

# Application News

## Analysis of Semivolatile Compounds by GCMS-TQ™8040 NX Triple Quadrupole Mass Spectrometer by EPA 8270E

### ■ Abstract

Eighty-four (84) Semivolatile Organic Compounds (SVOCs) listed in EPA 8270E were simultaneously analyzed with GC-MS/MS. All compounds, including known challenging polar compounds, such as benzoic acid, met the calibration criteria determined in EPA 8270E, with an optimal dynamic range.

### ■ Introduction

Semivolatile Organic Compounds (SVOCs) are currently regulated in multiple environmental matrices. Hence, environmental laboratories need to routinely analyze these contaminants. EPA 8270E is the first method published by the Environmental Protection Agency (EPA) allowing the use of GC-MS Triple Quadrupole. The superior selectivity in MS/MS results in better sensitivity than traditional GC-MS Single Quadrupole. In addition, when employing GC-MS/MS, a standard autotuning defined by GC-MS manufacturer is available for spectrum tuning validation, instead of manual tuning using DFTPP. In this study, the performance of Shimadzu's GCMS-TQ8040 NX for the simultaneous analysis of 84 SVOCs included in EPA 8270E was evaluated.

### ■ Experimental

#### Standard Solutions

Commercially available analytical standards were mixed to prepare stock analyte solution, Internal Standard (IS) solution, and surrogate solution. The standards purchased for this study are listed in Table 1.

#### Calibration Range and Method Conditions

Calibration standards were prepared to cover a wide calibration range (0.01 ppm to 50 ppm) with 12 calibration levels. To incorporate in a single run the compounds with drastically different responses, the detector voltage was set carefully to avoid saturation of the detector. Detailed GC and MS conditions are shown in Table 2. MRM transitions are shown in Supplemental Information.

Table 1: Standard products used in this study

Type	Manufacturer	P/N	Product name
Analyte	Restek	31850	8270 MegaMix Standard (76 compounds)
Analyte	Restek	31834	Benzidine Mix (2 components)
Analyte	o2si	110903-01	CLP 4.2 Additional Comp.
Analyte	Millipore Sigma	CRM48367	1,4-Dioxane solution
Analyte	Millipore Sigma	CRM47508	Benzoic Acid Solution
IS	Millipore Sigma	CRM48902	EPA 8270 Semivolatile Internal Standard Mix
Surrogate	Millipore Sigma	CRM47960	EPA 8270 Surrogate Standard

Table 2: Conditions of Shimadzu's GCMS TQ8040 NX

GC conditions	
Injection Temp.	275 °C
Injection Mode	Split (1:10)
Column Flow	1.2 mL/min
Flow Control Mode	Linear Velocity
Column	SH-I-5MS (30m x 0.25 mm, 0.25 µm)
Oven Temp.	40 °C (1 min) -> 20 °C/min to 280 °C -> 5 °C to 320 °C (1 min) Total run: 22 min
Carrier Gas	Helium
MS conditions	
Solvent Cut Time	2 min
Ion Source Temp.	200 °C
Interface Temp.	320 °C
Detector Voltage	1.3 kV (Absolute)

## ■ Results

### Validation of Calibration Curve

All the calibration curves for each compound were evaluated according to the Initial Calibration (ICAL) criteria from EPA 8270

- 1) Calibration points must be within  $\pm 30\%$  of the true value when recalculated with the calibration curve.
- 2) Relative Standard Deviation (RSD) of Response Factors (RFs) must be less than 20% for average RF calibration model.
- 3) Coefficient of determination R<sup>2</sup> must be more than 0.99 for linear and quadratic<sup>1</sup> calibration curve.

<sup>1</sup>Use of a quadratic calibration curve requires at least 6 calibration levels.

Acceptable calibration curves must meet criteria #1, and either criteria #2 or #3.

For some of the targeted compounds, a weighted calibration curve provides better results for meeting criteria #1. This approach is acceptable and recommended in EPA 8000D (Determinative Chromatographic Separations). Square value of concentration is applied as weight in this study (1/x<sup>2</sup>). Calibration standards with a calculated value that exceeded  $\pm 30\%$  of the true value (criteria #1) were omitted from the final calibration curve of each

Square value of concentration is applied as weight in this study (1/x<sup>2</sup>). Calibration standards with a calculated value that exceeded  $\pm 30\%$  of the true value (criteria #1) were omitted from the final calibration curve of each compound. Thus, the calibration range for each compound reported in Table 3 fulfills criteria #1.

