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

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Outline

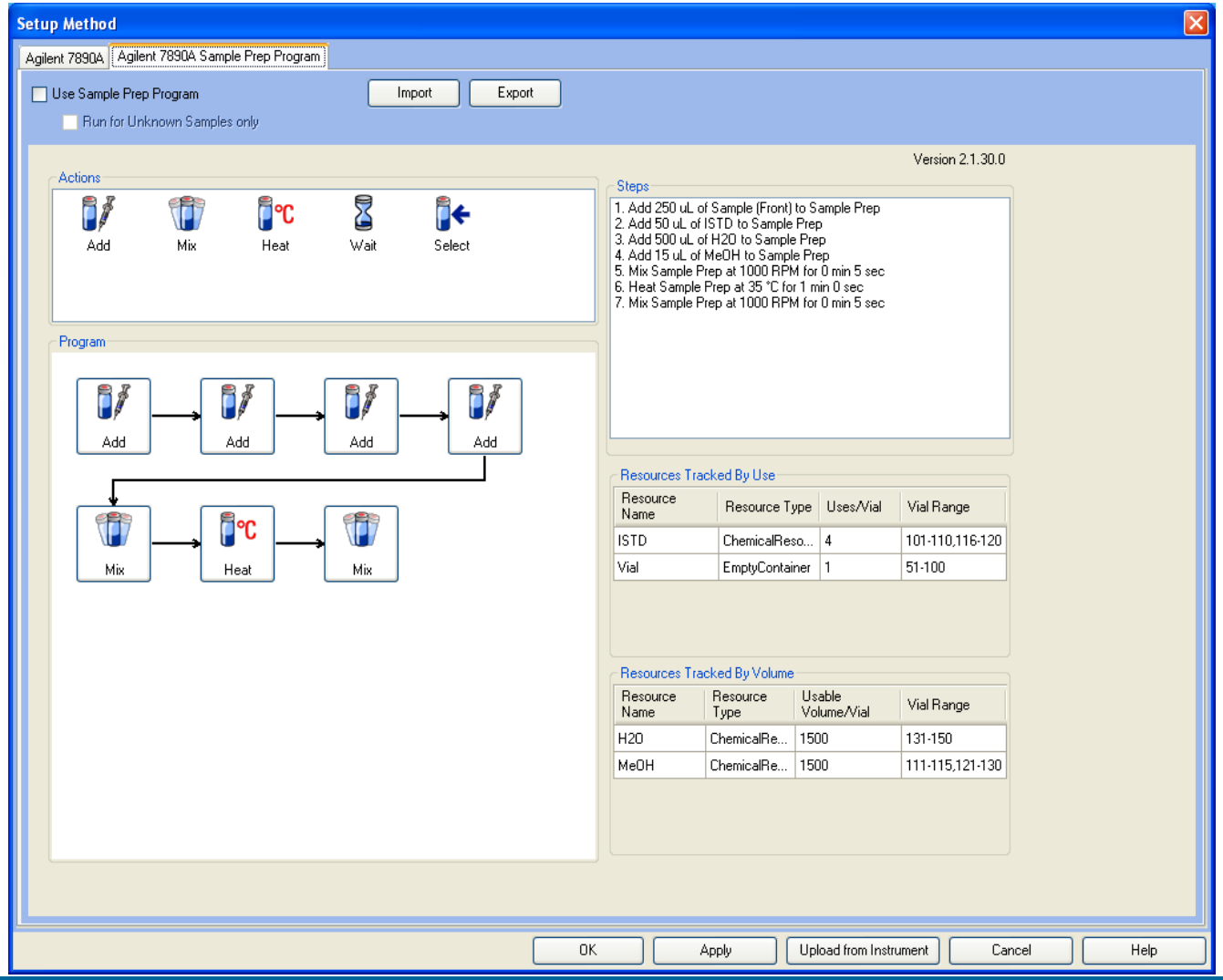
- A new sample preparation instrument for GC, GC/MS, LC, and LC/MS applications
- Features
- Performance
  - Sample dilution
  - ISTD additon
  - Derivatization
  - Calibration curve standard preparation
- Conclusions

