

## Methods 8260C and 8270D on a Single GCMS without Changing Columns

### ■ Introduction

USEPA Methods 8260C (volatile) and 8270D (semi-volatile) typically require dissimilar chromatographic columns with incompatible maximum temperatures. Historically, laboratories performing these analyses have found it necessary to allocate separate GCMS instrumentation for the two analysis modes, or perform frequent reconfiguration (column changing) to run these methods on a single GCMS unit. Breaking the MS vacuum to change analytical columns is time-consuming and inefficient, however productivity can be improved significantly using the Shimadzu Twin Line MS Kit. With this configuration, the user can easily switch between applications without breaking vacuum to change the analytical column.

The Twin Line MS Kit configuration incorporates two injectors and two chromatographic columns simultaneously connected to a single MS. Carrier gas flow is always supplied to both columns to prevent damage to the column not in use. The Twin Line MS Kit represents a

### ■ Instrument Configuration

The analyses were conducted using a Shimadzu GCMS-QP2010 SE shown in Figure 1. The GCMS was operated in full-scan mode according to the operational parameters specified in USEPA Methods 8260C and 8270D. The instrument was configured with the Shimadzu Twin Line MS Kit: two chromatographic columns were connected to two split-splitless injectors, and both of the

viable, simple option for improved efficiency for those laboratories with limited instrument resources; it is well suited to this combination of methods for several reasons:

- The GCMS-QP2010 SE high-capacity vacuum system can accommodate flows from two chromatographic columns simultaneously without impacting MS performance,
- The user can switch between USEPA Method 8260C and USEPA Method 8270D without breaking the MS vacuum to change columns, and
- The chromatographic column for volatile organics has a 320 °C maximum temperature that is compatible with the higher-temperature analysis conditions for semi-volatiles analysis.

Data are presented illustrating performance of USEPA Methods 8260C and 8270D when run on a single GCMS with the Twin Line MS Kit.

columns were interfaced to a single MS by means of a special adaptor and a two-hole ferrule. The Twin Line MS Kit column connection is shown in Figure 2. Volatile analysis by USEPA Method 8260C was performed using an EST Encon Evolution purge-and-trap concentrator with Centurion autosampler operated in the water mode.



Figure 1: GCMS-QP2010 SE

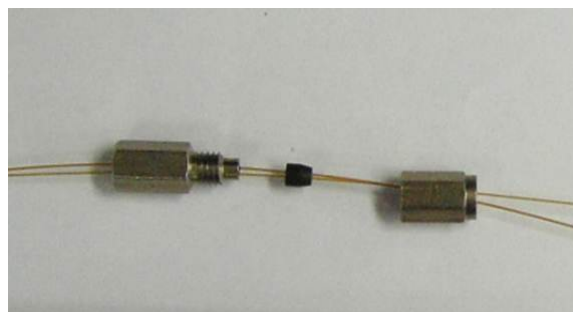


Figure 2: Twin Line MS Kit Column Connections with 2-Hole Ferrule

Instrument conditions for the analyses were based on those recommended for USEPA Methods 8260C and 8270D. Carrier gas flow was supplied to both analytical columns at all times to prevent column degradation; the column not being used for the analyses was operated in the constant linear velocity mode at 30 cm/sec. Narrow-bore columns (0.18-mm) were chosen for this application to minimize total column flow, but the use of

0.25-mm columns is also possible. The capacity of the GCMS-QP2010 SE vacuum system allows a total flow of 4 mL/min. Under the conditions specified below, total flow of both columns was slightly greater than 1 mL/min. Specific instrument conditions for the EPA Methods 8260C and 8270D analyses are shown in the respective sections below.

## Volatile Organics by USEPA Method 8260C

### ■ Experimental

Instrument configuration and operating conditions for analysis of Volatile Organics by USEPA Method 8260C are shown in Table 1, below.

**Table 1:** Instrument Operating Conditions for Volatile Analysis by USEPA Method 8260C

Gas Chromatograph	GC-2010 Plus
Column	RXI-624Sil MS 20 m x 0.18 mm x 1.0 µm (Restek Corp. #13865)
Oven Program	35 °C (hold 0.5 min.); 20 °C/min. to 220 °C (hold 2.5 min.)
Injector	Split mode, split ratio 75:1 225 °C Multi-purpose split liner with glass wool removed (Shimadzu 220-90784-00)
Primary Column Carrier Gas 8270 Column Carrier Gas	Helium; constant linear velocity 45 cm/sec Helium; constant linear velocity 30 cm/sec Maximum total flow for both columns = 1.3 mL/min
Interface Temperature	225 °C
Mass Spectrometer	GCMS-QP2010 SE
Ion Source Temperature	200 °C
MS Operating Mode	Full scan mode, m/z 36-260, scan rate 0.10 sec/scan (the scan rate was adjusted to provide a minimum of 10-12 spectra across all GC peaks)
Purge-and-Trap Concentrator	EST Encon Evolution with Centurion Autosampler
Sample Volume	5 mL
Trap	VOCARB 3000
Purge Flow Rate	Helium, 40 mL/min for 11 min
Dry Purge	Helium, 50 mL/min for 3 min
Desorb	260 °C for 0.5 min
Bake	260 °C for 10 min
Analysis Times	
GC Run Time	12.5 minutes
System Cycle Time	30 minutes

### ■ Results and Discussion

#### MS Tuning for Volatile Organics

The GCMS-QP2010 SE was tuned with perfluorotributylamine (PFTBA) using the Shimadzu GCMSsolution automatic tuning function; the tune evaluation standard of 25 ng 4-bromoflourobenzene (BFB)

was used to verify the tuning requirements specified in USEPA Method 8260C. All tune criteria for USEPA Method 8260C were met. Tuning results for BFB are shown below in Figures 3A and 3B.

