

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

Rebecca Veeneman
Riva ISCC
Agilent User Meeting

June 1, 2010



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

Setup Method

Agilent 7890A | Agilent 7890A Sample Prep Program

☐ Use Sample Prep Program Import Export

☐ Run for Unknown Samples only

Version 2.1.30.0

Actions

Add Mix Heat Wait Select

Program

1. Add 250 uL of Sample (Front) to Sample Prep
2. Add 50 uL of ISTD to Sample Prep
3. Add 500 uL of H2O to Sample Prep
4. Add 15 uL of MeOH to Sample Prep
5. Mix Sample Prep at 1000 RPM for 0 min 5 sec
6. Heat Sample Prep at 35 °C for 1 min 0 sec
7. Mix Sample Prep at 1000 RPM for 0 min 5 sec

Resources Tracked By Use

Resource Name	Resource Type	Uses/Vial	Vial Range
ISTD	ChemicalReso...	4	101-110,116-120
Vial	EmptyContainer	1	51-100

Resources Tracked By Volume

Resource Name	Resource Type	Usable Volume/Vial	Vial Range
H2O	ChemicalRe...	1500	131-150
MeOH	ChemicalRe...	1500	111-115,121-130

OK Apply Upload from Instrument Cancel Help

Easy Sample Prep – Resource Editor

Easy Sample Prep Resource Editor Version 2.1.36.0

Resource Name:

Resource Type:

Use Type: ☒ By Volume ☐ By Use

Usable Volume per Vial (μL):

Uses per Vial:

Display Color:

<< Resource Syringe Parameters

Syringe Parameters

Syringe Size (μL):

Number of Washes:

Number of Pumps:

Wash Volume (μL):

Draw Speed (μL/min):

Dispense Speed (μL/min):

Needle Depth Offset (0.1 mm steps):

Viscosity Delay (s):

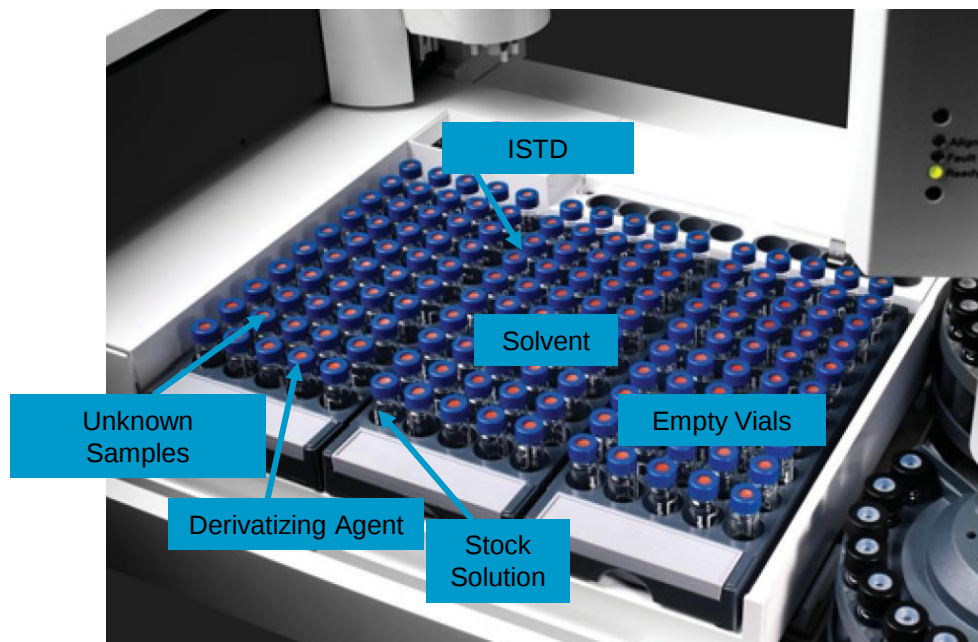
Air Gap (% Syr. Vol.):