The parameters for confirming criteria #2 and #3: %RSD of RF and R<sup>2</sup> are shown in Table 3. All the compounds included in the method optimized in this study fulfill the criteria and are suitable for quantification in the reported concentration range and 54 compounds fulfilled the criteria #2. More than a half of compounds shows excellent linearity, with calibration ranges between 0.01 – 50 ppm with 12 points or 0.01 – 20 ppm with 11 points. Challenging compounds such as Benzoic acid and nitro group compounds maintain a dynamic range over 2 orders of magnitude (from 0.2 to 50 ppm) when utilizing quadratic calibration curve despite their polarity. Moreover, all the target compounds show repeatability within acceptable levels, even at the lowest calibration point. As shown in Figure 1, %RSD is below 10% for most targeted compounds.

Table 3: Validation and repeatability result of each calibration curve

Compound Name	Dynamic Range (ppm)	The number of calibration points	RSD of RF (%)	R <sup>2</sup> value (Linear)	R <sup>2</sup> value (Quadratic)	RSD at the lowest calibration point (n=6) (%)
1,4-Dioxane	0.01 - 50	12	7.86	0.993	0.994	13.29
N-Nitrosodimethylamine	0.2 - 50	8	7.54	0.997	0.998	6.56
Pyridine	0.1 - 50	9	24.57	0.998	0.998	4.25
Benzaldehyde	0.02 - 50	11	20.89	0.995	0.996	10.54
Phenol	0.01 - 50	12	9.21	0.994	0.996	8.04
Aniline	0.01 - 20	11	12.11	0.990	0.996	4.44
Bis(2-chloroethyl)ether	0.01 - 50	12	6.88	0.997	0.997	8.36
2-Chlorophenol	0.01 - 50	12	5.96	0.996	0.997	8.61
1,3-Dichlorobenzene	0.01 - 50	12	9.21	0.996	0.996	3.39
1,4-Dichlorobenzene	0.01 - 50	12	8.27	0.992	0.992	7.70
Benzyl Alcohol	0.02 - 20	10	6.58	0.995	0.997	7.85
1,2-Dichlorobenzene	0.01 - 50	12	6.49	0.995	0.995	4.67
2-Methylphenol(o-Cresol)	0.01 - 50	12	9.91	0.991	0.994	9.02
Bis(2-chloro-1-methylethyl) ether	0.01 - 50	12	10.40	0.992	0.992	9.72
N-nitroso-di-n-propylamine	0.2 - 50	8	14.68	0.993	0.995	13.34
3-Methylphenol (m-Cresol)/4-Methylphenol(p-Cresol)	0.01 - 50	12	19.34	0.982	0.991	10.00
Hexachloroethane	0.01 - 50	12	5.89	0.998	0.998	9.38
Nitrobenzene	0.01 - 50	12	10.50	0.992	0.992	13.62
Isophorone	0.01 - 50	12	8.50	0.996	0.996	8.63
2-Nitrophenol	0.02 - 50	11	27.23	0.972	0.993	19.67
2,4-Dimethylphenol	0.01 - 50	12	9.00	0.994	0.996	14.85
Benzoic Acid	0.2 - 50	8	68.48	0.796	0.992	16.90
Bis(2-chloromethoxy)methane	0.02 - 50	11	7.41	0.996	0.997	4.32
2,4-Dichlorophenol	0.02 - 50	11	26.91	0.978	0.992	8.27
1,2,4-Trichlorobenzene	0.01 - 50	12	7.67	0.995	0.996	3.59

