Reducing Pressure on Operational Budgets: Helium Conservation Strategies for GC and GC/MSD

Agilent Science and Technology Symposium

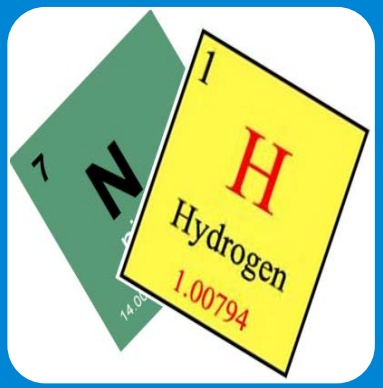
May 2014

Topics for Today

Reducing Pressure on Operations Related to Helium

Minimizing Helium Use

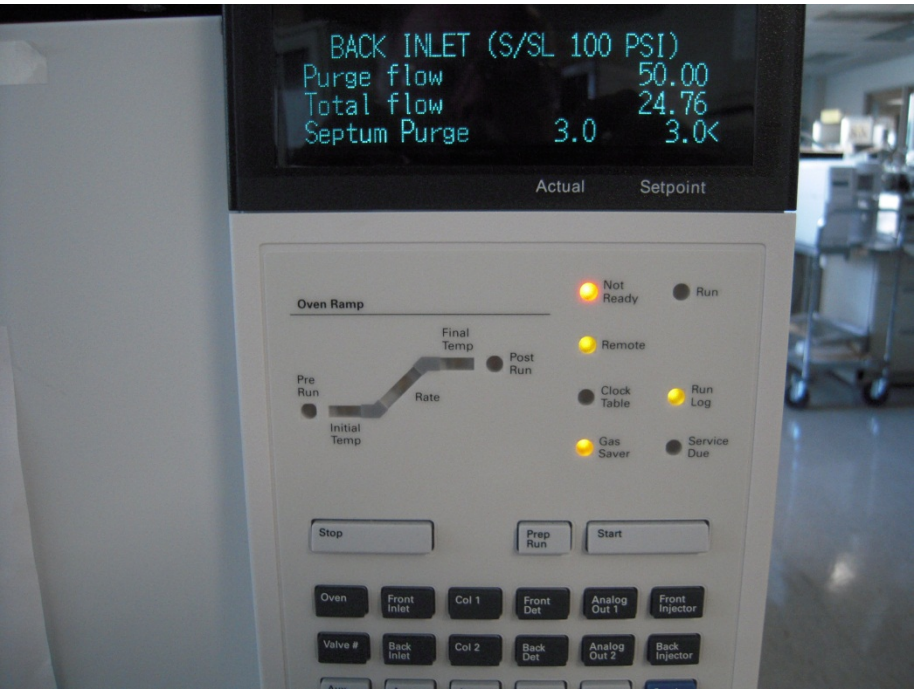
- Helium audit
- Identify leaks and other loss areas
- Optimize analyses to reduce helium consumption

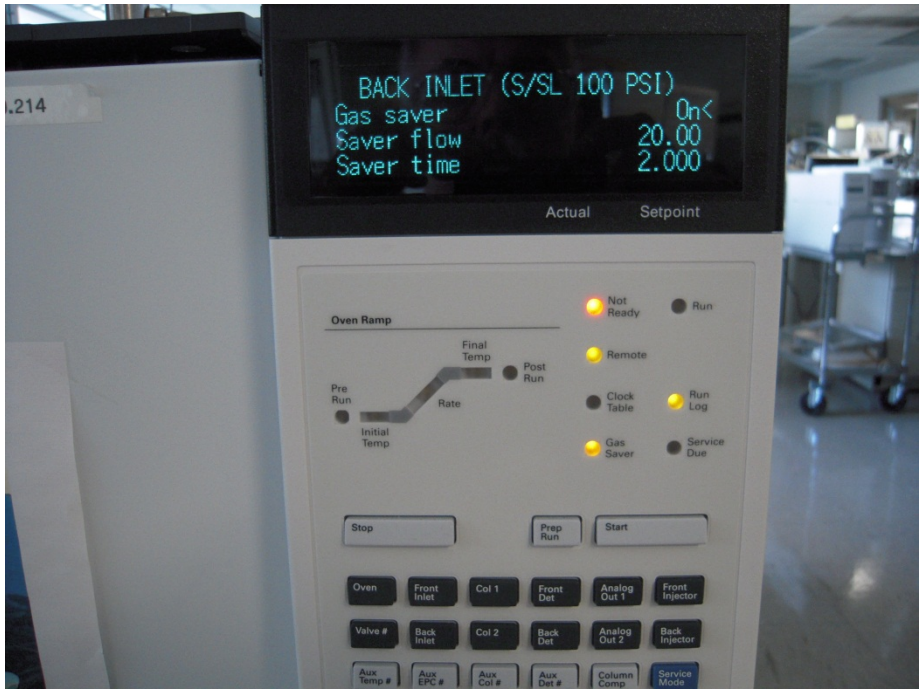


Adapting Methods to Alternate Carrier Gases

- Nitrogen Carrier Gas
- Hydrogen Carrier Gas

Helium Audit
Add Up All Uses of Helium and Compare It to Actual Usage





- Purge Flow is 50 mL/min for 2 min;
- Gas Saver drops it to 20 mL/min the remainder of run
- Column flow is 1.2 mL/min
- No helium is used as detector makeup gas (we use nitrogen)

Helium Audit for Agilent Little Falls Site

Example data from select parts of our facility

Laboraty	Number of GCs or Inlets	avg flow (ml/min)	Total Flow (ml/min)	Liters / day (minimum)	Liters / day (maximum)*	Cost/day	Cost/day (maximum)	Location (Floor)
SEM	2	40	80	115.2	172.8	1.38	2.06	1
GC area	15	50	750	1080	1620	12.9	19.34	1
GC pressure check				10	15	0.12	0.18	1
Arcon	5	50	250	360	540	4.3	6.45	1
NPD	4	70	280	403.2	604.8	4.81	7.22	1
ECD	5	50	250	360	540	4.3	6.45	1
Consumables	2	75	150	216	324	2.58	3.87	2
GCMS Training Lab	33	80	2640	3801.6	5702.4	45.39	68.09	2
Ken's Lab	9	70	630	907.2	1360.8	10.83	16.25	2
COE	6	60	360	518.4	777.6	6.19	9.28	2
Marketing Lab	35	50	1750	2520	3780	30.09	45.13	3
SW Validation 1	17	50	850	1224	1836	14.61	21.92	3
SW Validation 2	14	65	910	1310.4	1965.6	15.65	23.47	3
SW Validation 2	1	750	750	1080	1620	12.9	19.34	3
R&D	34	80	2720	3916.8	5875.2	46.77	70.15	3
Lab near Finance	7	50	350	504	756	6.02	9.03	3
FRC-GCs	5	70	350	504	756	6.02	9.03	3
FRC-GCMS	8	1.2	9.6	13.824	20.736	0.17	0.25	3
					cost/day	$219	$328	
					cost/yr	$79,870	$119,805	

Audit Results
Agilent Little Falls Site

- For entire facility, based on theoretical total flow of helium used, bill should be about **$170,000**
- Our actual bills were greater than **$500,000** per year

Action items:

- Look for leaks
- Raise awareness among users
- Where it makes sense, adjust instrument parameters to reduce helium usage

Leak Detectors

Agilent G3388B

Leak Detector

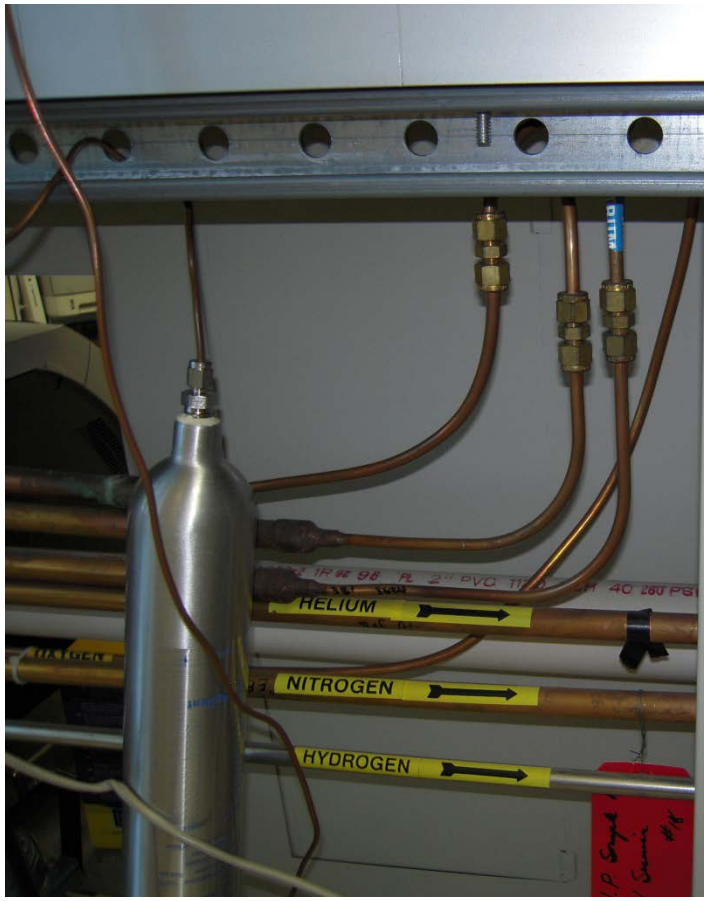
- Allows detection of **helium and hydrogen** to 0.0005 ml/min.
- Detects thermal conductivity differences
- Audible and visual alerts
- Small – about the size of a cell phone
- Recharge using USB to any PC
- Lithium ion battery, > 5 hours of life
- One year warranty from Agilent