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

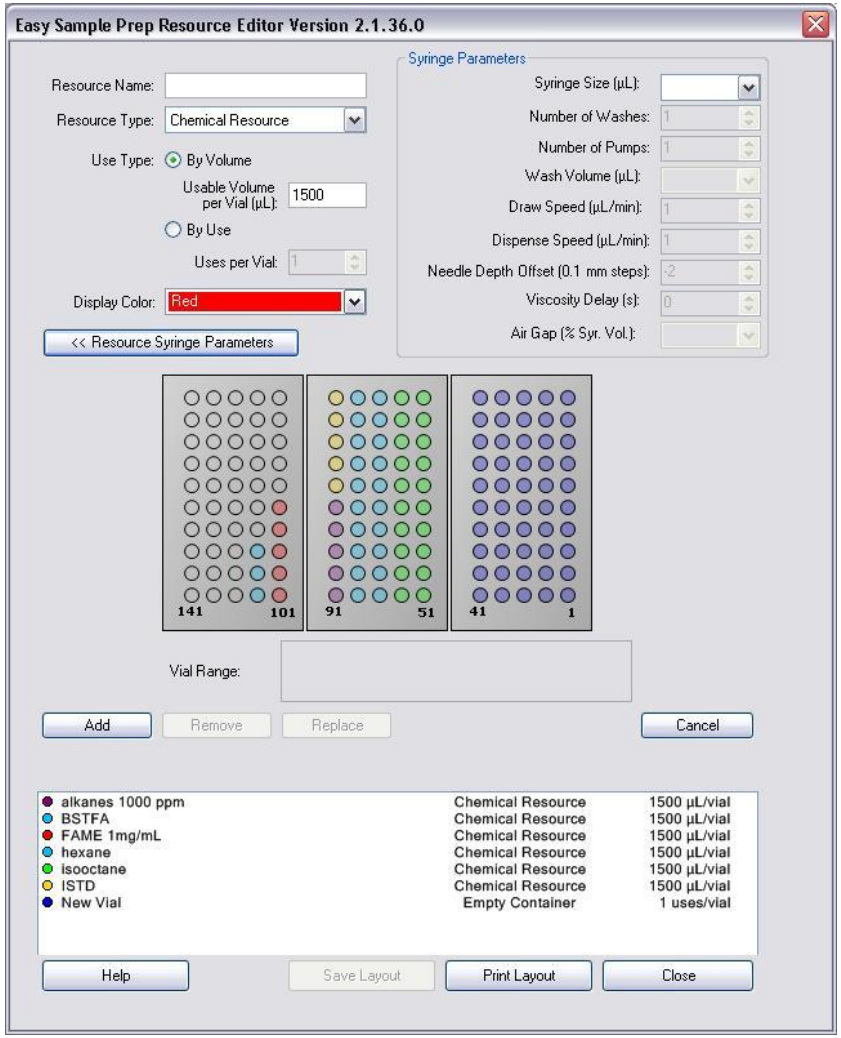
Easy Sample Prep – Icon Based Programming