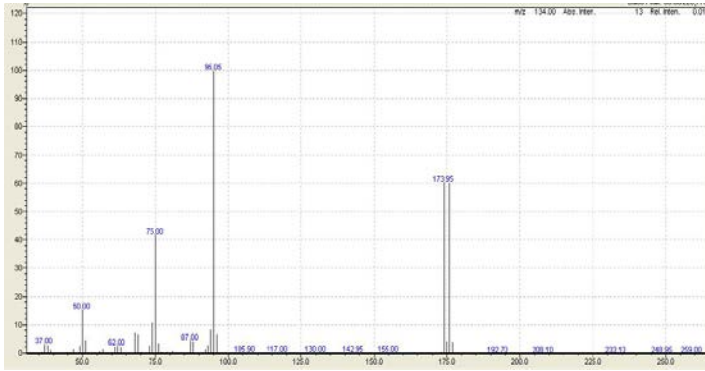


Figure 3A: BFB Mass Spectrum

Data		Data File Path	
Data: C:\GCMSsolution\Data\8260 Test for Twin-Line\VOA ICA			
4-Bromofluorobenzene			
m/z	Spectrum Check Criteria	Result	Status
50	15 to 40% of mass 95	15.325219	Pass
75	30 to 60% of mass 95	41.968955	Pass
95	Base Peak, 100% Relative Abundance	100.000000	Pass
96	5 to 9% of mass 95	6.736341	Pass
173	< 2% of mass 174	0.694586	Pass
174	> 50% of mass 95	60.567094	Pass
175	5 to 9% of mass 174	7.049975	Pass
176	> 95% but < 101% of mass 174	99.366573	Pass
177	5 to 9% of mass 176	6.571022	Pass

Figure 3B: BFB Tune Evaluation

### Calibration and Determination of Precision, Recovery, and MDLs for Volatile Organics

Calibration standards were prepared over the calibration range of 0.5-200 µg/L (parts-per-billion, ppb) and transferred to 40-mL VOA vials for sampling and analysis. Internal standards were held constant at 25 µg/L, and were added automatically with the Centurion autosampler. The initial calibration standards were analyzed using the instrument conditions outlined above. The detector (electron multiplier) voltage was adjusted to provide adequate response at the lowest calibration level and avoid saturation at the highest calibration level. Figure 4A shows the total ion chromatogram of a 10 µg/L standard; Figure 4B illustrates the chromatography of the early-eluting compounds in the 1.0 µg/L standard.

The data for the initial calibration were processed using GCMSsolution software. The calibration data were evaluated both by examination of the %RSD of the relative response factors and by the linearity (correlation coefficient) of the calibration curves for all of the target

analytes. For most of the target compounds, the %RSD of the relative response factors was 10-15% for the 0.2-200 µg/L calibration range. For those target compounds with %RSD >15%, the linear calibration was used. In all cases, the correlation coefficient was greater than 0.99, demonstrating linear response for the target compounds. The calibration data satisfied the USEPA Method 8260C calibration acceptance criteria. Calibration results are summarized in Table 2, below.

Analytical precision, method detection limits (MDLs), and compound recoveries were determined following the procedures referenced in USEPA Method 8260C. Eight aliquots of the 0.5 µg/L calibration standard were analyzed, analyte concentrations determined, and the MDLs calculated. Statistically-derived MDLs for most of the compounds were below 0.1 µg/L. Method detection limit results are presented in Table 2, below.