Liquid Leak Detector

- Works for all gases
- Good for checking tube fittings
- Must be applied to directly to connection
- Does not find leaks in the area
- Do not use on fittings with vacuum inside

Leak Investigation
Evaluating non-system areas for helium loss

Gas Towers
Easy access



Infrastructure Plumbing
Not so easy access

Leak Investigation
Results of Actions

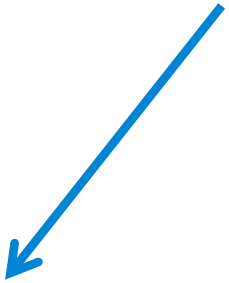
- Found and fixed many leaks
- Found helium plumbed to industrial device for purging where nitrogen should be used
- Raised awareness among users that reducing helium consumption is important
- Adjusted instruments to use less helium without compromising performance
- **After one year, our helium use dropped by a factor of 2.2**
- A customer, a large chemical company, reported similar results

Optimizing Methods To Save Helium

Looking at a Single Instrument:
GC/MSD Uses 32 L/day

GC Flow Conditions	
He Carrier Flow (mL/min):	1.2
He Split flow (mL/min):	50
Gas Saver Flow (mL/min):	20
Gas Saver On (min):	2
Run Time(min.):	20
Gas Volume in Cylinder (L):	8000
Runs per Day:	20

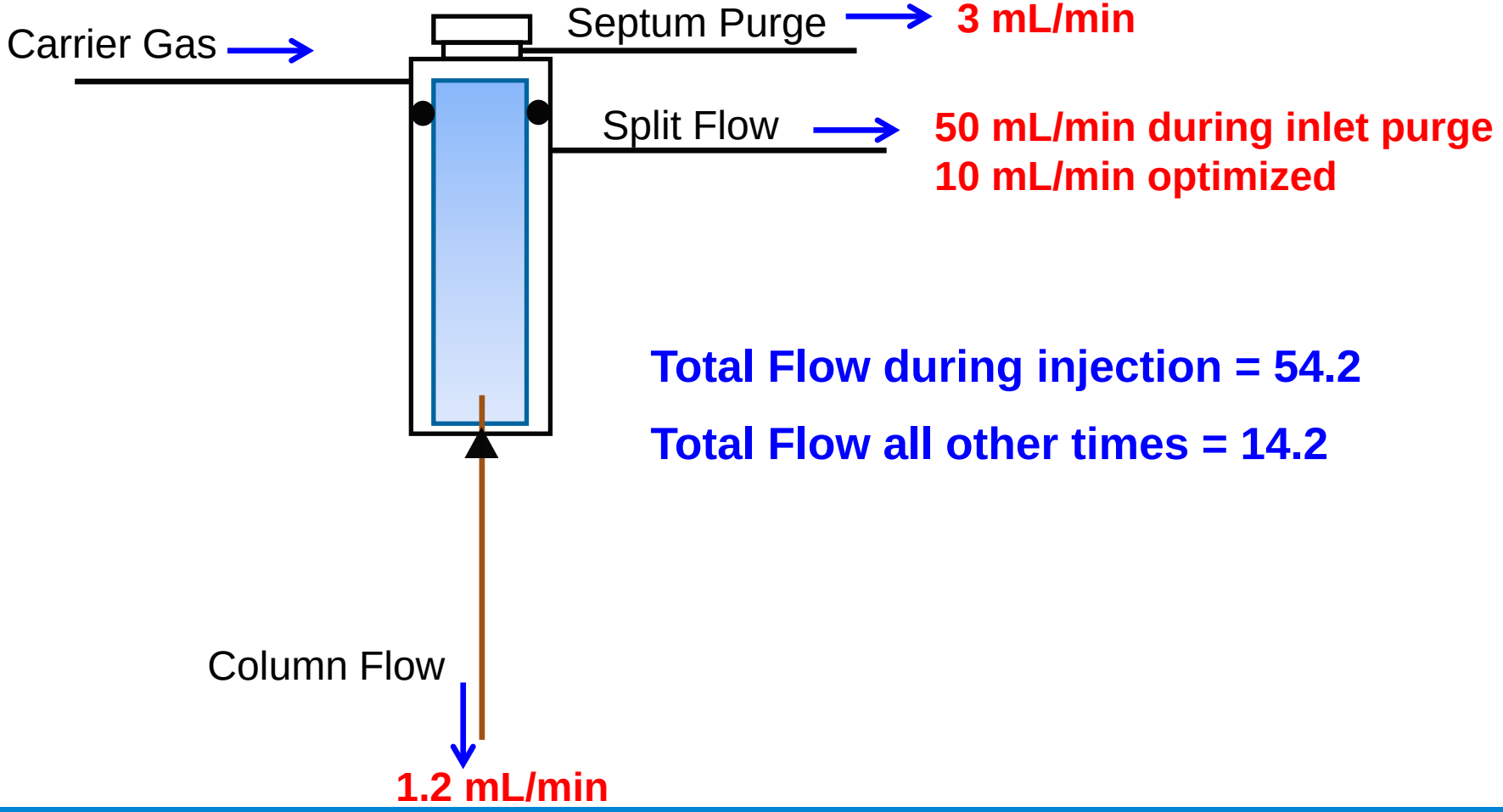
Note that Gas Saver offers significant savings with Split/Splitless and MMI inlets



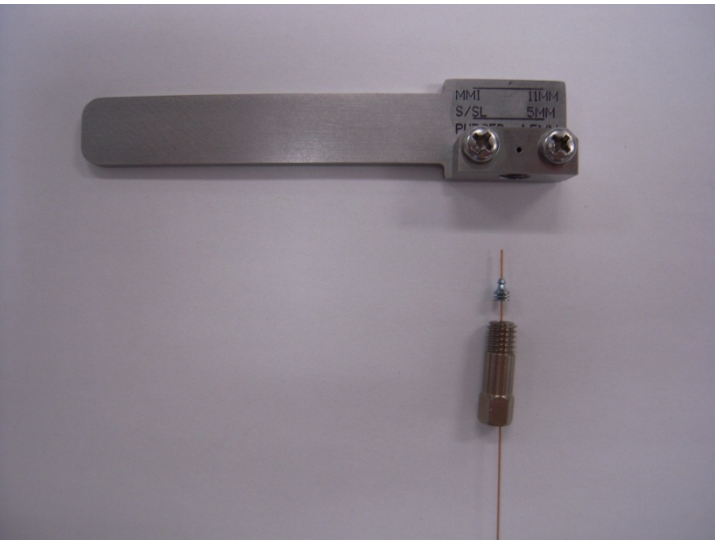
Parameter	No Gas Saver	Gas Saver
Daily He Usage (L)	74	32
He Cylinder Life (days)	109	252

Optimizing Split Flow to Lowest Value with MSD

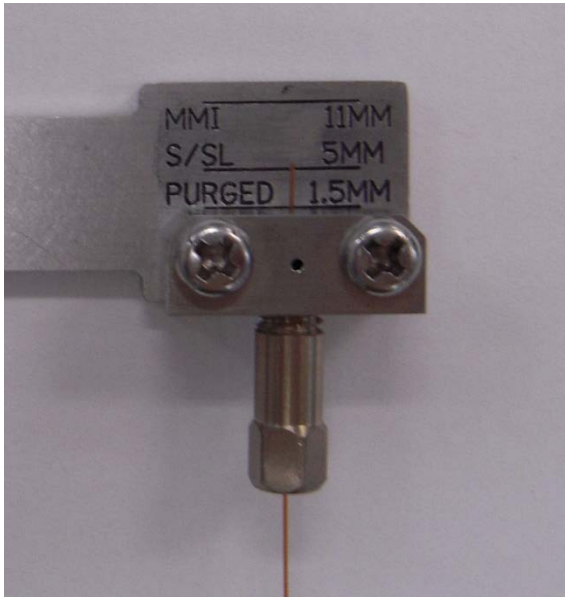
To optimize gas saver, reduce flow stepwise while monitoring m/z 28. Optimal flow is somewhat higher than flow where 28 abundance increases