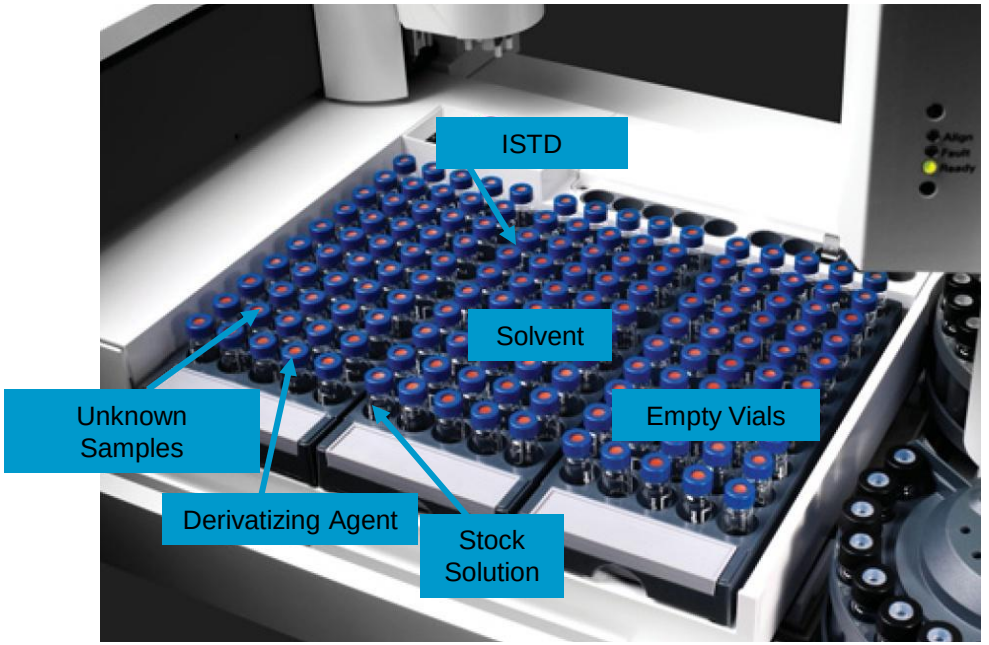


Sample Prep Method Editor

- 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





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

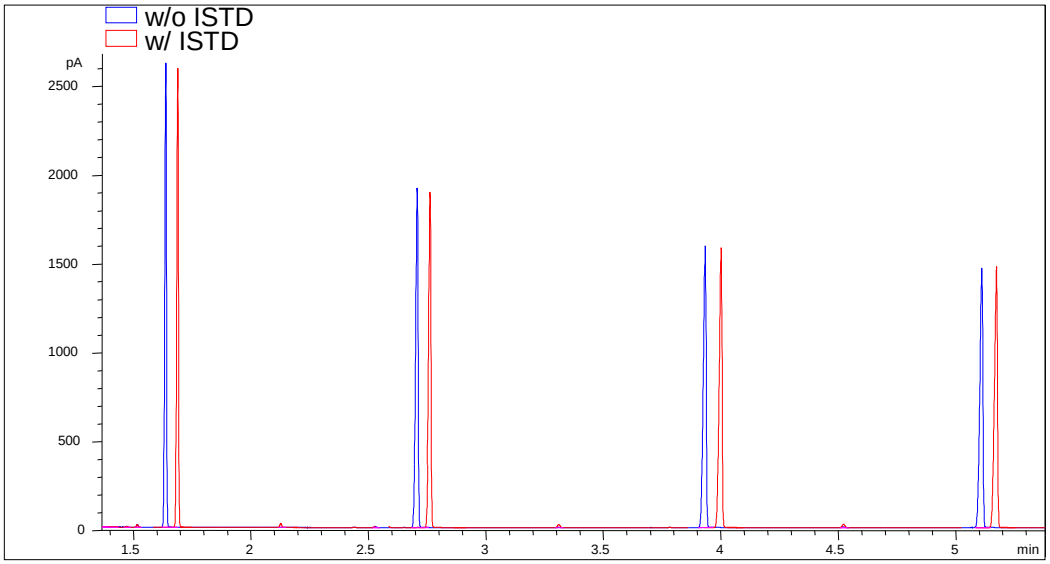
Heating/Mixing Bar Code

In-vial Extraction

Reproducible and Accurate Dilutions and ISTD Additions – For GC

Example 1

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



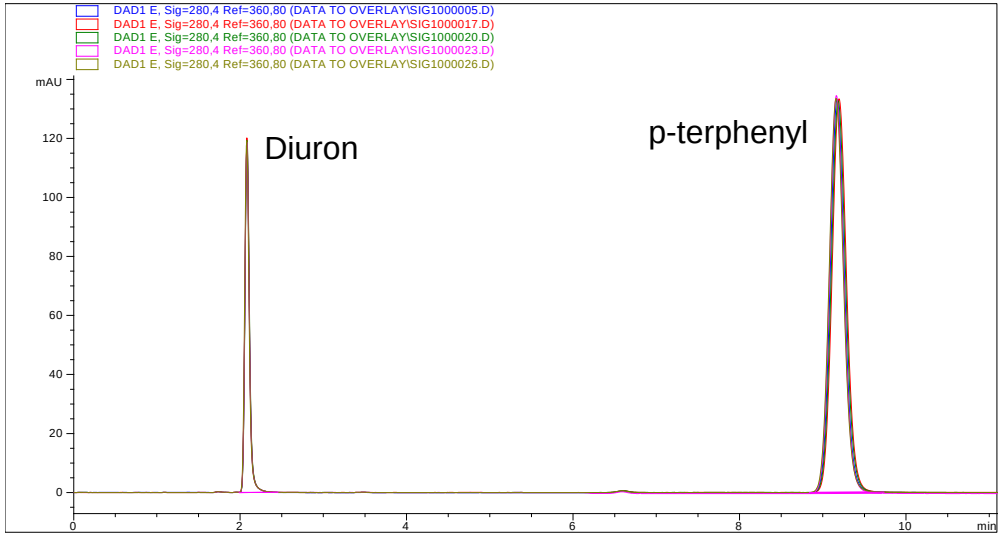
- Dispensing 50 µL gives ~0.5% RSD for 10 samples by weight