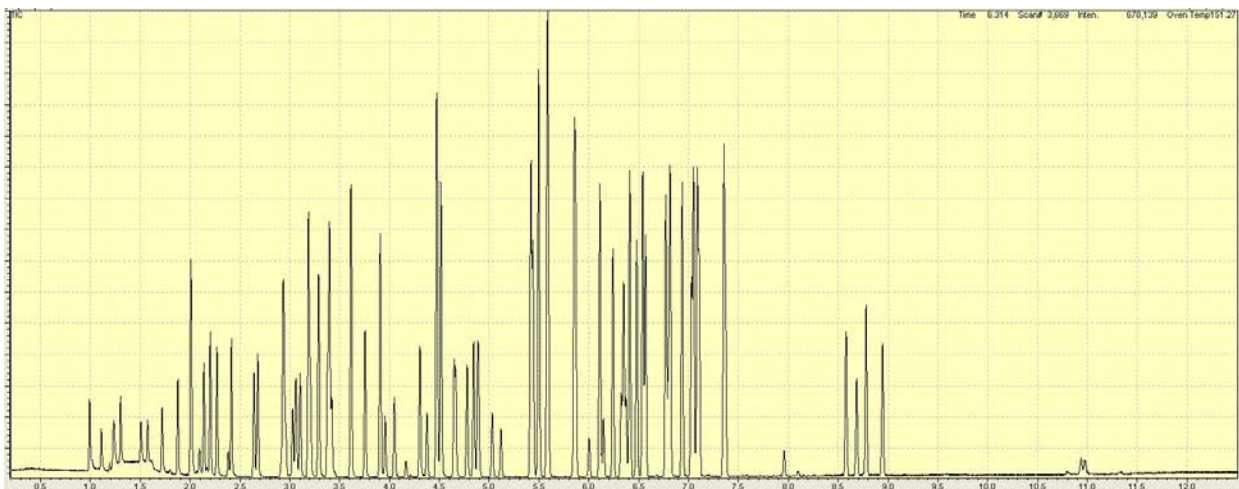
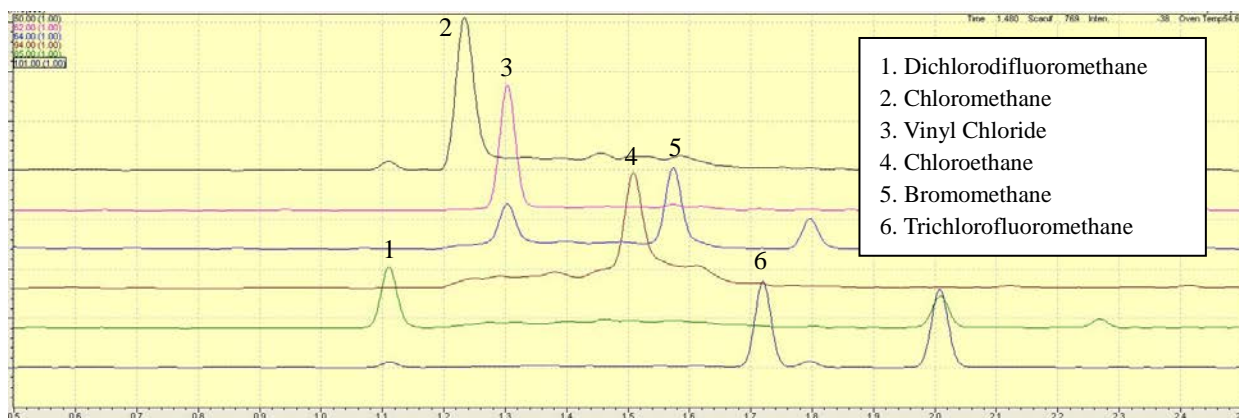


Figure 4A: Total Ion Chromatogram of the 10 µg/L Calibration Standard



**Figure 4B:** Mass Chromatograms of Early-eluting Compounds in 1.0 µg/L Calibration Standard

**Table 2:** Calibration Results, and Determination of Precision, Recovery, and Method Detection Limits for Volatile Organics by USEPA Method 8260C

Compound Name	Calibration Results			Determination of Precision, Recovery, and Method Detection Limits (n = 8)				
	Mean RRF	RSD (%)	r	Average Concentration (µg/L)	RSD (%)	Spike Level (µg/L)	Recovery (%)	MDL (µg/L)
Dichlorodifluoromethane	0.171		0.999	0.619	4.5	0.5	124	0.09
Chloromethane	0.343	3.8		0.600	5.5	0.5	120	0.11
Vinyl Chloride	0.308	9.2		0.614	7.3	0.5	123	0.15
Bromomethane	0.179		0.999	0.574	5.2	0.5	115	0.10
Chloroethane	0.184	7.4		0.574	6.0	0.5	115	0.11
Trichlorofluoromethane	0.244	12.9		0.565	6.0	0.5	113	0.11
1,1-Dichloroethene	0.256	13.4		0.483	5.3	0.5	97	0.08
Acetone	0.075		0.999	0.755	14.8	1.0	75	0.37
Iodomethane	0.195		0.999	0.485	16.3	0.5	97	0.26
Carbon Disulfide	0.830		0.999	0.530	5.9	0.5	106	0.10
Dichloromethane	0.306		0.999	0.443	13.6	0.5	89	0.20
trans-1,2-Dichloroethene	0.281	7.9		0.541	8.3	0.5	108	0.15
cis-1,2-Dichloroethene	0.310	5.7		0.568	5.4	0.5	114	0.10
2-Butanone (MEK)	0.122	9.2		0.842	9.6	1.0	84	0.27
1,1-Dichloroethane	0.143	4.7		0.605	4.2	0.5	121	0.08
2,2-Dichloropropane	0.325	6.5		0.539	10.1	0.5	108	0.18
Bromochloromethane	0.122	6.9		0.587	7.8	0.5	117	0.15
Chloroform	0.438	8.4		0.607	10.6	0.5	121	0.21
Carbon tetrachloride	0.271	10.7		0.639	3.1	0.5	128	0.07
1,1,1-Trichloroethane	0.321	7.8		0.593	6.0	0.5	119	0.12
2,3-Dichloropropene	0.351	5.8		0.524	7.1	0.5	105	0.12
1,1-Dichloropropene	0.351	5.8		0.644	8.1	0.5	129	0.17
Benzene	1.240	8.0		0.529	3.4	0.5	106	0.06
1,2-Dichloroethane	0.319	7.6		0.446	4.7	0.5	89	0.07
Trichloroethene	0.300	6.8		0.482	6.1	0.5	96	0.10

