EPA Method 8270 Semivolatile Organic Compounds Analysis on the Pegasus® BT: A Benchtop GC-TOFMS

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Key Words: EPA Method 8270, GC-TOFMS, Sensitivity, Linear Dynamic Range, Robust, Reproducible

1. Introduction

The United States Environmental Protection Agency (US EPA) Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from solid waste matrices, soils, air samples, and water samples by gas chromatography-mass spectrometry (GC-MS). The target compounds cover a large number of compound classes including acids, bases, and neutrals over a wide dynamic range typically from 0.1 to 50 ppm. The quality control criteria are quite rigorous, and in the majority of laboratories, must be met routinely for high throughput analysis. LECO has introduced a new GC Time-of-Flight Mass Spectrometer (GC-TOFMS) that exceeds the analytical demands of this method enabling the user to run split injections and still meet the sensitivity requirements of EPA Method 8270. A unique feature of the *Pegasus* BT is the patented open-style StayClean™ ion source that significantly increases instrument uptime by minimizing source maintenance. This application note details the performance of the *Pegasus* BT for EPA Method 8270, and demonstrates how to meet and exceed the method requirements.

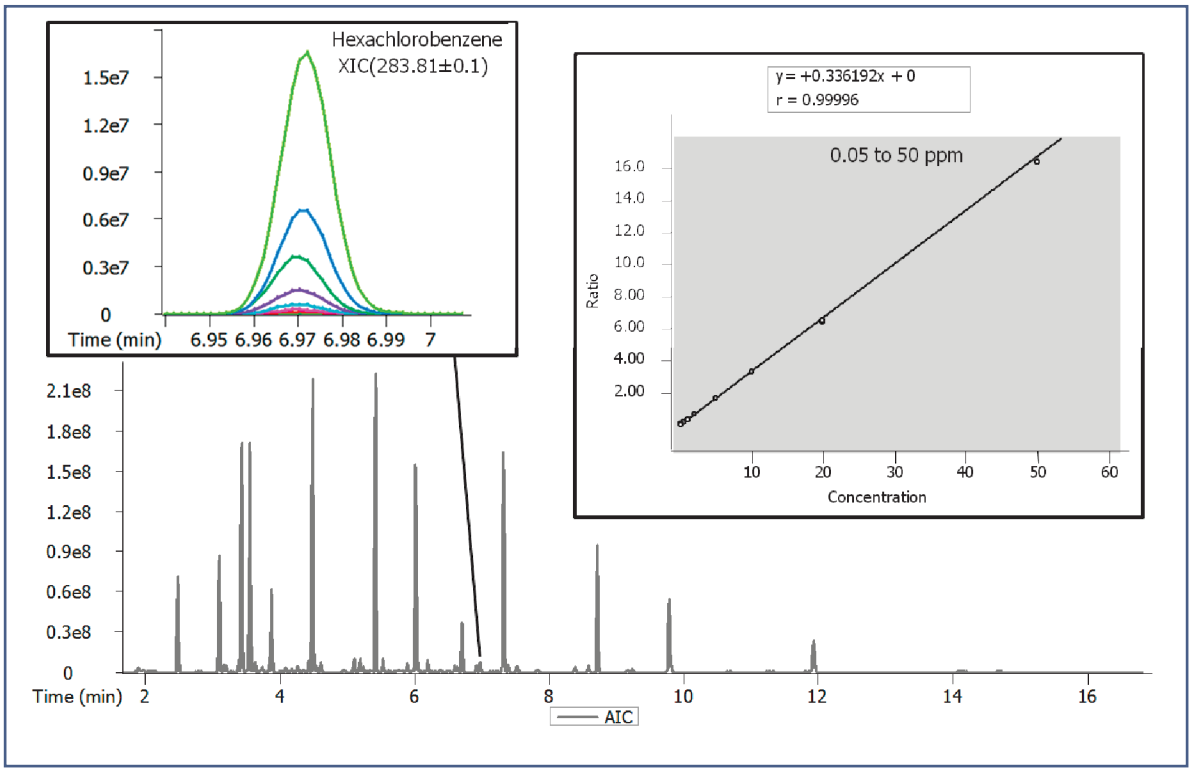

Figure 1. Extracted ion chromatogram (XIC) of the quant mass for hexachlorobenzene (HCB) from 0.05 to 50 ppm and its corresponding calibration curve, with the Analytical Ion Chromatogram (AIC) of a 1 ppm standard split 20:1 indicating where HCB elutes in the chromatogram.

