Mode:

- *DEA364310:* Ramped flow
- *DEA364308:* Constant flow

Oven Program Set Points:

- *DEA364310:* 210 °C for 0.5 min, ramp temperature 25 °C/min to 290 °C, hold for 2.8 min
- *DEA364308:* 200 °C for 0.5 min, ramp temperature 30 °C/min to 290 °C, hold for 5 min

Minimum Run Time:

- *DEA364310:* 6.5 min
- *DEA364308:* 8.5 min

Detector Temperature: See *DEA364310; DEA364308* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364310:* 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 4 weeks are within 1% of the values measured on week 1.
- *DEA364308:* 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.

SFL3

Method: DRUG3; DRUG3-15M

Instruments: DEA364354; DEA364355; DEA364376; DEA364360

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 7890/5977 GC/MSD

Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)

Inlet Temperature:

- *DEA364354; DEA364355; DEA364376*: 270 ºC
- *DEA364360*: 280°C

Minimum Injection Volume: 1 μL

Injection Mode: Split

Maximum Split Ratio:

- *DEA364354; DEA364355; DEA364376*: 60:1
- *DEA364360*: 50:1

Carrier Gas and Flow:

- *DEA364354; DEA364355; DEA364376*: Helium, 1 mL/min
- *DEA364360*: Helium, 1 mL/min for 1 min, ramp 0.25 mL/min per min to 1.75 mL/min

Control Mode:

- *DEA364354; DEA364355; DEA364376*: Constant flow
- *DEA364360*: Ramped flow

Oven Program Set Points:

- 250°C for 1 min, ramp temperature 20°C/min to 310°C, hold for 2 min
  - *DEA364354; DEA364355; DEA364376*:
- 240 °C for 1 min, ramp temperature 20 °C/min to 290 °C, hold for 1.5 min
  - *DEA364360*

Minimum Run Time:

- *DEA364354; DEA364355; DEA364376*: 6 min
- *DEA364360*: 5 min

Detector: See *DEA364354; DEA364355; DEA364376; DEA364360* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: DRUG3_30M; 30M_DRUG3

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/5977B GC/MSD

Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.250 mm I.D. × 0.25 µm film thickness (or equivalent)

Inlet Temperature:

- *DEA364310:* 280 ºC
- *DEA364308:* 270 ºC

Minimum Injection Volume: 1 µL

Injection Mode: Split

Maximum Split Ratio:

- *DEA364310:* 50:1
- *DEA364308:* 60:1

Carrier Gas and Flow:

- *DEA364310:* Helium, 1 mL/min for 1.5 min, ramp 1 mL/min per min to 1.6 mL/min
- *DEA364308:* Helium, 1 mL/min

Control Mode:

- *DEA364310:* Ramped flow
- *DEA364308:* Constant flow

Oven Program Set Points:

- *DEA364310:* 275 °C for 1 min, 20 °C/min to 295 °C, hold for 5.1 min
- *DEA364308:* 250 °C for 1 min, 20 °C/min to 310 °C, hold for 4 min

Minimum Run Time:

- *DEA364310:* 7.1 min
- *DEA364308:* 8.0 min

Detector: See *DEA364310; DEA364308* MSD1 Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: FENTISO_30M; 30M_FENTISOMER

Instruments: DEA364310; DEA364308

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose for furanyl fentanyl and 3-furanyl fentanyl

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/5977B GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature: 280 ºC
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow: Helium, 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 100 °C for 1 min, 12 °C/min to 280 °C, hold for 9 min
Minimum Run Time: 25.0 min
Detector: See *DEA364310; DEA364308* MSD1 Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: GENSCRN; GENSCRN-15; GENSCRN-15m

Instruments: DEA364354; DEA364355; DEA364376; DEA364357; DEA364358; DEA364359; DEA364360;

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 7890/5977 GC/MSD

Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)

Inlet Temperature:

- *DEA364354; DEA364355; DEA364376*: 270 ºC
- *DEA364357: DEA364358; DEA364359; DEA364360*: 280 ºC

Minimum Injection Volume: 1 μL

Injection Mode: Split

Maximum Split Ratio:

- *DEA364354; DEA364355; DEA364376; DEA364357; DEA364358:* 60:1
- *DEA364359; DEA364360*: 50:1

Carrier Gas and Flow:

- *DEA364354; DEA364355; DEA364376*: Helium, 1 mL/min
- *DEA 364357; DEA364358*: Helium, 1.5 mL/min
- *DEA364359:* Helium, 1 mL/min for 1 min, 0.25mL/min/min to 1.75 mL/min
- *DEA364360:* Helium, 1 mL/min for 1 min, 0.25 mL/min/min to 1.5 mL/min

Control Mode:

- *DEA364354; DEA364355; DEA364376; DEA 364357; DEA364358:* Constant flow
- *DEA364359; DEA364360:* Ramped flow

Oven Program Set Points:

