Benefits of Using Desorb Flow Control with the Encon Evolution

Application Note

Environmental

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Abstract:

Desorb flow control was developed in order to help manage the moisture associated with the four minute desorb time required for USEPA method 524.2. An added benefit to this process is the reduction in helium consumption when using this technique. This application will explain the patented process of Desorb Flow Control (DFC) (United States Patent Office numbers: 8062905, 7951609, 7803635) for Helium conservation and moisture control.

Introduction:

Helium has been an essential component in environmental testing for years. Since Helium is an inert gas with a similar diffusion speed as Hydrogen, it is ideal for using as a carrier gas in Gas Chromatography. In recent years, however, there has been an increasing shortage of Helium, leading laboratories to come up with different ways of conservation.

Desorb Flow Control was developed to assist laboratories routinely running for USEPA Method 524.2. Method 524.2 requires a four minute desorb and laboratories are reporting issues with water when using the long desorb time. By using DFC, labs are now able to keep the required four minute desorb time and control the amount of water transferred to the GC by decreasing the flow through the trap during desorb while maintaining the desired split ratio at the GC inlet. Subsequently, DFC not only controls the amount of water transferred, but also decreases Helium consumption. See Figures 1 and 2.

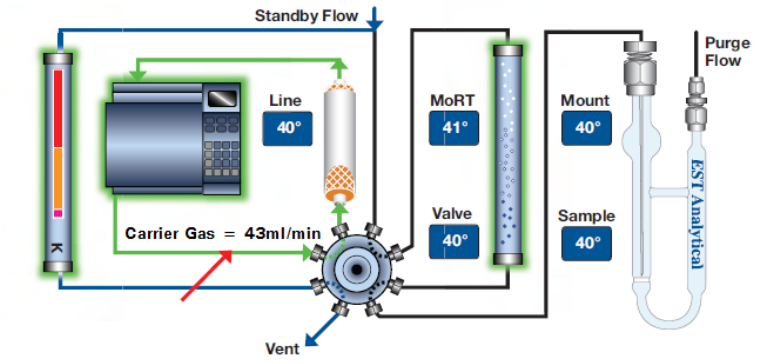

Figure 1: Traditional GC Flow

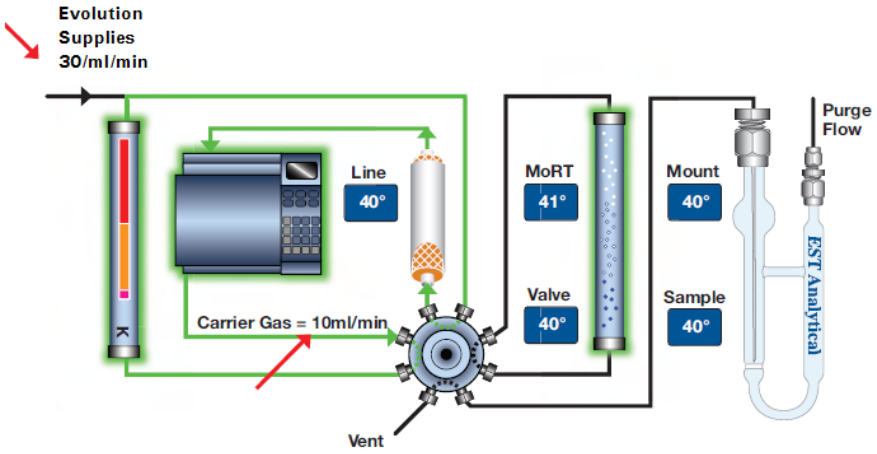

Figure 2: GC Flow with Encon Evolution using DFC

(NOTE: The Encon Evolution provides the increased flow after the trap during desorb at a set time in the desorb process.)

The advent of more efficient purge and trap systems and more sensitive GC/MS systems has aided in laboratories' production. However, water control is still an issue. Labs may pass an initial calibration curve, but as water builds up in the system, internal standard response drops over time, causing the laboratory to fail continuing calibration checks. In order to combat water, many labs use a high split rate. As a consequence, Helium consumption is much higher and sensitivity is decreased. Using DFC, labs can still achieve the moisture control required thus creating a more stable system and lowering Helium consumption by as much as 80%, depending on the experimental split rate. See Table 1. *The table below assumes a throughput of 72 samples per day.

Without DFC	Without DFC	With DFC
Desired Split Rate = 100:1	Desired Split Rate = 40:1	Desired Split Rate = 40:1 or100:1
Column Flow = 1ml/min	Column Flow = 1ml/min	Column Flow = 1ml/min
GC Split Flow = 100ml/min	GC Split Flow = 40ml/min	GC Split Flow = 43ml/min for 4.0min of Desorb
GC Total Flow = 103ml/min	GC Total Flow = 43ml/min	GC Total Flow = 13ml/min
20 min Cycle Time = 2060ml He/run	20 min Cycle Time = 860ml He/run	20 min Cycle Time = 380ml He/run
Daily Consumption = 148320ml	Daily Consumption = 61920ml	Daily Consumption = 27360ml

Table 1: Daily Helium Consumption

Experimental:

The sampling system used for this study was the EST Analytical Encon Evolution concentrator and the Centurion WS autosampler. The concentrator was affixed with a

Vocarb 3000 trap and connected to an Agilent 7890A GC and 5975C inert XL MS. The GC was configured with a Restek Rxi-624 Sil MS 30m x 0.25mm x 1.4μm column. Two different split ratios were used for comparison in this study, a 40:1 split rate was used for the baseline data and a 10:1 split rate was used for the DFC data. Refer to Table 2 for the sampling method parameters and Table 3 for GC/MS parameters.

