#### A New GC/MS System Designed for Helium Carrier Gas Conservation



Approaches for the Smarter Use of Helium

Dale R. Walker GCMS Applications Specialist August, 7, 2013



Agilent Technologies

The Measure of Confidence

#### **Helium Facts**

- How it's produced
- Where did it all go?
- What can you do conserve?
- Is there an alternative gas?



A brief history of Helium

Created by radioactive decay of heavy radioactive elements

- Primarily Thorium and Uranium
- Produces Alpha particles which form He nuclei
- Trapped in Natural Gas Fields mainly in American Great Plains
- Up to 7% by volume
- Separated from natural gas by low temperature fractional distillation



#### Helium Facts continued.

National Helium Reserve - Amarillo, Texas (Dept. of Interior)

- Fed by 425 mile pipeline from Kansas to Texas
- Five Helium plants feed the pipeline

1996 Congress decided to phase out the Helium Reserve

- Helium Reserve was \$1.4 Billion in debt in 1996
- Plan is to empty the reserve to recover the funds
- Expected to be empty by 2018
- As of 2012 the Reserve supplied 30% of the worlds Helium



For many years the U.S. supplied 90% of the world's Helium

- New plant in Algeria can supply all of Europe's demand
- Other plants are now on line in Canada and Poland

U.S. maintains reserves in Kansas, Oklahoma, and Texas

- New plants coming on line Wyoming, Qatar, and Russia
- New plants are not expected to ease the shortage



#### Helium Facts cont.

Most Helium is used for cryogenics (MRI's) and welding

- Also controlled atmospheres such as wind tunnels
- Gas Chromatography is a minor consumer

Prices are expected to continue to rise

- From 2002 to 2007 world prices doubled
- From 2007 to 2013 world prices have more than doubled again



#### **Helium Facts continued**

What can you do?

There are only two realistic choices:

<u>Option 1:</u> Implement Helium conservation measures. This approach extends the lifetime of existing Helium supplies, reduces operating costs, and ensures business continuity.

Option 2: Switch to a different carrier gas such as Hydrogen.



Environmental Protection Agency, Region 6 – Houston, Texas

The Environmental Protection Agency (EPA) Region 6 Laboratory is working with manufacturers to find solutions to the ongoing Helium shortage that is affecting many laboratories.



#### **Option 1: Helium Conservation**

Approach 1: Manual switching valve

A manual switching value is connected to both a Nitrogen and Helium cylinder and is manually switched at the end of each day, this requires someone to be physically present to switch the value.



#### **Option 1: Helium Conservation** An example installation





The EPA tested manual valve switching and found it suitable





#### **Option 1: Helium Conservation**

This simple cost saving alternative to switching carrier gases can be implemented immediately, with no time needed for method development, and without any noticeable effects on hardware or performance.

Depending on the total number of instruments and the time between sequences, this procedure has the potential to save laboratories thousands of dollars per year by conserving Helium.





#### **Option 1: Helium Conservation**

Approach 2: Automated valve

A better approach is to use an automated switching device that delivers Helium or Nitrogen to the GC flow control modules.

This type of automatic valve switching is preferred to the manual valve because it does not require user interaction. This switch may be programed to engage or disengage at the beginning or the end of your analytical run or sequence.



#### **Option 1: Helium Conservation Automated switching value: SW controlled**





#### **Option 1: Helium Conservation** Switching the Carrier with the *Carrier Saver switch*





#### **Option 1: Helium Conservation** Switching the Carrier with the *Carrier Saver switch*





#### **Option 1: Helium Conservation** Switching the Carrier with the *Carrier Saver switch*

#### Approach 3: Automated Helium conservation

Seamless integrated system includes GC hardware and software control which allows laboratories to integrate programmable Helium conservation into the daily operation of the GC or GC/MS system.



**Combines with 7890B Sleep-Wake feature** 

Automatically switches carrier gas supply to N<sub>2</sub> during GC idle time

**TWO Critical Components** 

- Carrier gas switching EPC module
- 7890B Sleep/Wake Feature

Plus ...

Chromatographic grade N<sub>2</sub> gas





#### Carrier Gas Switch EPC Module Ensures Business Continuity



- Built on 5<sup>th</sup> generation EPC
- Fully integrated and controlled by OpenLAB CDS
- Purge channel prevents cross contamination of gases
- Precise pressure control between tank and GC
- In AWAKE mode, switches between gases within 15-30 min for most detectors including MSD







#### **Greater reliability**

Based on proven 5<sup>th</sup> generation AUX EPC

Provides Nitrogen substitution when the GC is idle

It also provides additional warnings if set points are not reached.

This safety feature is very important when using Hydrogen. If a leak is detected an auto shutdown occurs and it remains in that state until reset by the user.





#### **Greater performance**

Purge channel prevents cross contamination of gases and acts as an intermediate pressure regulator from the tank to inlet EPC to ensure greater analytical precision.