Compound Name	Dynamic Range (ppm)	The number of calibration points	RSD of RF (%)	R <sup>2</sup> value (Linear)	R <sup>2</sup> value (Quadratic)	RSD at the lowest calibration point (n=6) (%)
Naphthalene	0.01 - 50	12	7.66	0.995	0.997	3.03
4-Chloroaniline	0.01 - 20	11	19.47	0.977	0.993	4.46
Hexachlorobutadiene	0.01 - 50	12	9.76	0.992	0.993	7.71
Caprolactam	0.1 - 20	8	32.36	0.970	0.994	7.34
4-Chloro-3-methylphenol	0.01 - 10	10	23.84	0.963	0.992	13.29
2-Methylnaphthalene	0.01 - 20	11	5.91	0.996	0.996	5.84
1-Methylnaphthalene	0.01 - 20	11	7.19	0.995	0.995	4.45
Hexachlorocyclopentadiene	0.01 - 50	12	14.70	0.991	0.996	17.98
2,4,6-Trichlorophenol	0.02 - 50	11	24.20	0.957	0.990	12.51
2,4,5-Trichlorophenol	0.02 - 50	11	27.61	0.976	0.991	15.99
1,1'-Biphenyl	0.01 - 50	12	5.85	0.997	0.998	16.41
2-Chloronaphthalene	0.01 - 50	12	6.13	0.997	0.997	6.56
2-Nitroaniline	0.02 - 20	10	29.85	0.956	0.992	12.39
1,4-Dinitrobenzene	0.2 - 20	7	35.44	0.957	0.990	10.63
Dimethyl phthalate	0.01 - 50	12	7.85	0.993	0.996	8.68
1,3-Dinitrobenzene	0.2 - 20	6	27.32	0.964	0.990	10.55
2,6-Dinitrotoluene	0.05 - 20	9	23.04	0.964	0.990	4.69
1,2-Dinitrobenzene	0.2 - 50	8	23.97	0.989	0.992	15.96
Acenaphthylene	0.02 - 50	11	12.01	0.995	0.995	3.37
3-Nitroaniline	0.1 - 50	9	18.96	0.977	0.990	5.22
Acenaphthene	0.01 - 50	12	11.25	0.993	0.993	7.79
2,4-Dinitrophenol	0.2 - 50	7	60.67	0.903	0.990	13.66
4-Nitrophenol	0.2 - 50	7	26.69	0.986	0.991	2.82
2,4-Dinitrotoluene	0.05 - 20	9	25.40	0.977	0.991	14.95
Dibenzofuran	0.01 - 20	11	7.32	0.997	0.997	2.19
2,3,5,6-Tetrachlorophenol	0.02 - 20	10	33.18	0.966	0.993	10.45
2,3,4,6-Tetrachlorophenol	0.02 - 10	9	22.50	0.958	0.993	16.22
Diethyl Phthalate	0.01 - 50	12	96.24	0.992	0.993	9.89
Fluorene	0.01 - 50	12	7.48	0.995	0.995	9.66
4-Chlorophenyl-phenyl ether	0.01 - 50	12	6.65	0.995	0.995	6.86
4-Nitroaniline	0.1 - 10	7	30.38	0.956	0.994	5.40
4,6-Dinitro-2-methylphenol	0.2 - 50	7	45.46	0.947	0.990	10.76
Diphenylamine	0.01 - 20	11	7.91	0.993	0.995	4.20
Azobenzene	0.01 - 20	11	7.83	0.995	0.995	8.40
4-Bromophenyl phenyl ether	0.01 - 50	12	9.72	0.995	0.995	7.20
Hexachlorobenzene	0.01 - 50	12	6.45	0.995	0.995	7.70
Atrazine	0.05 - 50	10	29.95	0.978	0.992	14.16
Pentachlorophenol	0.05 - 20	9	35.71	0.939	0.991	13.63
Phenanthrene	0.01 - 20	11	9.24	0.996	0.996	2.97
Anthracene	0.01 - 50	12	9.16	0.993	0.995	2.27
Carbazole	0.01 - 20	11	8.06	0.993	0.996	3.52
Di-n-butyl phthalate	0.02 - 20	10	12.42	0.983	0.995	3.66
Fluoranthene	0.01 - 20	11	7.19	0.996	0.996	4.96
Benidine	0.02 - 50	11	38.56	0.876	0.991	11.75
Pyrene	0.01 - 20	11	8.77	0.997	0.997	3.96
Butyl benzyl phthalate	0.01 - 20	11	26.78	0.955	0.992	10.35
Bis(2-ethylhexyl) adipate	0.01 - 20	11	35.94	0.930	0.991	11.83
3,3'-Dichlorobenzidine	0.1 - 50	9	26.14	0.972	0.991	7.06
Benzo[a]anthracene	0.01 - 20	11	27.81	0.988	0.991	5.72
Bis(2-ethylhexyl) phthalate	0.02 - 20	10	20.39	0.954	0.993	4.93
Chrysene	0.01 - 20	11	22.56	0.995	0.995	6.70
Di-n-octyl phthalate	0.01 - 20	11	26.45	0.940	0.993	7.37
Benzo(b)fluoranthene	0.05 - 20	9	10.15	0.991	0.997	3.32
Benzo(k)fluoranthene	0.05 - 20	9	13.06	0.994	0.996	6.62
Benzo(a)pyrene	0.05 - 20	9	15.65	0.990	0.996	3.21
Indeno(1,2,3-cd)pyrene	0.05 - 50	10	34.34	0.993	0.995	5.02
Dibenzo(a,h)anthracene	0.1 - 50	9	28.52	0.991	0.995	7.60
Benzo(g,h,i)pyrene	0.05 - 50	10	44.04	0.991	0.994	10.00

