

Abstract:

Standard preparation can often be a time consuming, tedious process. The opportunity for human error or inconsistencies between individual preparation techniques can inhibit curve linearity and/or continuing calibration reproducibility. This application note will explore water matrix standard preparation techniques for USEPA Method 8260b.

Discussion:

There have been many advances in purge and trap analysis in the past ten years. These improvements vary from water management to limiting the amount of sample carryover. However, in creating a more efficient concentrator, the ability of the concentrator to tolerate large amounts of methanol has become an issue. Environmental laboratories are required to write and follow standard operating procedures in order to prove that sample handling and analysis is consistent. Chief among these standard operating procedures is standard preparation. Since new purge and trap concentrators are no longer able to handle larger amounts methanol, the procedures for standard preparation may need to be altered to accommodate the concentrator's limitations. This application note will explore how methanol affects curve linearity and compound response.

Experimental:

The sampling system used for this study was the EST Analytical Centurion WS autosampler and Encon Evolution concentrator. The experiments were run in water mode with a 5mL purge volume. Coupled to the sampling system were an Agilent 7890A GC and 5975 inert XL MS. The GC was configured with a Restek Rtx-624 20m x 0.180mm x 1.0 μ m column. Experimental parameters for both the purge and trap and GC/MS are listed in Tables 1 and 2 respectively.



| Purge and Trap Concentrator | EST Encon Evolution |
|---|---------------------|
| Trap Type | Vocarb 3000 |
| Valve Oven Temp. | 130°C |
| Transfer Line Temp. | 130°C |
| Trap Temp. | 35°C |
| Moisture Reduction Trap (MoRT) Temp. | 39°C |
| Purge Time | 11 min. |
| Purge Flow | 40mL/min |
| Dry Purge Temp. | ambient |
| Dry Purge Flow | 40mL/min |
| Dry Purge Time | 1.0 min. |
| Desorb Pressure Control | On |
| Desorb Pressure | 12psi |
| Desorb Time | 1.0 min. |
| Desorb Temp. | 260°C |
| Moisture Reduction Trap (MoRT) Bake Temp. | 230°C |
| Bake Temp | 260°C |
| Sparge Vessel Bake Temp. | 110°C |
| Bake Time | 8 |
| Bake Flow | 40mL/min |
| Purge and Trap Auto-Sampler | EST Centurion WS |
| Sample Size | 5mL |
| Internal Standard Volume | 5µL |

Table 1: Purge and Trap Parameters

| GC/MS | Agilent 7890A/5975 inert XL |
|------------------------|---|
| Inlet | Split/Splitless |
| Inlet Temp. | 200°C |
| Inlet Head Pressure | 20.198 psi |
| Mode | Split |
| Split Ratio | 40:1 |
| Column | Rtx-624 20m x 0.18mm I.D. 1µm film thickness |
| Oven Temp. Program | 45°C hold for 1 min., ramp 18°C/min to 220°C, hold for 0.3 min., 11.02 min. runtime |
| Column Flow Rate | 0.8mL/min |
| Gas | Helium |
| Total Flow | 35.8mL/min |
| Source Temp. | 230°C |
| Quad Temp. | 150°C |
| MS Transfer Line Temp. | 180°C |
| Scan Range | m/z 35-265 |
| Scans | 3.12 scans/sec |
| Solvent Delay | 0.7 min |

Table 2: GC/MS Parameters

The USEPA Method 8260b standards were purchased from Restek. The compound concentration was 2000µg/ml for all of the analytes with the exception of tert-butyl alcohol which was at 10,000µg/ml and p&m-xylene which was at 4000µg/ml. The percent methanol in the standards was calculated using the 200ppb calibration standard as a reference due to the fact that the maximum amount of methanol was in this standard. These calculations are displayed in Table 3. Standards were prepared at two different concentrations in order to minimize the percent methanol differences seen when preparing a curve. Refer to Table 4 for standard concentrations.

| %Methanol Calculation | | | | | | |
|---------------------------------|----------|----------|----------|----------|-----------|-----------|
| Methanol in 200ppb Cal Standard | 1µl/ml | 2µl/ml | 5µl/ml | 10µl/ml | 20µl/ml | 50µl/ml |
| IS Methanol | 1µl/ml | 1µl/ml | 1µl/ml | 1µl/ml | 1µl/ml | 1µl/ml |
| Size Sample | 5ml | 5ml | 5ml | 5ml | 5ml | 5ml |
| Standard and IS Methanol | 10µl/5ml | 15µl/5ml | 30µl/5ml | 55µl/5ml | 105µl/5ml | 255µl/5ml |
| %Methanol in Purged Sample | 0.2% | 0.3% | 0.6% | 1.1% | 2.1% | 5.1% |