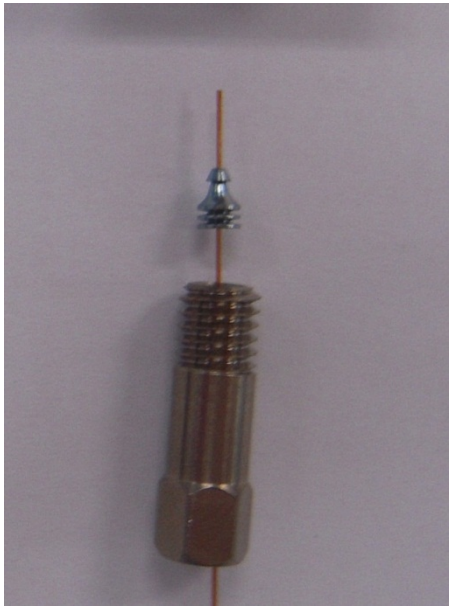


Minimizing Diffusion of Air Into Inlet
Use Flexible Metal Ferrule to Seal Column in Inlet



Pre-crimp deactivated ferrule to column





- Much easier to install column
- Does not loosen with oven cycling
- Doe not diffuse air like graphite

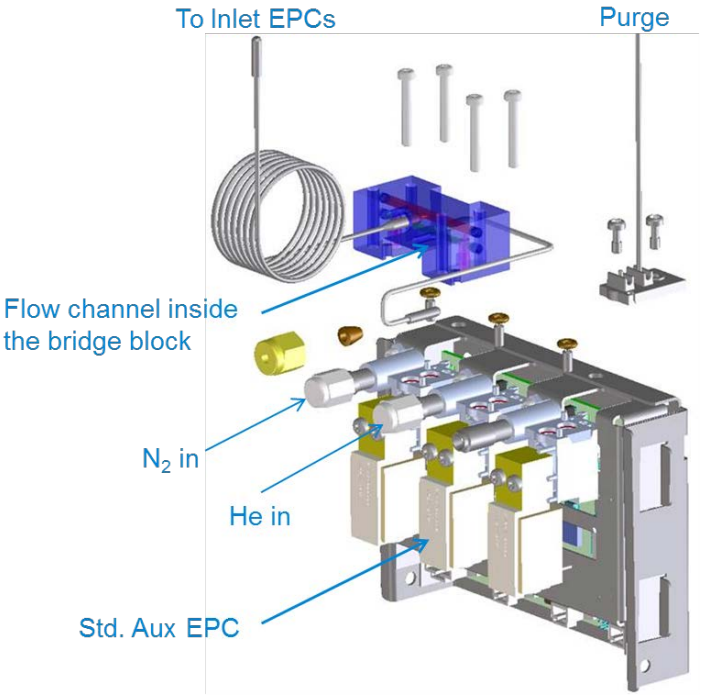
Helium Usage Comparison

Column (mL/min)	Split (mL/min)	Gas Saver (mL/min)	Detector (mL/min)	Runs per Day	Daily He Use (L)	
					No Gas Saver	Gas Saver
1.2	50	20	30	20	117	75
1.2	50	20	0	20	74	32
1.2	50	10	0	20	74	18
5	200	20	0	20	295	43

20 min run, 2 min gas saver time

By switching detector makeup to nitrogen, using gas saver, and optimizing it to 10 mL/min, **helium usage is reduced by 85%**

Reducing Helium Use Further…



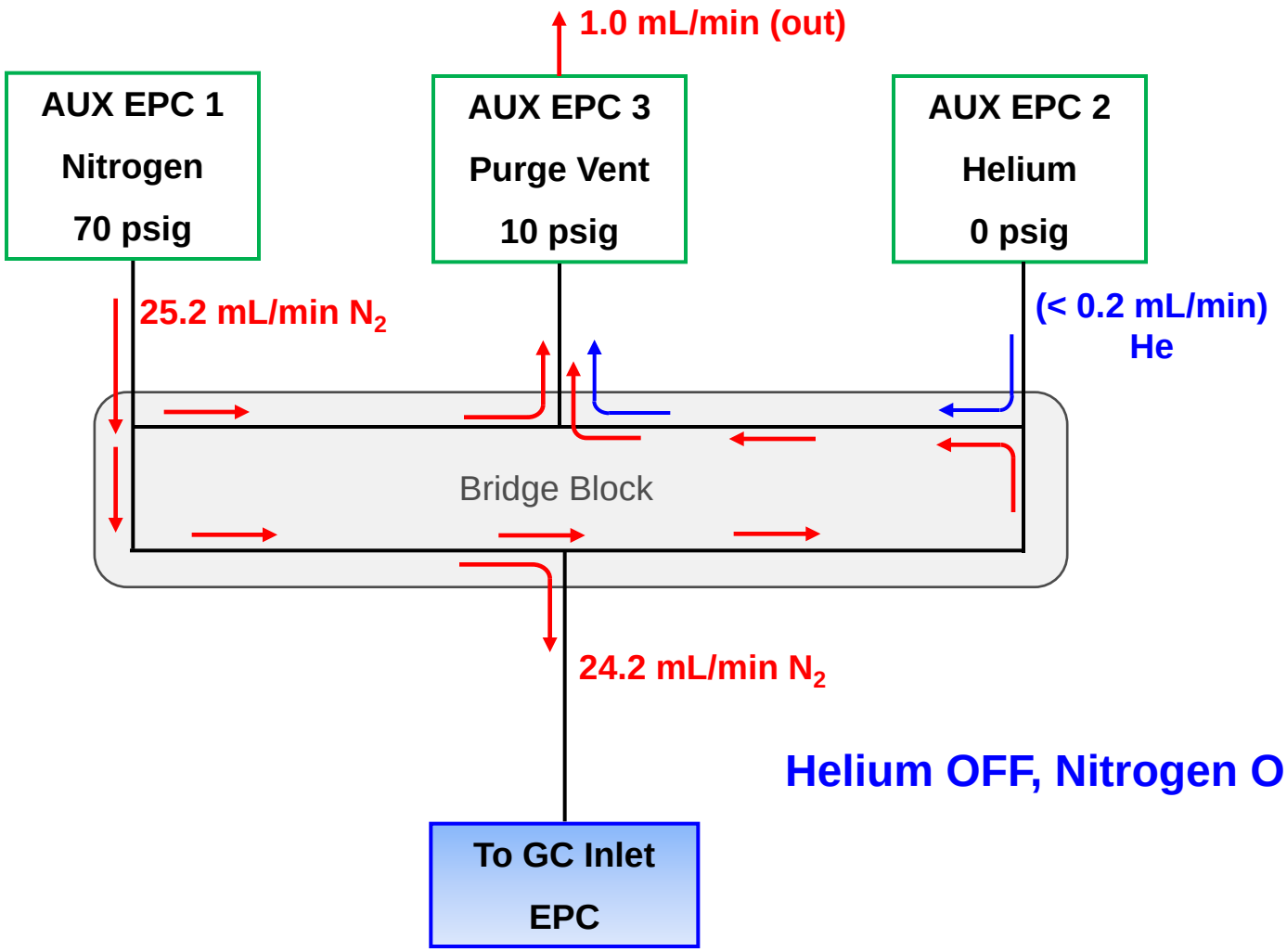
Switch between gases within 15-30 min for most detectors

Programmable Helium Conservation Module

- Fully controlled by Agilent data systems
  - Integrates into Sleep and Wake function
  - Automatically switches carrier gas supply to N_2 Standby during idle time
- Better alternative to just “shutting off the GC”
  - No system contamination due to ambient air exposure
  - Faster re-start of heated zones
  - Purge channel prevents cross contamination of gases
- Precise pressure control between tank and GC
- Combined with Helium Gas Saver to *GREATLY* reduce helium consumption

How Does It Work?

