SFL1

Method: SFL1 EARLY B

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent GC 7890B
Detector Temperature: 310°C
Column Type and Dimensions: HP-5 30m x 250µm x 0.25µm
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: H_2 at 1 mL/min
Make-up Gas: N_2 at 25 mL/min
Control Mode: Constant flow
Oven Program Set Points: 85°C for 2 min, 14°C/min to 300°C, hold 8 min
Minimum Run Time: 25.357 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 6 weeks are within 1% of the values measured on week 1.

SFL1

Methods: SFL1 SCRN 100A, SCRN 100B, SCRN 100B_F

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent GC 7890B
Detector Temperature: 310°C
Column Type and Dimensions: DB-5/HP-5 12m x 200µm x 0.33µm
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow:

- SCRN_100B: H_2 at 1.3101 mL/min
- SCRN_100A; SCRN_100B_F: H_2 at 1 mL/min

Make-up Gas: N_2 at 25 mL/min
Control Mode: Constant flow
Oven Program Set Points: 100°C for 1 min, 20°C/min to 275°C, hold 0.5 min, 35°C/min to 310°C, hold 3.25 min
Minimum Run Time: 14.5 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, including a low level (0.5%) marker compound. Earliest eluting compound met minimum retention factor criteria and/or fulfilled the repeatability and reproducibility acceptance criteria.

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

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

SFL1

Method: SFL1 SYNCANN B

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (FUB-AMB and ADB-FUBINACA)

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent GC 7890B
Detector Temperature: 310°C
Column Type and Dimensions: HP-5 12m x 200µm x 0.33µm
Inlet Temperature: 270°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: H_2 at 1.5 mL/min
Make-up Gas: N_2 at 25 mL/min
Control Mode: Constant flow
Oven Program Set Points: 165°C for 1 min, 30°C/min to 315°C, hold 6 min
Minimum Run Time: 12 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 6 weeks are within 1% of the values measured on week 1.

SFL1

Method: SFL1 EARLY

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent 7890A GC with Agilent 5975C MSD
Column Type and Dimensions: HP-5ms 30m x 250µm x 0.25µm
Inlet Temperature: 280°C
Minimum Injection Volume: 2 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow: H_2 at 1 mL/min
Control Mode: Constant flow
Oven Program Set Points: 90°C for 2 min, 14°C/min to 300°C, hold 10 min
Minimum Run Time: 27 min
Detector: See SFL1 EARLY 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/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.

SFL1

Method: SFL1 EARLY

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5975C
Mass Analyzer: Single quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 34-550
Scan Rate: 1,562 (n=2)
Source Temperature: 230°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.

SFL1

Method: SFL1 PCP

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (nicotine, 1-piperidinocyclohexanecarbontrile, phencyclidine, tenocyclidine)

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent 7890A GC with Agilent 5975C MSD
Column Type and Dimensions: HP-5ms 30m x 250µm x 0.25µm
Inlet Temperature: 280°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow: H_2 at 1 mL/min
Control Mode: Constant flow
Oven Program Set Points: 90°C for 2 min, 10°C/min to 190°C, hold 10 min, 20°C/min to 300°C, hold 0 min
Minimum Run Time: 27.5 min
Detector: See SFL1 PCP 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. 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.

SFL1

Method: SFL1 PCP

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

Scope: Limited purpose (nicotine, 1-piperidinocyclohexanecarbontrile, phencyclidine, tenocyclidine)

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5975C
Mass Analyzer: Single quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 34-550
Scan Rate: 1,562 (n=2)
Source Temperature: 230°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.

SFL1

Methods: SFL1 SCRN 250; SCRN 250A; SCRN 250B; SCRN 250C

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose

- *SFL1 SCRN 250; SCRN 250B; SCRN 250C*: Cannabidiol, Tetrahydrocannabinol and Cannabinol
- *SFL1 SCRN 250A*: Cannabidiol, Tetrahydrocannabinol, Cannabigerol and Cannabinol

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Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument:

- *SFL1 SCRN 250; SCRN 250A*: Agilent 7890B GC with Agilent 5977A MSD
- *SFL1 SCRN 250B; SCRN 250C*: Agilent 7890B GC with Agilent 5977B MSD

Column Type and Dimensions: DB-5ms 15m x 250µm x 0.25µm

Inlet Temperature:

- *SFL1 SCRN 250; SCRN 250C*: 250°C
- *SFL1 SCRN250A; SCRN 250 B*: 270°C

Minimum Injection Volume: 1 µL

Injection Mode: Split

Maximum Split Ratio: 60:1

Carrier Gas and Flow:

- *SFL1 SCRN 250*: H_2 at 0.80493 mL/min
- *SFL1 SCRN 250A; SCRN 250B*: H_2 at 1 mL/min
- *SFL1 SCRN250C*: H_2 at 1.2 mL/min

Control Mode: Constant flow

Oven Program Set Points:

- *SFL1 SCRN 250; SCRN 250A; SCRN 250B*: 250°C for 1 min, 20°C/min to 310°C, hold 2 min
- *SFL1 SCRN250C*: 250°C for 0 min, 50°C/min to 230°C, hold 3.4 min

Minimum Run Time:

- *SFL1 SCRN 250; SCRN 250A; SCRN 250B*: 6 min
- *SFL1 SCRN250C*: 5 min

Detector: See SFL1 SCRN 250, SCRN 250A, SCRN 250B and SCRN 250C 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. 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.

