SFL2

Method: DART-SCRN-OrbiTrapMS

Separation of Controlled and Non-controlled Substances by Mass Spectrometry DART

Scope: General purpose

Sample Preparation:

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

Method Parameters:

Instrument: ThermoFisher Scientific Exactive Plus HRAM DART-MS
Mass Analyzer: Orbitrap
Ionization Mode: DART
Drying Gas: Nitrogen
Ionization gas: Nitrogen
Capillary Temperature: 250°C
MS Scan Range: 50-750
MS Scan Rate: 7.2 scans/second
Collision Gas: Nitrogen
Collision Energy: 1V, 30V, 60V
Tune File: Exactive Plus calibration
Reference Masses: 195.087, 524.264, 1221.990, 1421.977, 1621.965
Activation Type: SID

Limitations: See individual instrument validation reports.

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

Selectivity and 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 measured monoisotopic pseudomolecular $(M+H)^+$ weight corresponds to the theoretical molecular weight of the substance. The measured m/z values for prominent ions in the sample spectrum were all within 5 ppm as those in the reference spectrum. No prominent unexplainable extraneous ions are observed in the sample spectrum.

SFL2

Method: AMINESRRT

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical sample concentration range is 0.5 – 10 mg/mL but sample concentration is not limited to this range. Appropriate solvents for this method include, but are not limited to, chloroform, methanol, hexane, or a combination of these solvents. An internal standard solution (ISS) of 0.05 mg/mL tetracosane (C24) in 4:1 chloroform (CHCl3): methanol (MeOH) is also suitable. Liquid-liquid extractions, such as basic extraction, into an appropriate solvent are also permissible. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar. It is also recommended that aqueous-extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent GC-FID 7890A
Detector Temperature: 280ºC
Column Type and Dimensions: DB-5 30m x 0.250mm x 0.25µm
Inlet Temperature: 280ºC
Minimum Injection Volume: 1µl
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: (H_2) 3.5ml/min (3min); ramp at 4ml/min/min to 6ml/min
Make-up Gas: N_2
Control Mode: Constant Flow
Oven Program Set Points: 150 ºC for 3 min the 45 ºC/min to 290 ºC for 0.5 min
Minimum Run Time: 6.61l 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 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.

SFL2

Method: HT30SCRN

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical sample concentration range is 0.5 – 10 mg/mL but sample concentration is not limited to this range. Appropriate solvents for this method include, but are not limited to, chloroform, methanol, hexane, or a combination of these solvents. An internal standard solution (ISS) of 0.05 mg/mL tetracosane (C24) in 4:1 chloroform (CHCl3): methanol (MeOH) or a marker solution containing C24 is also suitable. Liquid-liquid extractions, such as basic extraction, into an appropriate solvent are also permissible. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar. It is also recommended that aqueous-extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent GC-FID 7890A
Detector Temperature: 280ºC
Column Type and Dimensions: DB-5 30m x 0.320mm x 0.25µm
Inlet Temperature: 270ºC
Minimum Injection Volume: 1µl
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: (H_2) 1.5ml/min (2.4min); ramp at 45ml/min/min to 2.5ml/min (1 min); ramp at 45ml/min/min to 3.7ml/min (3.5min)
Make-up Gas: N_2
Control Mode: Constant Pressure
Oven Program Set Points: 240ºC for 3.5 min then 45ºC/min to 300ºC for 2.2min
Minimum Run Time: 7.0333 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 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.

SFL2

Methods: MTPA; MTPA5

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited Purpose

- MTPA: analysis of optical isomers of MPTA-derivatized substances
- MTPA5: analysis of optical isomers of methamphetamine

Sample Preparation:

A typical sample concentration range is 3 – 10 mg/mL but sample concentration is not limited to this range. Samples should be prepared by basic extraction (~10% sodium hydroxide, aqueous) into a suitable organic solvent. Appropriate solvents for this method include chloroform, methylene chloride, or hexane. The organic layer containing the target analyte(s) should be filtered through a cotton-plugged pipet, syringe filter, or similar with sodium sulfate or magnesium sulfate to remove residual water. Add approximately 2-5 drops of α-methoxy-α trifluoromethylphenylacetic acid reagent (MTPA) [0.1M in CHCl3] to the filtered sample.