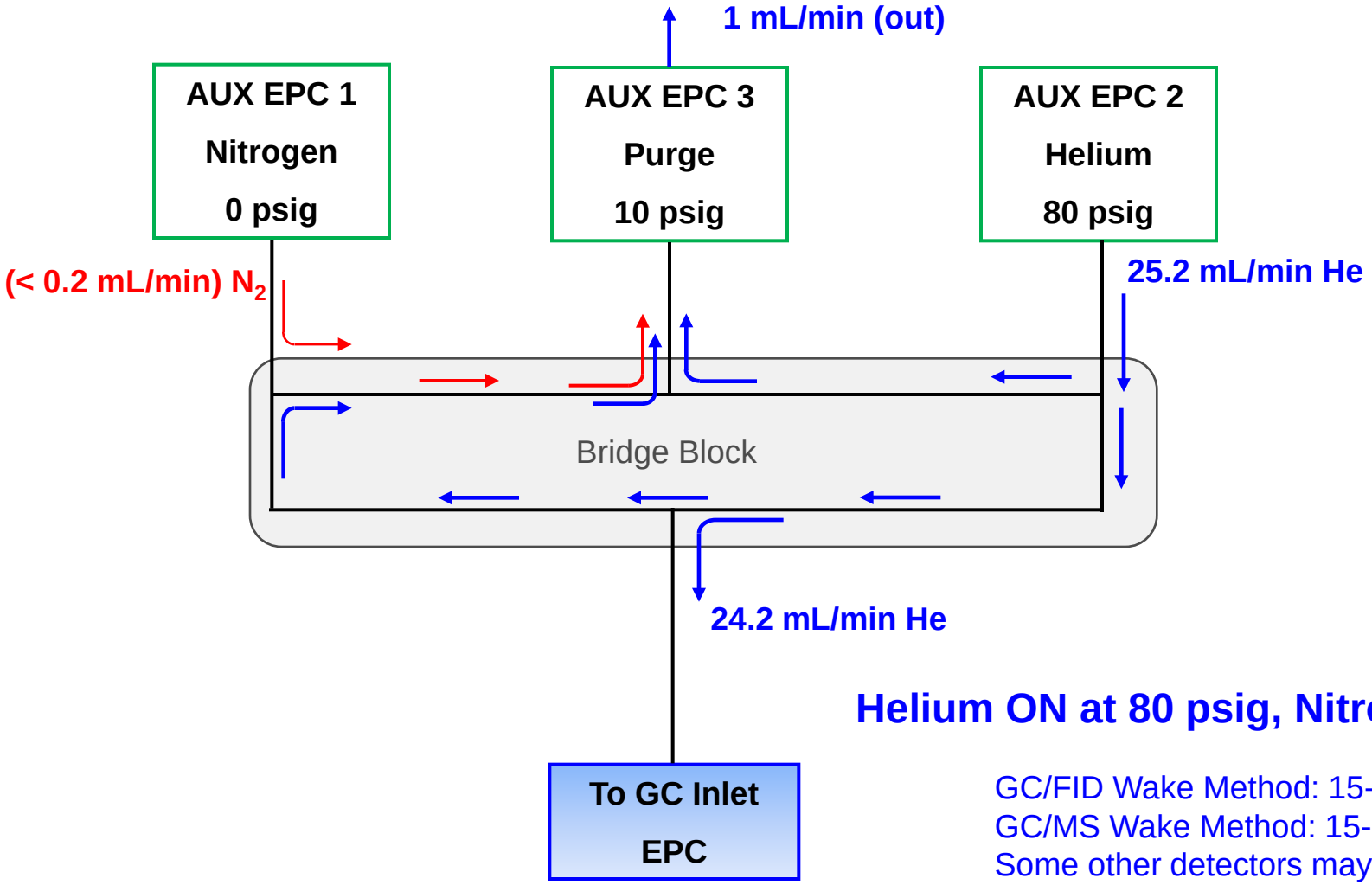
Helium Savings Mode (Nitrogen Carrier, or Sleep Mode)



Helium OFF, Nitrogen ON at 70 psig

How Does It Work?

Normal Operation Mode (Helium Carrier or Wake Mode)

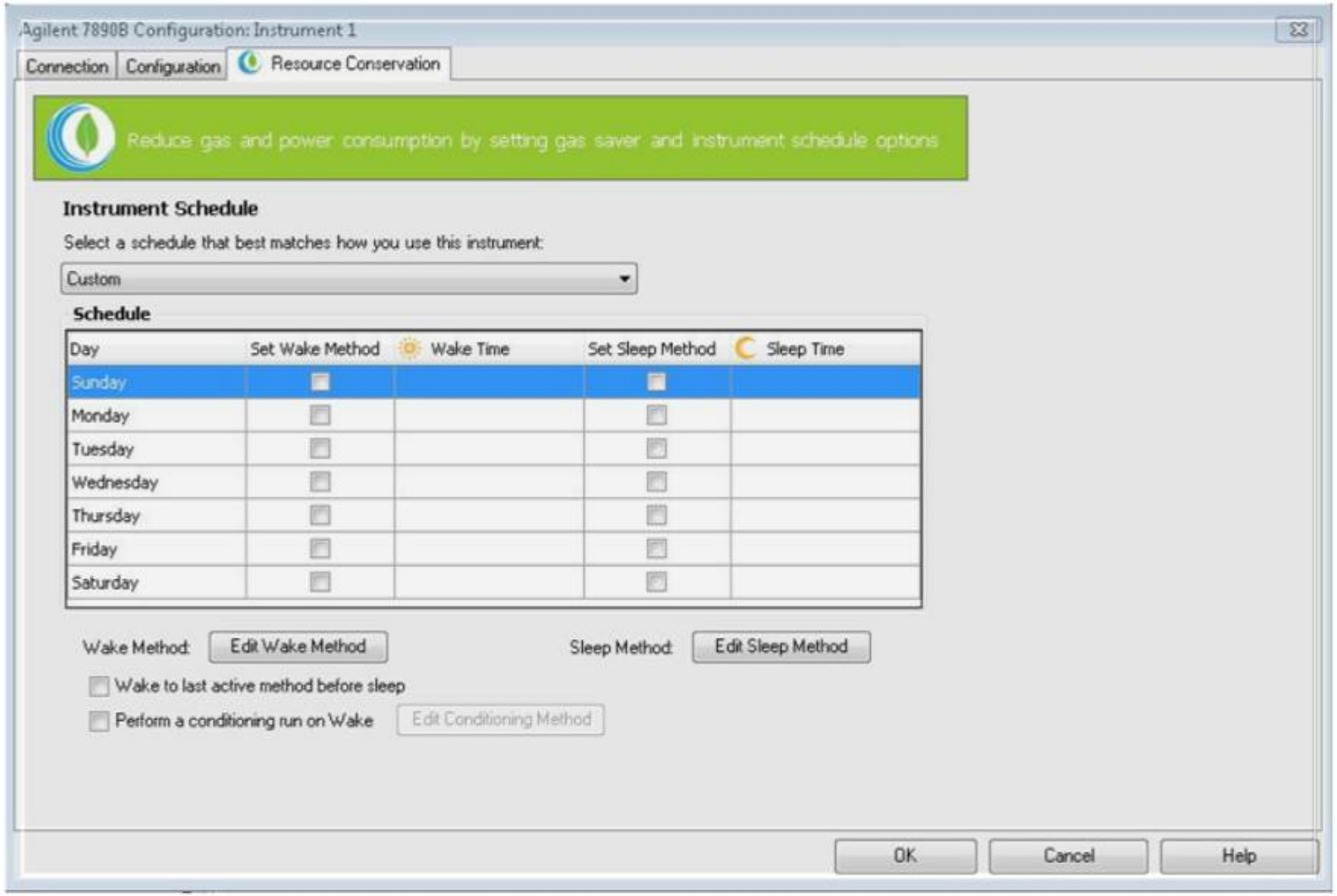


Helium ON at 80 psig, Nitrogen OFF

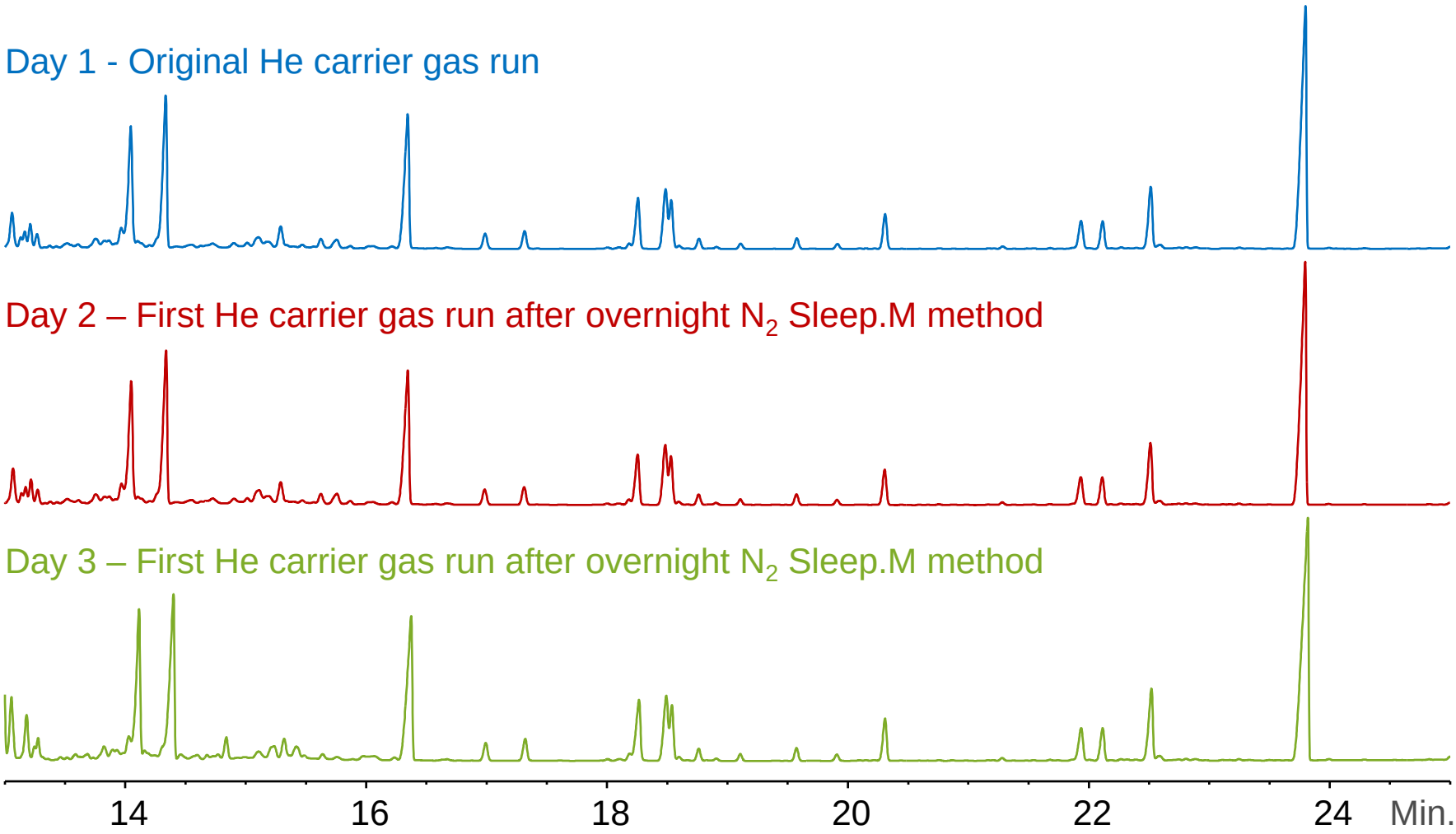
GC/FID Wake Method: 15-30 Min
GC/MS Wake Method: 15-30 Min
Some other detectors may need longer

