G-METHH-01 – Quantitation of Methamphetamine by Gas Chromatography

Scope

Samples containing methamphetamine hydrochloride

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:

1.0 mg/mL eicosane in chloroform.

Standard Solution:

Accurately weigh the Methamphetamine Hydrochloride reference material in Internal Standard Solution so that the concentration of the methamphetamine 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 (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 (or equivalent)
Inlet (Injector) Temperature: 280 °C
Mode: Split
Split Ratio: 60:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program Set Points: Isothermal at 165 °C for 2 min, Ramp temperature 30 °C/min to 250 °C
Total Run Time: 4.8 min
Detector Temperature: 280 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: chloroform

Limitations:

N/A

Acceptance Criteria:

Selectivity: Methamphetamine and eicosane 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:

0.100 – 1.00 mg/mL Methamphetamine

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

Scope

Samples containing PCP hydrochloride and/or base

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 PCP reference material in Internal Standard Solution so that the concentration of the PCP 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 (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 (or equivalent)
Inlet (Injector) Temperature: 230 °C
Mode: Split
Split Ratio: 50:1
Carrier Gas: Hydrogen
Carrier Gas Flow Rate: 1.0 mL/min
Oven Program Set Points: Isothermal at 200 °C for 1.2 min, Ramp temperature 30 °C/min to 270 °C, hold for 2 min.
Total Run Time: 5.5 min
Detector Temperature: 280 °C
Signal Data (Sampling) Rate: 50 Hz
Injection Volume: 1 µL
Injection Solvent: chloroform

Limitations:

N/A

Acceptance Criteria:

Selectivity: PCP and docanse 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:

0.10 – 3.00 mg/mL PCP

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METH-LC-1-6 – Quantitation of Methamphetamine by High Performance Liquid Chromatography

Scope

Samples containing methamphetamine hydrochloride

Procedure:

Accurately weigh the sample into a volumetric flask and dilute to volume using 0.1N HCl or other appropriate solvent. If necessary, perform dilutions so that the concentration of the target analyte is within the acceptable working range. Filter the solution through a 0.45 µm syringe filter.

Injection Solvent:

0.1N HCl or other appropriate solvent

Buffer Preparation:

4000mL distilled water, 10g sodium hydroxide, 30mL phosphoric acid, 8mL hexylamine, and 0.1g sodium azide

Standard Solution:

Accurately weigh the Methamphetamine Hydrochloride reference material in Injection Solvent so that the concentration of the oxycodone 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: HPLC equipped with photo diode array detector (or equivalent)
Column: C18, 5 micron, 150mm x 4.60mm
Column Temperature: 50 °C
Injection Parameters: 3.0 µL
Injection Solvent: Buffer
Mobile Phase: 90% buffer: 10% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.
Flow: 1.0 mL/min
Detection Wavelength: UV diode array @ 210nm, 10nm BW; 550nm Ref, 100nm BW
Total Run Time: 4.0 minutes

Limitations:

N/A

Acceptance Criteria:

Selectivity: Methamphetamine 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:

0.08088 – 1.6176 mg/mL Methamphetamine

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6OXY-LCM – Quantitation of Oxycodone by High Performance Liquid Chromatography

Scope

Samples containing oxycodone hydrochloride

Procedure:

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

Injection Solvent:

0.1N HCl

Buffer Preparation:

Accurately measure out 4000mL of Millipore water then add 10.00g of sodium hydroxide pellets. Measure 30mL of phosphoric acid. Add the phosphoric acid to the solution and do not rinse out the graduated cylinder. Using a pipette measure 8mL of hexylamine and add to the solution. Using a calibrated pH meter, measure the pH of the solution and adjust the pH to 2.5 with a sodium hydroxide solution or phosphoric acid.

Standard Solution:

Accurately weigh the Oxycodone Hydrochloride reference material in Injection Solvent so that the concentration of the oxycodone 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: HPLC equipped with photo diode array detector (or equivalent)
Column: 5µm C18, 150mm x 4.60mm
Column Temperature: 50 °C
Injection Parameters: 1.0 µL
Injection Solvent: Buffer
Mobile Phase: 85% buffer: 15% acetonitrile. Buffer prepared as listed above. Mobile phase constituents combined by instrument.
Flow: 1.0 mL/min
Detection Wavelength: Diode array@ 280nm, 4nm BW; 550nm Ref, 80nm BW
Total Run Time: 5.0 minutes

Limitations:

N/A

Acceptance Criteria:

Selectivity: Oxycodone resolved (R ≥1.5) from each compound.
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:

0.10 – 2.00 mg/mL Oxycodone

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