Method Parameters:

Instrument: Agilent GC-FID 7890A
Detector Temperature: 280ºC
Column Type and Dimensions: DB-5 30m x 320µm x 0.25µm
Inlet Temperature: 270ºC
Minimum Injection Volume: 1µl
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: (H_2) 2.0ml/min
Make-up Gas: N_2
Control Mode: Constant Flow
Oven Program Set Points: Isothermal at 220ºC
Minimum Run Time:

- *MTPA*: 9 min
- *MTPA5*: 5.6 min

Limitations: See individual instrument validation reports.

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

Selectivity: Target compounds are visually separated and observed as a single peak with a clear, non-splitting apex. Peak fronting/tailing, if observed, does not preclude the detection of closely eluting peaks. A minimum S/N =3 is observed. Earliest eluting compound met minimum retention factor criteria and fulfilled the repeatability and reproducibility acceptance criteria. Co-analysis (spiking) must be used for isomer determinations when more than one peak is within the acceptance criteria window of 0.1 minutes or 1% relative retention time, but is optional otherwise.

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.

SFL2 SCRN30

Method: SCRN30

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical sample concentration range is 0.5 – 10 mg/mL but sample concentration is not limited to this range. Appropriate solvents for this method include, but are not limited to, chloroform, methanol, hexane, or a combination of these solvents. An internal standard solution (ISS) of 0.05 mg/mL tetracosane (C24) in 4:1 chloroform (CHCl3): methanol (MeOH) or a marker solution containing C24 is also suitable. Liquid-liquid extractions, such as basic extraction, into an appropriate solvent are also permissible. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar. It is also recommended that aqueous-extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent GC-FID 7890A
Detector Temperature: 280ºC
Column Type and Dimensions: DB-5 30m x 0.320mm x 0.25µm
Inlet Temperature: 270ºC
Minimum Injection Volume: 1µl
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: (H_2) 2.5ml/min (5min); ramp at 45ml/min/min to 3.5ml/min (2.2min)
Make-up Gas: N_2
Control Mode: Constant Pressure
Oven Program Set Points: 175ºC for 1 min then 15ºC/min to 280ºC for 3.5min
Minimum Run Time: 11.5 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 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.

SFL2

Method: AMINES

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (Analysis of Amines)

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

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

Limitations: See individual instrument validation reports.

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

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

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

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

SFL2

Method: FASTGEN

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent 7890B (GC portion) and 5977A/B (MS portion)
Column Type and Dimensions: HP-5MS Ultra Inert 20m x 180 μm x 0.18μm
Inlet Temperature: 280ºC
Minimum Injection Volume: 1μL
Injection Mode: Split
Maximum Split Ratio: 100:1
Carrier Gas and Flow: Helium; 1.5 mL/min for 1.5 min, then 15mL/min per min to 2.5 mL/min for 5 min then 15mL/min per min to 3 mL/min
Control Mode: Ramped flow
Oven Program Set Points: 110°C for 0.7 min, then 66°C/min to 150°C for 0.3 min, then 90°C/min to 310°C for 5.2 min
Minimum Run Time: 8.583 min
Detector: See MSD Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

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

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

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

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

SFL2

Method: FASTSTEROID

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:
Instrument: Agilent 7890B (GC portion) and 5977A/B (MS portion)
Column Type and Dimensions: HP-5MS 20m x 180µm x 0.18µm film thickness
Inlet Temperature: 280ºC
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 30:1
Carrier Gas and Flow: Helium; 1 mL/min for 2.5 min, then 15 mL/min/min to 2 mL/min for 8 min, then 15 mL/min/min to 3 mL/min
Control Mode: Ramped flow
Oven Program Set Points: 90°C for 2 min, then 35°C/min to 200°C for 1.5 min, then 60°C/min to 310°C for 11.7 min
Minimum Run Time: 20.177 min
Detector: See MSD Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

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

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

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

Reproducibility: Individual retention times measured during 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.

