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.