**Table 2:** USEPA Method 8260C Calibration Results (continued)

1,2-Dichloropropane	0.314	6.5		0.561	4.7	0.5	112	0.09
Dibromomethane	0.144	8.4		0.646	6.0	0.5	129	0.13
Bromodichloromethane	0.314	5.7		0.545	6.3	0.5	109	0.11
cis-1,3-Dichloropropene	0.450	5.3		0.564	6.7	0.5	113	0.13
4-Methyl-2-pentanone (MIBK)	0.105	4.0		0.580	4.4	0.5	116	0.08
trans-1,3-Dichloropropene	0.402	4.0		0.508	5.7	0.5	102	0.10
2-Hexanone	0.112	6.4		0.509	5.3	0.5	102	0.09
Toluene	0.861	3.2		0.628	5.6	0.5	126	0.12
1,1,2-Trichloroethane	0.205	3.2		0.550	5.0	0.5	110	0.09
Tetrachloroethene	0.220	14.9		0.509	7.3	0.5	102	0.12
1,3-Dichloropropane	0.445	2.6		0.564	2.7	0.5	113	0.05
Dibromochloromethane	0.246	3.2		0.511	6.5	0.5	102	0.11
1,2-Dibromoethane (EDB)	0.239	8.0		0.564	4.6	0.5	113	0.08
Chlorobenzene	0.933		0.999	0.481	4.8	0.5	96	0.08
1,1,1,2-Tetrachloroethane	0.256	3.9		0.477	2.6	0.5	95	0.04
Ethylbenzene	0.499	4.4		0.571	4.5	0.5	114	0.09
m,p-Xylene	0.622	2.6		1.220	4.9	1.0	122	0.20
o-Xylene	0.589	3.2		0.569	6.2	0.5	114	0.12
Styrene	0.906	3.5		0.559	5.5	0.5	112	0.10
Bromoform	0.134	14.9		0.596	4.9	0.5	119	0.10
1,1,2,2-Tetrachloroethane	0.303	4.5		0.483	5.8	0.5	97	0.09
Isopropylbenzene	0.397	4.9		0.534	4.9	0.5	107	0.09
n-Propylbenzene	0.412	3.5		0.545	5.5	0.5	109	0.10
Bromobenzene	0.283	3.5		0.574	7.3	0.5	115	0.14
1,2,3-Trichloropropane	0.081	4.5		0.592	9.9	0.5	118	0.19
4-Chlorotoluene	0.348	4.1		0.614	7.6	0.5	123	0.15
2-Chlorotoluene	0.359	3.5		0.596	7.5	0.5	119	0.15
1,3,5-Trimethylbenzene	1.394	4.8		0.569	5.8	0.5	114	0.11
1,2,4-Trimethylbenzene	1.295	4.4		0.571	7.2	0.5	114	0.14
tert-Butylbenzene	0.624	7.6		0.589	8.3	0.5	118	0.16
sec-Butylbenzene	0.700	6.2		0.525	8.1	0.5	105	0.14
p-Isopropyltoluene	0.791	7.2		0.531	6.8	0.5	106	0.12
n-Butylbenzene	0.736	5.3		0.544	7.0	0.5	109	0.13
1,3-Dichlorobenzene	1.381	3.0		0.603	5.2	0.5	121	0.10
1,4-Dichlorobenzene	1.510	12.3		0.644	2.7	0.5	129	0.06
1,2-Dichlorobenzene	1.257	2.5		0.560	3.8	0.5	112	0.07
1,2-Dibromo-3-chloro-propane	0.117	5.3		0.506	9.8	0.5	101	0.16
Hexachlorobutadiene	0.325	5.1		0.509	5.7	0.5	102	0.10
Naphthalene	2.239	4.3		0.554	5.3	0.5	111	0.10
1,2,4-Trichlorobenzene	0.709	4.5		0.587	10.0	0.5	117	0.19
1,2,3-Trichlorobenzene	0.632	5.5		0.573	8.9	0.5	115	0.17

## Semi-volatile Organics by USEPA Method 8270D

### ■ Experimental

Instrument configuration and operating conditions for analysis of Semi-volatile Organics by USEPA Method 8270D are shown in Table 3 below.

**Table 3:** Instrument Operating Conditions for Semi-volatile Analysis by USEPA Method 8270D

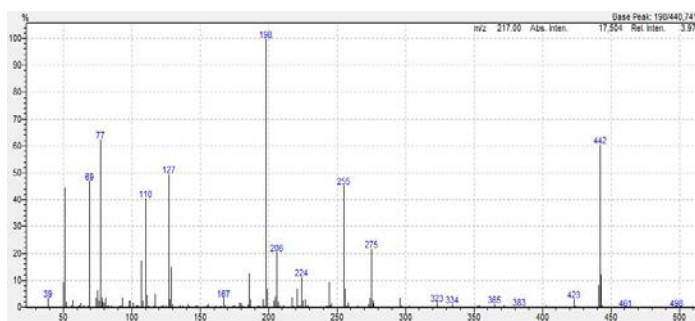
Gas Chromatograph	GC-2010 Plus
Column	RXI-5Sil MS 20 m x 0.18 mm x 0.18 μm (Restek Corp. #43602)
Oven Program	45 °C (hold 0.5 min.); 25 °C to 315 °C (hold 4.2 min.)
Injector	Split mode, split ratio 10:1 295 °C Multi-purpose split liner with glass wool (Shimadzu 220-90784-00)
Primary Column Carrier Gas 8260 Column Carrier Gas	Helium; constant linear velocity 40 cm/sec Helium; constant linear velocity 30 cm/sec Maximum total flow for both columns = 1.2 mL/min
Interface Temperature	320 °C
Mass Spectrometer	GCMS-QP2010 SE
Ion Source Temperature	220 °C
MS Operating Mode	Full scan mode, m/z 35-500, scan rate 0.10 sec/scan (the scan rate was adjusted to provide a minimum of 10-12 spectra across all GC peaks)
Analysis Times	
GC Run Time	16 minutes
System Cycle Time	22 minutes