Table 3: Percent Methanol Calculation

| Standard Preparation | | | | | | |
|--------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| Standard Concentrations | 0.2% Methanol | 0.3% Methanol | 0.6% Methanol | 1.1% Methanol | 2.1% Methanol | 5.1% Methanol |
| 0.5ppb to 10ppb cal. standards | 20ppm | 10ppm | 4ppm | 2ppm | 1ppm | 400ppb |
| 20ppb to 200ppb cal. standards | 200ppm | 100ppm | 40ppm | 20ppm | 10ppm | 4ppm |

*%Methanol includes the added methanol from the internal standard

Table 4: Stock Standard Preparation for Calibration Curves

A nine point calibration curve was prepared and run for each percent methanol level. The calibration curve preparation is presented in Table 5. A range of 0.5 to 200ppb was used to establish the curve and Agilent Chemstation software was employed to ascertain the linear calibration of the analytes. The analytes were also examined to establish compound response versus percent methanol in the standards; these results are listed in Table 6. The 5.1% methanol results were excluded from the results table due to the fact that all of the compounds needed to be linear regressed and still the results were not linear to 200ppb.

| Calibration Curve Preparation | | | | | | |
|------------------------------------|----------------------|----------------------|---------------------|---------------------|---------------------|----------------------|
| Calibration Standard Concentration | 0.2% Methanol | 0.3% Methanol | 0.6% Methanol | 1.1% Methanol | 2.1% Methanol | 5.1% Methanol |
| 0.5ppb | 2.5µl 20ppm std. | 5µl 10ppm std. | 12.5µl 4ppm std. | 25µl 2ppm std. | 50µl 1ppm std. | 125µl 400ppb std. |
| 1ppb | 5µl 20ppm std. | 10µl 10ppm std. | 25µl 4ppm std. | 50µl 2ppm std. | 100µl 1ppm std. | 250µl 400ppb std. |
| 2ppb | 10µl 20ppm std. | 20µl 10ppm std. | 50µl 4ppm std. | 100µl 2ppm std. | 200µl 1ppm std. | 500µl 4ppb std. |
| 5ppb | 25µl 20ppm std. | 50µl 10ppm std. | 125µl 4ppm std. | 250µl 2ppm std. | 500µl 1ppm std. | 1.25ml 4ppb std. |
| 10ppb | 50µl 20ppm std. | 100µl 10ppm std. | 250µl 4ppm std. | 500µl 2ppm std. | 1ml 1ppm std. | 2.50ml 4ppb std. |
| 20ppb | 10µl 200ppm std. | 20µl 100ppm std. | 50µl 40ppm std. | 100µl 20ppm std. | 200µl 10ppm std. | 500µl 4ppm std. |
| 50ppb | 25µl 200ppm std. | 50µl 100ppm std. | 125µl 40ppm std. | 250µl 20ppm std. | 500µl 10ppm std. | 1.25ml 4ppm std. |
| 100ppb | 50µl 200ppm std. | 100µl 100ppm std. | 250µl 40ppm std. | 500µl 20ppm std. | 1ml 10ppm std. | 2.50ml 4ppm std. |
| 200ppb | 100µl 200ppm std. | 200µl 100ppm std. | 500µl 40ppm std. | 1ml 20ppm std. | 2ml 10ppm std. | 5.00ml 4ppm std. |

* All standards diluted in De-ionized water into a 100ml volumetric flask.