Vial Range:

Add Remove Replace Cancel

alkanes 1000 ppm	Chemical Resource	1500 μL/vial
BSTFA	Chemical Resource	1500 μL/vial
FAME 1mg/mL	Chemical Resource	1500 μL/vial
hexane	Chemical Resource	1500 μL/vial
isooctane	Chemical Resource	1500 μL/vial
ISTD	Chemical Resource	1500 μL/vial
New Vial	Empty Container	1 uses/vial

Help Save Layout Print Layout Close



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*



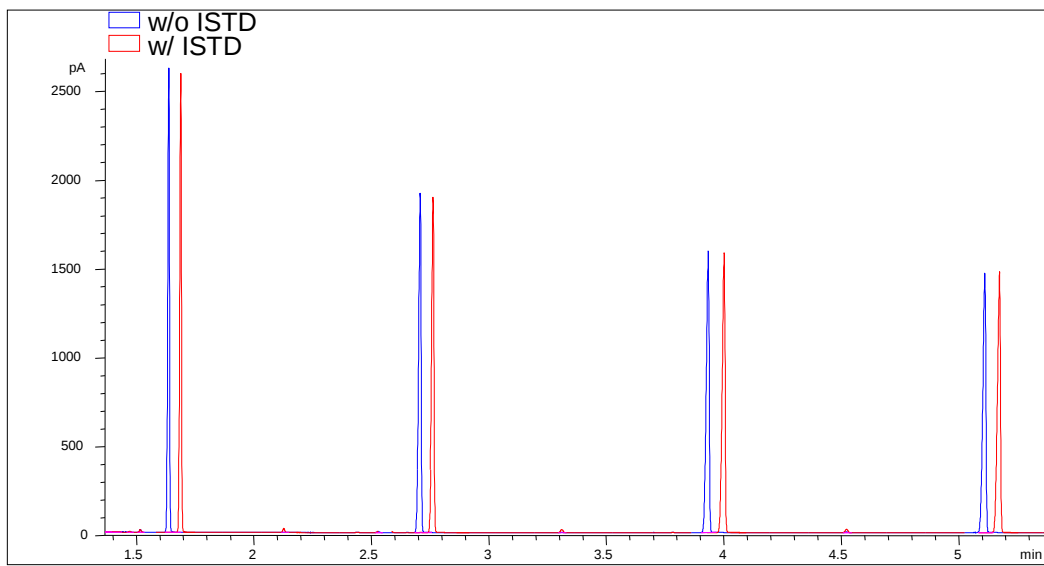
*In-vial
Extraction*



Reproducible and Accurate Dilutions and ISTD Additions – For GC

Example 1

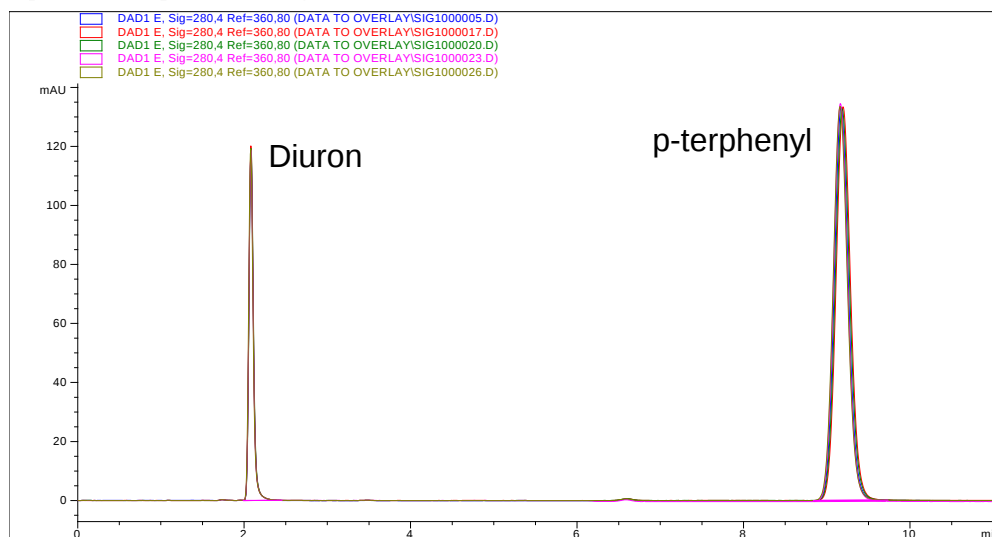
- Add 50 μL isooctane to empty vial
- Add 50 μL standard solution
- Add 0.5 μL ISTD
- Dispensing 50 μL gives ~0.5% RSD for 10 samples by weight
 - Accurate within 1%
- Dispensing 0.5 μL gives ~2% RSD for the 10 samples
 - Does not affect standard accuracy



