A Benchtop Sample Preparation Instrument: New Solutions for GC and GC/MS Applications

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Outline

• A new sample preparation instrument for GC, GC/MS, LC, and LC/MS applications

• Features

• Performance
  • Sample dilution
  • ISTD addition
  • Derivatization
  • Calibration curve standard preparation

• Conclusions
Agilent’s Sample Preparation Instrument

Features

• Dilution / Aliquoting
• Liquid Addition (standards, reagents, etc.)
• Heating (derivatization, digestion, etc.)
• Liquid/liquid extraction
• Sample mixing – vortex
• Sample tray heating
• Sample tray cooling
• Software based on Easy Sample Prep
  • Drag and drop method editor
Easy Sample Prep – Icon Based Programming

Sample Prep Method Editor

- Drag and drop programming
- Using Add, Mix, Heat and Wait steps to create a custom sample prep program
- Textual display of sample prep steps
Easy Sample Prep – Resource Editor

- Specify sample prep resources on tray
- Name resources, specify usage type
- Use colors to identify resources
- Provide default syringe parameters for resources
- Keeps track of resources based on volume allotted or number of uses
Sample Prep Programming Flexibility

- Examples of simple liquid manipulation
  - Reagent additions
  - Aliquoting / Dilutions
  - Mixing
  - Heating
  - L/L Extraction

Dilution  Internal Standard Addition  Small-Volume Sampling  Derivatization  Heating/Mixing Bar Code  In-vial Extraction
Reproducible and Accurate Dilutions and ISTD Additions – For GC

Example 1

- Add 50 μL isoctane to empty vial
- Add 50 μL standard solution
- Add 0.5 μL ISTD

Dispensing 50 μL gives ~0.5% RSD for 10 samples by weight
  - Accurate within 1%

Dispensing 0.5 μL gives ~2% RSD for the 10 samples
  - Does not affect standard accuracy
Reproducible Sample Dilutions and ISTD Additions – For LC

Example 2

- Add 187.5 μL acetonitrile to empty vial
- Add 62.5 μL Diuron standard
- Add 125 μL p-terphenyl

- Dispensing precision is ~0.5% for 10 samples measured gravimetrically
  - Accurate within 2%
Simulated Distillation Calibration Standard Dilution

Example 3

- Add 495 μL CS₂ to empty vial
- Heat SimDis sample (waxy)
- Mix SimDis sample
- Add 10 μL SimDis sample to CS₂

- 3 samples prepared
- 0.2% RSD by weight for CS₂
- Area repeatability between samples is typically < 5% RSD
Example 4

Calibration Curve Standards Diluted Linearly

- Add 100 μL iso-octane to empty vial
- Add varying amounts of stock solution
- Mix
Reproducible Calibration Curve Standards Preparation

• Preparing standards with the automated sample preparation instrument yields more reproducible results than standards prepared manually
  – Comparing 3 sets of standards
  – Manually made standards were prepared in volumetric flasks
EPA 8270 Standards Preparation

Example 5

• Add varying amounts (270-300 μL) of methylene chloride to empty vials
• Add varying amounts of stock solution (0.3-30 μL)
• Add 3 μL ISTD
• Mix
Relative Response Factor %RSDs were normalized to the manual method
- Manually prepared standards were prepared in autosampler vials
- If automated method is better than Manual → Normalized RRF RSD <1
- If manual method better than ALS → Normalized RRF RSD >1

Sample Preparation Instrument is as good as a skilled chemist when making a 7-level calibration set – results not significantly different
Faster Sample Preparation

- EPA 8270 calibration curve standards can be prepared in a third of the time using an automated procedure
- Generic standards can be prepared in half the time
  - Both achieve the same, if not better reproducibility and accuracy
Fatty Acid Derivatizations

Example 7

• Add 100 µL of BSTFA to 0.5 mL fatty acid solution
• Mix
• Heat at 70°C for 20 minutes

3 samples prepared

• Manual method RSD: 0.9%
• Automated method RSD: 0.7%

![Derivatization reactions yield the same results with less operator involvement](image-url)
Conclusions

- Samples prepared with an automated sample preparation instrument yield reproducible results
  - Results are as good, if not better than those obtained with manual methods
- Samples prepared with automated methods yield accurate data
  - Results achieve the same level of accuracy expected from manual methods
Increased Lab Productivity

- Automation of sample preparation frees lab personnel for other tasks
  - design experiments, work up data
- Improve quality of chromatographic results by providing better precision between samples
  - Less rework since autosamplers minimize human variability
- Samples take less time to make
Cost Effective Sample Preparation

- Liters of solvent can be saved per year by converting sample preparation steps to an automated method
  - Use 2 mL autosampler vials instead of larger volumetric flasks
  - Automating EPA 8270 saves 4 L of methylene chloride per analyst per year
  - Reduced exposure to hazardous chemicals
- Fewer mistakes mean more samples per day
Thank you for your attention!

Additional questions can be directed to:
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