

# Habits for a Healthy GC: Insider Tips

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### Is it time for maintenance?



# How often should I complete routine maintenance?

#### **Less Often**



#### Typically, how dirty or concentrated are your samples?



**More Often** 

#### Problem

- "Dirty" samples can contain
  - Nonvolatile/high-boiling material
  - Sediment and solids
  - Problematic matrix
- Don't always LOOK dirty
- Build up in liner, head of column

#### Prevention

- Sample cleanup and/or filtration
- Dilute
- Inject less



#### **Recommended Maintenance Tasks**

#### Often

- Replace wash solvents OFTEN
- Every day to once a week
- depending on number of samples/solvent run per day
- Clean out wash vials OR use new ones
- Replace inlet septum, liner, and trim column
- Depends on sample types
- Dirty matrices: 2 5 days
- Clean matrices: 2 weeks 1 month +
- Mixture of samples from clean to dirty: 1 2 weeks

#### Sometimes

- Clean the inlet body with cotton tipped swabs and solvents (do not use abrasives)
- Replace gold seal (and its washer)
- Depending on sample type
- Change the split vent trap

#### When Needed

- Replace the column
- Clean detector (if applicable/possible)
- Change gas filters



#### GC maintenance decision tree





# I'm so busy, I often forget to maintain my GC.



#### Solution: Use your software to set up maintenance counters & warnings Also known as EMF, or Early Maintenance Feedback

#### OpenLab CDS ChemStation Edition:

😐 C	urie (online): Method and Run Control	
File	RunControl Instrument Method Sequer	nce Backflush Wizard RTLock RTSearch View Abort Help
	Select Injection Source	Sequences 🔚 🛃 ECD_washes.S
	Edit Agilent 7890B Parameters	Last Run 0.0 Method: off 6
	Instrument Utilities	
	Columns	ent Control Run Queue Easy Sequence Easy Sequence Setup
	Snapshot	eport
	Instrument Configuration	a File C:\Chem32\1\Data\ECD_washes 2017-12-:
	Manage Rules and Alerts	ple Name: ED_washes_B_2
	Acquisition Method Viewer	
	Agilent 7890B Configuration	Acq. Operator : SYSTEM
	Unload method from Agilent 7800P	Acq. Instrument : Curie
	Start Calvera Componentian Run	Injection Date : 12/21/2017 11:40:37 AM
	Start Column Compensation Run	Sequence File : C:\Chem32\1\Data\ECD was}
	Sieep	Method : C:\Chem32\1\Data\ECD was}
_	Wake Up	Sequence Method)
_	Maintenance	Last changed : 12/21/2017 10:49:23 AM b
	Extend Run	Sample Info : 2 sample washes to look a
	Parts Finder	post DW align
		ECD2B, Back Signal (ED_washes_B_a2.D)
	· · · ·	16000 -
		14000 -
		12000 -

MassHunter Data Acquisition:



## Getting to know your GC's EMF

Note 3 primary tabs for counters, log, and actions

Maintenance tab for each of the GC's configured zones

Shows injection total, age, limits, and status for serviceable parts within configured zone

Sentum injections	160 Injections		
	100 11/001010		
Liner injections	168 Injections		·
Details Hide this EMF	Warning Threshold	Service due Threshold	
	✓ Enable	⊡ Enable	Remind yourself
Reset EMF			configuring warn
Set default recommended values for	or limits Set	200 Set	and service due l
Gold seal injections	168 Injections		<u>+</u>
Liner O-ring injections	168 Injections		<u>+</u>
Split vent trap injections	168 Injections		+
Gold seal age	3 : 7 (Months : Hrs)		+
Liner age	3 : 7 (Months : Hrs)		+
O-ring age	3 : 7 (Months : Hrs)		+

