Improvement of ASTM D-5769 by GCMS High Concentration Tuning and Automated Data Processing

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Method Challenges

- Quadratic calibration is implemented for these high concentration compounds
- MS detector saturates easily at high concentrations due to limited fragmentation
- Good linearity is difficult to obtain

Method ASTM D-5769 is a standard GCMS method to determine aromatics in finished motor gasoline or ethanol blends. However, the intrinsic high sensitivity of mass spectrometry usually gives rise to a constant linear velocity even if the columns used are different with constant linear velocity.

Optimum separation in one mouse-click
Transfer methods between GC's easily
Three tuning modes to select from

Eco-friendly
- Maintenance downtime reduced
- Analysis cycle shortened significantly
- Increased sensitivity
- Ultra-fast data acquisition speed for comprehensive 2-dimensional gas chromatography

Introduction

Method ASTM D-5769 is a standard GCMS method to determine aromatics in finished motor gasoline or ethanol blends. The intrinsic high sensitivity of mass spectrometry usually gives rise to a constant linear velocity even if the columns used are different with constant linear velocity. Therefore, it is difficult to meet the requirements. GCMS-QP2010 Ultra, a new single transmission quadruple GCMS, allows for tuning the instrument to a 'high concentration' mode that is suitable for ASTM D-5769 type of analysis. The addition of a new feature: high concentration tuning, greatly enhances method robustness and efficiency by expanding calibration linearity.

GCMS-QP2010 Ultra New Features

- Enhanced Performance
  - Maximum scan speed of 50,000 u/sec (100Hz)
  - ASPI function for greater sensitivity in fast scan
  - Ultra fast data acquisition speed for comprehensive 2-dimensional gas chromatography
  - Increased sensitivity
- Increased Productivity
  - Analyze cycle shortened significantly
  - Maintenance downtime reduced
- Eco-Friendly
  - 36% reduction of power consumption in analysis standby mode
  - 36% reduction of CO2 factory emission

Experimental – GCMS-QP2010 Ultra with AOC20i/s Autosampler

- GC
  - Inj: 250.0°C
  - Column: ZB-1 60 m x 0.25 mm x 1 m
  - injection: 0.1 µL
  - Carrier gas linear velocity: 35.0 cm/sec
- MS
  - ion source temp: 285.0°C
  - detector temp: 280.0°C
  - detector voltage: -0.1 kV relative to tune (0.98kV)
  - scan range: 45.0 – 300.00
  - scan interval: 0.10sec
  - scan range: 10-100scan/sec
  - software: Shimadzu Aromatics Report Software Ver: 2.0

Calibration Curve

- Linear velocity mode: 35.0 cm/sec
- Injection Volume: 0.1 µL
- Gas saver: On (split ratio 250)
- Split ratio: 250
- Linear velocity mode: 35.0 cm/sec
- INJ: 250.00°C
- Temperature: 10.0°C/min to 250.0°C
- Transfer methods between GC's easily
- Optimum separation in one mouse-click

Repeatability

Constant Linear Velocity

- Optimum separation in one mouse-click
- Transfer methods between GC's easily

Why fast cycle time necessary?

Precision / accuracy is poor with only 4-5 scans across a GC peak.
Sufficient data sampling points per peak is important to acquire chromatographic data accurately, especially for quantitation.

Compound Table

<table>
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<tr>
<th>Calibration Curve</th>
<th>TIC Chromatogram</th>
<th>AOC20i/s Autosampler</th>
<th>Very Fast Scan / Data Acquisition</th>
<th>Flexible Tuning Modes</th>
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<td>Repeatability</td>
<td>Aromatics Report</td>
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Conclusions

- High concentration tuning mode expands calibration linearity. Good linearity has been achieved across all concentrations of all compounds; no saturated integration was observed, therefore, no gain/factor 10 was necessary.
- Implementation of Shimadzu Aromatics report software has enabled automation of data acquisition, processing, and reporting.
- Linear velocity mode simplifies parameter settings and provides optimized separation.
- Fast scan rate (10 spectra/sec vs. 3 spectra/sec) improves both accuracy and precision.

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