How It Works: Configuring Sleep/Wake Operation

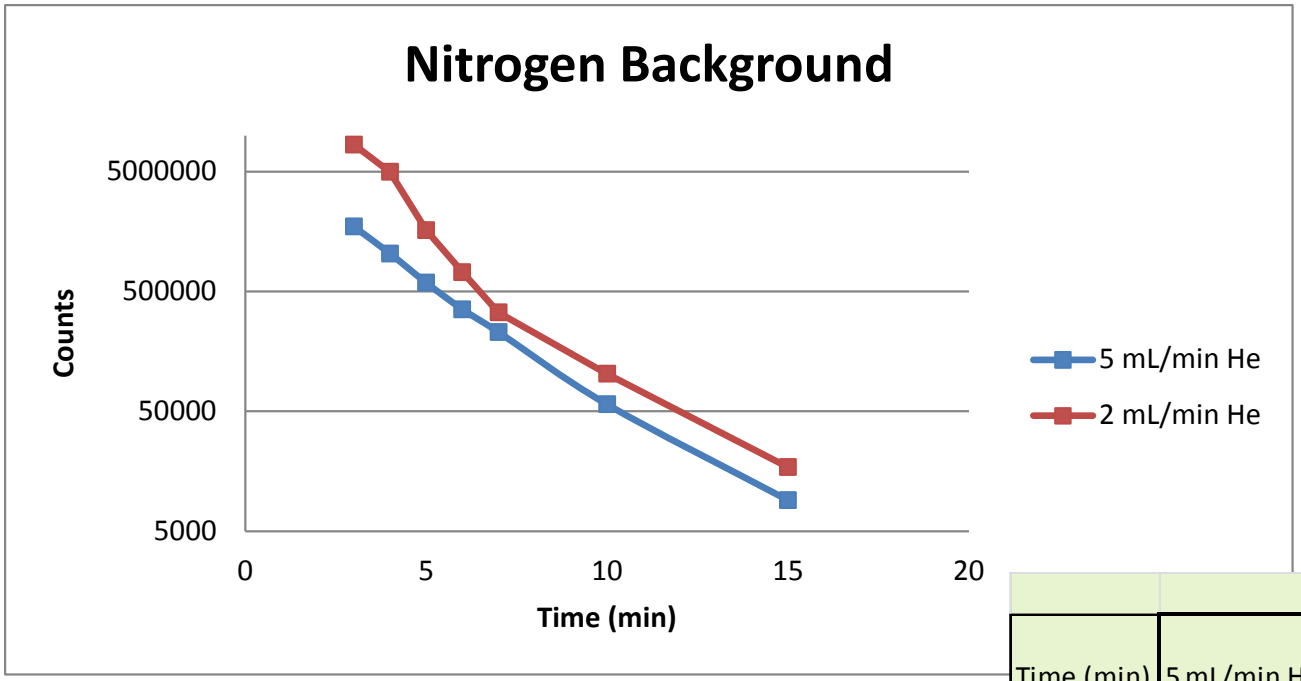
Simple, Straight Forward Setup



Performance: No Change in Chromatography After N_2 Carrier Sleep Method. GC/FID Analysis



Performance: Pass MS Tune within 15 min after Switching from N_2 to He as Carrier. GC/MSD



		Counts of Nitrogen Ion		
Time (min)	5 mL/min He	Relative to Saturation	2 mL/min He	Relative to Saturation
3	1735168	20.69%	8388096	100.00%
4	1033280	12.32%	4959232	59.12%
5	590080	7.03%	1618944	19.30%
6	354112	4.22%	722944	8.62%
7	228480	2.72%	333696	3.98%
10	56984	0.68%	102576	1.22%
15	9052	0.11%	17080	0.20%

Helium Usage with Helium Conservation Switch

Column (mL/min)	Split (mL/min)	Gas Saver (mL/min)	Detector (mL/min)	Runs per Day	Daily He Use (L)			He Cylinder Life (days)
					No Gas Saver	Gas Saver	He Conservation	
1.2	50	20	30	20	117	75	22	369
1.2	50	20	0	20	74	32	10	826
1.2	50	10	0	20	74	18	6	1316
5	200	20	0	20	295	43	17	465

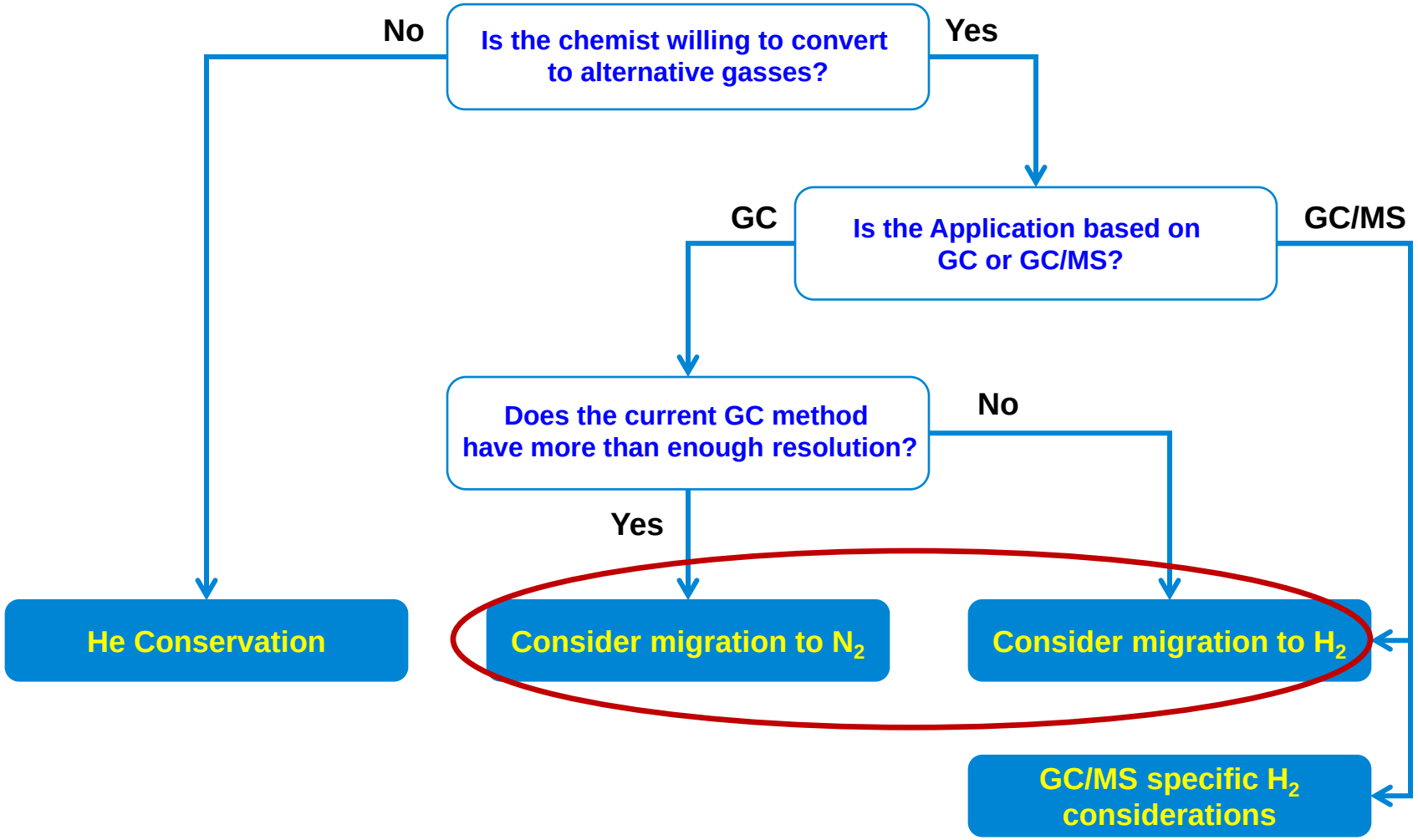
20 min run, 2 min gas saver time

By switching detector makeup to nitrogen, using gas saver, optimizing gas saver, and using the conservation switch, **helium usage is reduced by 95%**