- 85°C for 1 min, 30°C/min to 310°C, hold for 2.5 min
  - *DEA364354; DEA364355; DEA364376*
- 145°C for 1 min, 20°C/min to 280°C, hold for 0.25 min, 45°C/min to 295°C, hold for 3.167 min
  - *DEA364357; DEA364358*
- 90°C for 1 min, 45°C/min to 280 °C, hold for 5.778 min
  - *DEA364359*
- 135 °C for 1 min, 20 °C/min to 290 °C, hold for 3.25 min
  - *DEA364360*

Minimum Run Time:

- *DEA364354; DEA364355; DEA364376; DEA364359*: 11 min
- *DEA364357: DEA364358*: 11.5 min
- *DEA364360*: 12.0 min

Detector: See *DEA364354; DEA364355; DEA364376; DEA364357; DEA364358; DEA364359; DEA364360* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364354*: 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 4 weeks are within 1% of the values measured on week 1.
- *DEA364355*: 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 1 week are within 1% of the values measured on week 1.
- *DEA364376; DEA364357; DEA364360*: 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.
- *DEA364358*: 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 2 weeks are within 1% of the values measured on week 1.
- *DEA364359*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

SFL3

Method: GENSCRN-30; GENSCRN_30M; 30M_GENSCRN

Instruments: DEA364314; DEA364375; DEA364294; DEA364310; DEA364308

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 GC equipped with 5975/5977 MSD

Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 30 m × 0.25 mm I.D. × 0.25 μm film thickness (or equivalent)

Inlet Temperature:

- *DEA364314; DEA364375; DEA364294; DEA364310:* 280ºC
- *DEA364308:* 270ºC

Minimum Injection Volume: 1 μL

Injection Mode: Split

Maximum Split Ratio:

- *DEA364314; DEA364375;DEA364308*: 60:1
- *DEA364294; DEA364310*: 50:1

Carrier Gas and Flow:

- *DEA364314; DEA364375; DEA364310; DEA364308*: Helium, 1.5 mL/min
- *DEA364294*: Helium, 1.0 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.
  - *DEA364314; DEA364375*
- 125°C for 1.5 min, 45°C/min to 280°C, hold for 13.556 min
  - *DEA364294*
- 145 °C for 1.5 min, 25 °C/min to 280 °C, hold for 4.5 min, 30 ºC/min to 295 ºC, hold for 6.3 min
  - *DEA364310*
- 145 °C for 1.5 min, 20 °C/min to 280 °C, hold for 4.5 min, 30 ºC/min to 295 ºC, hold for 5.75 min
  - *DEA364308*

Minimum Run Time:

- *DEA364314; DEA364375; DEA364294:* 18.5 min
- *DEA364310:* 18.2 min
- *DEA364308:* 19.0 min

Detector: See *DEA364314; DEA364375; DEA364294; DEA364310; DEA364308* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364314; DEA364375; DEA364310; DEA364308*: 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.
- *DEA364294*: 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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: GENSCRNSHORT-15m

Instruments: DEA364359; DEA364360

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/5977B GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature: 280ºC
Minimum Injection Volume: 0.5 μL
Injection Mode: Split
Maximum Split Ratio: 40:1
Carrier Gas and Flow: Helium, 1.5 mL/min for 0.5 min, 99 mL/min/min to 3.0 mL/min for 6.5 min
Control Mode: Ramped flow
Oven Program Set Points: 120°C for 0.5 min, 40°C/min to 175°C, 30°C/min to 300°C, 20°C/min to 320°C
Minimum Run Time: 7.041 min
Detector: See *DEA364359* and *DEA364360* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: H2-FASTSCRN_DB35

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples are prepared in any appropriate non-chlorinated solvent. Internal standards may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977B GC/MSD
Column Type and Dimensions: DB-35 15 m × 0.25 mm I.D. × 0.25 μm film thickness
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen, 1.8 mL/min for 0.5 min then 2 mL/min per min to 2.5 mL/min
Control Mode: Ramped flow
Oven Program Set Points: 125°C for 0.5 min, 65°C/min to 175°C, 45°C/min to 300°C, 35°C/min to 330°C hold for 1.5958 min
Minimum Run Time: 6.5 minutes
Detector: See *DEA364405* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

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.

SFL3

Method: H2-FENT_DB35

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples are prepared in any appropriate non-chlorinated solvent. Internal standards may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977B GC/MSD
Column Type and Dimensions: DB-35 15 m × 0.25 mm I.D. × 0.25 μm film thickness
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 40:1
Carrier Gas and Flow: Hydrogen, 2.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 250°C for 5 min, 40°C/min to 330 °C, hold for 1 min
Minimum Run Time: 8.0 minutes
Detector: See *DEA364405* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

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.

SFL3

Method: H2-GENSCRN_DB35

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples are prepared in any appropriate non-chlorinated solvent. Internal standards may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977B GC/MSD
Column Type and Dimensions: DB-35 15 m × 0.25 mm I.D. × 0.25 μm film thickness
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 40:1
Carrier Gas and Flow: Hydrogen, 2.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 120°C for 1 min, 35°C/min to 310 °C, hold for 3 min
Minimum Run Time: 9.429 minutes
Detector: See *DEA364405* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

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.

