

SFL6

Method: GCLOWC-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.32 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Make-up Gas: Helium or Nitrogen

Control Mode: ramped flow

Oven Program Set Points: 100 °C initial temperature ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 6.28 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/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.

SFL6

Method: GCLOWD-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 12 m x 0.20 mm x 0.33µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Make-up Gas: Helium or Nitrogen

Control Mode: ramped flow

Oven Program Set Points: 100 °C initial temperature, ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 6.28 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/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.

SFL6

Method: GCMIDC-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.32 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min (0.5 min hold), ramped to 3 mL/min at 0.5 mL/min/min

Make-up Gas: Helium or Nitrogen

Control Mode: ramped flow

Oven Program Set Points: 170 °C initial temperature (0.5 min hold), ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 4.78 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/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.

SFL6

Method: GCHIGHC-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.32 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min ramped to 3 mL/min, Hold 0.5 minute ramp 0.5 mL/min/min

Make-up Gas: Helium or Nitrogen

Control Mode: ramped flow

Oven Program Set Points: 250 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 39 °C /min, 320 °C final temperature

Minimum Run Time: 2.00 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/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.

SFL6

Method: ISO-GC-1

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (for the qualitative analysis and determination of optical isomers of methamphetamine)

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the validation, samples of *d,l*-methamphetamine were prepared at approximately 0.6 mg/mL and samples of *d*-methamphetamine were prepared at approximately 0.3 mg/mL. Prior to each analysis, samples were base extracted into hexane with 0.1 mg/mL of tricosane.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.32 mm x 0.25 µm

Inlet Temperature: 280 °C

Minimum Injection Volume: 1.0 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Average Velocity: Hydrogen; 65 cm/sec

Make-up Gas: Helium or Nitrogen

Control Mode: constant flow

Oven Program Set Points: 220 °C initial temperature, hold initial for 0.25 minute, ramp to 265 °C at 15 °C /min, ramp to

300 °C at 35 °C /min, hold for 1.0 minute, 300°C final temperature

Minimum Run Time: 5.25 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/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.

SFL6

Method: ISO-GC-2

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: Limited purpose (for the qualitative analysis and determination of optical isomers of methamphetamine)

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the validation, samples of *d,l*-methamphetamine were prepared at approximately 0.6 mg/mL and samples of *d*-methamphetamine were prepared at approximately 0.3 mg/mL. Prior to each analysis, samples were base extracted into hexane with 0.1 mg/mL of tricosane.

Method Parameters:

Instrument: Agilent GC/FID 7890

Detector Temperature: 280 °C

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.25 mm x 0.25 µm

Inlet Temperature: 280 °C

Minimum Injection Volume: 1.0 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Average Velocity: Hydrogen; 65 cm/sec

Make-up Gas: Helium or Nitrogen

Control Mode: constant flow

Oven Program Set Points: 220 °C initial temperature, hold initial for 0.25 minute, ramp to 265 °C at 15 °C /min, ramp to

300 °C at 35 °C /min, hold for 1.0 minute, 300°C final temperature

Minimum Run Time: 5.25 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/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.

SFL6

Method: GENERAL PURPOSE

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC-IRD solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 2.0 mg/mL in chloroform with approximately 0.5 mg/mL of methamphetamine.

Method Parameters:

Instrument: Agilent/ASAP-GC/IRD 7890A/IRD II

Column Type and Dimensions: HP-5/DB-5; 30 m x 0.32 mm x 0.25µm

Inlet Temperature: 265 °C

Minimum Injection Volume: 0.2 µL

Injection Mode: Split

Maximum Split Ratio: 2:1

Carrier Gas and Flow: Helium; 2.0 mL/min

Control Mode: constant flow

Oven Program Set Points: 70 °C initial temperature, hold initial for 0.5 minute, ramp to 310 °C at 20 °C /min, 320 °C final temperature

Minimum Run Time: 13.50 min

Detector: see IRD Infrared Spectroscopy 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 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 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.