SFL2

Method: GENERAL

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent 7890A/B (GC portion) and 5975C or 5977A/B (MS portion)
Column Type and Dimensions: HP-5MS 30m x 250µm x 0.25µm
Inlet Temperature: 280ºC
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 75:1
Carrier Gas and Flow: Helium and 1.5 mL/min
Control Mode: Constant flow
Oven Program Set Points: 90°C for 1.35 min, then 35°C/min to 120°C for 0.55 min, then 45°C/min to 290°C for 8.5 min
Minimum Run Time: 15.035
Detector: See MSD Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

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

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

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

Reproducibility: Individual retention times measured during 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.

SFL2

Method: GENERAL-L

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent 7890B (GC portion) and 5977A (MS portion)
Column Type and Dimensions: Pre-column: DB-5 0.3m x 180µm x 0.18µm; DB-5MS (DB-CSI#6) 15m x 250µm x 0.25µm
Inlet Temperature: 280ºC
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 20:1
Carrier Gas and Flow: Helium and 2.2 mL/min
Control Mode: Constant flow
Oven Program Set Points: 280°C, then 20°C/min to 325°C for 1.4967 min
LTM Program Set Points: 75°C for 0.5 min, then 240°C/min to 140°C then 75°C/min to 340°C for 0.4 min
Minimum Run Time: 3.838 min
Detector: See MSD Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

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

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

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

Reproducibility: Individual retention times measured during 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.

SFL2

Method: HTGEN-L

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose

Sample Preparation:

Samples can be prepared in various solvents and at various concentrations. A typical range of concentration is 0.5-10 mg/mL, but can be more or less dependent upon the sample. Appropriate solvents for this method include, but are not limited to, methanol, chloroform, petroleum ether, hexanes, ethanol, and methylene chloride. A combination of these solvents or liquid-liquid extraction is also acceptable. A marker solution, such as tetracosane in solution, is permitted. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate insolubles. It is also recommended that aqueous extracted samples are passed through sodium sulfate or magnesium sulfate to remove residual water.

Method Parameters:

Instrument: Agilent 7890B (GC portion) and 5977A (MS portion)
Column Type and Dimensions: Pre-column: DB-5 0.3m x 180µm x 0.18µm; DB-5MS (DB-CSI#6) 15m x 250µm x 0.25µm
Inlet Temperature: 280ºC
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 75:1
Carrier Gas and Flow: Helium and 2mL/min
Control Mode: Constant flow
Oven Program Set Points: 280°C, then 20°C/min to 325°C for 2.2357 min
LTM Program Set Points: 90°C for 0.2 min, then 175°C/min to 280°C for 0.2 min then 75°C/min to 340°C for 2.2 min
Minimum Run Time: 4.486 min
Detector: See MSD Mass Spectrometry Method Parameters

Limitations: See individual instrument validation reports.

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

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

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

Reproducibility: Individual retention times measured during 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.

SFL2

Method: MSD

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 7890A/B (GC) and 5975C or 5977A/B (MS)
Mass Analyzer: Quadrupole
Ion Source: Electron Ionization (EI) (70eV)
Scan Range: 35-550
Scan Rate: 1,562 [N=2]
Source Temperature: 230ºC or 350 ºC
MS Quad Temperature: 150ºC
Transfer Line Temperature: 280ºC
Tune File: Stune.u

Limitations: See individual instrument validation reports.

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

Accuracy: Compounds are compared to a verified reference database, commercial library or published literature 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 (or pseudo-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.

SFL2

Method: SFL2

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

Scope: General purpose

Sample Preparation:

Sample will be analyzed directly or prepared for analysis by recovery of analyte(s) from a liquid, sublimation, or extraction with appropriate solvents including, but not limited to, chloroform, methanol, acetone, hexane, ethyl ether, petroleum ether, and recovery of analyte(s) from such extractions, with or without recrystallization.

Method Parameters:

Instrument: Thermo Scientific Nicolet IS10
Number of Background Scans: 4 Scans
Minimum Number of Sample Scans: 4 Scans
Scan Range: 600-4000 cm^{-1}
Sample Gain: 1.0-8.0 (Autogain)
Resolution: 4.000 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 150.00 (open)
Accessory: Smart iTX

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 corresponded to that of the reference spectrum; the prominent measured signals in the sample spectra were all within 4 cm^{-1} of those in the reference spectrum; and there were no prominent extraneous signals present in the sample spectrum.

