SFLT

Method: CSCRN

Instruments: DEA849792; DEA879793

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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Helium, 1.5 mL/min
Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 165°C for 0.2min, 30°C/min to 315°C, hold for 0.3min
- *DEA849793*: 165°C for 0.2min, 30°C/min to 315°C, hold for 1min

Minimum Run Time:

- *DEA849792***:** 5.5 min
- *DEA879793*: 6.2 min

Detector: See 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:

- *DEA849793*: 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.
- *DEA849794*: 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 3 weeks are within 1% of the values measured on week 1.

SFLT

Method: DRUG 1

Instruments: DEA849792; DEA849793

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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA849792*: Helium, 1.3 mL/min
- *DEA849793*: Helium, 1.27 mL/min

Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 100°C for 0.5min, 30°C/min to 310°C, hold for 1min
- *DEA849793*: 100°C for 0.5min, 30°C/min to 310°C, hold for 1.5min

Minimum Run Time:

- *DEA849792*: 8.5 min
- *DEA849793*: 9.0 min

Detector: See 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 with 1% of the average of 30 injections.

Reproducibility:

- *DEA849792*: 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 with 1% of the values measured on week 1.
- *DEA849793*: 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 4 weeks are within 1% of the values measured on week 1.

SFLT

Method: 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: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow: Helium, 3mL/min
Control Mode: constant flow
Oven Program Set Points: 240°C for 0.6min, 15°C/min to 320°C, hold for 0.66667min
Minimum Run Time: 6.6min
Detector: See 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 with 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 with 1% of the values measured on week 1.

SFLT

Method: TABSCRN

Instruments: DEA849792; DEA849793

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. Oxycodone used as fixed compound for selectivity during validation.

Method Parameters:

Instrument: Gas Chromatograph HP/Agilent 7890 equipped with Agilent 5977 mass selective detector
Column Type and Dimensions: ZB-1MS 15m x 0.25mm I.D. x 0.25µm film thickness (or equivalent)
Inlet Temperature: 280°C
Minimum Injection Volume: 1µL
Injection Mode: Split
Maximum Split Ratio: 50:1
Carrier Gas and Flow:

- *DEA849792*: Helium, 2.8 mL/min
- *DEA849793*: Helium, 2.7919mL/min

Control Mode: constant flow
Oven Program Set Points:

- *DEA849792*: 240°C for 0.6min, 30°C/min to 310°C, hold for 0.1min
- *DEA849793*: 240°C for 0.6min, 30°C/min to 310°C, hold for 0.6min

Minimum Run Time:

- *DEA849792*: 3.033min
- *DEA849793*: 3.533min

Detector: See 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/or the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

SFLT

Method: MSD1

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters:

Instrument: Agilent 5977 MSD or equivalent
Mass Analyzer: Quadrapole
Ionization Mode: Electron ionization
Scan Range: 37-500 m/z
Scan Rate: n = 3
Source Temperature: 320°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.