SFL6 - IRD

Method: IRD

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

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC-IRD solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL.

Method Parameters:

Instrument: Agilent/ASAP; GC/IRD 7890A/IRD II

Scan Range: 550 cm^{-1} to 4000 cm^{-1}

Transfer Line Temperature: 280 °C

Resolution: 16

Light Pipe Temperature: 280 °C

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.

SFL6

Method: GCLOWA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 100 °C initial temperature ramped to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 6.28 min

Detector: see MS 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 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.

SFL6

Method: GCLOW-LTMA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (hold 0.5 min) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 100 °C initial temperature, hold initial for 0.0 minute, ramp to 320 °C at 35 °C /min, 320 °C final temperature

LTM Program Set Points: 100 °C initial temperature, hold initial for 0.0 minute, ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 6.28 min

Detector: see MS 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 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.

SFL6

Method: GCLOWB

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol within approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35/DB-35; 15 m x 0.20 mm x 0.33 μ m

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 μ L

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 100 °C initial temperature, ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 6.28 min

Detector: see MS 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 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.

SFL6

Method: GC1-LTMF-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35 LTM/DB-35 LTM; 15 m x 0.18 mm x 0.20 µm

Inlet Temperature: 265 °C

Minimum Injection Volume: 1.0 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min

Control Mode: constant flow

Oven Program Set Points: 100 °C initial temperature (1.0 min hold), ramp to 310 °C at 35 °C /min, 310 °C final temperature

LTM Program Set Points: 100 °C initial temperature (1.0 min hold), ramp to 310 °C at 50 °C /min, hold for 1.0 minute, 310 °C final temperature

Minimum Run Time: 6.20 min

Detector: see MS 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 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.

SFL6

Method: GCMIDA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB-5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 170 °C initial temperature (0.5 min hold), ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 4.78 min

Detector: see MS 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 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.

SFL6

Method: GCMID-LTMA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB-5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (hold 0.5 min) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 170 °C initial temperature (0.5 min hold), ramp to 320 °C at 35 °C /min, 320 °C final temperature

LTM Program Set Points: 170 °C initial temperature (0.5 min hold), ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 4.78 min

Detector: see MS 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 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.

SFL6

Method: GCMIDB

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35/DB-35; 15 m x 0.20 mm x 0.33µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 170 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 35 °C /min, 320 °C final temperature

Minimum Run Time: 4.78 min

Detector: see MS 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 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.

SFL6

Method: GC2-LTMF-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35 LTM/DB-35 LTM; 15 m x 0.18 mm x 0.20 µm

Inlet Temperature: 265 °C

Minimum Injection Volume: 0.3 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min

Control Mode: constant flow

Oven Program Set Points: 180 °C initial temperature (0.3 min hold), ramp to 250 °C at 40 °C /min, 250 °C final temperature

LTM Program Set Points: 140 °C initial temperature (0.5 min hold), ramp to 310 °C at 200 °C /min, hold for 0.8 minute, 310 °C final temperature

Minimum Run Time: 3.05 min

Detector: see MS 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 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.

SFL6

Method: GCHIGHA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min ramped to 3 mL/min, Hold 0.5 minute ramp 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 250 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 39 °C /min, 320 °C final temperature

Minimum Run Time: 2.00 min

Detector: see MS 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 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.

SFL6

Method: GCHIGH-LTMA

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-5/DB5; 15 m x 0.25 mm x 0.25µm

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 µL

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min (0.5 min hold) ramped to 3 mL/min at 0.5 mL/min/min

Control Mode: ramped flow

Oven Program Set Points: 250 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 39 °C /min, 320 °C final temperature

LTM Program Set Points: 250 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 39 °C /min, 320 °C final temperature

Minimum Run Time: 2.00 min

Detector: see MS 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 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.

