

#### Forensic Blood Alcohol Determination with the Intuvo 9000 GC

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# The GC Usability Gap

**Usability lags features and performance** 



- Over the years, GC features and performance have matured and largely serve today's needs
- Usability has lagged and does not meet today's expectations, especially as expertise more scarce



#### **A Time for Change**



#### **Shifting Demographics**

Seasoned operators retiring; highly skilled replacements harder to find GC troubleshooting skills not necessarily located at point of use Operators assuming multiple responsibilities

#### **Challenging Economics**

- Budgets squeezed, expectations increased
- Capital purchase decisions made on business NOT technical basis





## **The GC Community Voice**

We listened and responded



#### **Improve the User Experience**

- Install and setup
- Use and maintain
- Make GC more practical for today's busy forensic lab enterprise

# **Prepare for Next Generation of Users**

Its not only about better analytical performance – its about better lab outcomes





# Make GC Easier



#### **Innovating a New Path to GC Productivity** A whole new way to GC

- Easier
- Faster
- Smaller
- Smarter
- Greener





#### **Innovating a New Path to GC Productivity** A whole new way to GC























## **Flexible Compatible Design**

**Configurable to any application** 



- SSL, MMI, GSV, LSV inlets
- FID, TCD, ECD, NPD, FPD, NCD, SCD detectors
- SQ and TQ mass spectrometers
- Headspace, thermal desorption, purge and trap samplers
- 16-, 50-, 150-position auto-injectors and trays
- Software: OpenLAB and MassHunter



## Little Falls Delaware Center of Excellence (COE)





## **Innovating the GC Flow Path**

# Conventional flow path





## **Innovating the GC Flow Path**







## **Innovating the GC Flow Path**







## **The Intuvo Chips Installed**





## **Click-and-Run Direct Connections**

**Eliminating connection uncertainty** 



- No more ferrules
- Direct face seal connections
- Audible and tactile click lets you know connection is made



- Easier to train
- Less unplanned downtime
- Fewer batch reruns and precious samples lost



#### **Intuvo Guard Chip and No-trim Columns Running more, maintaining less**



- Simple disposable design
- No column trimming
- Retention time reproducibility

- Less maintenance time
- Less recalibration and requalification
- Less unplanned downtime





## **Install The Guard Chip**

Replaces Gold Seal ~

Acts as a Guard Column -



Use two wrenches \_ for MMI **Use Torque Screwdriver** 



Agilent

## **Install The Column Gasket**

#### Install a new Gasket...





No hole. ≤ 350 °C



## **Install The Column**

- Place the column into position
- Insert the Smart ID in the lower Column 1 Connector
- Use the Torque screwdriver to tighten the "Click and Run" connectors
- Secure the Column Clamps









#### **Intuvo Guard Chip and No-trim Columns** No retention time shifts after maintenance





## **Autonomous Leak Checking**

**Avoiding unplanned downtime** 



Microfluidic-enabled 6<sup>th</sup> generation EPC modules allow hands-free leak checking to confirm and document leak-free operation autonomously.



System status





- System status
- Real-time chromatograms





- System status
- Real-time chromatograms
- Step-by-step user maintenance and troubleshooting





- System status
- Real-time chromatograms
- Step-by-step user maintenance and troubleshooting
- Finding parts fast





#### **Internet of Things A full suite of customer support**



- Intuvo serves up a web homepage
- User help for Maintenance, troubleshooting and diagnostics
- Accessible through any approved PC or mobile device securely on internal network



#### **Agilent CrossLab Services for Intuvo A streamlined service experience**

Senerated: Fri Jul 8 15	:46:04 2016					
System Information						
Hostname:	pfeifer2 A.00.01.094-201606212017		Serial Number: US0000002			
Firmware Version:						
System Configu	ration					
Injector:	G4513A		Serial Number:	CN10040090	Firmware:	A.10.08
Inlet:	SS		Serial Number:	SSL_Q000FD		
Detector 1:	FID		Serial Number:	3456101310315		
Headspace sampler:	[if present]		Mass Spectrometer:	[if present]		
Bus Type	D1_D2_SPLIT_1	10_TO_1 ( s	Carrier Board Type:	рр		
Bus Type Active Instrume State Date / Time t would be nice to be a	D1_D2_SPLIT_1	s Description me addition	Carrier Board Type:	рр		
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9000 GC Health Report

#### tion and familiarization

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## **BAC Dual FID configuration**







#### **Blood Alcohol Concentration Forensic Application Requirements**

Determination of blood alcohol concentration requires rigorous control.