Purge and Trap Concentrator	EST Encon Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	150ºC
Transfer Line Temp.	150ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp.	39ºC
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Flow Control	On (Program)
Desorb Pressure Control	On
Desorb Pressure	13.5psi
Desorb Time	4.0 min
Desorb Preheat Delay	10 sec
Desorb Temp.	250ºC
Moisture Reduction Trap (MoRT) Bake Temp.	210ºC
Bake Temp	260ºC
Sparge Vessel Bake Temp.	110ºC
Bake Time	8 min
Bake Flow	85mL/min
Purge and Trap Auto-Sampler	**EST Centurion WS**
Sample Type	Water
Water Volume	25ml
Internal Standard Vol.	5μl
Desorb Flow Control	**EST Encon Evolution**
Enable Ramp Control	On
Initial Pressure	13.5psi
Initial Hold Time	1.5min
Ramp Rate	10psi/min
Final Pressure	15.0psi

Table 2: Purge and Trap Parameters

GC/MS	Agilent 7890A/5975C inert XL
Inlet	Split/Splitless
Inlet Temp.	200°C
Inlet Head Pressure	7.45 psi
Mode	Split
Split Ratio	40:1 and 10:1
Column	Rxi-624Sil MS 30m x 0.25mm I.D. 1.4μm film thickness
Oven Temp. Program	40°C hold for 1.5 min, ramp 8°C/min to 100°C, ramp 20°C/min to 210°C, hold for 1.25 min, 16.5 min run time
Column Flow Rate	1mL/min
Gas	Helium
Total Flow	13.8mL/min and 43.8mL/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 35-300
Scans	3.12 scans/sec
Solvent Delay	1.0 min

Table 3: GC/MS Experimental Parameters

The USEPA Method 524.2 standards were acquired from AccuStandard. The linear ranges of the experiments were established by running eight point calibration curves from 0.5 to 100ppb. Table 4 displays curve linearity and compound response for the curves. Figure 3 displays chromatograms of the 20ppb calibration point with and without DFC. Finally, an experiment was run using Selective Ion Monitoring (SIM) of m/z 20 with and without DFC. This was done in order to show water control using D2O as the compound of interest so as not to SIM for water and overwhelm the MS. Figure 4 displays the results of this experiment.

Calibration Curve Results				
Compound	40:1 Split, No DFC		10:1 Split, DFC	
	Curve %RSD	Curve RF	Curve %RSD	Curve RF
dichlorodifluoromethane	9.67	1.373	13.86	1.272
chloromethane	5.64	1.679	8.28	1.555
vinyl chloride	5.02	1.591	6.07	1.432
bromomethane	9.39	0.791	10.86	0.890
chloroethane	6.12	0.772	6.02	0.723
trichlorofluoromethane	5.41	2.091	3.99	2.123
1,1-dichloroethene	5.55	1.021	5.18	1.042
methyl iodide	*0.998	1.117	*1.000	1.043
carbon disulfide	5.24	3.157	5.08	3.228
methylene chloride	8.36	0.904	9.15	0.917
methyl-t-butyl ether (MtBE)	6.21	1.601	8.12	1.517
1,1-dichloroethane	4.17	2.186	4.76	2.199
2-butanone	10.87	0.210	9.99	0.181
chloroform	6.72	1.878	10.36	1.935
2-chloroethylvinylether	6.25	1.079	5.62	1.061
benzene	5.20	4.162	4.18	4.151
1,2-dichloropropane	6.25	1.079	5.62	1.061
4-methyl-2-pentanone	10.98	0.383	6.30	0.346
toluene	6.39	2.571	7.29	2.681
2-hexanone	8.25	0.236	13.04	0.212
chlorobenzene	6.92	3.033	4.50	3.112
ethylbenzene	9.51	5.152	12.09	5.115
xylene (m + p)	7.45	4.077	8.37	4.117
xylene (o)	8.10	4.102	6.83	4.154
bromoform	8.62	0.401	6.06	0.394
1,1,2,2-tetrachloroethane	8.49	0.513	13.83	0.491
1,2-dibromo-3-chloropropane	10.04	0.098	12.29	0.091
1,2,4-trichlorobenzene	9.07	1.521	8.32	1.378
napthalene	12.73	1.789	14.83	1.547
hexachlorobutadiene	5.44	0.870	4.90	0.831
1,2,3-trichlorobenzene	8.56	1.229	8.52	1.120
Average	7.55	1.70	8.14	1.67