#### I don't see the EMF I need Solution: Add a custom EMF to meet your needs

Maintenance	Add a new user defined counter	y Sequence Setup
Early Maintenance Feedback Counters Maintenance Log Maintenance Action	Name	
Front Inlet V Back Inlet Front Detector Back Detector	Syringe life Names can contain A-Z, a-z, and 0-9 only.	Sequences (The current method has been modified. Save or discard the changes.)
injections 166 Injections	Should track	Time entered into Queue     Estimated Completion Time     Status
Hide this EMF Warning Threshold	Add Cancel	
Reset EMF 2000	D.M	Inlet Inlet Back Detector Back Injector Column 1 Column 2 Instrument
Oven cycles 177 Injections	injections Details Hide this EMF	166     Injections       Warning Threshold     Service due Threshold       Image: Color package     Image: Color package
Length     30     m       Add User Defined Counter     Hide selected counters on this page	C_washes.M Reset EMF	2000 Set 2500 Set
	Oven cycles	177 Injections +
	Conline Plot	30 m +
	ECD ECD Hz - -	er Hide selected counters on this Show all hidden counters Print Options
	e000-	
	5000-	



## Instrument EMF tab

	Maintenance					
	Early Maintenance Feedback Counters Maintenance Log Maintenance Actions					
Set EMF for gas filters,	Front Inlet V Back Inlet V Front Detector V Back Detector V Back Injector V Column 1 V Column 2 Instrument					
filters	Instrument On Time 1 : 1 (Months : Weeks) +					
	Instrument Run Count 179 Injections +					
	Gas Filter Maintenance age 3 : 1 (Months : Days) +					
	Add User Defined Courter         Hide selected counters on this page         Show all hidden counters         Print         Options					
	Be careful with these buttons – They reset the counters!					

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#### When you reach or pass the warning level...





## After maintaining your system...

	Maintenance Early Maintenance Feedback Counters Maintenance Log Maintenance Actions			
	Front Inlet Back Inlet From SS Inlet	ont Detector 🥥 Back Detector 🌒 Back Inje	ctor 🥥 Column 1 🎱 Column 2 🌒 Instrument	
	Septum injections	168 Injections		+
Remember to reset your EMF	Liner injections	168 Injections		·
	Details	Warning Threshold	Service due Threshold	
		Enable	Imable	
	Reset EMF	limits Set	200 Set	
	Gold seal injections	168 Injections		+
	Liner O-ring injections	168 Injections		+
	Split vent trap injections	168 Injections		+
	Gold seal age	3 : 7 (Months : Hrs)		+
	Liner age	3 : 7 (Months : Hrs)		+
	O-ring age	3 : 7 (Months : Hrs)		+
	Split vent Trap age	3:7 (Months:Hrs)		+
	Add User Defined Counter	Hide selected counters on this Show all hi	idden counters Print Optio	ons
	C			



• Whether split or splitless injection type, all noninjected sample effluent exits via split vent

Mode: Splitless	Purge Flow to Split Vent: 100 mL/min	at 1 min	Splitless: This method specifies after 1 min. of injection, the split vent opens and any remaining sample purged
Mode: Split	Split Ratio: sample 50 Unused sample :1 S	Split Flow 55.198 mL/min	Split: sample effluent purged via split vent

Purged sample exits via split vent line, filtered by split vent trap





- Dirty samples and high boiling material can clog line and/or saturate split vent trap
- Recommend changing every 6 months, more frequently if running dirty samples
- Symptoms of a clogged split vent trap/contaminated split vent line:
- GC stuck in stand-by or "not ready"
- Elevated baseline
- Contamination, ghost, and bonus peaks





T20 Torx Driver - - <u>5182-3465</u> **Split vent trap-**1pk: <u>5188-6495</u> 2pk: <u>G1544-80530</u>









#### Other good habits

- Don't Overload your Column!
  - Watch for highly concentrated samples
  - Don't inject too much sample
- How to tell?
  - What is your injection volume?
  - Look at peaks in the chromatogram
    - (and/or EIC, if using GC/MS)



#### Important, good habits for a happy GC and analyses



- Check installation depth for Split/Splitless inlets
- 4-6 mm
- Check installation depth for MMI inlets
  - 12-14 mm (updated recommendation from 11mm)