### ■ Results and Discussion

#### MS Tuning and System Performance Checks for Semi-volatile Organics

The GCMS-QP2010 SE was tuned using the Shimadzu GCMSsolution automated tuning function; the tune evaluation standard of 25 ng decafluorotriphenylphosphine (DFTPP) was used to verify that the instrument met the tuning requirements specified in USEPA Method 8270D (Figures 5A and 5B).

Chromatographic column performance was evaluated by inspecting the tailing of pentachlorophenol and benzidine (Figures 5C and 5D), and measuring the breakdown of DDT to DDE and DDD (Figures 5E and 5F). The assessment of column performance was performed concurrently with the DFTPP tuning verification test.



**Figure 5A:** DFTPP mass spectrum

#### Spectrum Check

• Count 1

Data	Data File Path
Data1:	C:\GCMSsolution\Data\New SV Files\STune 11062012 - 1.QGI

• DFTPP

m/z	Spectrum Check Criteria	Result	Status
51	30 - 60 percent of mass 198	45.018730	Pass
68	Less than 2 percent of mass 69	1.805383	Pass
70	Less than 2 percent of mass 69	0.430219	Pass
127	40 - 60 percent of mass 198	49.672030	Pass
197	Less than 1 percent of mass 198	0.597403	Pass
198	Base peak, 100 percent relative abundance	100.000000	Pass
199	5 - 9 percent of mass 198	7.178139	Pass
275	10 - 30 percent of mass 198	21.701181	Pass
365	Greater than 1 percent of mass 198	1.324587	Pass
441	Present but less than mass 443	67.596655	Pass
442	Greater than 40 percent of mass 198	60.447292	Pass
443	17 - 23 percent of mass 442	20.019068	Pass

**Figure 5B:** DFTPP spectrum check

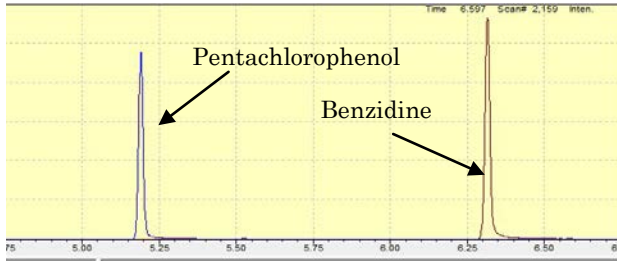


Figure 5C: Tailing of pentachlorophenol and benzidine

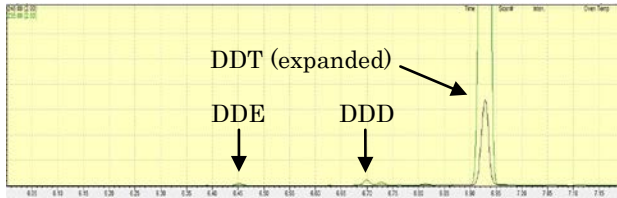


Figure 5E: DDT Breakdown

**Column Performance**

Count 1

Data	Data File Path	Sample Name	Sample ID
Data1	C:\GCMSolutionData\New SV Files\STune 11062012 - 1.QGD	STune	STune

[Results]

ID	Compound Name	Tailing F.	Tailing F. L. Limit	Tailing F. U. Limit	Tailing F. Status
1	DFTPP	0.861071	-	-	-
2	4,4'-DDE	0.725914	-	-	-
3	4,4'-DDD	0.912226	-	-	-
4	4,4'-DDT	1.093114	-	-	-
5	Pentachlorophenol	0.942667	0.900000	2.000000	Pass
6	Benzidine	0.957531	0.900000	2.000000	Pass

Figure 5D: Column performance check

**Degradation Check**

Count 1

Data	Data File Path	Sample Name	Sample ID
Data1	C:\GCMSolutionData\New SV Files\STune 11062012 - 1.QGD	STune	STune

Analyte#	ID	Compound Name	Area	m/z	%breakdown	%breakdown U. Limit	Status
4	4	4,4'-DDT	765677	235.02	0.428235	10.000000	Pass
4	2	4,4'-DDE	877	246.02			
4	3	4,4'-DDD	2416	235.02			

Figure 5F: Degradation check

### Calibration and Determination of Precision, Recovery, and IDLs for Semi-volatile Organics

Calibration standards were prepared over the calibration range of 0.4-160 µg/mL and transferred to 2-mL autosampler vials for analysis. Internal standards were held constant at 40 µg/mL. Data for the initial calibration standards were acquired using the instrument conditions outlined above. The detector (electron multiplier) voltage was adjusted to provide adequate response at the lowest calibration level and avoid saturation at the highest calibration level. Figure 6 shows the total ion chromatogram of a 50 µg/mL standard. Selected chromatograms of individual analytes which may be subject to non-ideal chromatography, are shown in Figures 7A-7F.