Reproducible Sample Dilutions and ISTD Additions – For LC

Example 2

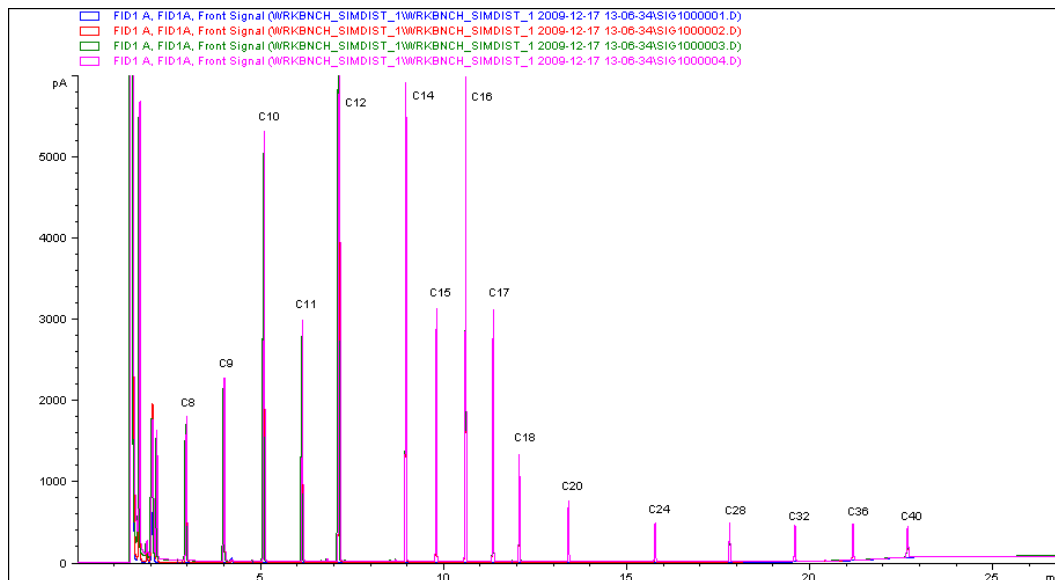
- Add 187.5 μ L acetonitrile to empty vial
- Add 62.5 μ L Diuron standard
- Add 125 μ L p-terphenyl
- Dispensing precision is ~0.5% for 10 samples measured gravimetrically
 - Accurate within 2%