SFL3

Method: H2-GENSCRN-30m

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples are prepared in any appropriate non-chlorinated solvent. Internal standards may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977A GC/MSD
Column Type and Dimensions: DB-5MS, 5% phenyl methylpolysiloxane stationary phase 30m × 0.25mm I.D. × 0.25µm film thickness
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen, 0.8 mL/min for 1.5 min, 1 mL/min/min to 3 mL/min for 8 min
Control Mode: Ramped flow
Oven Program Set Points: 120°C for 1.5 min, 40°C/min to 280 °C, hold for 7.5 min
Minimum Run Time: 13.0 minutes
Detector: See *DEA364374* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

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.

SFL3

Method: H2-HITEMP_DB35

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples are prepared in any appropriate non-chlorinated solvent. Internal standards may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977B GC/MSD
Column Type and Dimensions: DB-35 15 m × 0.25 mm I.D. × 0.25 μm film thickness
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: split
Maximum Split Ratio: 40:1
Carrier Gas and Flow: Hydrogen, 2.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 260°C for 4 min, 40°C/min to 330 °C, hold for 5 min
Minimum Run Time: 10.75 minutes
Detector: See *DEA364405* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

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.

SFL3

Method: HFASTSCRN; HFASTSCRNEXT

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 non-chlorinated appropriate solvent, including but not limited to methanol, acetone, petroleum ether, ethyl ether, ethyl acetate, xylenes and hexane, or any combination of these. Internal standards, such as diheptyl phthalate, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Agilent 7890B/5977B GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature:

- *HFASTSCRN*: 270 ºC
- *HFASTSCRNEXT*: 280°C

Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Hydrogen, 1.8 mL/min
Control Mode: Constant flow
Oven Program Set Points:

- *HFASTSCRN*: 120°C for 0.2 min, ramp temperature 40°C/min to 310°C, hold for 1.05 min
- *HFASTSCRNEXT*: 120°C for 0.2 min, ramp temperature 40°C/min to 310°C, hold for 3.05 min

Minimum Run Time:

- *HFASTSCRN*: 6 min
- *HFASTSCRNEXT*: 8 min

Detector: See DEA364404 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

Reproducibility:

- *HFASTSCRN*: Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.
- *HFASTSCRNEXT*: Individual retention times measured for representative compounds during 5 weeks are within 0.1 minutes of the values measured on week 1.

SFL3

Method: LONGSCRN

Instrument: DEA364354; DEA364355

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/5977A GC/MSD
Column Type and Dimensions: DB-5 5% phenyl methylpolysiloxane stationary phase, 15 m × 0.250 mm I.D. × 0.25 μm film thickness (or equivalent)
Inlet Temperature: 270 ºC
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 30:1
Carrier Gas and Flow: Helium, 1 mL/min
Control Mode: Constant flow
Oven Program Set Points: 90°C for 2 min, 15°C/min to 310 °C, hold for 5.33 min
Minimum Run Time: 21.997 min
Detector: See *DEA364354; DEA364355* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364354:* Individual retention times measured for representative compounds during 6 weeks are within 0.1 minutes of the values measured on week 1.

- *DEA364355:* 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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: LTM-GENSCRN-15

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 7890 equipped with Agilent 5977 MSD
Column Type and Dimensions: Inlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
LTM: DB-5 MS 15 m x 0.25 mm I.D. x 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent) Outlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
Inlet Temperature: 280ºC
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 280ºC
LTM Program Set Points: 145ºC for 1.0 min, 20 ºC/min to 280 ºC, hold for 0.75 min, 45 ºC/min to 295 ºC, hold for 2.667 min
Minimum Run Time: 11.5 min
Detector: See *DEA364356* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: LTM-SCREEN1-15

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 7890 equipped with Agilent 5977 MSD
Column Type and Dimensions: Inlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
LTM: DB-5 MS 15 m x 0.25 mm I.D. x 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent) Outlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
Inlet Temperature: 280ºC
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 280ºC
LTM Program Set Points: 160ºC for 0.5 min, 25 ºC/min to 260 ºC, hold for 0 min, 20 ºC/min to 280 ºC, hold for 0.5 min, 30 ºC/min to 295 ºC, hold for 1.5 min
Minimum Run Time: 8.0 min
Detector: See *DEA364356* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: LTM-SCREEN2-15

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 7890 equipped with Agilent 5977 MSD
Column Type and Dimensions: Inlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
LTM: DB-5 MS 15 m x 0.25 mm I.D. x 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent) Outlet segment: 1 m x 0.25 mm I.D. deactivated fused silica
Inlet Temperature: 280ºC
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 280ºC
LTM Program Set Points: 160ºC for 0.5 min, 35 ºC/min to 295 ºC, hold for 1.1429 min
Minimum Run Time: 5.5 min
Detector: See *DEA364356* MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: SCREEN1-15

Instrument: DEA364357; DEA364358

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 15 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 160°C for 0.5 min, 25°C/min to 260°C, 20°C/min to 280°C, hold for 0.5 min, 30°C/min to 295°C, hold for 1 min.
Minimum Run Time: 8.0 min
Detector: See *DEA364357; DEA364358* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364357*: 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.
- *DEA364358*: 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 2 weeks are within 1% of the values measured on week 1.