Because there are no changes during the analytical sequence this eliminates the need for method revalidation.



#### **Seamless integration**

Fully integrated within the 7890B GC (Open LAB, Chem Station, Mass Hunter)

Method identifies both gases and uses the appropriate flows for each gas.

Switching between each gas is easily implemented using new OpenLAB Sleep/Awake functions.



The new Agilent Open Lab CDS software provides many possible Sleep/Wake program configurations and operates much like a programmable home heating system.



You program the 7890B GC to load the Sleep method when not in operation, this eliminates the use of Helium by using Nitrogen as a carrier gas.



## How Does It Work?

Helium Conservation Mode (Sleep Mode with Nitrogen Carrier)





You program the Wake method to load just before the start of the workday, this restores helium as the carrier gas so the GC system is completely ready and waiting to run samples.



# How Does It Works ?

Normal Operation Mode (Wake Mode with Helium Carrier)





## **Option 1: Helium Conservation**

**Introducing the Programmable Helium Conservation Module** 

For GC and GC/MS systems, it only takes 15 minutes to completely remove all of the Nitrogen from the internal GC flow paths. The GCMS will pass the Nitrogen background test and be available to start the analytical sequence within 20 minutes of loading the wake method.



#### Programmable Helium Conservation Module <u>Perfomrance Testing</u>: Switching from Helium to Nitrogen

#### Running Helium only

Running Nitrogen in the injection port overnight



Running over a weekend with Nitrogen purge in the inlet shows no backdiffusion of Nitrogen into the MS

The Measure of Confidence



# Performance: No Change in Chromatography After N<sub>2</sub> Carrier Sleep Method (GC/FID)





#### Performance: Pass MS Tune Within 15min After Switching From N<sub>2</sub> to He as Carrier (GC/MSD)



		Counts of Nitrogen Ion		
		Relative to		Relative to
Time (min)	5 mL/min He	Saturation	2 mL/min He	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%



#### **Carrier Gas Savings Calculator**

Save Helium by > 90% compared to a typical use

Helium supplies last MUCH longer

- ✓ Gas saver mode: 9 times longer
- $\checkmark$  N<sub>2</sub> standby: another 3.3 times more
- ✓ Total save: last 30 times longer





#### **Carrier Gas Savings Calculator**

Change values in gray boxes to calculate savings for your operating parameters

Method: ASTM D7504 - Analysis of Trace Impurities in Monocyclic Column: HP-INNOWax, 30 m x 0.32mm ID x 0.25 um

#### **Gas Flow Conditions**

He Carrier Flow (mL/min):	1.2
He Split flow (mL/min):	190
Gas Saver Flow (mL/min):	20
Gas Saver On (min):	3
Run Time(min.):	30
Gas Volume in Cylinder (L):	8000
Runs per Day:	15
He Cylinder Cost (\$):	300
N2 Cylinder Cost (\$):	60

Parameter	No Gas Saver	Gas Saver On	Gas Saver + N₂Standby
Daily He Usage (L)	275	30	9
He Cylinder Life (days)	29	265	873
Daily N₂ Usage (L)	0	0	21
N <sub>2</sub> Cylinder Life (days)	0	0	381
Yearly He Cost (\$)	3,769	413	125
Yearly N2 Cost (\$)	0	0	57
Yearly Total Gas Cost (\$)	3,769	413	183



## **Option 2: Hydrogen Carrier**

Hydrogen is great for many GC methods such as ECD and FID.

Many people have been successful switching their GCMS methods to Hydrogen carrier gas as well

- Vince Marti at Region 8 lab for 8270 and 8260
  Vince uses cylinder Hydrogen
- Lisa Wool at Region 6 for Pesticide's, PCB's, TPH
  Lisa uses a Hydrogen generator



## **Option 2: Hydrogen Carrier**

However, Hydrogen is not the best gas for GCMS methods. Depending on the method requirements

- The main issue with Hydrogen carrier is that it is not inert
- There is some reactivity with specific compounds
- Reactivity with Dichloromethane forms HCL in the GC inlet (Vince Marti in Region 8 discovered cooler inlet temps avoid this problem)
- BFB tuning anomalies can occur (ion 96)
- DFTPP tuning seems to be less effected



#### **Pertinent Application Notes**

# Reducing Helium Costs more than 10-fold with the PCT Gas Saver mode

Agilent Technologies, Inc., April 1, 2010

5990-5444EN

#### **Prevent Business Disruptions by Managing your Helium Usage**

Agilent Technologies, Inc., April 30, 2013 5991-2256EN

# Successfully converting from Helium to alternate gases when analyzing VOC's following EPA 524.2 methodology

Agilent Technologies, Inc., August 2013



#### **Original Designers of Each System**

Fred Feyerherm, PhD

Harry Prest, PhD

Jim McCurry, PhD

**Bruce Quimby, PhD** 