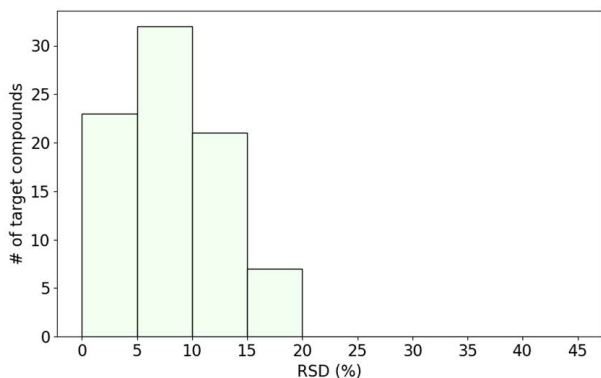


Figure. 1: Distribution of %RSD of RF

## ■ Conclusions

A method was optimized for the simultaneous analysis of 84 SVOCs listed in EPA 8270E by Shimadzu's GCMS-TQ8040 NX. All compounds displayed valid calibration curves within a suitable dynamic range for the analysis of these chemicals in environmental samples. Simply performed tuning validation is an additional benefit of using GC-MS/MS. This work demonstrates how GC-MS/MS can enhance workflows for the analysis of SVOCs with EPA 8270E.

## ■ Supplemental Information

Table S1: RT and MRM transition of target compound

Compound	Type	RT	Quantifying Transition	CE	Reference Transition	CE	Ratio
1,4-Dioxane	Target	2.519	88.00>58.10	9	58.00>56.00	42	1.29
N-Nitrosodimethylamine	Target	2.779	74.00>59.00	18	74.00>71.00	39	36.44
Pyridine	Target	2.827	79.10>52.10	15	79.10>50.10	21	44.56
Benzaldehyde	Target	4.917	105.00>77.00	15	106.00>77.00	21	81.78
Phenol	Target	5.001	94.00>66.00	9	66.00>40.00	12	43.83
Aniline	Target	5.038	93.00>66.10	18	93.00>51.10	30	21.30
Bis(2-chloroethyl)ether	Target	5.102	93.00>63.10	9	63.00>61.10	30	3.62
2-Chlorophenol	Target	5.167	128.00>64.00	18	128.00>91.90	15	40.44
1,3-Dichlorobenzene	Target	5.342	146.00>111.10	18	146.00>75.20	30	97.97
1,4-Dichlorobenzene	Target	5.420	146.00>111.10	21	146.00>75.10	33	98.33
Benzyl Alcohol	Target	5.549	79.00>77.10	12	107.00>79.10	9	89.15
1,2-Dichlorobenzene	Target	5.592	146.00>111.10	21	146.00>75.10	27	88.96
2-Methylphenol(o-Cresol)	Target	5.670	108.00>77.00	27	108.00>79.00	18	98.90
Bis(2-chloro-1-methylethyl) ether	Target	5.704	121.05>77.00	9	121.05>62.20	42	5.55
N-nitroso-di-n-propylamine	Target	5.851	70.00>55.10	18	70.00>54.00	24	90.96
3-Methylphenol (m-Cresol)/4-Methylphenol(p-Cresol)	Target	5.851	107.10>77.10	15	107.10>79.10	6	28.61
Hexachloroethane	Target	5.981	117.00>81.90	30	119.00>83.80	33	51.48
Nitrobenzene	Target	6.050	77.05>51.00	21	123.05>77.00	15	90.36
Isophorone	Target	6.331	82.00>54.10	9	138.00>82.00	18	79.75
2-Nitrophenol	Target	6.435	139.00>109.10	9	139.00>81.00	12	87.32
2,4-Dimethylphenol	Target	6.476	107.00>77.10	18	122.00>107.10	18	95.31
Benzoic Acid	Target	6.540	122.10>105.10	9	105.10>77.10	15	87.84
Bis(2-chloromethoxy)methane	Target	6.636	93.00>63.10	9	63.00>61.00	18	2.00
2,4-Dichlorophenol	Target	6.716	162.00>63.10	33	164.00>63.10	30	83.05
1,2,4-Trichlorobenzene	Target	6.821	180.00>109.00	30	180.00>145.10	18	96.74
Naphthalene	Target	6.913	128.10>102.10	20	128.10>78.00	20	58.05
4-Chloroaniline	Target	6.981	127.00>65.10	27	127.00>92.10	18	60.06
Hexachlorobutadiene	Target	7.078	225.00>189.80	21	225.00>155.00	30	73.54
Caprolactam	Target	7.370	113.10>85.10	9	113.10>56.10	15	69.75
4-Chloro-3-methylphenol	Target	7.573	107.00>77.10	18	142.00>107.00	18	99.52
2-Methylnaphthalene	Target	7.765	142.10>115.10	28	115.10>89.00	16	29.45
1-Methylnaphthalene	Target	7.884	142.10>115.10	28	115.10>89.00	16	30.16
Hexachlorocyclopentadiene	Target	7.976	237.00>141.00	27	237.00>143.00	27	87.95
2,4,6-Trichlorophenol	Target	8.116	196.00>97.00	33	198.00>97.00	30	80.59
2,4,5-Trichlorophenol	Target	8.158	196.00>97.10	30	198.00>97.00	30	83.95
1,1'-Biphenyl	Target	8.339	154.10>128.10	22	154.10>115.10	24	74.03
2-Chloronaphthalene	Target	8.366	162.00>127.20	30	162.00>77.10	30	94.59
2-Nitroaniline	Target	8.492	138.00>92.00	15	138.00>65.10	33	115.05
1,4-Dinitrobenzene	Target	8.650	168.00>75.10	30	168.00>92.00	15	31.67

