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Gas chromatography calibration procedure

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Calibration is a vital step in ensuring the accuracy and reliability of gas chromatography (GC) results. Here's a step-by-step guide on how to calibrate the carrier gas flow, column oven temperature, and detector calibration. Millimeter QUALITY CONTROL LABORATORY CALIBRATION CHECK REPORT OF GAS CHROMATOGRAPH CALIBRATION DATE: N/A LAST CALIBRATION DONE: N/A NEXT DUE FOR CALIBRATION: N/A INSTRUMENT DETAILS: Instrument Name: Gas Chromatograph Inj. Method: Manual Injection G.C. Parameters: - Column: DB-5 Capillary (0.32mm x 30m x 0.25micron) - Range: 2 - Oven Temp.: 300C - Split Ratio: 1:20 - Detector Temp.: 1800C - Nitrogen: 100 kPa - Inj. Temp.: 1400C - Carrier: 25Kpa (Helium) - Inj. Volume: 1.0µl - Hydrogen: 55Kpa - Air: 85 Kpa Preparation of internal Standard: - 25 ml Ethanol in 1000 ml with water Test Preparation: - Methanol in different ratios with Internal Std. - Ratio 1.0 (2 samples) - Ratio 2.0 (2 samples) - Ratio 3.0 (2 samples) - Ratio 4.0 (2 samples) - Ratio 5.0 (2 samples) Correlation Coefficient: N/A NAME SIGN DATE NAME SIGN DATE Approaches to gas chromatography calibration differ based on specific analytical goals and experiment setups. These can be categorized as external calibration, internal standard calibration, or standard addition. Before beginning the process, ensure that the GC instrument is turned on and the carrier gas cylinders are opened or run your gas mixer if using one. Adjust the pressure and check the carrier gas flow rate against setpoint and observed flows at different rates. Validate the gas flow rates against pre-defined acceptance criteria. Begin running dilutions of the compound/s of interest and plot response times versus concentration. Construct calibration curves for each analyte of interest using precise calibration standards. This article serves as a brief guide on how to conduct precise gas chromatography calibration. We touched on the use of either premixed cylinders or bespoke gas mixers for validating flow rates, and there are various considerations to make about which solution is more valuable to you. To find out which solution suits your GC calibration needs best, contact a member of the Environics team today. SOP covers: * Calibration of Gas Chromatography * Safety And Precautions * Gas Flow Rate Calibration * Precision Of Injector And Linearity Of Detector Response * Calibration Of Head Space * Calibration For Syringe To lay down a procedure for calibration of Gas Chromatography (Head Space GC-HS-10). This SOP is applicable for operation and calibration of Gas Chromatography in the Quality Control Laboratory. Firstly, preheat the oven to a temperature that is 5°C per minute higher than its maximum capacity, but not exceeding it by more than 10°C, and maintain this temperature for 30 minutes. Be extremely careful when handling the column, especially if it's capillary. Do not connect or disconnect the column when the oven temperature exceeds ambient. Ensure that all solvents and reagents used to prepare solutions are of high purity (spectroscopic grade, GC grade, or HPLC grade). After each use, rinse syringes thoroughly to avoid carryover contamination. If using capillary columns, replace ferrules when they become too flat to form a proper seal. Next, secure the column ends with adapters to the injection and detector ports inside the oven. Check for any leaks by applying IPA:water (1:1) mixture to the joints. Turn on the nitrogen, hydrogen, and air gas cylinders and their respective knobs located at the back of the instrument. Recognize the color-coded tubing - red for hydrogen, yellow for nitrogen, and blue for air. Verify that the carrier gas connections, specifically nitrogen, adhere to the specifications in section 5.4. Perform calibration using methanol, isopropyl alcohol, methylene chloride, ethyl acetate, and dimethyl formamide on a half-yearly basis ± 5 days. Recalibrate when major maintenance or changes are made to significant parts or if parameters of calibration are replaced. For gas flow rate calibration: - Set the required flow rate for carrier gas (nitrogen) at 2.0 ml/min, then 5.0 ml/min, and finally 10.0 ml/min. - Check fittings for leaks and maintain each flow rate for 5 minutes to equilibrate before observing on a digital flow meter. Repeat similar procedures for air and hydrogen gas flows, adjusting the rates accordingly. Document