Response factors were tabulated and percent relative standard deviation (%RSD) of response factors were determined as described in USEPA Method 8270D; the response factors for the initial calibration are presented in Table 4. Response of selected analytes varied considerably, especially at low concentrations, so

response factors for the lowest calibration points are not included for all of the analytes. Most analytes showed % RSD less than 15%. Where specific analytes had % RSD greater than 15%, the linear calibration was used for calculations, and the correlation coefficient (r) was used to evaluate the validity of the calibration.

Replicate injections of the four low-level calibration standards (0.4, 1.0, 2.0, and 5.0 µg/mL) were used to assess analytical precision and calculate instrument detection limits in a manner analogous to the procedure referenced in Method 8270D. (Sample extractions were not performed, so the statistical values are presented as "instrument detection limits", expressed in µg/mL). The statistical values and determination of instrument detection limits were based on data from injection of the standard concentration corresponding to the lowest point in the initial multi-point calibration for a given analyte. Data for assessment of analytical precision are summarized in Table 4.

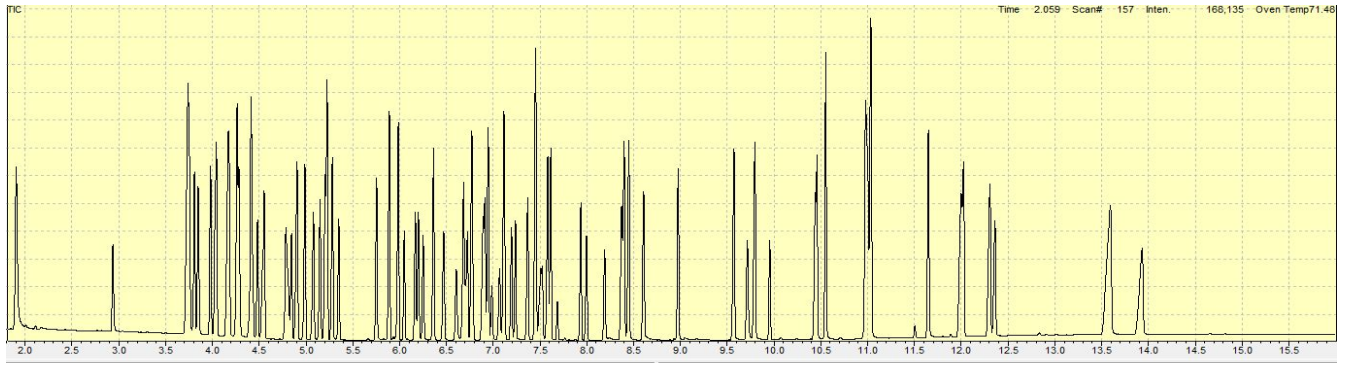


Figure 6: Total Ion Chromatogram of 50 µg/mL Semi-volatile Calibration Standard

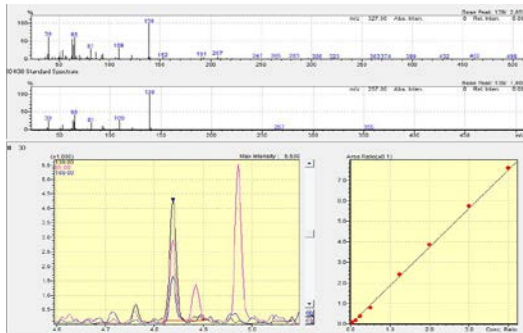


Figure 7A: 2-Nitrophenol (2 µg/mL)

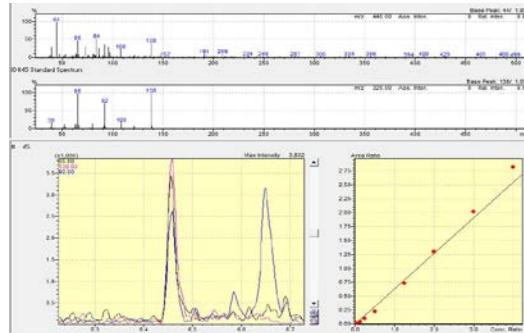


Figure 7B: 2-Nitroaniline (1 µg/mL)

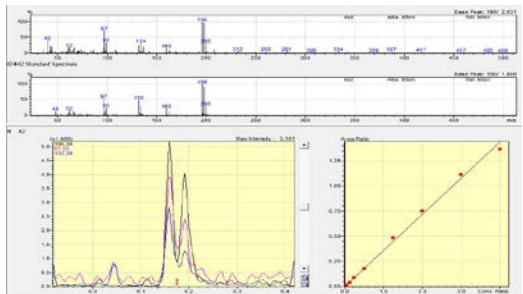


Figure 7C: 2,4,5- and 2,4,6-Trichlorophenols (1 µg/mL)

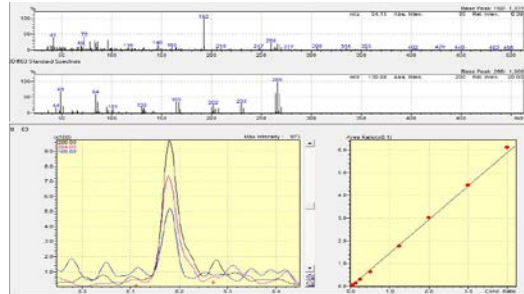


Figure 7D: Pentachlorophenol (1 µg/mL)

