SFL3

Method: ISOMERSCRN

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 ether, hexane or chloroform. Internal standards, such as tetracosane, may be used. Perform filtration, centrifugation, derivatization and acid/base extraction as necessary.

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B equipped with Analytical Solutions and Providers, Model IRD3
Column Type and Dimensions: HP-5 30 m × 0.32 mm I.D. × 0.25 µm film thickness, 5% phenyl methylpolysiloxane stationary phase (or equivalent)
Inlet Temperature: 280ºC
Minimum Injection Volume: 2 µL
Injection Mode: Split
Maximum Split Ratio: 5:1
Carrier Gas and Flow: Helium at 2.0 mL/min
Control Mode: Constant flow
Oven Program Set Points: 70ºC, hold for 2 min, 10ºC/min to 280ºC, hold for 4 min, 35ºC/min to 310ºC, hold for 3 min
Minimum Run Time: 30.857 min
Detector Temperature: see IRD1 method for DEA364380

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. This method is not intended for the use of identifying substances at low concentrations. Substances at concentrations of 0.5% and lower are not within the scope of this method. 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 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 the individual relative retention times during 6 weeks are within 1% of the values measured on week 1.

SFL3 IRD1

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

Scope: General purpose

Sample Preparation:

GC effluent

Method Parameters

Instrument: Analytical Solution and Providers, Model IRD3
Scan Range: 4000 – 550 cm^{-1}
Transfer Line Temperature: 280ºC
Resolution: 8 cm^{-1}
Light Pipe Temperature: 280ºC

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 spectra 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.