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2. Experimental

Analytical standards were purchased from Restek (Bellefonte, PA) including 8270 MegaMix Standard (Cat # 31850), 8270 Matrix Spike Mix (Cat # 31687), CLP 04.1 BNA Surrogate Mix (Cat # 31493), SV Internal Standard (Cat # 31206), and GC-MS Tuning Mixture (Cat # 31615). A 10 point dilution series of the 8720 MegaMix Standard was prepared from 0.05 ppm to 50 ppm, spiked with 20 to 30 ppm of the BNA Surrogate Standard, and 20 ppm of the SV Internal Standard. The GC-MS Tuning Mixture was prepared at 50 ppm, and the 8270 Matrix Spike Mix was prepared at 20 ppm.

Table 1. GC-TOFMS (*Pegasus* BT) Conditions

Gas Chromatograph	**LECO L-Pal3 Autosampler with Agilent 7890B GC**
Injection	GC inlet, split 20:1 @ 270 °C
Carrier Gas	He @ 1.4 ml/min, Constant Flow
Column	ZB-SemiVolatiles, 30 m x 0.25 mm ID x 0.25 µm df (Phenomenex, Torrance, CA)
Oven Program	70 °C (1 min), to 285 °C @ 28 °C/min, to 305 °C @ 3 °C/min, to 320 °C @ 30 °C/min (1 min); Total time: 16.5 min
Transfer Line	300 °C
Mass Spectrometer	**LECO *Pegasus* BT**
Ion Source Temperature	250 °C
Mass Range	30-650 m/z
Acquisition Rate	10 spectra/s

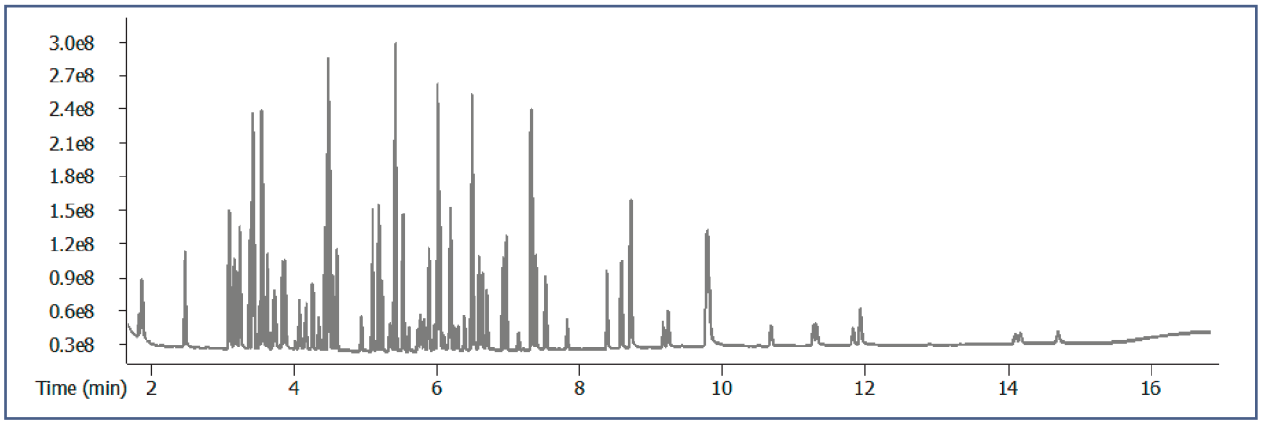

Figure 2. Total ion chromatogram (TIC) of a 5 ppm standard containing 90 components.