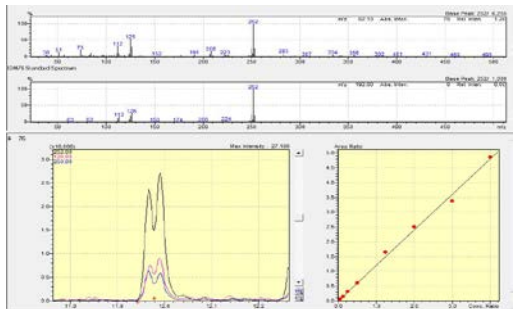


Figure 7E: Benzo(b&k)fluoranthenes (1 µg/mL)

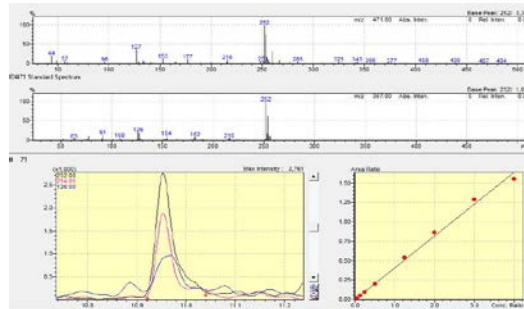


Figure 7F: 3,3'-Dichlorobenzidine (1 µg/mL)



**Table 4:** Calibration Results, and Determination of Precision, Recovery, and Instrument Detection Limits for Semi-volatile Organics by USEPA Method 8270D

Compound Name	Calibration Results			Determination of Precision, Recovery, and Instrument Detection Limits (n = 8)				
	Mean RRF	RSD (%)	r	Average Concentration (µg/L)	RSD (%)	Spike Level (µg/L)	Recovery (%)	IDL* (µg/L)
N-Nitrosodimethylamine	1.578	11.0		0.342	9.0	0.40	85.4	0.09
Phenol	3.558	11.2		1.087	5.8	1.00	108.7	0.19
Bis(2-chloroethyl) ether	2.687	8.1		0.396	7.6	0.40	98.9	0.09
2-Chlorophenol	1.855	10.4		1.025	2.4	1.00	102.5	0.08
1,3-Dichlorobenzene	1.826	9.0		0.350	7.1	0.40	87.5	0.07
1,4-Dichlorobenzene	1.835	9.0		0.347	4.0	0.40	86.7	0.04
1,2-Dichlorobenzene	1.740	8.4		0.339	3.5	0.40	84.6	0.04
Benzyl Alcohol	1.513	6.9		1.050	4.8	1.00	105.0	0.15
3&4-Methylphenol	2.057	13.2		1.108	3.4	1.00	110.8	0.11
2-Methylphenol	2.055	11.0		1.014	5.0	1.00	101.4	0.15
Bis(2-Chloroisopropyl) ether	3.966	8.2		0.399	4.9	0.40	99.8	0.06
N-Nitrosodi-n-propylamine	1.524	12.5		0.358	4.3	0.40	89.5	0.05
Hexachloroethane	0.756	8.8		0.431	7.4	0.40	107.7	0.09
Nitrobenzene	0.477	10.1		0.947	3.3	1.00	94.7	0.10
Isophorone	1.041	3.7		0.338	2.7	0.40	84.5	0.03
2-Nitrophenol	0.165		0.999	1.721	3.5	2.00	86.1	0.18
2,4-Dimethylphenol	0.387	9.7		1.102	10.0	1.00	110.2	0.33
Bis(2-chloroethoxy)methane	0.658	10.0		0.368	5.9	0.40	92.0	0.07
2,4-Dichlorophenol	0.216	9.1		0.749	5.2	1.00	74.9	0.12
1,2,4-Trichlorobenzene	0.260	10.9		0.407	4.7	0.40	101.8	0.06
Naphthalene	1.258	12.3		0.395	2.0	0.40	98.7	0.02
4-Chloroaniline	0.476	11.7		0.934	5.8	1.00	93.4	0.16
Hexachlorobutadiene	0.135		0.999	0.433	2.8	0.40	108.2	0.04
4-Chloro-3-methylphenol	0.360	13.7		1.000	5.1	1.00	100.0	0.15
2-Methylnaphthalene	0.768	8.3		0.329	6.7	0.40	82.1	0.07
Hexachlorocyclopentadiene	0.300	12.0		0.430	6.3	0.40	107.5	0.08
2,4,6-Trichlorophenol,	0.342	11.4		0.864	3.4	1.00	86.4	0.09
2,4,5-Trichlorophenol,	0.330	14.4		0.877	7.0	1.00	87.7	0.18
2-Chloronaphthalene	1.244	6.0		0.356	3.8	0.40	88.9	0.04
2-Nitroaniline	0.574		0.999	1.540	3.7	2.00	77.0	0.17
Dimethyl phthalate	1.222	5.9		0.337	6.6	0.40	84.3	0.07
2,6-Dinitrotoluene	0.253	12.3		1.705	4.4	2.00	85.2	0.23
Acenaphthylene	2.143	8.3		0.337	1.2	0.40	84.3	0.01
3-Nitroaniline	0.749		0.998	1.284	4.2	2.00	64.2	0.16
Acenaphthene	1.314	3.2		0.360	5.1	0.40	89.9	0.06
2,4-Dinitrophenol	0.122		0.997	2.901	8.2	5.00	58.0	0.71
4-Nitrophenol	0.339		0.998	2.749	4.0	5.00	55.0	0.33
Dibenzofuran	1.705	11.0		0.394	3.6	0.40	98.6	0.04
2,4-Dinitrotoluene	0.290	14.7		0.609	5.3	1.00	60.9	0.10
Diethyl phthalate	1.241	8.4		0.411	5.0	0.40	102.7	0.06
4-Chlorophenyl phenyl ether	0.621	13.0		0.432	7.3	0.40	108.0	0.10