SFL3

Method: SCREEN1-30

Instruments: DEA364314; DEA364375

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 30 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 210°C for 1.5 min, 20°C/min to 280°C, hold for 4 min, 30°C/min to 295°C, hold for 3.0 min.
Minimum Run Time: 12.5 min
Detector: See *DEA364314; DEA364375* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: SCREEN2-15

Instrument: DEA364357; DEA364358

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 15 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 160°C for 0.5 min, 35°C/min to 295°C, hold for 1.1429 min.
Minimum Run Time: 5.5 min
Detector: See *DEA364357; DEA364358* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: SCREEN2-30

Instruments: DEA364314; DEA364375

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 30 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 220°C for 0 min, 30°C/min to 295°C, hold for 3.5 min.
Minimum Run Time: 6.0 min
Detector: See *DEA364314; DEA364375* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA364314*: 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 4 weeks are within 1% of the values measured on week 1.

- *DEA364375*: 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.

SFL3

Method: SCREEN3-15

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 15 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 200°C for 1.0 min, 30°C/min to 295°C, hold for 1.8333 min.
Minimum Run Time: 5.991 min
Detector: See *DEA364357* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: SCREEN3-30

Instruments: DEA364314

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 GC equipped with Agilent 5977 MSD
Column Type and Dimensions: DB-5 MS 30 m × 0.25 mm I.D. × 0.25 μm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1 μL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: Helium at 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 250°C for 1.0 min, 30°C/min to 295°C, hold for 5.5 min
Minimum Run Time: 8.0 min
Detector: See *DEA364314* MSD1 Mass Spectrometer Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: MSD1

Instruments: DEA364354; DEA364355; DEA364376; DEA364404; DEA364356; DEA364357; DEA364358; DEA364314; DEA364375; DEA364294; DEA364310; DEA364359; DEA364360; DEA364374; DEA364405; DEA364308

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5977
Mass Analyzer: Quadrupole
Ionization Mode: Electron Ionization
Scan Range:

- *DEA364354; DEA364355; DEA364376; DEA364404; DEA364356; DEA364357; DEA364358; DEA364314; DEA364308*
  - 40-600 m/z
- *DEA364294; DEA364310; DEA364375*
  - 40-500 m/z
- *DEA364359; DEA364360; DEA364374; DEA364405*
  - 40-550 m/z

Scan Rate: N=2
Source Temperature: 230ºC
MS Temperature: 150ºC
Transfer Line Temperature: 280ºC
Tune Type: Standard

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Methods: DEA-FTIR-2; DEA364317_IR1; DEA364368_IR1; DEA-FTIR-1

Instruments: DEA364303; DEA364367; DEA364317; DEA364368; DEA364369; DEA364370

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

Scope: General purpose

Sample Preparation:

Sample will be analyzed either by direct analysis, sublimation, or extraction with any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexane, or any combination of these.

Method Parameters:

Instrument: Thermo-Nicolet iS10
Number of Background Scans: 8 Scans
Minimum Number of Sample Scans:

- *DEA364303; DEA364367; DEA364369; DEA364370***:** 8 Scans
- *DEA364317; DEA364368*: 1 scan

Scan Range:

- *DEA364303; DEA364367*: 399-4000 cm^{-1}
- *DEA364317*: 400-4000 cm^{-1}
- *DEA364368; DEA364369; DEA364370*: 525-4000 cm^{-1}

Sample Gain: Autogain
Resolution: 4.000 cm^{-1}
Optical Velocity:

- *DEA364303; DEA364367; DEA364368; DEA364369; DEA364370*: 0.4747 cm/s
- *DEA364317*: 0.6329 cm/s

Aperture: 150.00 (open)
Accessory:

- *DEA364303; DEA364317***:** Smart Golden Gate KRS-5
- *DEA364367; DEA364368; DEA364369; DEA364370*: Smart iTX diamond crystal

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: DEA-FTIR-1

Instrument: DEA364382

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

Scope: General purpose

Sample Preparation:

Sample will be analyzed either by direct analysis, sublimation, or extraction with any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexane, or any combination of these.

Method Parameters:

Instrument:	Thermo-Nicolet iS50
Number of Background Scans:	8
Minimum Number of Sample Scans:	8
Scan Range:	525-4000 cm^{-1}
Sample Gain:	Autogain
Resolution:	4.000
Optical Velocity:	0.4747
Aperture:	150.00 (open)
Accessory:	Smart iTX diamond crystal

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL3

Method: DEA-RAMAN-1

Instrument: DEA364382

Identification of Controlled and Non-controlled Substances by RAMAN Spectroscopy

Scope: General purpose

Sample Preparation:

Sample will be analyzed either by direct analysis, sublimation, or extraction with any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexane, or any combination of these.