EPA Method 8270 allows users to use split injections if the mass spectrometer is sensitive enough to meet the method requirements. Figure 2 shows the TIC of a 5 ppm 8270 MegaMix standard split 20:1. There are many advantages of using a split injection instead of splitless, including improved chromatographic peak shape, less reactivity in the injection port, and improved uptime as less inlet maintenance and column trimming are required.

Table 2. DFTPP Tuning Results for 10 Replicate Injections

Mass	Ion Abundance Criteria	Mean	STDEV	Min	Max	Criteria
51	10-80% of Base Peak	64.5	0.25	64.1	64.9	PASS
68	< 2% of mass 69	1.93	0.04	1.87	1.98	PASS
70	< 2% of mass 69	0.38	0.01	0.37	0.40	PASS
127	10-80% of Base Peak	52.1	0.18	51.9	52.4	PASS
197	< 2% of mass 69	0.22	0.03	0.19	0.28	PASS
198	Base Peak, or > 50% of Mass 442		Base Peak			PASS
199	5-9% of mass 198	6.73	0.04	6.68	6.77	PASS
275	10-60% of Base Peak	23.0	0.08	22.9	23.1	PASS
365	> 1% of mass 198	1.90	0.02	1.86	1.92	PASS
441	Present but < 24% of mass 442	13.2	0.03	13.15	13.25	PASS
442	Base Peak, or > 50% of mass 198	76.5	0.3	76.1	76.9	PASS
443	15-24% of mass 442	19.5	0.08	19.4	19.6	PASS

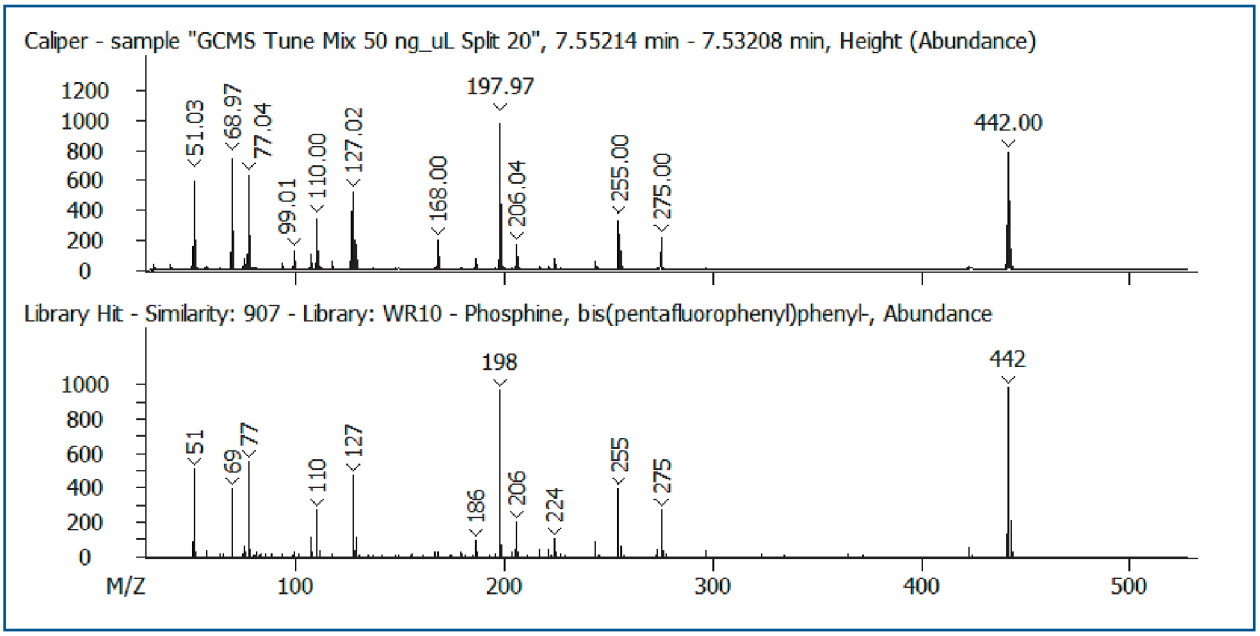

Figure 3. The Caliper (raw) mass spectrum at the apex of the DFTPP chromatographic peak with a one point background subtraction, and the corresponding library match spectrum for DFTPP, show that the Pegasus BT routinely passes DFTPP tuning.