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

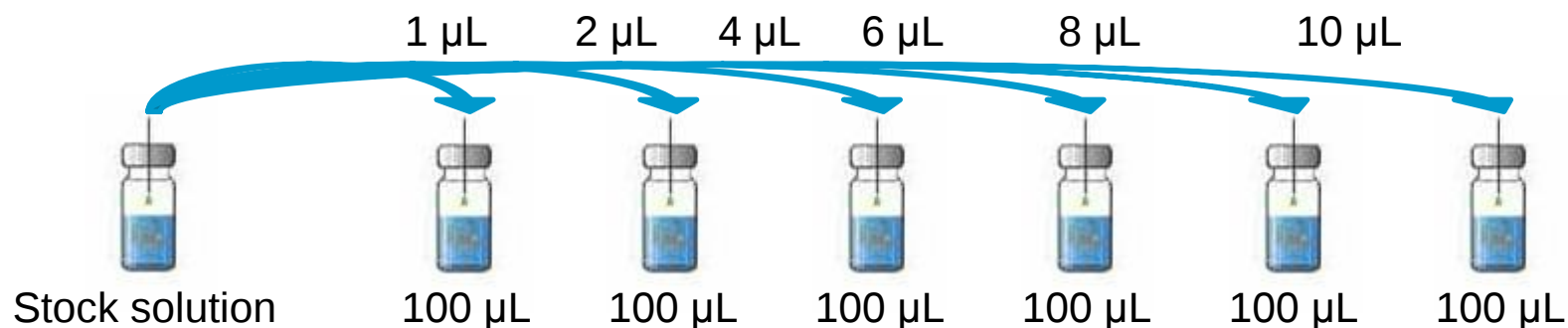


Calibration Curve Standard Preparation

Example 4

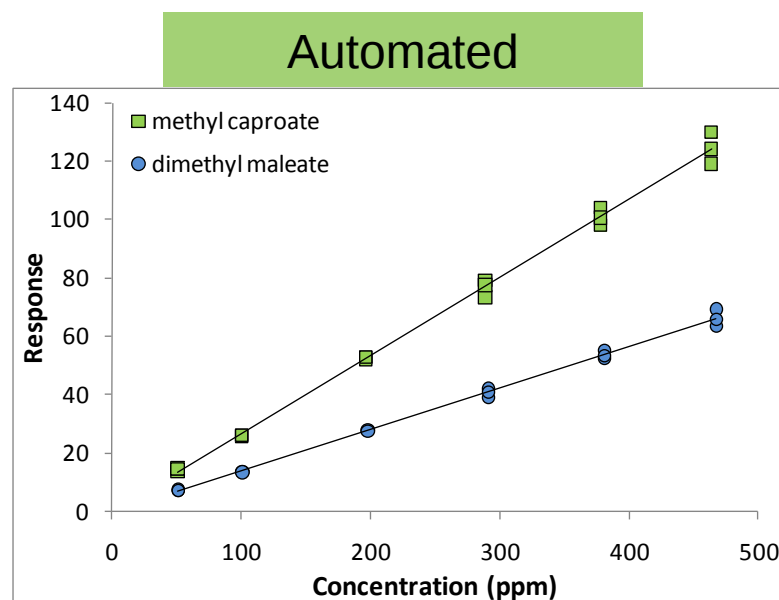
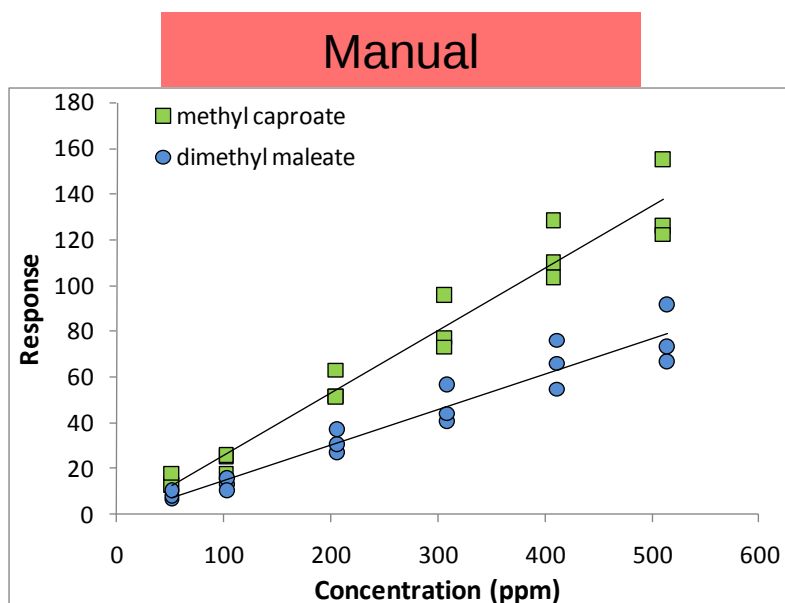
Calibration Curve Standards Diluted Linearly