- Ferrule keep slipping?
- Use a column installation tool to increase ferrule "bite"
  - Flexi-metal ferrules pre-swage and hold correct inlet depth
  - Use with deep well column nut at inlet and detector interfaces
  - Install with GC column installation/metal ferrules pre-swaging tool (G3440-80218)
  - Using graphite ferrules?
  - Use GC column installation/graphite ferrules pre-swaging tool (G3440-80217)





#### Nonpolar Column Test Mix



Name	Exp. RT	Act RT	Area	Amount	Peak Symmetry	СТQ
Total Area Count		0		100		
Decane %	1.49	1.506	790.25	28.056	1.038	
1-Octanol %	1.92	1.89	640.94	22.755	0.987	
2,6-DMP %	2.43	2.378	660.99	23.467	0.877	
2/6 DMA Retention min	2.83	2.849	724.48	25.721	0.981	
		Total Area Count	2816.668			

- QC standard containing 4 analytes, approximately equal quantities
- Auto-scheduled weekly via LIMs
- Monitor over time:
  - Retention time
    - Look for retention shifts
  - Area count
    - Watch for response loss
  - Peak symmetry
    - (1 = symmetric, no tailing)
- Custom report alerts operator of problematic results
- Use LIMs to control chart results
  - Monitor performance over time
  - Establish upper/lower control limits
  - Take action when parameter out of specification
- Rerun standard after maintenance to reestablish expected performance values



Calendar

#### Nonpolar Column Test Mix- Out of Spec Results



Name	Exp. RT	Act RT	Area	Amount	Peak Symmetry	СТQ
Total Area Count		0		100		
Decane %	1.49	1.492	365.51	2 0	1.058	
1-Octanol %	1.92	1.86	309.36	22.823	0.822	
2,6-DMP %	2.43	2.334	331.43	24.451	0.650	Bad Symmetry - Pass Scan to Day Tech
2/6 DMA Retention min	2.83	2.796	349.19	25.761	0.640	Bad Symmetry - Pass Scan to Day Tech
		Total Area Count	1355.493			

			Before- 7/2017
Act RT	Area	Amour	it Peak Symmetry
0		100	
1.506	790.25	23.056	1.038
1.89	640.94	22.755	0.987
2.378	660.99	23.467	0.877
2.849	724.48	25.721	0.981
Total Area Count	2816.668		

Retention time earlier- column trim between then and now?

~50% loss in response- perhaps poorly installed column?

2,6-DMP tailing peak- time to change inlet liner, gold seal?



#### Environmental Pesticides Probes: Endrin/DDT breakdown





#### **Environmental Probes: Endrin DDT breakdown**



#### Environmental Probes: Endrin DDT breakdown

Pre maintenance Endrin % breakdown: **22.5** % DDT % breakdown: **3.3** %

Post maintenance Endrin % breakdown: **5.7%** DDT % breakdown: **1.1%** 



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#### Addressing Endrin DDT Breakdown

- Complete routine inlet maintenance
  - Replace liner and septum and especially trim head of column if you observe increased DDT breakdown



# Particles Particles







#### Addressing Endrin DDT Breakdown

- Check vial caps is the syringe puncture near the center of vial cap? Or closer to the edge?
- Check alignment of ALS tower and look at syringe needle
- Do you see black/gray color around the puncture mark in vial cap? syringe may be impacting septum nut needle guide cone
  - Replace syringe guide ("foot"), syringe and/or septum nut

Part	Agilent Part Number	Take Action/Replace If
Septum Nut	<u>18740-60835</u>	<ul><li>Gouge marks are observed</li><li>Contamination buildup</li></ul>
ALS syringe	10 μL tapered- <u>5181-1267</u> 10 μL tapered, PTFE tip- <u>5181-</u> <u>3354</u>	<ul> <li>Gouge marks observed on septum nut</li> <li>Needle bent or looks pitted</li> </ul>
ALS needle guide	7693 ALS- <u>G4513-40525</u>	<ul><li>Plastic looks worn or deformed</li><li>Contamination buildup</li></ul>





#### **Troubleshooting starting points**





# Thank you for your attention!