Ten replicate injections of the GC-MS Tuning Mixture under the experimental conditions described in Table 1 were used to show that all of the instrument tuning and performance criteria for the method were met (Table 2, and Figures 3 and 4). DFTPP passed using the single point approach, but also passed using the average over the entire peak (not shown). The sensitivity of the instrument enabled split injections, allowing the injection port inertness and column performance checks to pass without difficulty.

The average tailing factor at 10% peak height for ten replicate injections of pentachlorophenol and benzidine was 1.13 ±0.02 and 1.30 ±0.02, respectively, both less than the required value of 2. Degradation of dichlorodiphenyltrichloroethane (DDT) to dichlorodiphenyldichloroethane (DDD) and dichlorodiphenyl-dichloroethylene (DDE) should be less than 20%, and was less than 1% in this study, which highlights the benefits of a split injection for injection port inertness and column performance.

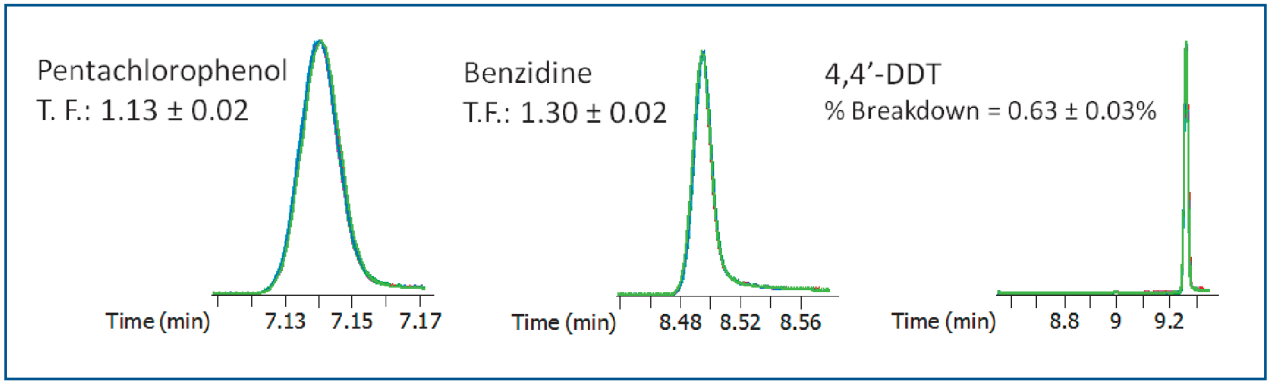

Figure 4. Check of the GC column performance and injection port inertness.

4. Quantitation in ChromaTOF® 5.0: Initial Calibration

A multipoint calibration curve was generated for the 84 compounds in the 8270 MegaMix Standard from 0.05 ppm to 50 ppm, injected in triplicate (Figure 5). Average response factor (AvgRF) was used as the default mode of quantitation as long as the relative standard deviation of the RF (% RSD RF) was less than 20% and the percent difference of the calculated to the theoretical value (%D) was less than 20%. Alternatively, linear regression with a correlation coefficient (r) greater than 0.995 can be used, provided the %D is less than 20%. The results are shown in Table 3. Average RF was used for all but one compound (benz[a]anthracene).

Figure 5. Quantitation using ChromaTOF 5.0 to evaluate calibration data for 8270 compounds. The highlighted compound is 2-Nitrophenol.