Method Parameters:

Instrument:	Thermo-Nicolet iS50
Number of Background Scans:	0
Minimum Number of Sample Scans:	8
Detector:	InGaAs
Sample Gain:	1.0
Resolution:	4.000
Optical Velocity:	0.3165
Aperture:	75.00
Min/Max Range:	350-3600 cm^{-1}

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFL 3 LSD Screen

Separation of Controlled and Non-controlled Substances by Liquid Chromatography

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

Sample Preparation:

Samples must be prepared in water, methanol, acetonitrile, or buffer. It is recommended that samples prepared in methanol or acetonitrile be diluted into water or buffer.

Method Parameters:

Instrument: Waters I-Class UPLC equipped with a PDA detector
Column: Waters Acquity UPLC BEH C18, 1.7 µm, 2.1 x 100 mm
Column Temperature: 40°C
Buffer/Mobile Phase: A: 20 mM Phosphate Buffer, pH 2.3 with 0.2% hexylamine and 25 mg/L of sodium azide; B: Acetonitrile
Minimum Injection Volume: 0.5 µL
Gradient Set Points: Isocratic (85% A, 15% B)
Flow Rate: 0.5 mL/min
Detection Wavelength (if used in method validation): 210 nm(+/- 1.2 nm)
Minimum Run Time: 5.5 minutes

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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/or 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/or the individual relative retention times during 6 weeks are with 1% of the values measured on week 1.

SFL 3 Phenethylamine 1

Separation of Controlled and Non-controlled Substances by Liquid Chromatography

Scope: Limited purpose (Pseudoephedrine, Methamphetamine, 3,4-MDMA, Caffeine)

Sample Preparation:

Samples must be prepared in water, methanol, acetonitrile, or buffer. It is recommended that samples prepared in methanol or acetonitrile be diluted into water or buffer.

Method Parameters:

Instrument: Waters I-Class UPLC equipped with a PDA detector
Column: Waters Acquity UPLC BEH C18, 1.7 µm, 2.1 x 100 mm
Column Temperature: 50°C
Buffer/Mobile Phase: A: 20 mM Phosphate Buffer, pH 2.3 with 0.2% hexylamine and 25 mg/L of sodium azide; B: Acetonitrile
Minimum Injection Volume: 0.5 µL
Gradient Set Points: Isocratic (94% A, 6% B)
Flow Rate: 0.5 mL/min
Detection Wavelength: 210 nm(+/- 1.2 nm)
Minimum Run Time: 3.5 minutes

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 retention times measured 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 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.

SFL3

Method: NMR_1H_1

Identification of Controlled and Non-controlled Substances by Nuclear Resonance Spectroscopy

Scope: General purpose (Proton)

Procedure:

Sample dissolved in deuterated solvents. Insoluble material filtered or removed from sample. Recommended sample concentration: 5-25 mg/mL in a 5 mm ID NMR tube.

Method Parameters:

Instrument: Varian 400-MR
MHz: 400
Minimum Spectral Range (ppm): 1-13
Minimum Delay between Pulses (seconds): 1
Minimum Pulse Angle (degrees): 1-90
Minimum Acquisition Time: 2 seconds
Minimum Number of Scans: 8

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra, or spectra from another ISO/IEC 17025. Overall sample spectral pattern corresponds to that of the reference spectrum acquired using the same solvent. All signals in the sample spectra were within 0.2ppm (^{1}H-NMR) of those in the reference spectrum. No unexplainable 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.

SFLT

Method: CHIRAL

Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope: Limited purpose (Analysis of methamphetamine and amphetamine)

Sample Preparation:

Samples will be dissolved in water, methanol or 0.1N HCl and diluted to an appropriate concentration using an internal standard solution containing phenylpropanolamine.

Method Parameters:

Instrument: Capillary Electrophoresis HP/Agilent 7100 equipped with an UV/Vis detector
Capillary Type and Dimensions: 64.5cm (56.0cm LEF) x 50µm ID bare fused silica capillary
Capillary Temperature: 20°C
Injection Parameters: Sample for 2 seconds at 50mbar; Water for 1 second at 35mbar
Buffer: Custom Chiral Buffer for Phenethylamines and Propoxyphene CE buffer with HP-B Cyclodextrin
Voltage: 30kV
Detection Wavelength: 200nm
Minimum Run Time: 17.3min

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 within 1% of the average of all injections.

Reproducibility: Individual migration times measured during 6 weeks. The individual relative retention times are within 1% of the values measured on week 1.

SFLT

Method: DEXTROMETHORPHAN

Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope: Limited purpose (levomethorphan and dextromethorphan)

Sample Preparation:

Samples will be dissolved in water, methanol or 0.1N HCl and diluted to an appropriate concentration using an internal standard solution containing phenylpropanolamine.