Table 5: Calibration Curve Preparation

| Compound | 0.2% Methanol | | 0.3% Methanol | | 0.6% Methanol | | 1.1% Methanol | | 2.1% Methanol | |
|-------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF |
| Dichlorodifluoromethane | 6.10 | 0.597 | 6.76 | 0.440 | 4.77 | 0.368 | 11.00 | 0.197 | 11.54 | 0.255 |
| Chloromethane | 9.88 | 0.626 | 4.89 | 0.456 | 4.10 | 0.426 | 11.74 | 0.245 | 9.76 | 0.289 |
| Vinyl Chloride | 7.23 | 0.578 | 3.08 | 0.428 | 3.26 | 0.408 | 7.99 | 0.239 | 7.55 | 0.266 |
| Bromomethane | 8.64 | 0.313 | 4.26 | 0.233 | 12.02 | 0.247 | 11.75 | 0.145 | 0.995* | 0.143 |
| Chloroethane | 8.20 | 0.353 | 5.83 | 0.267 | 3.66 | 0.265 | 7.74 | 0.160 | 8.13 | 0.166 |
| Trichlorofluoromethane | 2.88 | 0.837 | 4.18 | 0.636 | 4.57 | 0.625 | 5.36 | 0.378 | 6.62 | 0.389 |
| diethyl ether | 3.68 | 0.416 | 3.81 | 0.323 | 5.24 | 0.331 | 5.42 | 0.194 | 2.84 | 0.202 |
| 1,1,2-trichlorofluoroethane | 5.71 | 0.495 | 4.09 | 0.379 | 5.05 | 0.383 | 8.96 | 0.225 | 6.74 | 0.227 |
| 1,1-Dichloroethene | 5.42 | 0.449 | 3.52 | 0.347 | 3.48 | 0.350 | 4.61 | 0.204 | 6.84 | 0.215 |
| Acetone | 11.10 | 0.192 | 7.07 | 0.127 | 14.52 | 0.165 | 11.53 | 0.092 | 0.995* | 0.402 |
| Iodomethane | 13.31 | 0.427 | 9.38 | 0.370 | 10.79 | 0.365 | 7.56 | 0.211 | 15.04 | 0.202 |
| Carbon Disulfide | 10.55 | 1.248 | 5.69 | 0.934 | 8.69 | 0.919 | 6.70 | 0.512 | 12.70 | 0.556 |
| Carbon Disulfide | 4.56 | 0.774 | 8.22 | 0.577 | 6.58 | 0.597 | 4.71 | 0.348 | 7.39 | 0.361 |
| Methylene Chloride | 8.72 | 0.500 | 7.83 | 0.386 | 6.04 | 0.387 | 9.75 | 0.237 | 12.90 | 0.246 |
| acetonitrile | 8.65 | 0.062 | 9.02 | 0.035 | 13.64 | 0.041 | 7.54 | 0.026 | 9.08 | 0.031 |
| Tert Butyl Alcohol | 9.01 | 0.066 | 14.90 | 0.030 | 12.82 | 0.045 | 13.76 | 0.022 | 0.998* | 0.038 |
| MTBE | 3.75 | 1.623 | 1.72 | 1.218 | 2.81 | 1.266 | 3.56 | 0.745 | 2.99 | 0.764 |
| cis-1,2-Dichloroethene | 5.43 | 0.499 | 4.86 | 0.388 | 3.19 | 0.384 | 9.17 | 0.236 | 10.65 | 0.246 |
| acrylonitrile | 12.01 | 0.224 | 10.17 | 0.176 | 9.56 | 0.197 | 9.54 | 0.110 | 6.28 | 0.116 |
| Isopropylether | 7.23 | 1.774 | 4.14 | 1.293 | 4.49 | 1.344 | 4.86 | 0.791 | 4.89 | 0.797 |
| Vinyl acetate | 8.71 | 1.283 | 4.24 | 1.000 | 2.81 | 1.060 | 9.25 | 0.616 | 7.18 | 0.576 |
| 1,1-Dichloroethane | 5.05 | 1.012 | 3.18 | 0.738 | 2.73 | 0.764 | 4.85 | 0.447 | 5.70 | 0.460 |
| Ethyl Tert Butyl Ether (ETBE) | 2.60 | 1.813 | 2.28 | 1.310 | 3.49 | 1.340 | 2.68 | 0.805 | 2.45 | 0.818 |
| trans-1,2-Dichloroethene | 7.80 | 0.594 | 3.21 | 0.440 | 2.82 | 0.442 | 7.60 | 0.275 | 7.15 | 0.284 |