SFL6

Method: GCHIGHB

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35/DB-35; 15 m x 0.20 mm x 0.33 μ m

Inlet Temperature: 270 °C

Minimum Injection Volume: 0.5 μ L

Injection Mode: Split

Maximum Split Ratio: 50:1

Carrier Gas and Flow: Helium; 1.0 mL/min ramped to 3 mL/min, Hold 0.5 minute ramp 0.5 mL/min/min

Control Mode: constant pressure, ramped flow

Oven Program Set Points: 250 °C initial temperature, hold initial for 0.5 minute, ramp to 320 °C at 39 °C /min, 320 °C final temperature

Minimum Run Time: 2.00 min

Detector: see MS 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 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.

SFL6

Method: GC3-LTMF-H

Separation of Controlled and Non-controlled Substances by Gas Chromatography

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL in methanol with approximately 0.05 mg/mL of fentanyl and approximately 0.05 mg/mL of methyl palmitate.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Column Type and Dimensions: HP-35 LTM/DB-35 LTM; 15 m x 0.18 mm x 0.20 µm

Inlet Temperature: 280 °C

Minimum Injection Volume: 0.2 µL

Injection Mode: Split

Maximum Split Ratio: 100:1

Carrier Gas and Flow: Hydrogen; 1.0 mL/min

Control Mode: constant flow

Oven Program Set Points: 300 °C initial temperature, hold initial for 0.0 minute, ramp to 325 °C at 39 °C /min, 325 °C final temperature

LTM Program Set Points: 270 °C initial temperature, hold initial for 0.0 minute, ramp to 340 °C at 175 °C /min, hold for 0.5 minute, 325 °C final temperature

Minimum Run Time: 0.90 min

Detector: see MS 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 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.

SFL6

Method: MS

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

Scope: General purpose

Sample Preparation:

Samples may be prepared using any generally accepted GC-MS solvent and sample preparation procedure. For the initial validation, all samples were prepared at approximately 1.0 mg/mL.

Method Parameters:

Instrument: Agilent GC/MS 7890/5977 or equivalent

Mass Analyzer: Quadrapole

Ionization Mode: EI

Scan Range: 40 m/z – 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 (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.

SFL6

Methods: ATR

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

Scope: General purpose

Sample Preparation:

Sample will be analyzed either by direct analysis, sublimation, or extraction with any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexane, or any combination of these.

Method Parameters:

Instrument: Thermo Fisher Scientific / Thermo Nicolet iS10 FT-IR

Number of Background Scans: 8 Scans

Minimum Number of Sample Scans: 8 Scans

Scan Range: 400 cm^{-1} to 4000 cm^{-1}

Sample Gain: Autogain

Resolution: 4 cm^{-1}

Optical Velocity: 0.4747 cm/s

Aperture: Open

Accessory: ATR

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 spectrum 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.

SFL6

Method: ATR50

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

Scope: General purpose

Sample Preparation:

Sample will be analyzed either by direct analysis, sublimation, or extraction with any appropriate solvent, including but not limited to methanol, chloroform, acetone, petroleum ether, ethyl ether, methylene chloride, water, ethyl acetate, xylenes and hexane, or any combination of these.

Method Parameters:

Instrument: Thermo Fisher Scientific / Thermo Nicolet iS50 FT-IR

Number of Background Scans: 8 Scans

Minimum Number of Sample Scans: 8 Scans

Scan Range: 400 cm^{-1} to 4000 cm^{-1}

Sample Gain: Autogain

Resolution: 4 cm^{-1}

Optical Velocity: 0.4747 cm/s

Aperture: 100

Accessory: Raman

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 spectrum 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.

G-PCPH-01 – Quantitation of Phencyclidine by Gas Chromatography

Scope

Samples containing phencyclidine

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 docosane in chloroform.

