GCHIGH-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Cocaine, Heroin, Fentanyl, and Other Late Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, and methylene chloride. Samples should be filtered prior to analysis to remove any insoluble material. Tetracosane is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Tetracosane in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.7 mL/min
Control Mode: Constant flow
Oven Program: 210 °C, hold 0.1 min, ramp 30 °C/min to 300 °C, hold 0.4 min
Total Run Time: 3.50 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

GCHIGH-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Cocaine, Heroin, Fentanyl, and Other Late Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, and methylene chloride. Samples should be filtered prior to analysis to remove any insoluble material. Tetracosane is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Tetracosane in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Helium
Carrier Gas Flow Rate: 1.7 mL/min
Control Mode: Constant flow
Oven Program: 210°C, hold 0.1 min, ramp 30°C/min to 300°C, and hold 0.4 min
Total Run Time: 4.10 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

GCLOW-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Dimethyl phthalate in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.7-2.0 mL/min
Control Mode: Constant flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C
Total Run Time: 3.00 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

GCLOWX-H – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Dimethyl phthalate in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.7-2.0 mL/min, hold 3 min, ramp at 10 mL/min to 2.5 mL/min, hold 0.1 min
Control Mode: Ramped flow
Oven Program: 75°C, hold 0.5 min, ramp at 40°C/min to 175°C, no hold, ramp at 30°C/min to 300°C, hold 0.1 min
Total Run Time: 7.27 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

GCLOW-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Dimethyl phthalate in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Helium
Carrier Gas Flow Rate: 1.7 mL/min
Control Mode: Constant flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C, hold 0.5 min
Total Run Time: 3.50 min

Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

GCLOWX-He – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine and Other Early Eluting Compounds)

Sample Preparation:

Samples can be prepared with organic solvents such as acetone, acetonitrile, chloroform, ethanol, ethyl acetate, hexane, methanol, methylene chloride, ammoniacal chloroform, and ammoniacal hexane. Samples should be filtered prior to analysis to remove any insoluble material. Dimethyl phthalate is used as the internal standard (reference compound), if necessary.

Solubility limitations and instrumental response differences should be considered when choosing solvents for analysis.

Sample can be base-extracted into organic solvent. Base-extracted samples should be centrifuged and/or passed through sodium sulfate prior to analysis to remove residual water. Samples should be analyzed soon after preparation when base-extracting into ethyl acetate; otherwise acetylation by-products can be observed over time.

Internal Standard Solution:

Dimethyl phthalate in selected solvent (optional)

Method Parameters:

Instrument: Gas Chromatograph Agilent 7890B
Column: DB-5MS Ultra Inert 15 m × 0.25 mm I.D. × 0.25 µm film thickness, [(5% phenyl)-methylpolysiloxane] stationary phase
Inlet (Injection) Temperature: 280 °C
Injection Volume: 1.0 µL
Mode: Split
Split Ratio: 60:1
Carrier Gas: Helium
Carrier Gas Flow Rate: 1.7 mL/min, hold 3.5 min, ramp at 10 mL/min to 2.5 mL/min, hold 1 min
Control Mode: Ramped flow
Oven Program: 75°C, hold 0.5 min, ramp 40°C/min to 175°C, hold 0.5 min, ramp 30°C/min to 300°C, hold 1 min
Total Run Time: 8.67 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

IR01 – Identification of Controlled and Non-controlled Substances by Solid Phase Infrared Spectroscopy

Scope:

General Purpose

Sample Preparation:

Powder and liquid samples are analyzed by direct analysis. If needed to separate mixtures, samples may be extracted using solid-or liquid based procedures, or by sublimation.

Method Parameters:

Instrument: Thermo Scientific Nicolet iS10 Infrared Spectrometer
Number of Background Scans: 8 scans
Minimum Number of Sample Scans: 8 scans
Scan Range: 650-4000 cm^{-1}
Sample Gain: Autogain
Resolution: 4.000 cm^{-1}
Optical Velocity: 0.4747 cm/s
Aperture: 150 (Open)
Accessory: Smart Golden Gate ATR

Limitations:

See individual instrument validation reports

Acceptance Criteria:

Selectivity: 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 withinm^{-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 are established via evaluation of system-wide historical spectral data.

ISOM01 – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Methamphetamine Diastereoisomers)

Sample Preparation:

6-8 mg of sample is weighed into a test tube. 1 mL of 1 N NaOH is added and mixed thoroughly. 2 mL of hexane is added and mixed thoroughly to extract. Transfer the top (hexane) sample layer to a new test tube and discard the bottom basic layer. Three drops of MTPA-Cl reagent is added to sample and vortexed for 5 seconds. Filter and transfer to an autosampler vial.