| ethyl acetate | 10.66 | 0.104 | 9.96 | 0.077 | 11.82 | 0.090 | 8.21 | 0.050 | 11.27 | 0.053 |
| 2-Butanone | 10.01 | 1.039 | 10.48 | 0.790 | 8.71 | 0.907 | 13.64 | 0.535 | 0.998* | 1.053 |
| 2,2-Dichloropropane | 3.83 | 0.859 | 2.54 | 0.635 | 2.84 | 0.629 | 9.12 | 0.354 | 5.36 | 0.324 |
| Bromochloromethane | 5.99 | 0.355 | 5.10 | 0.257 | 2.09 | 0.262 | 5.30 | 0.161 | 9.51 | 0.172 |
| propionitrile | 12.27 | 0.096 | 14.39 | 0.067 | 11.56 | 0.075 | 11.03 | 0.040 | 6.37 | 0.042 |
| methacrylonitrile | 12.14 | 0.567 | 13.52 | 0.439 | 12.52 | 0.484 | 10.01 | 0.280 | 8.07 | 0.284 |
| THF | 7.66 | 0.227 | 11.59 | 0.171 | 6.94 | 0.195 | 8.92 | 0.110 | 13.30 | 0.116 |
| Chloroform | 7.61 | 1.055 | 13.75 | 0.831 | 7.37 | 0.812 | 9.59 | 0.497 | 9.83 | 0.504 |
| methyl acrylate | 8.24 | 0.628 | 5.41 | 0.480 | 5.15 | 0.518 | 4.49 | 0.294 | 5.29 | 0.313 |
| Dibromofluoromethane SUR | 5.67 | 0.537 | 13.58 | 0.434 | 7.47 | 0.418 | 14.23 | 0.272 | 14.00 | 0.277 |
| 1,1,1-Trichloroethane | 2.66 | 0.926 | 2.48 | 0.704 | 3.27 | 0.704 | 3.47 | 0.430 | 5.34 | 0.433 |
| 2-Chloroethylvinylether | 4.87 | 0.409 | 3.74 | 0.319 | 4.08 | 0.330 | 7.86 | 0.195 | 4.36 | 0.208 |
| Carbon Tetrachloride | 8.34 | 0.707 | 13.76 | 0.521 | 13.79 | 0.526 | 10.18 | 0.320 | 10.58 | 0.328 |
| 1,1-Dichloropropene | 2.47 | 0.818 | 2.43 | 0.617 | 2.22 | 0.618 | 4.69 | 0.382 | 6.95 | 0.392 |
| methyl acetate | 4.59 | 1.892 | 2.75 | 1.424 | 2.49 | 1.515 | 2.73 | 0.911 | 3.35 | 0.915 |
| isobutyl alcohol | 10.38 | 0.031 | 0.998* | 0.014 | 0.998* | 0.021 | 8.15 | 0.009 | 14.64 | 0.010 |
| Tert Amyl Methyl Ether | 2.89 | 1.771 | 2.74 | 1.291 | 3.54 | 1.334 | 3.57 | 0.817 | 3.85 | 0.825 |
| Benzene | 3.84 | 2.330 | 2.52 | 1.716 | 2.33 | 1.751 | 1.99 | 1.066 | 4.92 | 1.109 |
| 1,2-Dichloroethane | 3.23 | 0.826 | 2.28 | 0.620 | 1.89 | 0.636 | 4.85 | 0.394 | 9.43 | 0.414 |
| propyl acetate | 9.17 | 0.630 | 4.39 | 0.451 | 3.82 | 0.513 | 5.97 | 0.316 | 4.19 | 0.309 |
| Trichloroethene | 6.06 | 0.378 | 2.40 | 0.265 | 2.82 | 0.282 | 3.04 | 0.178 | 6.65 | 0.181 |
| 1,2-Dichloropropane | 3.01 | 0.354 | 4.57 | 0.247 | 3.08 | 0.261 | 2.97 | 0.164 | 3.8 | 0.161 |
| methyl methacrylate | 5.77 | 0.302 | 3.50 | 0.223 | 3.34 | 0.243 | 6.18 | 0.150 | 6.42 | 0.153 |
| Dibromomethane | 6.12 | 0.213 | 2.70 | 0.157 | 3.96 | 0.168 | 3.82 | 0.105 | 5.31 | 0.108 |
| Bromodichloromethane | 4.28 | 0.281 | 1.98 | 0.200 | 4.62 | 0.217 | 4.05 | 0.135 | 3.93 | 0.130 |
| 2-chloroethanol | 7.67 | 0.154 | 7.48 | 0.113 | 7.09 | 0.118 | 13.25 | 0.078 | 8.51 | 0.074 |
| 2-nitropropane | 6.00 | 0.144 | 11.49 | 0.104 | 10.58 | 0.117 | 7.52 | 0.064 | 11.15 | 0.062 |