Compound	Type	RT	Quantifying Transition	CE	Reference Transition	CE	Ratio
Dimethyl phthalate	Target	8.723	163.00>77.20	15	163.00>133.10	15	92.01
1,3-Dinitrobenzene	Target	8.749	168.00>75.00	30	168.00>122.00	12	69.14
2,6-Dinitrotoluene	Target	8.789	165.00>90.00	15	165.00>63.10	33	115.02
1,2-Dinitrobenzene	Target	8.855	92.05>63.10	24	168.05>77.90	12	50.12
Acenaphthylene	Target	8.882	152.10>150.10	28	152.10>126.10	28	93.06
3-Nitroaniline	Target	8.999	92.05>65.00	12	138.05>65.00	27	98.60
Acenaphthene	Target	9.099	152.10>150.10	28	152.10>126.10	28	93.06
2,4-Dinitrophenol	Target	9.130	184.05>107.00	12	154.05>107.00	6	72.78
4-Nitrophenol	Target	9.211	109.05>81.00	12	109.05>53.10	18	61.04
2,4-Dinitrotoluene	Target	9.293	89.05>63.10	18	165.05>119.00	6	95.08
Dibenzofuran	Target	9.318	168.00>139.10	24	139.00>89.10	21	10.55
2,3,5,6-Tetrachlorophenol	Target	9.417	230.00>130.90	36	232.00>132.90	36	76.86
2,3,4,6-Tetrachlorophenol	Target	9.474	230.00>131.00	39	232.00>131.00	33	86.92
Diethyl Phthalate	Target	9.604	149.00>65.00	30	177.00>149.10	12	98.21
Fluorene	Target	9.740	165.10>163.10	28	165.10>115.10	28	72.43
4-Chlorophenyl-phenyl ether	Target	9.740	141.00>115.20	21	204.00>141.20	21	97.09
4-Nitroaniline	Target	9.758	138.00>108.10	12	108.00>80.10	12	55.91
4,6-Dinitro-2-methylphenol	Target	9.799	198.05>121.00	12	198.05>53.00	27	68.79
Diphenylamine	Target	9.888	169.00>167.20	27	168.00>139.00	39	93.85
Azobenzene	Target	9.941	77.00>51.20	15	77.00>74.10	33	7.01
4-Bromophenyl phenyl ether	Target	10.352	250.00>141.10	21	248.00>141.10	18	97.78
Hexachlorobenzene	Target	10.436	283.80>248.80	24	283.80>213.80	28	70.16
Atrazine	Target	10.554	215.10>58.00	14	215.10>173.10	6	91.50
Pentachlorophenol	Target	10.678	265.90>164.90	26	265.90>166.90	26	97.20
Phenanthrene	Target	10.942	178.10>176.10	28	178.10>152.10	20	133.23
Anthracene	Target	11.006	178.10>176.10	28	178.10>152.10	20	109.95
Carbazole	Target	11.204	167.00>139.20	27	166.00>140.00	18	75.40
Di-n-butyl phthalate	Target	11.644	149.00>93.10	18	149.00>65.10	27	99.94
Fluoranthene	Target	12.425	202.10>200.10	30	200.10>198.10	30	12.83
Benzidine	Target	12.592	184.00>156.10	24	184.00>167.10	24	72.37
Pyrene	Target	12.708	202.10>200.10	30	200.10>198.10	30	13.73
Butyl benzyl phthalate	Target	13.533	149.00>65.10	24	149.00>93.10	18	94.39
Bis(2-ethylhexyl) adipate	Target	13.643	129.00>55.10	21	129.00>101.10	9	93.52
3,3'-Dichlorobenzidine	Target	14.294	252.05>154.10	27	254.00>154.30	30	67.69
Benzo[a]anthracene	Target	14.330	228.10>226.10	32	226.10>224.10	32	28.94
Bis(2-ethylhexyl) phthalate	Target	14.377	149.00>65.10	27	167.00>149.10	15	99.32
Chrysene	Target	14.387	228.10>226.10	32	226.10>224.10	32	28.84
Di-n-octyl phthalate	Target	15.469	149.00>65.10	24	149.00>93.20	18	99.52
Benzo(b)fluoranthene	Target	16.163	252.10>250.10	36	250.10>248.10	36	26.05
Benzo(k)fluoranthene	Target	16.215	252.10>250.10	36	250.10>248.10	36	25.97
Benzo(a)pyrene	Target	16.785	252.10>250.10	36	250.10>248.10	36	25.54
Indeno(1,2,3-cd)pyrene	Target	19.092	276.10>274.10	36	274.10>272.10	36	24.87
Dibenzo(a,h)anthracene	Target	19.150	278.10>276.10	36	278.10>274.10	60	21.38
Benzo(g,h,i)pyrene	Target	19.654	276.10>274.10	36	274.10>272.10	36	24.66