Internal Standard Solution:

N/A

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph with Flame Ionization Detector (GC/FID)
Column Type and Dimensions: HP-5 30 m x 0.32 mm I.D. x 0.25 µm film thickness
Inlet Temperature: 260 °C
Minimum Injection Volume: 1 µL
Injection Mode: Split
Maximum Split Ratio: 35:1
Carrier Gas and Flow: Hydrogen, 1.75 mL/min
Make-up Gas: Nitrogen
Control Mode: Constant flow
Oven Program Set Points: 220 °C isothermal
Minimum Run Time: 5.5 min
Detector Temperature: 300 °C

Limitations:

See individual instrument validation reports

Acceptance Criteria:

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/or 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.

MS01 – Identification of Controlled and Non-controlled Substances by Mass Spectrometry (EI)

Scope:

General Purpose

Sample Preparation:

GC effluent

Internal Standard Solution:

N/A

Method Parameters:

Instrument: Agilent Technologies 5977 Mass Spectrometer
Mass Analyzer: Quadruple
Ionization Mode: Positive Electron Ionization
Scan Range: 40-500 m/z
Scan Rate: N = 2
Source Temperature: 230 °C
MS Temperature: 150 °C
Transfer Line Temperature: 280 °C
Tune Type: Standard Tune

Limitations:

See individual instrument validation reports.

Acceptance Criteria:

Selectivity: 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 are established via evaluation of system-wide historical spectral data.

THCSCRN – Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope:

Limited Purpose (Separation of Cannabinoids)

Sample Preparation:

Weigh approximately 50 mg of plant material (excluding seeds, stems, stalks and roots) into a test tube or beaker. Add 5 mL of internal standard solution and extract at room temperature for at least 10 minutes. Vortex at least twice for 10-15 seconds during the extraction period. Filter into autosampler vial prior to analysis.

Internal Standard Solution:

0.05 mg/mL 4-androsten-3,17-dione in 9:1 Methanol/Chloroform, or 0.05 mg/mL testosterone in 9:1 Methanol/Chloroform

Method Parameters:

Instrument: Agilent 7890B Gas Chromatograph System
Column Type and Dimensions: DB-5MS 15 m x 0.25 mm x 0.25 µm
Inlet (Injection) Temperature: 250 °C
Injection Volume: 1 µL
Mode: Split
Split Ratio: 50:1
Carrier Gas: Helium
Carrier Gas Flow Rate: 1.2 mL/min, hold 4.3 min, ramp at 2 mL/min to 1.5 mL/min
Control Mode: Ramped flow
Oven Program: 210 °C, ramp at 30 °C/min to 235 °C, hold 3 min, ramp at 30 °C/min to 280 °C
Minimum Run Time: 5.3 min
Detector: See MS01 Mass Spectrometer Method Parameters

Limitations:

See individual instrument validation reports.

Acceptance Criteria:

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/or 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.

DEA 108 – Quantitation of Hydrocodone Bitartrate by Gas Chromatography

Scope:

Samples containing Hydrocodone Bitartrate

Procedure:

Accurately weigh the sample and dissolve in internal standard solution so that the concentration of the target analyte is bracketed between the high- and low-concentration QC solutions. For samples containing a large amount of insoluble material, it may be necessary to deliver the required amount of internal standard solution via volumetric pipet instead of diluting to volume using the indicator line on a volumetric flask. Centrifuge or filter sample solutions containing appreciable amounts of insoluble material prior to removing aliquot portions for serial dilutions or transferring solutions to injection vials.

Internal Standard Solution:

0.4 mg/mL tetracosane in 1:1 Chloroform/Methanol solution.

Standard Solution:

Accurately weigh the Hydrocodone Bitartrate reference material and dissolve into the Internal Standard Solution. Prepare the solution such that the concentration of target analyte is within the method's working range.

Quality Control Solutions:

Prepare two QC solutions 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 Agilent 7890A equipped with an FID detector (or equivalent)
Column: DB-5 12 m long × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenylmethylpolysiloxane stationary phase
Inlet (Injector) Temperature: 280 °C
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program: 250 °C for 2 min, ramp 30 °C/min to 300 °C, hold 1 min
Total Run Time: 4.6 min
Detector Temperature: 280 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: 1:1 Chloroform/Methanol

Limitations:

N/A

Acceptance Criteria:

Selectivity: Hydrocodone and tetracosane resolved (R ≥1.5) from each compound tested.
Linearity: At least seven 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:

0.11 -1.99 mg/mL

DEA 250 – Quantitation of Δ^9-Tetrahydrocannabinol (THC) and Δ^9-Tetrahydrocannabinolic Acid (THCA) by Liquid Chromatography

Scope:

DEA 250 is an external standard, multi-point calibration method used for the quantitation of Δ^9-Tetrahydrocannabinol (THC) and Δ^9-Tetrahydrocannabinolic Acid (THCA)

Procedure:

1. Grind at least 200 mg of dry plant material and then sieve the ground material through a 40-mesh screen (425 µm particle size)
2. Weigh two separate portions of 100 mg of the material from step 1 into two separate centrifuge tubes
3. Add 5mL of 80:20 ACN: MEOH into each centrifuge tube, and vortex for 2-3 seconds
4. Sonicate for 15 minutes
5. Centrifuge at 1000 rpm for 2 minutes
6. Transfer each supernatant into a 10 mL volumetric flask and dilute to mark using 80:20 ACN: MeOH
7. If necessary, performed a second dilution using 80:20 ACN: MeOH to attain target concentration.
8. Pass the final solutions via a 0.45 µm filter and into an autosampler vial.