Table 3. EPA Method 8270 Calibration Curve Data for 84 Compounds from 0.05 to 50 ppm

Name	Type	R.T. (min)	Avg RF	% RSD RF	*r*
N-Nitrosodimethylamine	Analyte	1.837	0.577	8.425	------
Pyridine*	Analyte	1.975	1.174	7.698	------
2-Fluorophenol	Surrogate	2.475	0.367	5.14	------
Phenol-d6	Surrogate	3.090	0.581	3.92	------
Phenol*	Analyte	3.100	0.314	7.758	------
Aniline	Analyte	3.163	1.304	3.669	------
Bis(2-chloroethyl) ether	Analyte	3.198	1.025	5.732	------
2-Chlorophenol-D4	Surrogate	3.242	0.373	3.895	------
2-Chlorophenol	Analyte	3.253	0.398	7.524	------
1,3-Dichlorobenzene	Analyte	3.380	1.032	6.147	------
1,4-Dichlorobenzene	Analyte	3.437	1.075	7.583	------
Benzyl alcohol	Analyte	3.513	0.234	5.162	------
1,2-Dichlorobenzene-D4	Surrogate	3.547	0.98	0.914	------
1,2-Dichlorobenzene	Analyte	3.558	1.01	7.825	------
2-Methylphenol	Analyte	3.587	0.423	2.733	------
Bis(2-chloro-1-methylethyl)ether	Analyte	3.625	1.943	5.703	------
3/4-Methylphenol	Analyte	3.712	0.378	3.9	------
N-Nitrosodi-n-propylamine	Analyte	3.729	0.508	7.917	------
Hexachloroethane	Analyte	3.837	0.429	5.399	------
Nitrobenzene-D5	Surrogate	3.867	0.522	3.019	------
Nitrobenzene	Analyte	3.882	0.216	6.85	------
Isophorone	Analyte	4.079	1.03	6.196	------
2-Nitrophenol	Analyte	4.150	0.098	5.204	------
2,4-Dimethylphenol	Analyte	4.167	0.289	5.908	------
Bis(2-chloroethoxy)methane	Analyte	4.257	0.8	3.017	------
2,4-Dichlorophenol	Analyte	4.345	0.19	5.907	------
1,2,4-Trichlorobenzene	Analyte	4.427	0.335	2.007	------
Naphthalene	Analyte	4.499	1.146	4.684	------
4-Chloroaniline	Analyte	4.537	0.772	7.899	------
Hexachlorobutadiene	Analyte	4.600	0.156	1.946	------
4-Chloro-3-methylphenol	Analyte	4.944	0.251	5.033	------
2-Methylnaphthalene	Analyte	5.097	0.588	5.282	------
1-Methylnaphthalene	Analyte	5.184	0.578	5.149	------
Hexachlorocyclopentadiene	Analyte	5.232	0.326	2.02	------
2,4,6-Trichlorophenol	Analyte	5.337	0.089	6.534	------
2,4,5-Trichlorophenol	Analyte	5.364	0.101	9.49	------
2-Fluorobiphenyl	Surrogate	5.415	1.771	3.367	------
2-Chloronaphthalene	Analyte	5.524	1.554	3.336	------
2-Nitroaniline	Analyte	5.607	0.127	7.753	------
1,4-Dinitrobenzene	Analyte	5.729	0.056	7.153	------
Dimethyl phthalate	Analyte	5.767	0.552	6.982	------
1,3-Dinitrobenzene	Analyte	5.790	0.104	7.962	------
2,6-Dinitrotoluene	Analyte	5.819	0.171	7.38	------
1,2-Dinitrobenzene	Analyte	5.864	0.072	8.875	------
Acenaphthylene	Analyte	5.889	2.2	7.71	------

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Table 3. EPA Method 8270 Calibration Curve Data for 84 Compounds from 0.05 to 50 ppm (Continued)