SFL1

Methods: SFL1 SCRN 250, SCRN 250A, SCRN 250B and SCRN 250C

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

Scope: Limited purpose

SFL1 SCRN 250; SCRN 250B and SCRN 250C: Cannabidiol, Tetrahydrocannabinol and Cannabinol

SFL1 SCRN 250A: Cannabidiol, Tetrahydrocannabinol, Cannabigerol and Cannabinol

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Sample Preparation:

GC effluent

Method Parameters:

Instrument:

- *SFL1 SCRN 250; SCRN 250A*: Agilent 5977A
- *SFL1 SCRN 250B; SCRN 250C*: Agilent 5977B

Mass Analyzer: Single quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 40-550
Scan Rate:

- *SFL1 SCRN 250; SCRN 250A*: 3,125 (n=1)
- *SFL1 SCRN 250B; SCRN 250C*: 1,562 (n=2)

Source Temperature:

- *SFL1 SCRN 250*: 300°C
- *SFL1 SCRN 250A*: 325°C
- *SFL1 SCRN 250B; SCRN 250C*: 230°C

MS Temperature: 150°C
Transfer Line Temperature:

- *SFL1 SCRN 250; SCRN 250A*; : 250°C
- *SFL1 SCRN 250B; SCRN 250C*: 280°C

Tune Type: Standard

Limitations summary: 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.

SFL1

Methods: SFL1 SCRN 90 and SCRN 90T

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument:

- *SFL1 SCRN 90*: Agilent 7890B GC with Agilent 5977B MSD
- *SFL1 SCRN 90T*: Agilent 7890B GC with Agilent 5977A MSD

Column Type and Dimensions: DB-5ms 15m x 250µm x 0.25µm
Inlet Temperature: 250°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 60:1
Carrier Gas and Flow: H_2 at 1.2 mL/min
Control Mode: Constant flow
Oven Program Set Points:

- *SFL1 SCRN 90***:** 90°C for 1 min, 30°C/min to 310°C, hold 2 min
- *SFL1 SCRN 90T*: 90°C for 1 min, 30°C/min to 310°C, hold 2.5 min

Minimum Run Time:

- *SFL1 SCRN 90***:** 10.333 min
- *SFL1 SCRN 90T*: 10.833 min

Detector: See SFL1 SCRN 90 and SCRN 90T 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/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.

SFL1

Methods: SFL1 SCRN 90 and SCRN 90T

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument:
- *SFL1 SCRN 90*: Agilent 5977B
- *SFL1 SCRN 90T*: Agilent 5977A

Mass Analyzer: Single quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 40-550
Scan Rate:
- *SFL1 SCRN 90*: 1,562 (n=2)
- *SFL1 SCRN 90T*: 3,125 (n=1)

Source Temperature:
- *SFL1 SCRN 90*: 230°C
- *SFL1 SCRN 90T*: 325°C

MS Temperature: 150°C
Transfer Line Temperature: 250°C
Tune Type: Standard

Limitations summary: 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.

SFL1

Method: SFL1 SYNCANN

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (FUB-AMB and ADB-FUBINACA)

Sample Preparation:

Prepare solutions in appropriate solvent at approximately 0.5-1.0 mg/mL concentration and filter prior to analysis. Sample may be base extracted, acid extracted, or derivatized prior to addition of solvent. Tetracosane is the internal standard used in the validation.

Method Parameters:

Instrument: Agilent 7890B GC with Agilent 5977B MSD
Column Type and Dimensions: DB-5ms 15m x 250µm x 0.25µm
Inlet Temperature: 250°C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 25:1
Carrier Gas and Flow: H_2 at 1.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 90°C for 1 min, 30°C/min to 310°C, hold 2 min
Minimum Run Time: 14.5 min
Detector: See SFL1 SYNCANN 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. 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.

SFL1

Method: SFL1 SYNCANN

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

Scope: Limited purpose (FUB-AMB and ADB-FUBINACA)

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5977B
Mass Analyzer: Single quadrupole
Ionization Mode: Negative Electron Ionization
Scan Range: 40-550
Scan Rate: 1,562 (n=2)
Source Temperature: 230°C
MS Temperature: 150°C
Transfer Line Temperature: 250°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.

SFL1

Method: SFL1 16Scans

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 Scientific, Nicolet iS10 FT-IR
Number of Background Scans: 16 Scans
Minimum Number of Sample Scans: 16 Scans
Scan Range: 4000-400 cm^{-1}
Sample Gain: 8.0
Resolution: 4 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 150.00
Accessory: Smart-Golden Gate ATR

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.

SFL1

Method: SFL1 32Scans

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 Scientific, Nicolet iS10 FT-IR
Number of Background Scans: 32 Scans
Minimum Number of Sample Scans: 32 Scans
Scan Range: 4000-400 cm^{-1}
Sample Gain: 8.0
Resolution: 4 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 150.00
Accessory: Smart-Golden Gate ATR

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.

TCPPCP – Quantitation of Phencyclidine by Gas Chromatography

Scope

Samples containing phencyclidine base and/or phencyclidine hydrochloride in the presence of tenocyclidine.

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 phencyclidine hydrochloride reference material in Internal Standard Solution so that the concentration of the phencyclidine 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 detector (or equivalent)
Column: DB-1 (Agilent Part No. 122-1032); 30 m × 0.25 mm I.D. × 0.25 µm film thickness, 100% dimethylpolysiloxane stationary phase
Inlet (Injector) Temperature: 270 °C
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program: Isothermal at 125 °C, Ramp temperature 6 °C/min to 165 °C, 4 °C/min to 200 °C, 30 °C/min to 240 °C, hold for 4 min.
Total Run Time: 20.75 min
Detector Temperature: 310 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: Chloroform

Limitations:

This method has no known limitations.

Acceptance Criteria:

Selectivity: Phencyclidine and docosane 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:

0.1141 – 0.9512 mg/mL Phencyclidine