Table 4: Curve Linearity and Compound Response Summary

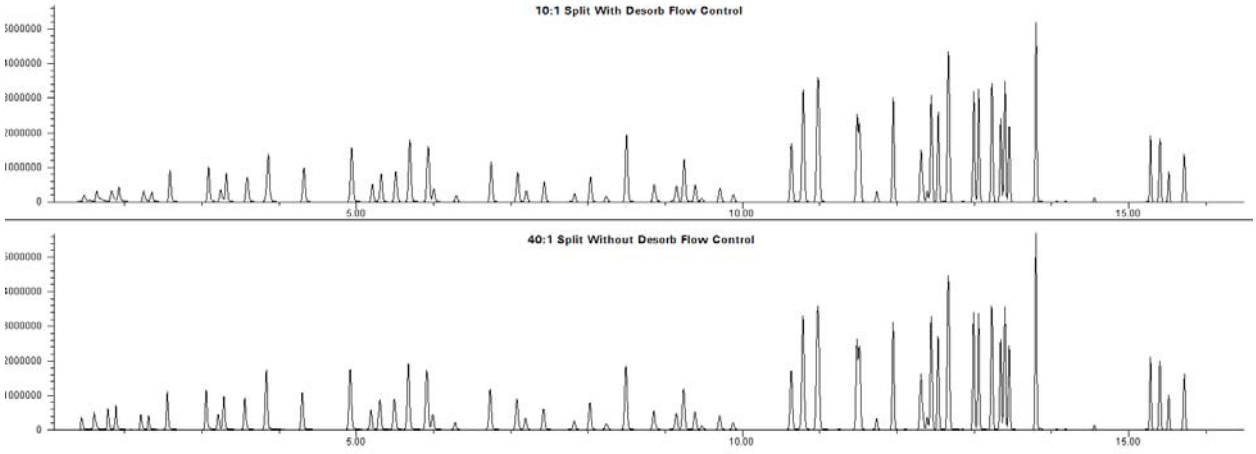

Figure 3: 20ppb Chromatograms With and Without DFC

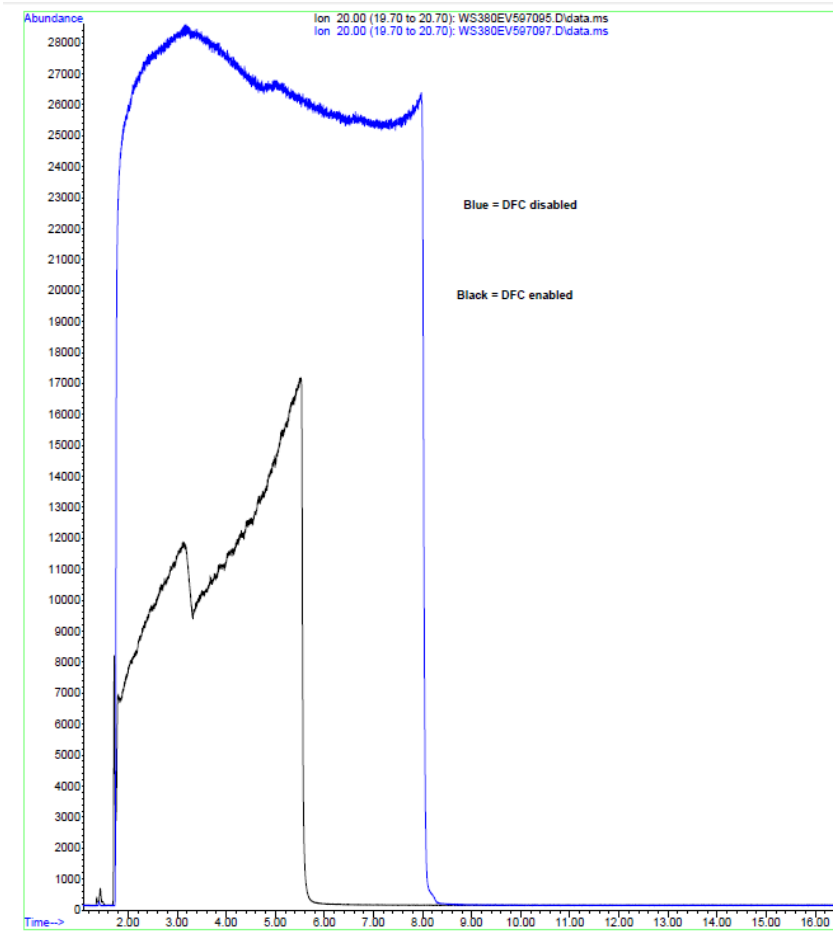

Figure 4: Overlay of D2O m/z 20 With and Without DFC

Conclusions:

The results of this study show the patented Desorb Flow Control is an exceptional tool for conserving helium and creating a more stable system. The option of maintaining the desired split ratio at the GC inlet while decreasing the flow through the trap during desorb provides sensitivity while controlling the amount of moisture being sent onto the GC column. Furthermore, the split rate during the GC/MS run time is substantially lower than during the desorb time. Laboratories can have the advantage of running a higher split rate during the desorb process then maintaining a lower split during the rest of the GC/MS separation and analysis, thus providing laboratories with up to an 80% reduction in GC/MS helium use.

For More Information

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