Method Parameters:

Instrument: Capillary Electrophoresis HP/Agilent 7100 equipped with an UV/Vis detector
Capillary Type and Dimensions: 64.5cm (56.0cm LEF) x 50µm ID bare fused silica capillary
Capillary Temperature: 20°C
Injection Parameters: Sample for 2 seconds at 50mbar; Water for 1 second at 35mbar
Buffer: Custom Buffer for d,l Methorphan
Voltage: 30kV
Detection Wavelength: 200nm
Minimum Run Time: 17.3min

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 within 1% of the average of all injections.

Reproducibility: Individual migration times measured during 6 weeks. The individual relative retention times are within 1% 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.

SFLT

Method: PSILOCIN-PSILOCYBIN

Separation of Controlled and Non-controlled Substances by Capillary Electrophoresis

Scope: Limited purpose (Psilocin-Psilocybin)

Sample Preparation:

Samples will be dissolved in an appropriate solvent, such as methanol, and diluted to an appropriate concentration. An internal standard solution containing phenylpropanolamine may be utilized but is not required.

Method Parameters:

Instrument: Capillary Electrophoresis HP/Agilent 7100 equipped with an UV/Vis detector
Capillary Type and Dimensions: 64.5cm (56.0cm LEF) x 50µm ID bare fused silica capillary
Capillary Temperature: 25°C
Injection Parameters: Sample for 2 seconds at 50mbar; Water for 1 second at 35mbar
Buffer: Accelerator B
Voltage: 30kV
Detection Wavelength: 220nm
Minimum Run Time: 15.0min

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 29 injections.

SFLT

Method: LTM MTPA FRONT

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (All isomers of methamphetamine)

Sample Preparation:

Samples will be based extracted into a suitable 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, and pyridine derivatized with MTPA.

Method Parameters:

Instrument: Gas Chromatograph HP/Agilent 7890B LTM equipped with an FID detector
Column Type and Dimensions: DB-CSI 15mx0.250mmI.D.x0.25µm film thickness in LTM; 1mx0.18mm segment heated in oven, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 270°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen 1.5mL/min
Make-up Gas: Nitrogen
Control Mode: constant flow
Oven Program Set Points: 270-300°C at 10°C/min
LTM Program Set Points: ramp at 10°C/min from 220°C to 250°C
Minimum Run Time: 3.0min
Detector Temperature: 300°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFLT

Method: LTM SCREEN FRONT

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: Gas Chromatograph HP/Agilent 7890B LTM equipped with an FID detector
Column Type and Dimensions: DB-CSI 15mx0.250mmI.D.x0.25µm film thickness in LTM; 1mx0.18mm segment heated in oven, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Hydrogen 2.0mL/min
Make-up Gas: Nitrogen
Control Mode: constant flow
Oven Program Set Points: 270-315°C at 15°C/min
LTM Program Set Points: ramp at 60°C/min from 95°C hold 0.2min to 310°C
Minimum Run Time: 5.5min
Detector Temperature: 300°C

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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 1 week are within 1% of the values measured on week 1.

SFLT

Method: CSCRN

Instruments: DEA849792; DEA879793

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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Helium, 1.5 mL/min
Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 165°C for 0.2min, 30°C/min to 315°C, hold for 0.3min
- *DEA849793*: 165°C for 0.2min, 30°C/min to 315°C, hold for 1min

Minimum Run Time:

- *DEA849792***:** 5.5 min
- *DEA879793*: 6.2 min

Detector: See MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA849793*: 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.
- *DEA849794*: 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 3 weeks are within 1% of the values measured on week 1.

SFLT

Method: DRUG 1

Instruments: DEA849792; DEA849793

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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA849792*: Helium, 1.3 mL/min
- *DEA849793*: Helium, 1.27 mL/min

Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 100°C for 0.5min, 30°C/min to 310°C, hold for 1min
- *DEA849793*: 100°C for 0.5min, 30°C/min to 310°C, hold for 1.5min

Minimum Run Time:

- *DEA849792*: 8.5 min
- *DEA849793*: 9.0 min

Detector: See MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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:

- *DEA849792*: 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.
- *DEA849793*: 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 4 weeks are within 1% of the values measured on week 1.

SFLT

Method: 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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Helium, 3mL/min
Control Mode: constant flow
Oven Program Set Points: 240°C for 0.6min, 15°C/min to 320°C, hold for 0.66667min
Minimum Run Time: 6.6min
Detector: See MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFLT

Method: TABSCRN

Instruments: DEA849792; DEA849793

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. Oxycodone used as fixed compound for selectivity during validation.