| Compound | 0.2% Methanol | | 0.3% Methanol | | 0.6% Methanol | | 1.1% Methanol | | 2.1% Methanol | |
|-----------------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF | Curve %RSD | Ave. Curve RF |
| cis-1,3-Dichloropropene | 3.13 | 0.581 | 2.53 | 0.406 | 4.66 | 0.419 | 5.07 | 0.262 | 6.75 | 0.253 |
| 4-methyl-2-pentanone | 6.49 | 0.432 | 5.00 | 0.300 | 5.24 | 0.353 | 4.83 | 0.213 | 7.31 | 0.213 |
| Toluene-d8 SUR | 3.41 | 1.292 | 3.33 | 0.895 | 4.63 | 0.942 | 2.47 | 0.623 | 5.23 | 0.611 |
| Toluene | 3.80 | 0.899 | 2.70 | 0.639 | 2.54 | 0.679 | 2.97 | 0.435 | 4.96 | 0.426 |
| ethyl methacrylate | 4.01 | 0.542 | 5.75 | 0.384 | 4.36 | 0.421 | 5.69 | 0.261 | 5.99 | 0.251 |
| trans-1,3-Dichloropropene | 4.61 | 0.565 | 6.16 | 0.383 | 5.76 | 0.404 | 6.96 | 0.249 | 8.28 | 0.244 |
| 1,1,2-Trichloroethane | 3.29 | 0.322 | 2.88 | 0.232 | 2.40 | 0.246 | 3.74 | 0.158 | 5.36 | 0.154 |
| Tetrachloroethene | 6.73 | 0.357 | 6.97 | 0.231 | 8.96 | 0.243 | 9.42 | 0.160 | 4.67 | 0.143 |
| 1,3-Dichloropropane | 2.93 | 0.569 | 2.09 | 0.408 | 2.91 | 0.430 | 1.98 | 0.278 | 3.97 | 0.274 |
| isopropyl acetate | 6.50 | 0.097 | 1.69 | 0.071 | 10.40 | 0.077 | 8.93 | 0.049 | 6.58 | 0.046 |
| butyl acetate | 4.48 | 0.279 | 4.18 | 0.198 | 2.92 | 0.221 | 6.21 | 0.137 | 4.98 | 0.130 |
| Dibromochloromethane | 6.27 | 0.358 | 8.32 | 0.249 | 9.40 | 0.265 | 11.76 | 0.163 | 13.39 | 0.157 |
| 2-Hexanone | 5.04 | 0.307 | 8.80 | 0.220 | 7.11 | 0.263 | 5.05 | 0.153 | 10.98 | 0.156 |
| 1,2-Dibromoethane | 5.30 | 0.331 | 2.72 | 0.240 | 2.49 | 0.258 | 2.92 | 0.163 | 4.73 | 0.161 |
| Chlorobenzene | 5.66 | 1.118 | 1.78 | 0.738 | 3.79 | 0.744 | 6.39 | 0.555 | 8.40 | 0.539 |
| 1,1,1,2-Tetrachloroethane | 3.89 | 0.376 | 6.68 | 0.250 | 6.69 | 0.248 | 7.26 | 0.178 | 9.29 | 0.169 |
| Ethylbenzene | 3.75 | 1.964 | 2.54 | 1.322 | 2.38 | 1.323 | 2.55 | 0.963 | 4.32 | 0.931 |
| Xylene (m+p) | 4.02 | 3.096 | 3.23 | 1.027 | 2.56 | 1.027 | 3.02 | 0.756 | 4.88 | 0.734 |
| Styrene | 5.03 | 1.242 | 3.59 | 0.829 | 4.48 | 0.818 | 4.47 | 0.592 | 5.25 | 0.579 |
| Xylene (o) | 4.32 | 1.566 | 3.38 | 1.035 | 2.02 | 1.036 | 2.96 | 0.757 | 3.52 | 0.722 |
| n-amyl acetate | 3.70 | 0.850 | 2.24 | 0.567 | 3.75 | 0.588 | 6.81 | 0.408 | 7.01 | 0.367 |
| Bromoform | 8.78 | 0.273 | 11.04 | 0.186 | 13.72 | 0.184 | 14.76 | 0.129 | 14.61 | 0.117 |
| Isopropylbenzene | 5.01 | 1.945 | 3.8 | 1.292 | 4.19 | 1.279 | 3.16 | 0.933 | 4.22 | 0.899 |
| cis-1,4-dichloro-2-butene | 8.18 | 0.189 | 12.37 | 0.126 | 12.50 | 0.127 | 0.999* | 0.081 | 0.997* | 0.076 |