Internal Standard Solution:

N/A

Standard Solution:

Cayman Phytocannabinoid Mixture 5 (CRM)

Quality Control Solutions:

Prepare two QC solutions 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: Shimadzu LC-2030C Plus Cannabis Analyzer
Column: Shimadzu Nexleaf CBX for Potency: 150 mm x 4.6 mm, 2.7 µm
Column Temperature: 35 °C
Injection Volume: 5 µL
Injection Solvent: 80% Acetonitrile (ACN) / 20% Methanol
Autosampler Temperature: 4 °C
Flow: 1.6 ml/min
Mobile Phase: A: 0.085% H_3PO_4 in water; B: 0.085% H_3PO_4 in Acetonitrile
Gradient Program:

0.00-3.00 min: 30:70 A/B
3.00-7.00 min: 30:70 A/B to 15:85 A/B
7.00-7.01 min: 15:85 A/B to 5:95 A/B
7.01-8.00 min: 5:95 A/B
8.00-8.01 min: 5:95A/B to 30:70 A/B
8:01-10.0 min: 30:70 A/B

Detection: 220 nm
Sampling Period: 200 msec
Peak Width: > 5 s

Limitations:

Δ^8- THC and Δ^9-THC are not baseline resolved when both compounds are present in similar concentration. If Δ^8- THC is present in the sample at a high concentration, resolution between Δ^8-THC and Δ^9-THC should be greater than 1.3.

Acceptance Criteria:

Selectivity: Δ^9-THC and Δ^9-THCA are resolved (R ≥1.5) from each compound tested.
Linearity: At least seven concentrations were within 95-105% overall average sensitivity (response/concentration) limits.
Repeatability: Relative Standard Deviation (RSD) for each concentration tested was less than 2%.
Recovery: Experimentally measured within 92-103%.

Working Range:

Δ^9-THC: 29.2 to 125 µg/mL

Δ^9-THCA: 13.1 to 250 µg/mL

DEA 503 – Quantitation of Methamphetamine HCl by UV/Vis Spectroscopy

Scope:

DEA 503 applies to samples containing methamphetamine hydrochloride (HCl), and samples containing mixtures of methamphetamine HCl, dimethyl sulfone, boric acid, or sodium chloride.

Procedure:

Dissolve samples in DI H20 using class-A glassware or calibrated micro pipettes. Solutions shall be prepared so that final target analyte concentrations are bracketed by simultaneously analyzed quality control (QC) solutions.

Standard Solution:

Stock solutions are prepared by dissolving methamphetamine HCL certified reference material in DI H20. Two separate stock solutions are prepared for 18-Cell Cuvette Changer and the Fiber Optic Dip Probe configurations, respectively.

Quality Control Solutions:

Prepare two QC solutions 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.

Method Parameters:

Instrument: Agilent Cary 60 UV/Vis Spectrometer
Configurations: 18-Cell Cuvette Chamber or Fiber Optic Dip Probe
Detection wavelength: 267 nm
Collection time: 0.5 seconds
Replicates: 5
Total Run Time: 2.5 seconds

Limitations:

Accuracy Limitations: DEA 503 is accurate for quantitation of methamphetamine HCl in deionized water using 267 nm as the detection wavelength, when there are no other compounds with significant absorption at that wavelength, such as caffeine, cocaine HCl, creatine, α-Benzyl-N-methylphenethylamine (B-compound), or phenethylamine.
Solution Storage: Solutions should be analyzed within 24 hours of preparation. If solutions cannot be analyzed within such time frame, then refrigeration is recommended. The absorbance of the solutions should be evaluated upon preparation and during refrigerated storage in order to monitor any response changes.

Acceptance Criteria:

Selectivity: The selectivity of DEA 503 was evaluated by collecting the UV/Vis spectra (200-300 nm) for negative controls, individual component solutions, and mixtures of methamphetamine HCl with selected compounds. The absorption of each tested solution at 267 nm was compared to the absorption measured for methamphetamine hydrochloride at similar concentration.
Linearity: The linear range of DEA 503 was determined separately for each of the two instrument configurations.

- Accepts linearity data for concentrations that fall within the 95 - 105% sensitivity limits.
- Ensures the final accepted linear range is comprised of at least seven concentrations.

Repeatability: Accepts repeatability data that does not exceed 2% calculated RSD for each concentration level tested.
Accuracy: Accepts accuracy data where the experimentally measured purity (expressed in % w/w) is within ± 5% relative to the known prepared purity.

Working Range:

18 Cell Cuvette Chamber: 1.37 – 4.97 mg/mL

Fiber Optic Dip Probe: 0.54– 3.98 mg/mL