  - Accurate within 1%
- Dispensing 0.5 µL gives ~2% RSD for the 10 samples
  - Does not affect standard accuracy

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%

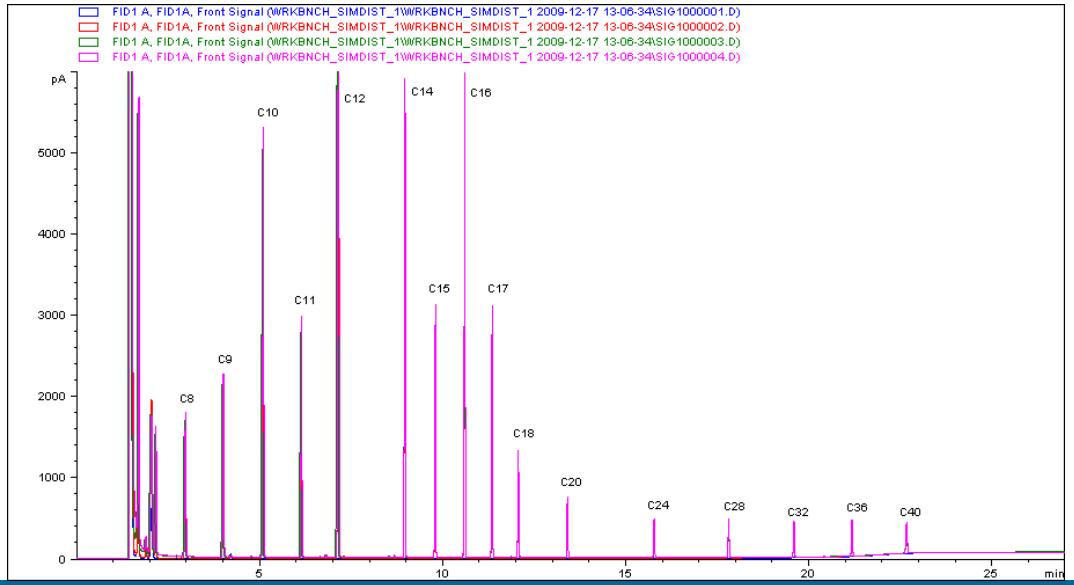


Simulated Distillation Calibration Standard Dilution

Example 3

- Add 495 µL CS_2 to empty vial
- Heat SimDis sample (waxy)
- Mix SimDis sample
- Add 10 µL SimDis sample to CS_2

- 3 samples prepared
- 0.2% RSD by weight for CS_2
- Area repeatability between samples is typically < 5% RSD