- Many forensic labs use flame ionization detection (FID) which lacks identification capabilities
- Often a second system with a column having different retentive properties is used to confirm analyte identification

Blood alcohol concentration determination and confirmation can be achieved simultaneously with the Intuvo 9000 GC.

- An Intuvo 9000 GC system was equipped with a 7697A Headspace Sampler and configured with an inlet splitter allowing dual column, dual FID analysis.
- NEW BAC UI columns (123-9334UI-INT and 123-9434UI-INT) were used which improve resolution of critical analytes





#### **Intuvo Dual Flow Path**





#### 7697 Headspace parameters

7697 Headspace Sampler	Set point	
Oven	70°C	
Loop	70°C	
Transfer line	90°C	
Vial equilibration time	7min	
Injection duration	0.5min	
Vial size	20mL	
Vial shaking	off	
Vial fill mode	Default (50mL/min to 15psi (0.1min))	
Vial fill pressure	15psi	
Loop ramp rate	30psi/min	
Loop final pressure	1.5psi	
Loop equilibration time	0.05min	





#### **Intuvo Parameters**

Intuvo 9000 GC	Set point				
Oven	40°C (6.5min)				
Split/Splitless Inlet	Split 10:1, 110°C				
DB-BAC1 UI (123-9334UI-INT) 30m x 320µm x 1.8µm	Constant pressure 21psi				
DB-BAC2 UI (1230-9434UI-INT) 30m x 320µm x 1.2µm	Controlled by column 1				
FID (Front and Back)	250°C				
H2	30mL/min				
Air	400mL/min				
N2 (makeup)	25mL/min				
Jumper chip	110°C Jump				
Bus	Default (On 200°C) Bus temp				
Front/Back Signal	20Hz set to de				





#### **Calibration Results**

#### Calibration standards were made in house

- 0.8% to 0.01%
- methanol, acetaldehyde (not quantified), acetone, ethanol, and isopropanol.
- Headspace vials were made in triplicate at each level consisting of 450 µL of internal standard (0.3% npropanol) and 50µL of standard.

Calibration curves for methanol, ethanol, acetone, and isopropanol are shown right.

- Ethanol calibration curves yield
   0.9996 or better for the dual column ensemble.
- Slope difference between the two column/detector pairs is 6.3%




# **Calibration Accuracy Verification**

- Agilent Ethanol Standards were evaluated on the Intuvo 9000 GC.
- The concentration of the ethanol standards were calculated based on the calibration curves and compared to the expected concentration.
- Pass or fail was determined with ± 6% error tolerance.

Ethanol Standard	Calculated Concentration DB-BAC1 UI	Pass/Fail	Calculated Concentration DB-BAC2 UI	Pass/Fail
20mg/dL (5190-9756)	19.8mg/dL	Pass	19.3mg/dL	Pass
50mg/dL (5190-9757)	50.0mg/dL	Pass	47.1mg/dL	Pass
80mg/dL (5190-9758)	79.3mg/dL	Pass	76.8mg/dL	Pass
100mg/dL (5190-9759)	96.7mg/dL	Pass	94.4mg/dL	Pass
150mg/dL (5190-9760)	152mg/dL	Pass	149mg/dL	Pass
200mg/dL (5190-9761)	197mg/dL	Pass	193mg/dL	Pass
300mg/dL (5190-9762)	302mg/dL	Pass	302mg/dL	Pass
400mg/dL (5190-9763)	384mg/dL	Pass	386mg/dL	Pass





# **Area Repeatability**

Area repeatability for the 80mg/dL standard as well as the Agilent Blood Alcohol Checkout mix (5190-9765) was determined for five replicate headspace vials.



Analyte	DB-BAC1 UI	DB-BAC2 UI
Ethanol 80 mg/dL standard	3.70%	2.80%
Methanol	4.10%	1.40%
Acetaldehyde	2.80%	3.00%
Ethanol	2.30%	1.10%
Isopropanol	3.30%	1.90%
T-butanol	2.80%	2.70%
Propanal	3.40%	3.00%
N-propanol	3.10%	2.10%
Acetone	3.40%	2.90%
Acetonitrile	2.30%	2.80%
2-butanol	2.00%	3.00%
Ethyl acetate	3.20%	3.10%
2-butanone	3.10%	3.00%



# **Retention Time Repeatability**

Retention time repeatability for the 80mg/dL standard and the Agilent Blood Alcohol Checkout mix (5190-9765) was determined for five replicate headspace vials.