Table S2: RT and MRM transition of ISTD compound

Compound	Type	RT	Quantifying Transition	CE	Reference Transition	CE	Ratio
1,4-Dichlorobenzene-d4	ISTD	5.420	115.00>78.10	18	115.00>76.10	27	42.56
Naphthalene-d8	ISTD	6.913	136.00>108.10	27	136.00>134.10	27	51.87
Acenaphthene-d10	ISTD	9.093	162.00>160.10	30	164.00>160.10	36	94.31
Phenanthren-d10	ISTD	10.948	188.00>160.10	30	188.00>158.10	33	81.37
Chrysene-d12	ISTD	14.397	236.00>232.10	30	236.00>234.10	24	59.10
Perylene-d12	ISTD	16.961	260.00>256.10	39	260.00>258.10	30	67.91

Table S3: RT and MRM transition of surrogate compound

Compound	Type	RT	Quantifying Transition	CE	Reference Transition	CE	Ratio
2-Fluorophenol	Surrogate	4.038	112.00>64.10	18	112.00>92.10	9	32.82
Phenol-d6	Surrogate	5.001	99.00>71.10	12	99.00>69.10	27	33.22
Nitrobenzene-d5	Surrogate	6.050	82.00>54.10	18	128.00>82.10	18	65.66
2-Fluorobiphenyl	Surrogate	8.250	172.00>151.20	27	172.00>146.10	27	70.37
2,4,6-Tribromophenol	Surrogate	10.077	332.00>142.80	39	330.00>143.10	33	78.01
p-Terphenyl-d14	Surrogate	12.942	244.00>240.10	30	244.00>226.20	18	51.00

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