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.