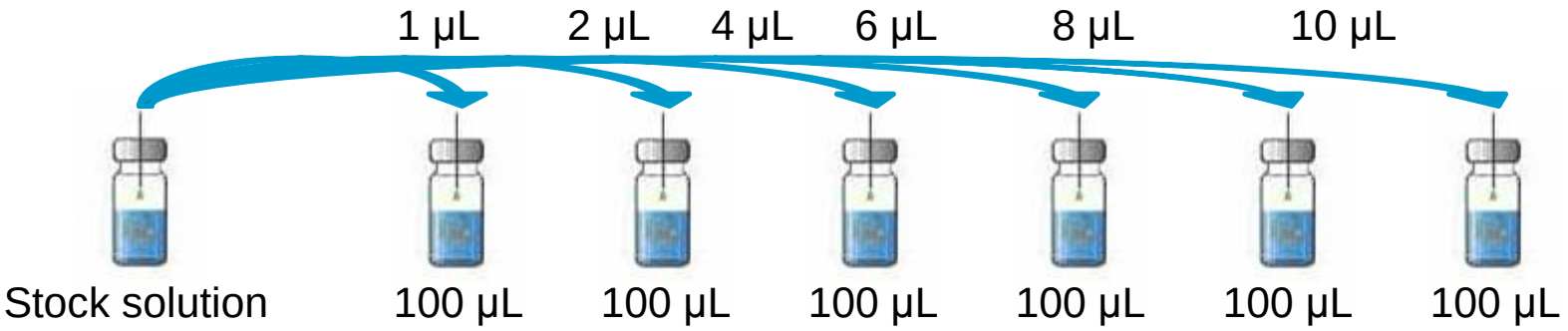


Calibration Curve Standard Preparation

Example 4

Calibration Curve Standards Diluted Linearly

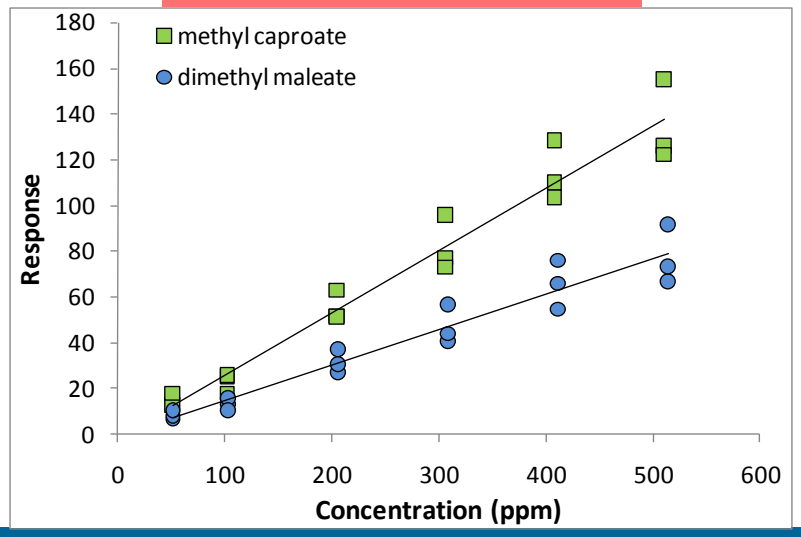
- Add 100 µL isooctane to empty vial
- Add varying amounts of stock solution
- Mix



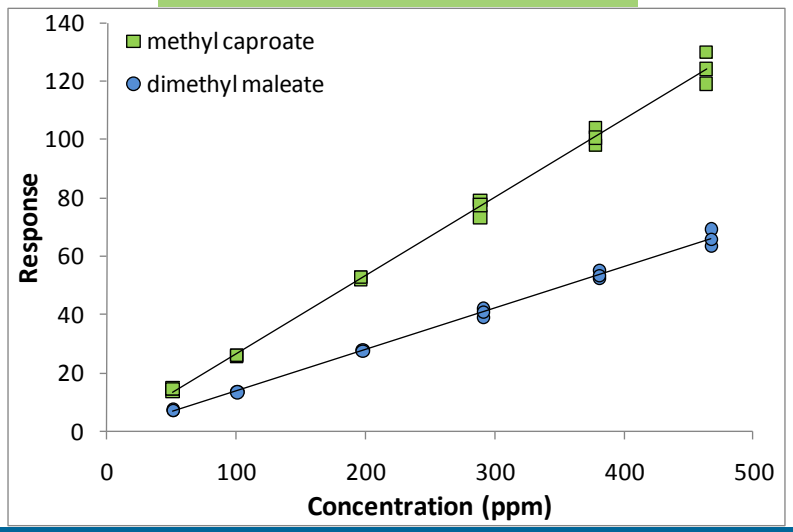
Reproducible Calibration Curve Standards Preparation