- 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



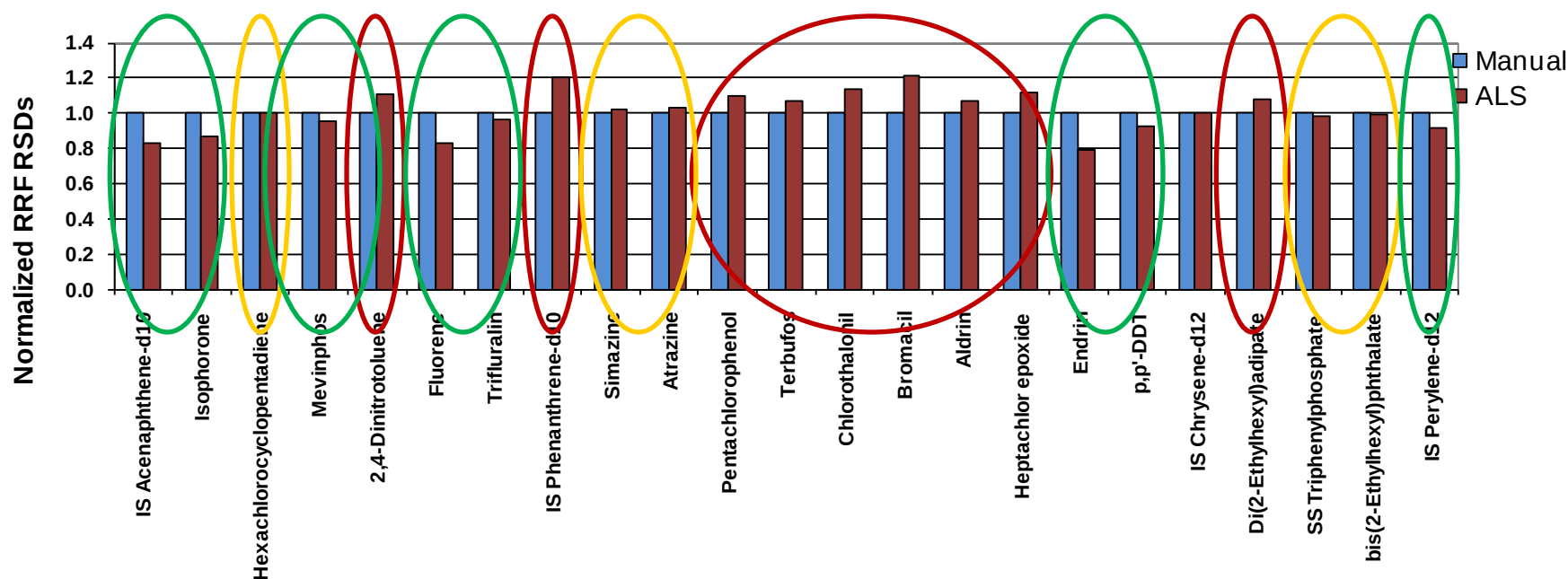
EPA 8270 Standards Preparation

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

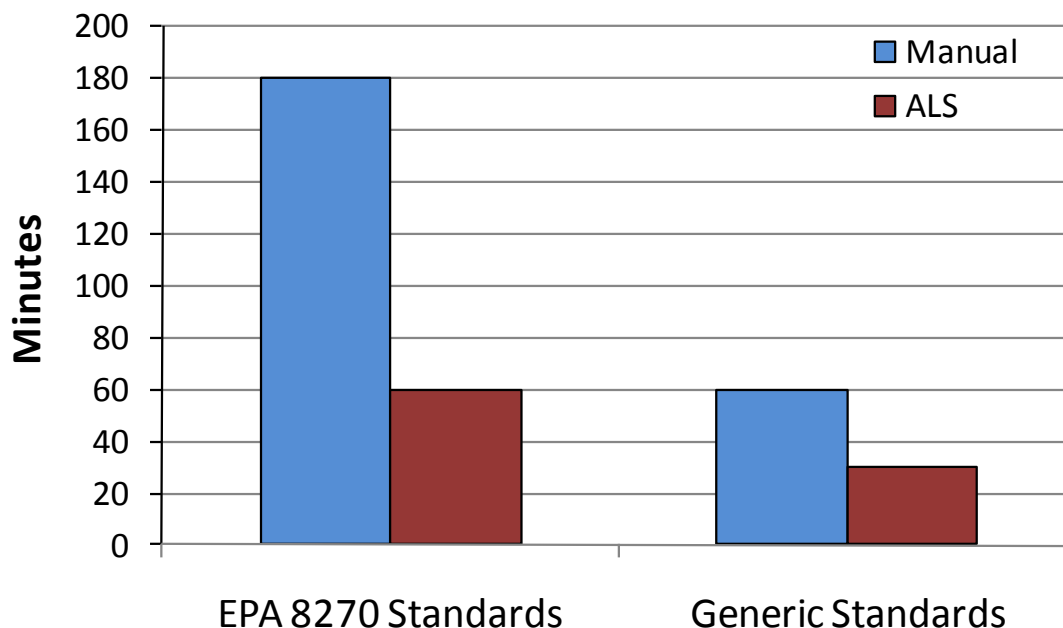


- Relative Response Factor %RSDs were normalized to the manual method
 - Manually prepared standards were prepared in autosampler vials
- If automated method is better than Manual → Normalized RRF RSD < 1