Name	Type	R.T. (min)	Avg RF	% RSD RF	*r*
3-Nitroaniline	Analyte	5.965	0.173	6.112	------
Acenaphthene	Analyte	6.040	1.341	2.539	------
2,4-Dinitrophenol‡	Analyte	6.057	0.04	10.238	------
4-Nitrophenol	Analyte	6.095	0.112	5.813	------
2,4-Dinitrotoluene	Analyte	6.170	0.166	7.711	------
Dibenzofuran	Analyte	6.192	2.148	1.868	------
2,3,5,6-Tetrachlorophenol	Analyte	6.255	0.08	10.589	------
2,3,4,6-Tetrachlorophenol	Analyte	6.292	0.089	11.549	------
Diethyl Phthalate	Analyte	6.385	0.483	8.33	------
4-Chlorophenyl phenyl ether	Analyte	6.490	0.671	3.345	------
Fluorene	Analyte	6.492	1.249	6.793	------
4-Nitroaniline	Analyte	6.497	0.186	5.994	------
4,6-Dinitro-2-methylphenol	Analyte	6.525	0.052	8.015	------
Diphenylamine	Analyte	6.592	1.097	4.717	------
Azobenzene	Analyte	6.630	0.289	7.106	------
2,4,6-Tribromophenol	Surrogate	6.700	0.079	6.93	------
4-Bromophenyl phenyl ether	Analyte	6.920	0.438	8.076	------
Hexachlorobenzene	Analyte	6.970	0.336	2.276	------
Pentachlorophenol*	Analyte	7.140	0.038	11.186	------
Phenanthrene	Analyte	7.337	1.335	4.781	------
Anthracene	Analyte	7.382	1.828	3.421	------
Carbazole	Analyte	7.519	1.436	3.42	------
Dibutyl phthalate	Analyte	7.820	0.357	10.856	------
Fluoranthene	Analyte	8.379	1.479	1.563	------
Pyrene	Analyte	8.579	1.542	3.832	------
p-Terphenyl-d14	Surrogate	8.712	0.52	8.99	------
Benzyl butyl phthalate	Analyte	9.164	0.141	9.953	------
Bis(2-ethylhexyl) adipate	Analyte	9.234	0.131	9.617	------
Benz[a]anthracene	Analyte	9.767			0.9987
Bis(2-ethylhexyl)phthalate*	Analyte	9.791	0.239	10.425	------
Chrysene	Analyte	9.811	1.379	4.165	------
Di-n-octyl phthalate	Analyte	10.676	0.279	7.858	------
Benzo[b]fluoranthene	Analyte	11.264	1.041	6.505	------
Benzo[k]fluoranthene	Analyte	11.309	1.114	7.535	------
Benzo[a]pyrene	Analyte	11.821	0.896	7.607	------
Indeno[1,2,3-cd]pyrene	Analyte	14.094	0.633	8.146	------
Dibenz[a,h]anthracene	Analyte	14.164	0.704	7.091	------
Benzo[ghi]perylene	Analyte	14.691	0.926	7.95	------

**The 0.05 ppm standard was not included in the calibration curve because the %D was greater than 20%.*
‡The detection limit was 0.2 ppm for 2,4-Dinitrophenol.

The calibration data demonstrate that the instrument meets the method requirements, and is linear over 4 orders of dynamic range. Concentrations were determined for an 8270 Matrix Spike using this calibration data and the absolute mean %D was 5.7% with a range from -14.5 to 18.5% for all 84 compounds, validating the calibration data acquired.

5. Conclusion

This application note demonstrates that LECO's *Pegasus* BT meets or exceeds the EPA Method 8270 requirements for instrument performance. The sensitivity surpasses what most laboratories are currently achieving for this method, expanding the typical linear dynamic range, and enabling split injections for increased uptime and sample throughput. The *StayClean* ion source will further increase uptime as it is extremely low maintenance. Another added benefit occurs because the *Pegasus* BT is a Time-of-Flight MS, so a user also has the ability do non target or retrospective analysis at the same time without spending time and resources reacquiring data.

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