- 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

Manual



Automated

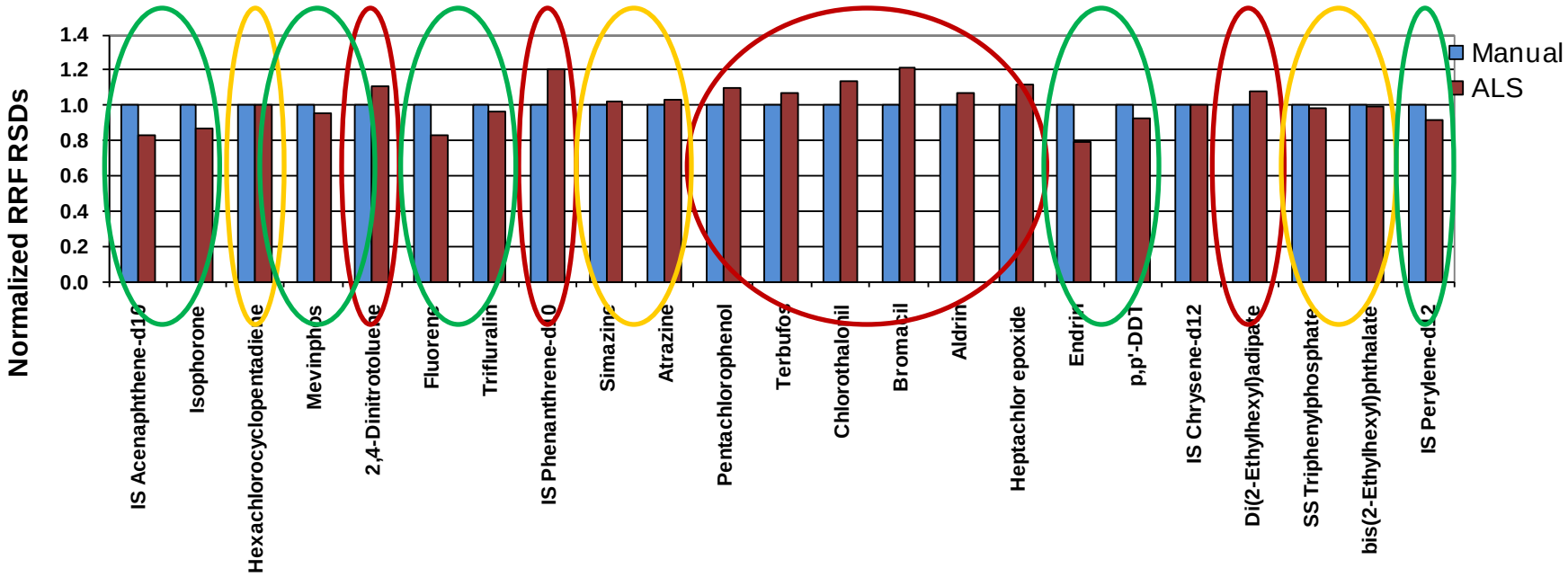


EPA 8270 Standards Preparation

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

EPA 8270 Calibration Curve Standards

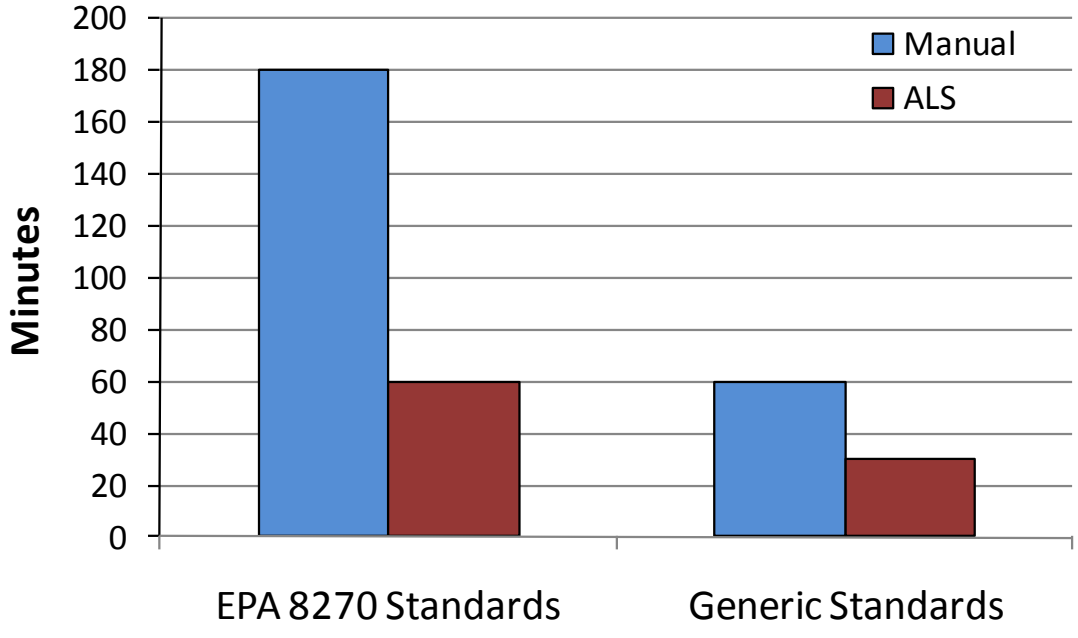


- Relative Response Factor %RSDs were normalized to the manual method
  - Manually prepared standards were prepared in autosampler vials
- If automated method is better than Manual → Normalized RRF RSD <1
- If manual method better than ALS → Normalized RRF RSD >1