Standard Solution:

Accurately weigh the Phencyclidine Base reference material in Internal Standard Solution so that the concentration of the phencyclidine is within the 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 HP/Agilent 7890 equipped with an FID detector (or equivalent)

Column: HP-5 (Agilent Part No. 19091J-101 SN USB409713B); 12 m × 0.20 mm I.D. × 0.33 µm film thickness, 5% phenylmethylpolysiloxane stationary phase

Inlet (Injector) Temperature: 230 °C

Mode: Split

Split Ratio: 50:1

Carrier Gas: Hydrogen

Carrier Gas Flow Rate: 1.0 mL/min

Oven Program: 200 °C for 1.2 min., ramp temperature 30 °C/min to 270 °C, hold for 2 min

Total Run Time: 5.5333 min

Detector Temperature: 280 °C

Signal Data (Sampling) Rate: 50 Hz

Injection Volume: 1 µL

Injection Solvent: chloroform

Limitations:

N/A

Acceptance Criteria:

Selectivity: Phencyclidine and docosane 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:

0.1168 mg/mL – 2.9359 mg/mL Phencyclidine

METH-LC – Quantitation of Methamphetamine by High Performance Liquid Chromatography

Scope

Samples containing methamphetamine hydrochloride and/or methamphetamine base

Procedure:

Accurately weigh the sample into a volumetric flask and dilute to volume using Injection Solvent. If necessary, perform a serial dilution using Injection Solvent so that the concentration of the target analyte is within the acceptable working range. Filter the solution through a 0.2-0.45 µm filter.

Injection Solvent:

0.1 N hydrochloric acid

Buffer Preparation:

To 4 L of water, add 30 mL phosphoric acid, 10.0 g NaOH, 8 mL hexylamine, and 100 mg sodium azide (pH 2.5).

Standard Solution:

Accurately weigh the Methamphetamine Hydrochloride reference material in Injection Solvent so that the concentration of the methamphetamine is approximately 0.6 mg/mL.

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: High Performance Liquid Chromatograph HP/Agilent 1200 Series (or equivalent)

Column: Phenomenex Luna C18 Column: 75 mm x 4.6 mm x 3 µm, or 3.5 µm

Column Temperature: 50 °C

Mobile Phase: 95% buffer: 5% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.

Flow: 1.5 mL/min

Gradient Program: Isocratic

Total Run Time: 3 min

Detection: 210 nm (10 nm bandwidth); reference: 360 nm (100 nm bandwidth); peak width > 0.05 min

Injection Volume: 3 µL

Injection Solvent: 0.1 N hydrochloric acid

Limitations:

This method is not suitable for samples containing acetaminophen.

Acceptance Criteria:

Selectivity: Methamphetamine resolved ($R \geq 1.5$) from each compound tested except for the compounds noted in Limitations.

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 $\pm 5\%$ relative to the known prepared purity.

Working Range:

0.11672 mg/mL – 1.25039 mg/mL Methamphetamine

MDMA-LC – Quantitation of Methylenedioxyamphetamine by High Performance Liquid Chromatography

Scope

Samples containing ephedrine, amphetamine, methamphetamine, methylenedioxyamphetamine, methylenedioxyamphetamine, and ketamine

Procedure:

Accurately weigh the appropriate standard(s) into a volumetric flask and dilute to volume using Injection Solvent. If necessary, perform a serial dilution using Injection Solvent so that the concentration of the target analyte is within the acceptable working range. Filter the solution through a 0.2-0.45 µm filter.

Injection Solvent:

0.1 N hydrochloric acid

Buffer Preparation:

To 4 L of water, add 30 mL phosphoric acid, 10.0 g NaOH, 8 mL hexylamine, and 100 mg sodium azide (pH 2.5).

Standard Solution:

Accurately weigh the appropriate reference material(s) in Injection Solvent so that the concentration of the appropriate standard is approximately 0.5 mg/mL (Ephedrine 0.25 mg/mL).

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: High Performance Liquid Chromatograph HP/Agilent 1200 Series (or equivalent)

Column: Phenomenex Luna C18 Column: 75 mm x 4.6 mm x 3 µm, or 3.5 µm

Column Temperature: 50 °C

Mobile Phase: 95% buffer: 5% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.