Alternate Carrier Gases

Carrier Gas Decision Tree

Migrating GC methods to nitrogen and hydrogen



Use of N_2 as Carrier Gas

Many helium GC methods are suited to nitrogen conversion

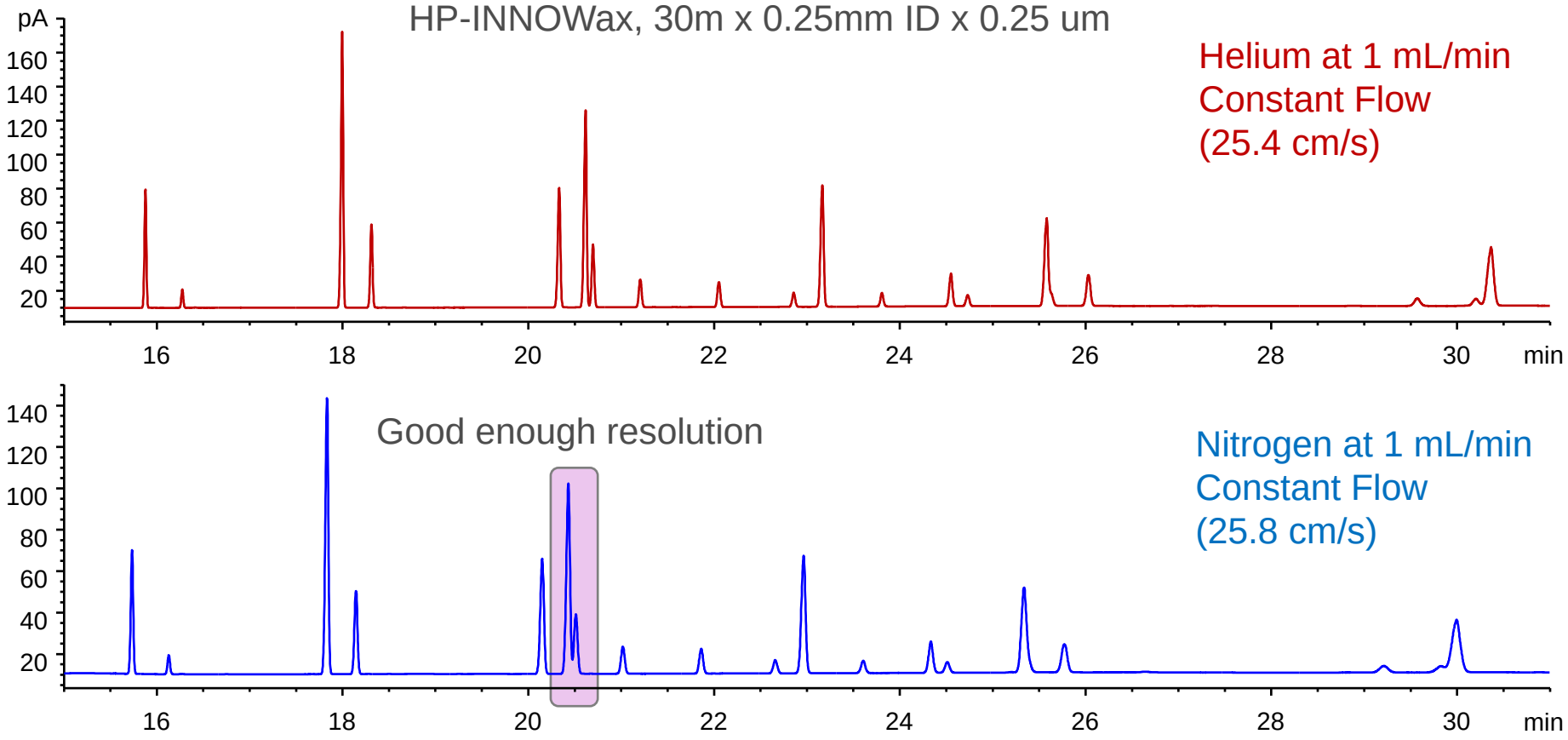
- Readily available and less expensive gas
- No safety concerns
- Suitable for simple routine analysis (with sufficient resolution)
- More inert than H_2, especially with PLOT/Micropacked columns
  - Some compounds catalytically reduced in H_2
- 2-D GC ideally suited to nitrogen
  - Resolution issues solved by using 2 different columns

Potential issues

- Reduced chromatographic resolution at higher flows
- Not suitable for GC/MSD and certain GC detector applications

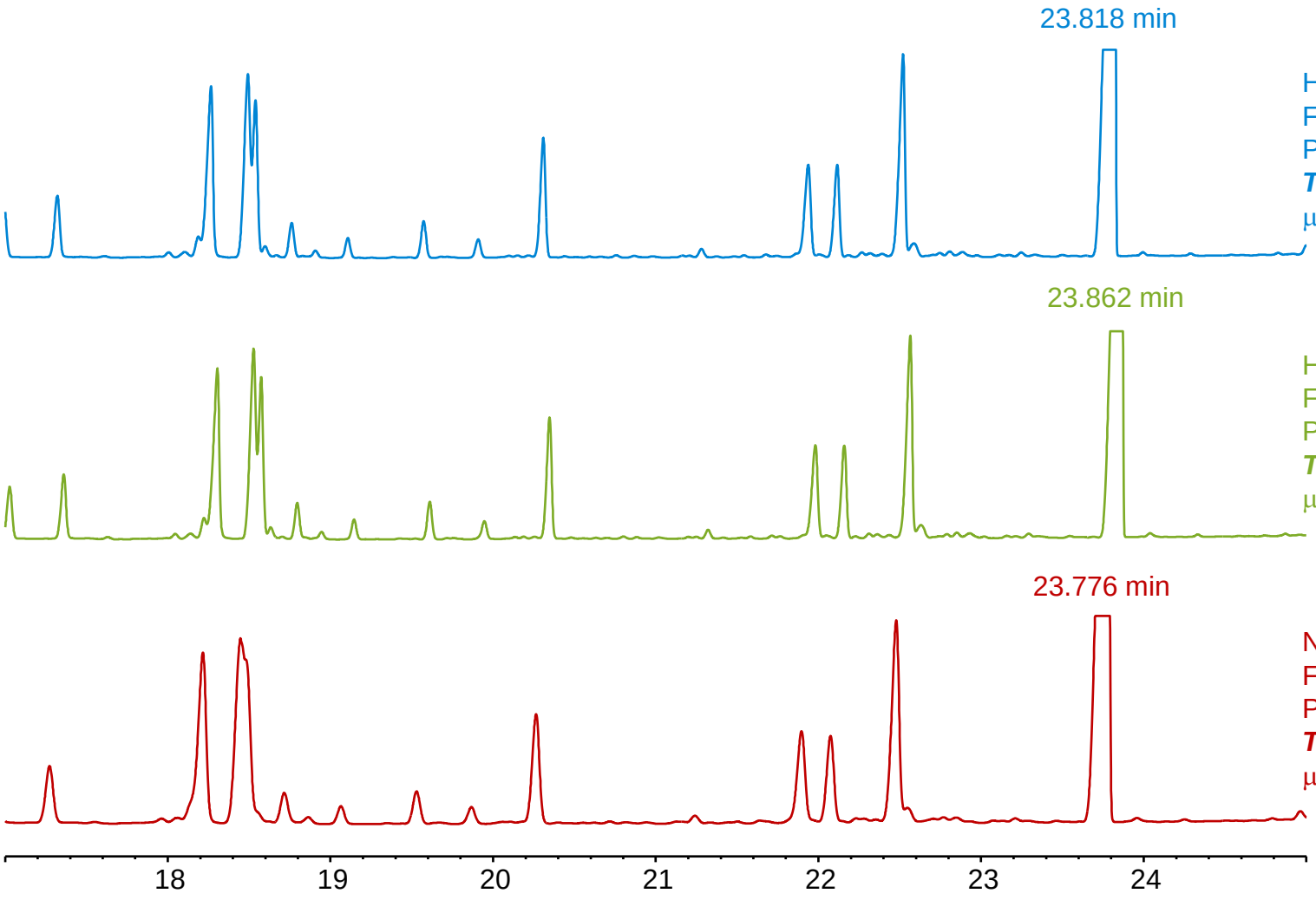
Many Helium GC Have Excess Resolution

EN14103 – GC Analysis of FAME content in biodiesel



Same Holdup Time (T_r) Gives Consistent Retention

Times Compared to Original Helium Method



Use of H_2 as Carrier Gas

Advantages to hydrogen conversion

- Readily available, less expensive, can be generated in lab
- Same or better chromatographic resolution per unit time
- Only alternative to He for GC/MSD
  - Reduces or eliminates source cleaning