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

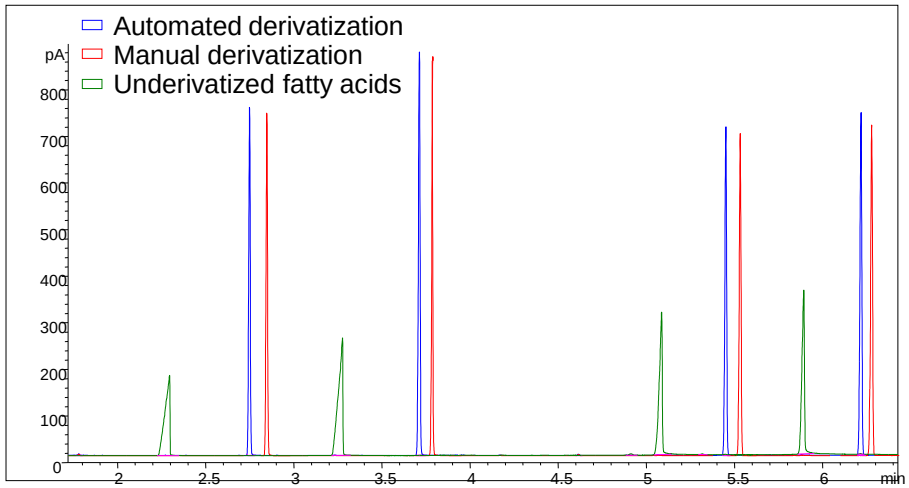
Example 7

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

Analyte	Ratio-Manual	Ratio-Automated
Capric acid	0.92	0.92
Capric acid	1.2	1.2
Myristic acid	1.0	1.0
Palmitic acid	1.1	1.1

<u>3 samples prepared</u>

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



Derivatization reactions yield the same results with less operator involvement

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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