Method Parameters:

Instrument: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA849792*: Helium, 2.8 mL/min
- *DEA849793*: Helium, 2.7919mL/min

Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 240°C for 0.6min, 30°C/min to 310°C, hold for 0.1min
- *DEA849793*: 240°C for 0.6min, 30°C/min to 310°C, hold for 0.6min

Minimum Run Time:

- *DEA849792*: 3.033min
- *DEA849793*: 3.533min

Detector: See MSD1 Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

SFLT

Method: MSD1

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5977 MSD or equivalent
Mass Analyzer: Quadrapole
Ionization Mode: Electron ionization
Scan Range: 37-500 m/z
Scan Rate: n = 3
Source Temperature: 320°C
MS Temperature: 150°C
Transfer Line Temperature: 280°C
Tune Type: Standard

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFLT

Method: DEA360968-IR1

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

Scope: General purpose

Sample Preparation:

Samples will be analyzed by direct placement on the ATR. The may be done by direct analysis or after sublimation or extraction with any appropriate solvent including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexanes, or any combination of these.

Method Parameters:

Instrument: Thermo-Nicolet iS10 IR
Number of Background Scans: 8
Minimum Number of Sample Scans: 8 scans
Scan Range: 525-4000 cm-1
Sample Gain: Autogain
Resolution: 4.000 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 80.00 (Medium Resolution)
Accessory: ATR

Limitations: N/A

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

SFLT

Method: LSDSCRN

Separation of LSD and related 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: High Performance Liquid Chromatograph HP/Agilent 1200 series equipped with a DAD
Column: XDB C18 150mm x 4.6mm x 5µm (or equivalent)
Column Temperature: 50°C
Buffer/Mobile Phase: 85Mm Sodium Phosphate buffer w/ Hexylamine and sodium azide (pH ~2.5)
Minimum Injection Volume: 5µL
Gradient Set Points: Isocratic 80% Phosphate Buffer: 20% Acetonitrile
Flow Rate: 1.0mL/min
Detection Wavelength: 210nm
Minimum Run Time: 7.5 min.

Limitations: See individual instrument validation reports.

Acceptance Criteria (unless otherwise noted in individual validation report):

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.

G-METHH-01 – Quantitation of Methamphetamine by Gas Chromatography

Scope

Samples containing methamphetamine hydrochloride

Procedure:

Accurately weigh the sample and dissolve in internal standard solution so that the concentration of the target analyte is bracketed between the high- and low-concentration QC solutions. For samples containing a large amount of insoluble material, it may be necessary to deliver the required amount of internal standard solution via volumetric pipet instead of diluting to volume using the indicator line on a volumetric flask. Centrifuge or filter sample solutions containing appreciable amounts of insoluble material prior to removing aliquot portions for serial dilutions or transferring solutions to injection vials.

Internal Standard Solution:

1.0 mg/mL eicosane in chloroform.

Standard Solution:

Accurately weigh the Methamphetamine Hydrochloride reference material in Internal Standard Solution so that the concentration of the methamphetamine is within the working range.

Quality Control Solutions:

Prepare two QC solutions for use as positive controls during quantitative analysis. These two solutions are prepared such that their target analyte concentrations represent the low and high ends of the method's working range.

Chromatographic System:

Instrument: Gas Chromatograph HP/Agilent 7890 equipped with an FID (or equivalent)
Column: HP-5 (Agilent Part No. 19091J-101 SN USB409713B); 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenylmethylpolysiloxane stationary phase (or equivalent)
Inlet (Injector) Temperature: 280 °C
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program: Isothermal at 165 °C for 2 min, Ramp temperature 30 °C/min to 250 °C
Total Run Time: 4.8 min
Detector Temperature: 280 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: chloroform

Limitations:

N/A

Acceptance Criteria:

Selectivity: Methamphetamine and eicosane resolved (R ≥1.5) from each compound tested.
Linearity: At least seven concentrations were within 95-105% overall average sensitivity (response/concentration) limits.
Repeatability: Relative Standard Deviation (RSD) for each concentration tested was less than 2%.
Accuracy: Experimentally measured purity (expressed in % w/w) within ± 5% relative to the known prepared purity.

Working Range:

0.100 – 1.00 mg/mL Methamphetamine

G-PCPH-01 – Quantitation of PCP by Gas Chromatography

Scope

Samples containing PCP hydrochloride and/or base

Procedure:

Accurately weigh the sample and dissolve in internal standard solution so that the concentration of the target analyte is bracketed between the high- and low-concentration QC solutions. For samples containing a large amount of insoluble material, it may be necessary to deliver the required amount of internal standard solution via volumetric pipet instead of diluting to volume using the indicator line on a volumetric flask. Centrifuge or filter sample solutions containing appreciable amounts of insoluble material prior to removing aliquot portions for serial dilutions or transferring solutions to injection vials.

Internal Standard Solution:

0.4 mg/mL docosane in chloroform.

Standard Solution:

Accurately weigh the PCP reference material in Internal Standard Solution so that the concentration of the PCP is within the working range.

Quality Control Solutions:

Prepare two QC solutions for use as positive controls during quantitative analysis. These two solutions are prepared such that their target analyte concentrations represent the low and high ends of the method's working range.