- If manual method better than ALS → Normalized RRF RSD > 1

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

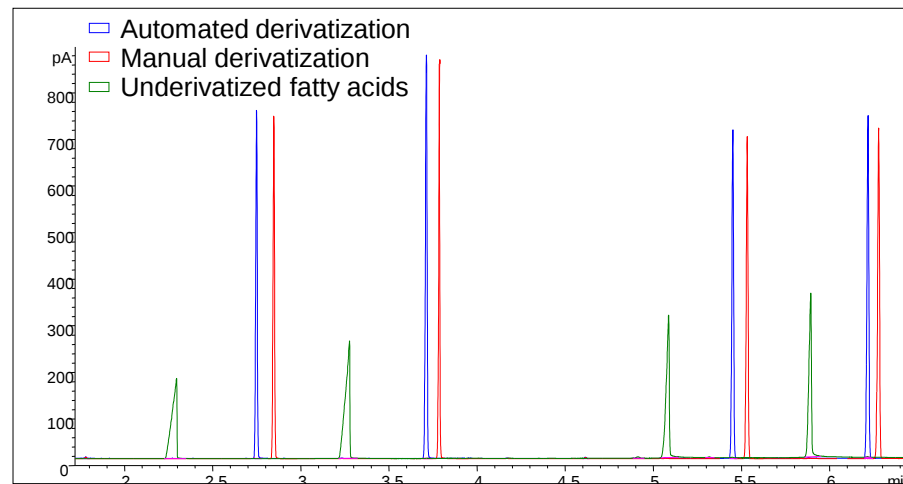
- Add 100 μL of BSTFA to 0.5 mL fatty acid solution
- Mix
- Heat at 70°C for 20 minutes

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

3 samples prepared

- 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:

Peter Mrozinski

peter_mrozinski@agilent.com