Flow: 2.0 mL/min

Gradient Program: Isocratic

Total Run Time: 5 min

Detection: 210 nm (4 nm bandwidth); reference: 360 nm (4 nm bandwidth); 234 nm (4 nm bandwidth); reference: 360 nm (4 nm bandwidth); peak width > 0.05 min

Injection Volume: 2.5 µL

Injection Solvent: 0.1 N hydrochloric acid

Limitations:

This method parameters will not resolve mixtures of methylenedioxyamphetamine and methamphetamine.

Acceptance Criteria:

Selectivity: Ephedrine, Amphetamine, Methamphetamine, Methylenedioxyamphetamine, Methylenedioxyamphetamine and Ketamine resolved ($R \geq 1.5$) from each compound tested except for the compounds noted in Limitations.

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 $\pm 5\%$ relative to the known prepared purity.

Working Range:

0.06 mg/mL – 0.5 mg/mL Ephedrine

0.10 mg/mL – 0.82 mg/mL Amphetamine

0.24 mg/mL – 1.02 mg/mL Methamphetamine

0.22 mg/mL – 1.09 mg/mL
Methylenedioxyamphetamine (210 nm)

0.07 mg/mL – 1.09 mg/mL
Methylenedioxyamphetamine (234 nm)

0.24 mg/mL – 1.00 mg/mL
Methylenedioxyamphetamine (210 nm)

0.05 mg/mL – 1.00 mg/mL
Methylenedioxyamphetamine (234 nm)

0.05 mg/mL – 1.09 mg/mL Ketamine

PSEUDO-UPLC – Quantitation of Pseudoephedrine by Ultra-High Performance Liquid Chromatography

Scope

Samples containing pseudoephedrine

Procedure:

Accurately weigh the sample using a five decimal place analytical balance into a volumetric flask and dilute to volume using Injection Solvent. If necessary, perform a serial dilution using Injection Solvent so that the concentration of the target analyte is within 25% of the standard concentration. Filter the solution through a 0.2-0.45 µm filter.

Injection Solvent:

Potassium hydroxide buffer (pH ~ 11.0 – 11.5).

Buffer Preparation:

To 1 L of water, add 1 mL of 10% potassium hydroxide.

Standard Solution:

Accurately weigh the Pseudoephedrine Hydrochloride using a five place analytical balance reference material in Injection Solvent so that the concentration of the methamphetamine is within the working range.

Quality Control Solutions:

Prepare two QC solutions using a five place decimal place analytical balance for use as positive controls during quantitative analysis. These two solutions are prepared such that their target analyte is within 25% of the standard concentration. The QC low is prepared by gravimetrically diluting the QC high solution until the desired concentration is reached.

Chromatographic System:

Instrument: Waters Acquity UPLC equipped with photo diode array detector (or equivalent)

Column: Waters BEH C18, 100 mm, 2.1 mm id, 1.7 µm particle size

Column Temperature: 65 °C

Injection Solvent: Buffer

Injection Volume: 5 µL

Gradient Program:

- 0.0 – 3.0 min: 70:30 to 50:50 buffer:methanol
- 3.0 – 3.5 min: 50:50 to 5:95 buffer:methanol
- 3.6 – 4.0 min: 5:95 to 70:30 buffer:methanol

Flow: 0.600 mL/min

Detection Wavelength: 210 - 350 nm, collected data at 220 nm

Total Run Time: 4.0 minutes

Limitations:

This method will not separate benzylpiperazine and pseudoephedrine.

Acceptance Criteria:

Selectivity: Pseudoephedrine resolved ($R \geq 1.5$) from each compound tested from each compound tested except for the compounds noted in Limitations.

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 $\pm 5\%$ relative to the known prepared purity.

Working Range:

0.0998808 mg/mL – 0.209164 mg/mL
Pseudoephedrine