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.