Analyte	DB-BAC1 UI	DB-BAC2 UI
Ethanol 80 mg/dL standard	0.04%	0.10%
Methanol	0.01%	0.02%
Acetaldehyde	0.01%	0.02%
Ethanol	0.02%	0.05%
Isopropanol	0.02%	0.04%
T-butanol	0.03%	0.04%
Propanal	0.01%	0.02%
N-propanol	0.03%	0.04%
Acetone	0.02%	0.03%
Acetonitrile	0.02%	0.03%
2-butanol	0.04%	0.04%
Ethyl acetate	0.02%	0.03%
2-butanone	0.02%	0.03%





# **DB-BAC1 UI Blood Alcohol Checkout Mix**

New columns resolve t-butanol and n-propanol from forensic analytes of interest





# **DB-BAC2 UI Blood Alcohol Checkout Mix**

Elution order changes allow confirmation of forensic analyte identification





# Conclusions

- Forensic Analysis of blood alcohol can be easily accomplished with Intuvo
  - Inlet splitting capability enables analysis and confirmation in a single run on two columns of different phases
  - Can be configured as an Agilent Analyzer as well
    - Includes a factory method and report template
- New blood alcohol columns (DB-BAC 1 UI and DB-BAC 2 UI) yield excellent resolution of all forensic analytes of interest in the sample
- Excellent linearity, retention time repeatability, and area repeatability were achieved







# 5977B/9000GC Intuvo

Bulk Drug Analysis

#### **Kirk E. Lokits, Ph.D GCMS Applications Chemist**



December 5, 2017

# Intuvo Parameter Optimization

- > Starting point for new temperature zones (Guard Chip, Chip Buss)
- How inert is the flow path
- > Semi-fast GC method for sample throughput
- General conditions (inlet temp, flow, column type)
- > Does atune.u and stune.u respond similarly with Intuvo
- > Can background/carryover be reduced via draw out diameter
- > How does source temperature affect Intuvo chromatography



# **Fast Method Parameters for Drugs on Intuvo**

Ramp Initial Temperature Ramp 1	°C/min 150	°C 100 325	Hold min 1.0 2.5	MSD Solvent Delay Acquisition Mode Scan Range	5977B Extractor Source 1.5 min Scan 40 to 500
Runtime	5.0 min			I hreshold	150
Inlet Temperature Mode Flow rate Head pressure Average velocity	Split/Spli 250 °C Split, Cor 1.2 mL/m 7.1 psi 49.63 cm/	tless hstant   hin /sec	Flow	TID Quad Temp Source Temp Transfer Line Tune	2 OFF 150 °C 320 °C 280 °C Stune.u Gain = 1.0
Split Flow Split Ratio	48 mL/mi 40:1	n		Guard Chip	250 °C Isothermal
Column	DB-5MSU 15m x 0.2	JI part 25 mm	# (122-5512UI) id x 0.25 μm film	Flow Chip Bus I	sothermal 300 °C
Liner	Split, stra 5190-229	aight 99 4	90 μL ultra inert		

Injection volume 1.0 µL

## Intuvo Guard Chip Temperature Settings

#### **Tracking Oven MCH**

#### 250 °C Isothermal MCH



# Intuvo GCMS Interface Temperature Settings





#### 5977B El Extractor Source Tune Settings Atune.u

Tune timestamp: 6/21/2017 9:48 AM (UTC-04:00) D:\MASSHUNTER\GCMS\1\5977\atune\_320.u

MS Source

MS Quad



٦	Ion Polarity	Pos	PFTBA	Open						
	Emission	34.6	Mass Gain	137						
	Electron Energy	70.0	Mass Offset	-26						
	Filament	1	Amu Gain	2533						
	Repeller	32.11	Amu Offset	137.13						
	Ion Focus	90.3	Width219	-0.018						
	Entrance Lens	20.2	DC Polarity	Pos						
	Ent Lens Offset	13.23	HED Enable	On						
	Ion Body	0.00	EM Volts	907.9						
	Post Extractor 1	0	Extractor Lens	0.00						
۰.	Post Extractor 2	0	Scan Speed	3						
	JetClean Flow	0	Averages	3						

320 Turbo Speed

150 Hi Vac

Intuvo 5977

US1711M035

100.0

N/C

#### Rel Abund Pw50 Actual m/z Abund 69.00 456,555 100.0% 0.60 218.90 386,520 84.7% 0.60 501.90 14,517 3.2% 0.58

Low High Step Speed Threshold Peaks Base Abundance Total Ion 10.00 701.00 0.10 3 100 145 69.00 441,344 1,575,380



Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	441,344	100.0%	70.00	4,944	1.1%
219.00	219.00	359,616	81.5%	220.00	15,211	4.2%
502.00	502.10	13,097	3.0%	503.10	1,247	9.5%

Air/Water Check: H20 ~0.6% N2 ~0.6% O2 ~0.2% CO2 ~0.2% N2/H20 ~98.6% Column(1) Flow: 1.20 Column(2): 0.00 ml/min Interface Temp: 280

#### Ramp Criteria:

Ion Focus maximum 90 volts using ion 502; Electron Multiplier Gain 44432.000 Repeller maximum 35 volts using ion 219; Gain Factor 0.4443

Mass Gain Values(Scan Speed): 150(3) 149(2) 162(1) 173(0) 226(FS1) 225(FS2)

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset	137.1	137.1	137.1	137.1	137.1	137.1	137.1
Entrance Lens Offset	13.2	13.2	13.2	13.2	13.2	13.2	13.2

#### Stune.u



Air/Water Check: H20 ~0.8% N2 ~0.9% O2 ~0.2% CO2 ~0.3% N2/H20 ~122.7% Column(1) Flow: 1.20 Column(2): 0.00 ml/min Interface Temp: 280

502.00

#### Ramp Criteria:

502.00

Ion Focus maximum 90 volts using ion 502; Electron Multiplier Gain 48004.858 Repeller maximum 20 volts using ion 219; Gain Factor 0.4800

Mass Gain Values(Scan Speed): 147(3) 157(2) 173(1) 208(0) 269(FS1) 339(FS2)

5,388

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset	141.3	141.3	141.3	141.3	141.3	141.3	141.3
Entrance Lens Offset	5.6	5.0	6.4	6.9	13.1	17.6	17.6
Target Abund (%)	1.0	100.0	55.0	45.0	3.5	2.5	
Actual Tune Abund (%)	0.9	100.0	56.5	45.1	3.8	2.2	

2.2%

503.00

636

11.8%

Agilent

Autotune - 5977

# **Draw Out Lens Comparison 3, 6, and 9 mm Diameters**



# **Draw Out Lens Comparison 3, 6, and 9 mm Diameters**

![](_page_49_Figure_1.jpeg)

#### Source Temperature Comparison (225, 275, 325°C) Cannabinoid Mix

![](_page_50_Figure_1.jpeg)

![](_page_50_Picture_3.jpeg)

# **Better chromatography**

![](_page_51_Figure_1.jpeg)

![](_page_51_Picture_2.jpeg)

#### Source Temperature Comparison (225, 275, 325°C) Toxicology Checkout Standard 5 ng/µL on column

![](_page_52_Figure_1.jpeg)

![](_page_52_Picture_3.jpeg)

![](_page_53_Figure_0.jpeg)

![](_page_53_Picture_2.jpeg)

# **Fire Debris Analysis with the Intuvo 9000 GC and 5977B MSD**

#### **Kirk E. Lokits, Ph.D GCMS Applications Chemist**

![](_page_54_Picture_2.jpeg)

![](_page_54_Picture_3.jpeg)

## **Method Parameters for Accelerants on Intuvo**

Ramp Initial	°C/min	°C 40	Hold min 2.0	MSD Solvent Delav	5977B Extractor Source 3.0 min
Ramp 1	5	120	0.0	Acquisition Mode	Scan
Ramp 2	12	300	5.0	Scan Range	33 to 300 a.m.u.
Runtime	38 min			Threshold Sampling	150 2
Inlet	Split/Spli	tless		TID	OFF
Temp	250 °C			Quad Temp	150 °C
Mode	Split, Co	nstant	Flow	Source Temp	230 °C
Flow	1.2 mL/n	nin		Transfer Line	280 °C
Inlet Press	11.06 psi			Tune	etune.u
Septum Purge	3.0 mL/m	in			Gain = 1.0
Purge Flow	24 mL/m	in (Sp	lit Ratio 20:1)	Guard Chip	Tracking Oven Ramp
Column	DB-1MSU	JI part	t # (122-0132UI)		
	30m x 0.2	25 mm	id x 0.25 µm film	Flow Chip Bus	Isothermal 300 °C
Liner	Single tap 5190-2293	er w/wo	ool 900µL ultra inert		
Injection volume	1.0 µL	Co	mplete cycle time	between injections	2 minutes