Chromatographic System:

Instrument: Gas Chromatograph HP/Agilent 7890 equipped with an FID (or equivalent)
Column: HP-5 (Agilent Part No. 19091J-101 SN USB409713B); 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenylmethylpolysiloxane stationary phase (or equivalent)
Inlet (Injector) Temperature: 230 °C
Mode: Split
Split Ratio: 50:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program: Isothermal at 200 °C for 1.2 min, Ramp temperature 30 °C/min to 270 °C, hold for 2 min.
Total Run Time: 5.5 min
Detector Temperature: 280 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: chloroform

Limitations:

N/A

Acceptance Criteria:

Selectivity: PCP and docanse resolved (R ≥1.5) from each compound tested.
Linearity: At least seven concentrations were within 95-105% overall average sensitivity (response/concentration) limits.
Repeatability: Relative Standard Deviation (RSD) for each concentration tested was less than 2%.
Accuracy: Experimentally measured purity (expressed in % w/w) within ± 5% relative to the known prepared purity.

Working Range:

0.10 – 3.00 mg/mL PCP

METH-LC-1-6 – Quantitation of Methamphetamine by High Performance Liquid Chromatography

Scope

Samples containing methamphetamine hydrochloride

Procedure:

Accurately weigh the sample into a volumetric flask and dilute to volume using 0.1N HCl or other appropriate solvent. If necessary, perform dilutions so that the concentration of the target analyte is within the acceptable working range. Filter the solution through a 0.45 µm syringe filter.

Injection Solvent:

0.1N HCl or other appropriate solvent

Buffer Preparation:

4000mL distilled water, 10g sodium hydroxide, 30mL phosphoric acid, 8mL hexylamine, and 0.1g sodium azide

Standard Solution:

Accurately weigh the Methamphetamine Hydrochloride reference material in Injection Solvent so that the concentration of the oxycodone is within the working range.

Quality Control Solutions:

Prepare two QC solutions for use as positive controls during quantitative analysis. These two solutions are prepared such that their target analyte concentrations represent the low and high ends of the method's working range.

Chromatographic System:

Instrument: HPLC equipped with photo diode array detector (or equivalent)
Column: C18, 5 micron, 150mm x 4.60mm
Column Temperature: 50 °C
Injection Parameters: 3.0 µL
Injection Solvent: Buffer
Mobile Phase: 90% buffer: 10% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.
Flow: 1.0 mL/min
Detection Wavelength: UV diode array @ 210nm, 10nm BW; 550nm Ref, 100nm BW
Total Run Time: 4.0 minutes

Limitations:

N/A

Acceptance Criteria:

Selectivity: Methamphetamine resolved (R ≥1.5) from each compound tested.
Linearity: At least seven concentrations were within 95-105% overall average sensitivity (response/concentration) limits.
Repeatability: Relative Standard Deviation (RSD) for each concentration tested was less than 2%.
Accuracy: Experimentally measured purity (expressed in % w/w) within ± 5% relative to the known prepared purity.

Working Range:

0.08088 – 1.6176 mg/mL Methamphetamine

6OXY-LCM – Quantitation of Oxycodone by High Performance Liquid Chromatography

Scope

Samples containing oxycodone hydrochloride

Procedure:

Accurately weigh the sample into a volumetric flask and dilute to volume using Injection Solvent. If necessary, perform dilutions using Injection Solvent so that the concentration of the target analyte is within the acceptable working range. Filter the solution through a 0.45 µm syringe filter.

Injection Solvent:

0.1N HCl

Buffer Preparation:

Accurately measure out 4000mL of Millipore water then add 10.00g of sodium hydroxide pellets. Measure 30mL of phosphoric acid. Add the phosphoric acid to the solution and do not rinse out the graduated cylinder. Using a pipette measure 8mL of hexylamine and add to the solution. Using a calibrated pH meter, measure the pH of the solution and adjust the pH to 2.5 with a sodium hydroxide solution or phosphoric acid.

Standard Solution:

Accurately weigh the Oxycodone Hydrochloride reference material in Injection Solvent so that the concentration of the oxycodone is within the working range.

Quality Control Solutions:

Prepare two QC solutions for use as positive controls during quantitative analysis. These two solutions are prepared such that their target analyte concentrations represent the low and high ends of the method's working range.

Chromatographic System:

Instrument: HPLC equipped with photo diode array detector (or equivalent)
Column: 5µm C18, 150mm x 4.60mm
Column Temperature: 50 °C
Injection Parameters: 1.0 µL
Injection Solvent: Buffer
Mobile Phase: 85% buffer: 15% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.
Flow: 1.0 mL/min
Detection Wavelength: Diode array@ 280nm, 4nm BW; 550nm Ref, 80nm BW
Total Run Time: 5.0 minutes

Limitations:

N/A

Acceptance Criteria:

Selectivity: Oxycodone resolved (R ≥1.5) from each compound.
Linearity: At least seven concentrations were within 95-105% overall average sensitivity (response/concentration) limits.
Repeatability: Relative Standard Deviation (RSD) for each concentration tested was less than 2%.
Accuracy: Experimentally measured purity (expressed in % w/w) within ± 5% relative to the known prepared purity.

Working Range:

0.10 – 2.00 mg/mL Oxycodone