calibration results in a Calibration TDS. Preparation of Standard Stock Solution: Weigh approximately 0.1 g each of methanol, isopropyl alcohol, methylene chloride, and ethyl acetate into a 100-mL volumetric flask containing about 30 mL of Dimethylformamide (for gas chromatograph) or Dimethyl sulphoxide (for head space). Mix well and dilute to volume with the solvent. Calibration Levels: 1. Level 5 (250 ppm): Dilute 25 mL of standard stock solution to 100 mL. 2. Level 4 (200 ppm): Dilute 20 mL of standard stock solution to 100 mL. 3. Level 3 (150 ppm): Dilute 15 mL of standard stock solution to 100 mL. 4. Level 2 (100 ppm): Dilute 10 mL of standard stock solution to 100 mL. 5. Level 1 (50 ppm): Dilute 5 mL of standard stock solution to 100 mL. Precision Preparation: Transfer 100 ppm standard solution into a clean, dry vial. Inject 1µL of this standard solution in six replicates. The acceptance criteria for precision is that the %RSD of area should not exceed 15% and the %RSD of retention time should be less than 2%. Linearity Preparation: Inject 1.0, 2.0, 3.0, 4.0, and 5.0 levels of standard solution in three replicates respectively. The acceptance criteria for linearity is that the %RSD of retention time should not exceed 2% and the %RSD of area should not exceed 15%. The correlation coefficient 'r' obtained from the linearity level for each solvent should be at least 0.99. Calibration of Head Space: Chromatographic Conditions: Equipment: Gas chromatograph GC-2010 PLUS & Headspace HT3 or equivalent Column: DB-624, 30 m x 0.53 mm x 3.0 µm or equivalent Carries gas: Nitrogen Column pressure/Flow rate: 3.4 psi / 5 mL/min Detector: FID Injector temperature: 150°C Injection mode Split ratio: 1 : 5 Detector temperature/Signal acquire/H2 flow/Zero air flow: 240°C, 40 msec, 40 mL/min, and 400 mL/min respectively Headspace Conditions: Constant heat time: On GC cycle time: 35.0 min Valve oven temperature: 105°C Transfer line temperature: 110°C Platen/sample temperature: 110°C Platen temperature equilibration time: 30 min Mixer: On Mixing level: Level 5 Mixing time: 1 min Mixer stabilize time: 0.5 min Pressurize: 10 psi Pressurize time: 1.0 min Pressurize equilibrium time: 0.5 min Loop fill pressure: 5 psi Loop fill time: 1 min Inject time: 1 min Preparation of Standard Stock Solution for Head Space: Weigh approximately 0.1 g each of methanol, isopropyl alcohol, methylene chloride, and ethyl acetate into a 100-mL volumetric flask containing about 30 mL of Dimethyl sulphoxide. Mix well and dilute to volume with the solvent. The acceptance criteria for precision is that the %RSD of area should not exceed 15% and the %RSD of retention time should be less than 2%. Given article text here To perform detector linearity and precision, follow these steps: 1. Prepare individual vials with 2 ml each of 50 ppm, 100 ppm, 150 ppm, 200 ppm, and 250 ppm solutions in triplicate. 2. Seal the vials using Teflon-coated septum and metallic caps with a crimper. 3. Inject blank samples (using the same DMSO) and the prepared solutions into the gas chromatograph under headspace conditions. 4. Record the chromatogram, peak area response, and retention time for each injection. 5. Plot a graph showing the concentration of each solvent on the x-axis and average area on the y-axis. Acceptance criteria: * % RSD of area: not more than 15.0% * % RSD of retention time: less than 2.0% * Theoretical plate: not less than 2,000 for the first replicate * Resolution: not less than 1.0 for the first replicate * Tailing factor: not more than 2.00 for the first replicate * Correlation coefficient (r): not less than 0.99 To perform static carryover analysis: 1. Use a 250 ppm standard solution. 2. Sequence of injections: three blanks, one standard solution, and two blanks. 3. Calculate the percentage of carryover using the average area of the last two blank injections. Acceptance criteria: not more than 1.0% To perform syringe calibration: 1. Sonicate a methanol-filled syringe. 2. Wash the syringe with purified water, then rinse with methanol and dry it in an oven. 3. Fill an empty 5 ml volumetric flask with purified water using the calibrated syringe. 4. Calculate the net volume by weighing the collected volume. Acceptance criteria: the net volume should be within ± 1.0% of the actual volume. To perform maintenance: 1. When the instrument is under repair, place a tag stating "OUT OF ORDER". 2. Inform the Head QC/Designee. 3. The Head QC/Designee will inform the instrument manufacturer/service person.