**Table 4:** USEPA Method 8270D Calibration Results (continued)

Fluorene	1.376	14.1		0.395	3.0	0.40	98.8	0.04
4-Nitroaniline	0.352		0.999	1.301	6.3	2.00	65.1	0.24
2-Methyl-4,6-dinitrophenol	0.108		0.999	3.509	4.7	5.00	70.2	0.50
N-Nitrosodiphenylamine	0.737	12.8		0.348	10.2	0.40	87.0	0.11
4-Bromophenyl phenyl ether	0.252	14.4		0.375	9.9	0.40	93.8	0.11
Hexachlorobenzene	0.254		0.998	0.371	6.4	0.40	92.6	0.07
Pentachlorophenol	0.132	13.9		1.391	4.7	2.00	69.6	0.20
Phenanthrene	1.225	13.4		0.390	4.0	0.40	97.4	0.05
Anthracene	1.213	10.5		0.360	2.4	0.40	90.1	0.03
Carbazole	1.100	5.8		0.340	2.9	0.40	85.1	0.03
Di-n-butyl phthalate	1.344	6.7		0.283	5.5	0.40	70.8	0.05
Fluoranthene	1.160	8.0		0.369	5.0	0.40	92.3	0.05
Pyrene	1.373	5.5		0.353	7.9	0.40	88.2	0.08
Butylbenzyl phthalate	0.590		0.999	1.569	2.7	2.00	78.4	0.13
3,3'-Dichlorobenzidine	0.386	13.5		1.808	1.6	2.00	90.4	0.09
Bis(2-ethylhexyl) phthalate	0.830		0.999	1.575	3.1	2.00	78.8	0.14
Benzo[a]anthracene	1.214	9.1		0.441	6.7	0.40	110.1	0.09
Chrysene	1.168	12.6		0.424	3.9	0.40	106.1	0.05
Di-n-octyl phthalate	1.531		0.999	1.342	3.0	2.00	67.1	0.12
Benzo[b]fluoranthene	1.203	6.1		0.385	4.8	0.40	96.1	0.06
Benzo[k]fluoranthene	1.221	12.0		0.403	5.6	0.40	100.6	0.07
Benzo[a]pyrene	1.158	4.6		0.353	8.0	0.40	88.2	0.08
Indeno[1,2,3-cd]pyrene	1.303	5.6		0.317	4.8	0.40	79.3	0.05
Dibenzo(a,h)anthracene	1.070	6.9		0.329	6.7	0.40	82.3	0.07
Benzo(g,h,i) perylene	1.085	5.7		0.354	6.4	0.40	88.6	0.07

\*Statistically-derived instrument detection limits (IDLs) were determined by multiple injections of low level standards, and did not include the sample preparation (extraction and clean-up) steps.

### ■ Summary and Conclusions

The Twin Line MS Kit allows the user to install two chromatographic columns in the same mass spectrometer, providing greater flexibility for those laboratories wishing to perform volatile and semi-volatile analyses on a single GCMS unit without breaking the MS vacuum. This configuration incorporates two injection ports and two chromatographic columns simultaneously connected to a single MS. With this configuration, the user can easily switch between volatile and semi-volatile applications without venting the MS vacuum system to change the analytical column.

The analytical data presented here meet all method performance criteria established for both USEPA Methods 8260C and 8270D when run using the Twin Line MS Kit and the GCMS-QP2010 SE.

Combining volatile and semi-volatile applications on a single GCMS requires that the maximum temperatures of both chromatographic columns allow temperature

programming for both applications. In addition, carrier gas flow is always supplied to both columns to prevent damage of the columns when not in use, so the MS vacuum system was capable of supporting the flow from both analytical columns simultaneously. The Twin Line MS Kit configuration is optimum for combination of these two applications for several reasons:

- The GCMS-QP2010 SE high-capacity vacuum system can accommodate flows from two chromatographic columns simultaneously without impacting MS performance,
- The user can switch between USEPA Method 8260C and USEPA Method 8270D without venting the MS vacuum system to change columns, and
- A chromatographic column for volatile organics is available with a 320 °C maximum temperature that is compatible with the higher-temperature analysis conditions for semi-volatiles analysis.

■ **References**

1. USEPA Method 8260C Volatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS), Revision 3, August, 2006.
2. USEPA Method 8270D Semi-volatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS), Revision 4, February, 2007.
3. 40 CFR Appendix B to Part 136 - Definition and Procedure for the Determination of the Method Detection Limit.

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