Potential issues

- Safety concerns
- Some compounds react/decompose in presence of H_2
- Not all detectors can be used with H2

Introduction: Converting from He to H_2 Carrier Gas

Methods that will generally require less optimization include analytes that are:

- “durable” compounds
- at higher concentrations
- analyzed with split injections
- derivatized

Methods that will generally require more optimization include analytes that are:

- “fragile” compounds
- at trace concentrations

Allow time for necessary updates to SOPs and validation

Designed for Reliability – H_2 Safety

Safety Shutdown

When gas pressure set points are not met, the valve and heater are shut off to prevent explosion

Flow Limiting Frit

If valve fails in open position, inlet frit limits the flow

Oven ON/OFF Sequence

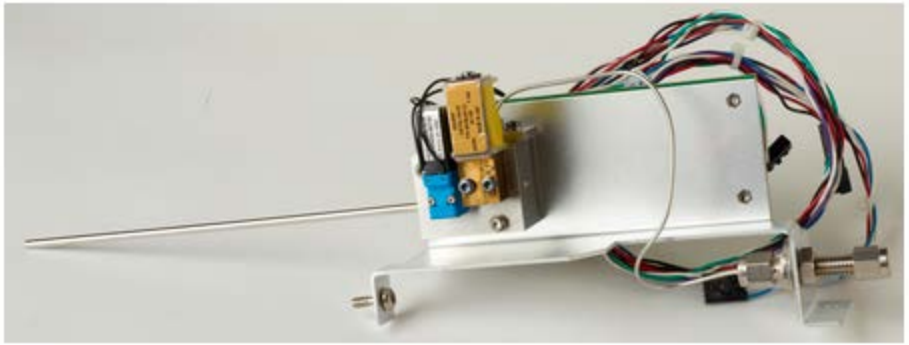
Fan purges the oven before turning on heater to remove any collected H_2

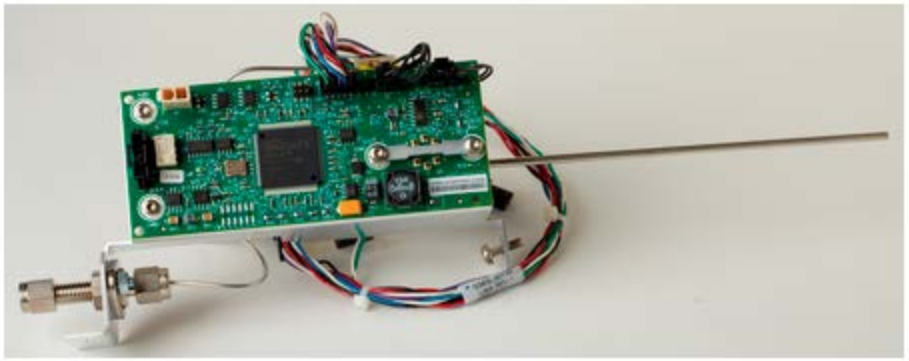
Explosion Test

GC and MS designed to contain parts in case of explosion

Hydrogen Sensing Module for 7890 GC Oven

- Complete GC shutdown when 1% H_2 is detected in oven (4% H_2 is LEL)
  - Open flaps, oven vents, turns off ignition sources and puts GC in shutdown state requiring user interaction
- Fully integrated into 7890A+/B GC
- Ability to calibrate on a set schedule or instantly when deemed necessary
  - Ability to print calibration report on demand





For more information:
http://www.chem.agilent.com/en-US/products-services/Instruments-Systems/Gas-Chromatography/7890B-GC/Pages/H2Sensor.aspx

First, Listen to Agilent Webinar on Details of Conversion of GC/MSD Method from He to H_2

Go To This URL for recorded webinar:

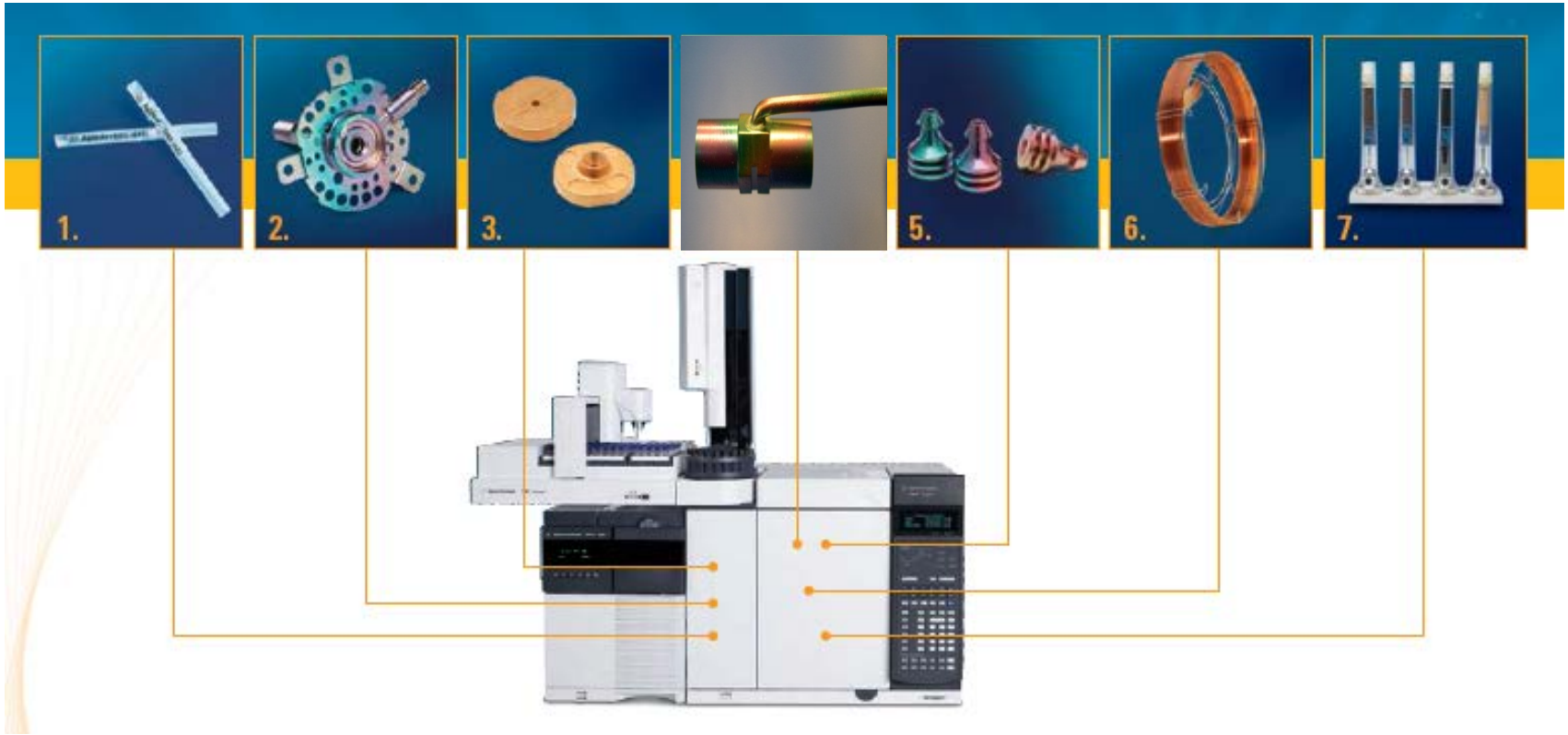
http://www.agilent.com/chem/heliumupdate

Topics Covered:

- H_2 Safety
- Source of H_2 Carrier and Plumbing
- MS Components Required: Magnet and Draw Out Lens
- Choosing a Column and Method Conditions
- Initial Startup with Hydrogen
- H_2 Conversion Considerations for Success
- Performance Expectations