GC Equilibration Time 0.1 min

![](_page_56_Figure_0.jpeg)

![](_page_56_Picture_2.jpeg)

![](_page_57_Figure_0.jpeg)

![](_page_57_Picture_2.jpeg)

#### **Extracted Ion Profiles**

![](_page_58_Figure_1.jpeg)

![](_page_58_Picture_3.jpeg)

![](_page_59_Figure_0.jpeg)

![](_page_59_Picture_2.jpeg)

#### **Extracted Ion Profiles**

![](_page_60_Figure_1.jpeg)

![](_page_60_Picture_3.jpeg)

# RT Reproducibility of 6 alkylbenzenes on multiple instruments, guard chips, and column installations

Castle Peaks	Co	onventional Re	etention Time		<u>Fast Retention</u> Time	
n=8	Range	Average	%RSD	Range	Average	%RSD
n-propylbenzene	9.3340 - 9.3660	9.3493	0.14	4.7280 - 4.7790	4.7486	0.48
3-ethyltoluene	9.5640 - 9.6030	9.5828	0.15	4.8430 - 4.8890	4.8615	0.42
4-ethyltoluene	9.6190 - 9.6580	9.6375	0.15	4.8700 - 4.9150	4.8883	0.42
trimethylbenzene	9.8060 - 9.8870	9.8460	0.32	4.9560 - 4.9990	4.9739	0.39
2-ethyltoluene	10.0590 - 10.1500	10.0901	0.26	5.0870 - 5.1280	5.1036	0.35
1,2,4- trimethylbenzene	10.5090 - 10.5570	10.5321	0.15	5.3100 - 5.3440	5.3258	0.27

![](_page_61_Picture_2.jpeg)

![](_page_62_Picture_0.jpeg)

# **Energetics Analysis with the Intuvo 9000 GC and 5977B MSD**

#### **Kirk E. Lokits, Ph.D GCMS Applications Chemist**

![](_page_62_Picture_3.jpeg)

December 5, 2017

## AccuStandard Energetics Sample A Extractor Source/7890

![](_page_63_Figure_1.jpeg)

![](_page_63_Picture_2.jpeg)

![](_page_63_Picture_3.jpeg)

### AccuStandard Energetics Sample B Extractor Source/7890

![](_page_64_Figure_1.jpeg)

![](_page_64_Picture_3.jpeg)

#### Extractor Source/7890 compared to High Efficiency Source (HES)/Intuvo

![](_page_65_Figure_1.jpeg)

![](_page_65_Picture_3.jpeg)

#### 7 ppm Cerilliant energetics standard with varying Guard Chip

![](_page_66_Figure_1.jpeg)

![](_page_66_Picture_3.jpeg)

# **Restek Energetic Standard @ 100 ppm H<sub>2</sub> carrier gas**

![](_page_67_Figure_1.jpeg)

![](_page_67_Picture_3.jpeg)

## Calibration Curve of Cyclonite (RDX) 5 -150 ppm H<sub>2</sub> carrier gas

![](_page_68_Figure_1.jpeg)

![](_page_68_Picture_3.jpeg)

## Cotton Swab Sample Matrix Spiked (RDX, TNT, PETN) Extracted 1 mL Acetone

![](_page_69_Figure_1.jpeg)

![](_page_69_Picture_3.jpeg)

## **Summary**

Intuvo was able to achieve separation/resolution, reproducibility, and sensitivity, analyzing components of forensic interest (volatiles BAC, street drugs, ignitable liquids, and energetics)

Guard chip doesn't interfere with early eluting peaks or peak shape but can be controlled to produce better chromatography on early eluting peaks similar to a retention gap or pre-column

Intuvos (Multi-mode inlet) MMI cycle time reached 35°C equilibration in 4 minutes and ready for the next injection and the 7890 MMI took 7 minutes to come ready

Intuvo was able to achieve separation of energetics in a helium and hydrogen carrier gas environment

# Intuvo was able to run under fast chromatographic conditions (150°C oven ramp) without additional voltage or special oven shroud (120 Vac 15 amp plug)

Retention time differences of 6 alkylbenzene compounds were monitored and found to have maintained <0.5% RSD while being analyzed on 2 different Intuvo 9000 GCs, multiple column installations multiple guard chip replacements, as well as routine inlet maintenance replacing septa and liners

![](_page_70_Picture_7.jpeg)

# Thank you for your attention

![](_page_71_Picture_1.jpeg)