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

SFL2

Method: SFL2-Transmittance

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

Scope: Limited purpose (analysis of phenformin HCl)

Sample Preparation:

Sample will be analyzed by placing directly on salt plate (invisible to IR) or prepared for analysis by recovery of analyte(s) from a liquid, sublimation, or extraction with appropriate solvents including, but not limited to, chloroform, methanol, acetone, hexane, ethyl ether, petroleum ether, and recovery of analyte(s) from such extractions, with or without recrystallization.

Method Parameters:

Instrument: Thermo Scientific Nicolet IS10
Number of Background Scans: 4 Scans
Minimum Number of Sample Scans: 4 Scans
Scan Range: 400-4000 cm^{-1}
Sample Gain: 1.0
Resolution: 4.000 cm^{-1}
Optical Velocity: 0.4747
Aperture: 80.00 (Medium Resolution)
Accessory: Smart Omni-Transmission Accessory

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 corresponded to that of the reference spectrum; the prominent measured signals in the sample spectra were all within 4 cm^{-1} of those in the reference spectrum; and there were no prominent extraneous signals present in the sample spectrum.

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

SFL2

Method: Proton

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

Scope: General purpose (Proton)

Procedure:

Samples should be prepared using an appropriate solvent to dissolve the analyte of interest (ie: maleic acid in deuterium oxide (D2O)) containing 4,4-dimethyl-4-silapentane-1-sulfonic acid (DSS), tetramethylsilane (TMS), or trimethylsilylpropanoic acid (TSP) for 0 ppm reference at a recommended concentration ranging from 5-30mg/mL, but can be more or less depending on the sample. Vortex the sample for several seconds. If insolubles are present, additional solvent (not containing maleic acid or the reference compound) may be added. It is recommended that samples are filtered through a cotton-plugged pipet, syringe filter, or similar, to eliminate additional insolubles. Place sample into an NMR tube, cap it and place it in an NMR turbine properly.

Method Parameters:

Instrument: Varian/400MR
Minimum Spectral Width (sw): at least containing -1 ppm through 13 ppm
Minimum Delay Between Pulses (d): ≤45 seconds
Minimum Pulse Angle: ≤ 90 degrees
Minimum Acquisition Time: ≥ 2.5 seconds
Minimum Number of Scans: ≥ 1 scan (as necessary to enhance signal to noise)

Limitations: See individual instrument validation reports.

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

Accuracy: Analyte spectra are compared to a verified reference database, commercial library, published literature spectra, or spectra from another ISO/IEC 17025. Overall sample spectral pattern corresponds to that of the reference spectrum acquired using the same solvent. Prominent measured signals in the sample spectra were all within 0.2ppm of those in the reference spectrum. No unexplainable extraneous signals are observed in the sample spectrum.

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

PCPQ1 – Quantitation of Phencyclidine (PCP) by Gas Chromatography

Scope

Samples containing phencyclidine (PCP)

Procedure:

Accurately weigh the sample and dissolve in internal standard solution. If necessary, perform a serial dilution using internal standard solution so that the concentration of the target analyte is within the low and high concentration QC solutions. Filter the solution if an appreciable amount of insoluble material is present.

Internal Standard Solution:

0.9507 mg/mL Eicosane in chloroform/methanol (80:20)

Standard Solution:

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

Quality Control Solutions:

Prepare two QC solutions for PCP 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: HP-5 30 m × 0.320 mm I.D. × 0.25 µm film thickness,
Inlet (Injector) Temperature: 220 °C
Mode: Split
Split Ratio: 100:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 2ml/min(1.65min);ramp@1.5ml/min/min to 3ml/min(4min)
Oven Program: 165C for 1.5min then 40C/min to 250C for 1.5 min
Total Run Time: 5.125 min
Detector Temperature: 275°C
Signal Data (Sampling) Rate: 20 Hz
Injection Volume: 1 µL
Injection Solvent: chloroform/methanol 80:20)

Limitations:

There are no known limitations associated with this method.

Acceptance Criteria:

Selectivity: Selectivity was determined for the critical pair of PCP and lidocaine. Resolution between the PCP/Lidocaine critical pair is > 1.5.
Linearity: Eight 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:

0.16 mg/mL – 3.01 mg/mL PCP