Inert Flow Path Items Help with H2 Conversion

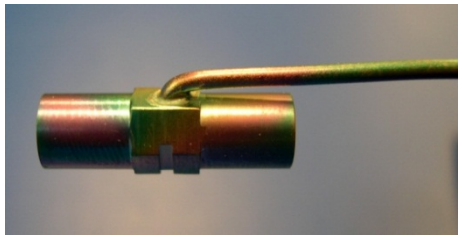
Reliability, Durability, Speed and Ease of Use



Configuration of Controlled Substance Analyzer

Post column Capillary Flow Technology (PUU) device provides:

A) Column Backflush
B) No vent column change
C) Convert from H2 to He with same retention times

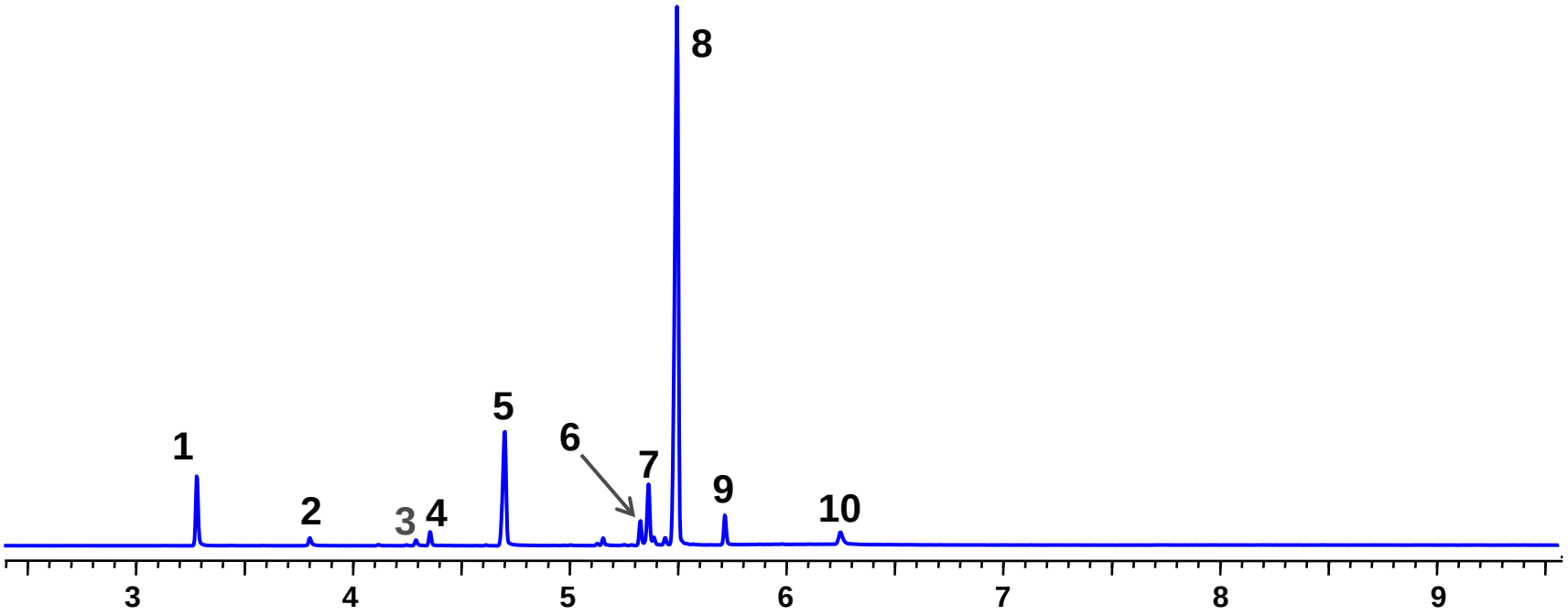


Purged Ultimate Union (PUU)

Example: Street Heroin

Controlled Substances Analyzer

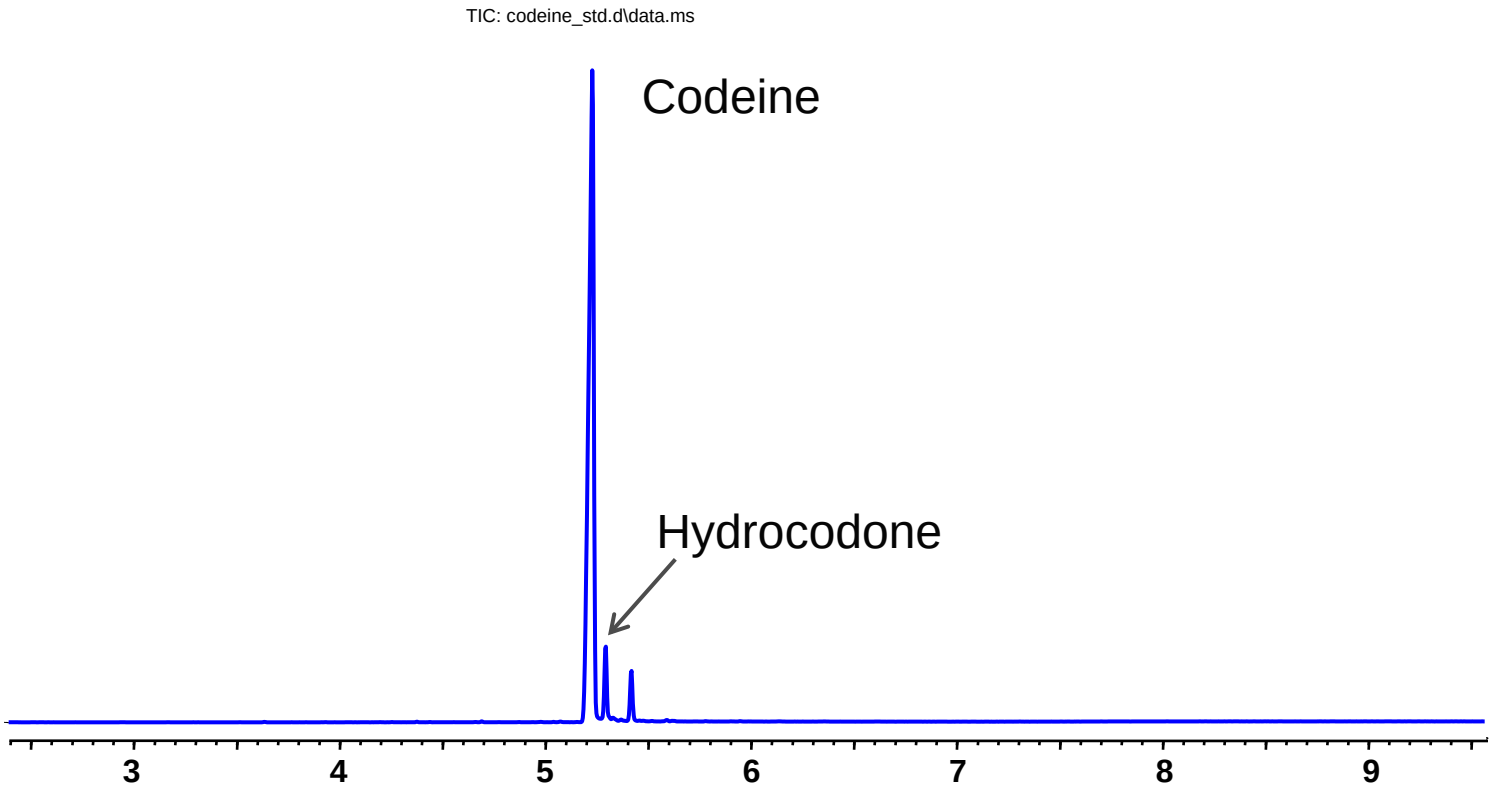
1 N-Propylamphetamine (ISTD)
2 Benzocaine
3 Caffeine
4 Lidocaine
5 10,11-Dihydrodibenz(b,f)(1,4)oxazepin-11-one (ISTD)
6 Acetylcodeine
7 6-Monoacetylmorphine
8 Heroin
9 Papaverine
10 Noscapine



Codeine, H_2 Instrument

Some hydrocodone is formed when codeine is injected.

- About 6% is converted.



Presentation Summary

- Do a helium audit. You might be amazed at how much He is being wasted
- Review you GC and GC/MS methods to see if there are opportunities to save helium
- Determine if alternate carrier gases can be used for some methods

Helpful Links
Alternate Carrier Gas

Agilent Website for Alternative Carrier Gases

http://www.chem.agilent.com/en-US/Promotions/pages/alternate-carrier-gas.aspx

Link to World He Shortage Information:

http://www.chem.agilent.com/Library/flyers/Public/Introducing%20the%20Programmable%20Helium%20Conservation%20Module.pdf

Agilent 7890B Gas Chromatograph and Related Accessories:

http://www.chem.agilent.com/en-US/products-services/Instruments-Systems/Gas-Chromatography/7890B-GC/Pages/default.aspx

Thank you

Let's Continue the Conversation

www.agilent.com

Access Agilent

Facebook  @Agilent.Tech

Twitter  @Agilent

LinkedIn