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

			Syring	e Parameters		
lesource Name:			Gynnig	Syringe Size (µL):		
Resource Tupe:	Chemical Resource		Number of Washes:		/ashes:	1 0
riesource rype.			Number of Pumps:		Pumps:	1 6
Use Type:	 By Volume Usable Volume per Vial (μL): By Use 			Wash Volum	e (μL):	
			Draw Speed (µL/min): Dispense Speed (µL/min):		./min):	1 2
					1 5	
	Uses per Vial: 📋	\$	Ne	edle Depth Offset (0.1 mm	steps):	-2 0
Display Color:	Red			Viscosity Del	ay (s):	0 0
(C Deserves 1	Curiners Decementary		Air Gap (% Syr. Vol.):		Vol.):	v
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	Vial Range:					
Add	- Pemeuro	Poplaga	3			Canaal
Adu	L nemove	neplace				Caricei
alkanes 1000	opm			Chemical Resource	1	500 µL/vial
BSTFA EAME 1mg/ml				Chemical Resource	1	500 µL/vial
hexane				Chemical Resource	i	500 µL/vial
isooctane				Chemical Resource	1	500 µL/vial
New Vial				Empty Container	1	1 uses/vial
				and and the state of the second s		
					68	
Hale		Couplance		Print Lougut		Class



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

- 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

- 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

- 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





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

- 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 \rightarrow Normalized RRF RSD <1
- If manual method better than ALS \rightarrow 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:

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