| BFB SUR | 10.69 | 1.051 | 4.73 | 0.667 | 7.01 | 0.631 | 10.72 | 0.528 | 14.71 | 0.509 |
| Bromobenzene | 5.97 | 1.379 | 3.43 | 0.880 | 5.06 | 0.846 | 4.64 | 0.664 | 9.88 | 0.657 |
| 1,2,3-Trichloropropane | 3.57 | 1.183 | 4.66 | 0.785 | 4.03 | 0.779 | 6.86 | 0.581 | 4.59 | 0.556 |
| 1,1,2,2-Tetrachloroethane | 6.44 | 0.909 | 3.72 | 0.617 | 3.38 | 0.603 | 4.5 | 0.462 | 3.39 | 0.446 |
| n-Propylbenzene | 3.05 | 4.183 | 2.02 | 2.707 | 2.54 | 2.551 | 2.99 | 2.033 | 4.21 | 1.940 |
| trans-1,4-dichloro-2-butene | 6.87 | 0.327 | 5.76 | 0.221 | 6.54 | 0.225 | 6.55 | 0.158 | 5.10 | 0.143 |
| 2-Chlorotoluene | 4.22 | 0.788 | 2.12 | 0.514 | 3.51 | 0.487 | 2.57 | 0.386 | 5.77 | 0.374 |
| 4-Chlorotoluene | 4.54 | 0.824 | 1.79 | 0.535 | 2.77 | 0.509 | 3.38 | 0.406 | 10.01 | 0.402 |
| 1,3,5-Trimethylbenzene | 2.70 | 2.832 | 2.95 | 1.805 | 3.07 | 1.717 | 3.34 | 1.352 | 3.99 | 1.305 |
| tert-Butylbenzene | 2.80 | 2.430 | 2.63 | 1.558 | 2.96 | 1.475 | 3.02 | 1.176 | 4.67 | 1.130 |
| pentachloroethane | 9.48 | 0.360 | 8.07 | 0.285 | 6.74 | 0.272 | 0.997* | 0.199 | 9.08 | 0.216 |
| sec-Butylbenzene | 3.27 | 0.732 | 3.76 | 0.469 | 2.42 | 0.449 | 2.98 | 0.352 | 7.23 | 0.339 |
| 1,2,4-Trimethylbenzene | 4.20 | 2.917 | 2.97 | 1.865 | 2.76 | 1.778 | 2.92 | 1.395 | 4.56 | 1.349 |
| nitrobenzene | 8.91 | 0.077 | 11.32 | 0.029 | 14.98 | 0.031 | 0.997* | 0.016 | 14.74 | 0.015 |
| 1,3-Dichlorobenzene | 5.52 | 1.558 | 2.32 | 1.018 | 2.89 | 0.947 | 5.94 | 0.764 | 13.10 | 0.771 |
| 1,4-Dichlorobenzene | 8.12 | 1.620 | 5.59 | 1.072 | 9.00 | 1.019 | 11.54 | 0.818 | 7.56 | 0.768 |
| Isopropyltoluene | 3.75 | 3.001 | 4.36 | 1.915 | 4.46 | 1.818 | 3.89 | 1.437 | 5.63 | 1.375 |
| 1,2-Dichlorobenzene | 4.87 | 1.510 | 1.845 | 0.992 | 3.38 | 0.930 | 4.16 | 0.739 | 7.02 | 0.719 |
| n-Butylbenzene | 4.31 | 2.643 | 3.83 | 1.722 | 4.10 | 1.618 | 6.08 | 1.257 | 7.05 | 1.225 |
| 1,2-Dibromo-3-chloropropane | 8.86 | 0.204 | 7.93 | 0.135 | 7.02 | 0.135 | 10.46 | 0.096 | 7.87 | 0.091 |
| 1,2,4-Trichlorobenzene | 4.50 | 0.945 | 3.02 | 0.633 | 8.22 | 0.608 | 4.12 | 0.458 | 12.11 | 0.465 |
| Naphthalene | 8.98 | 3.167 | 4.73 | 2.097 | 2.22 | 2.056 | 5.48 | 1.528 | 5.75 | 1.499 |
| Hexachlorobutadiene | 5.07 | 0.334 | 11.38 | 0.237 | 5.40 | 0.211 | 7.45 | 0.161 | 14.66 | 0.170 |
| 1,2,3-Trichlorobenzene | 7.84 | 0.903 | 2.55 | 0.600 | 4.15 | 0.570 | 4.21 | 0.428 | 8.93 | 0.437 |
| Average | 6.15 | 0.907 | 5.40 | 0.619 | 5.60 | 0.619 | 6.50 | 0.422 | 7.52 | 0.427 |

*Linear Regression Value

Table 6: Data Summary

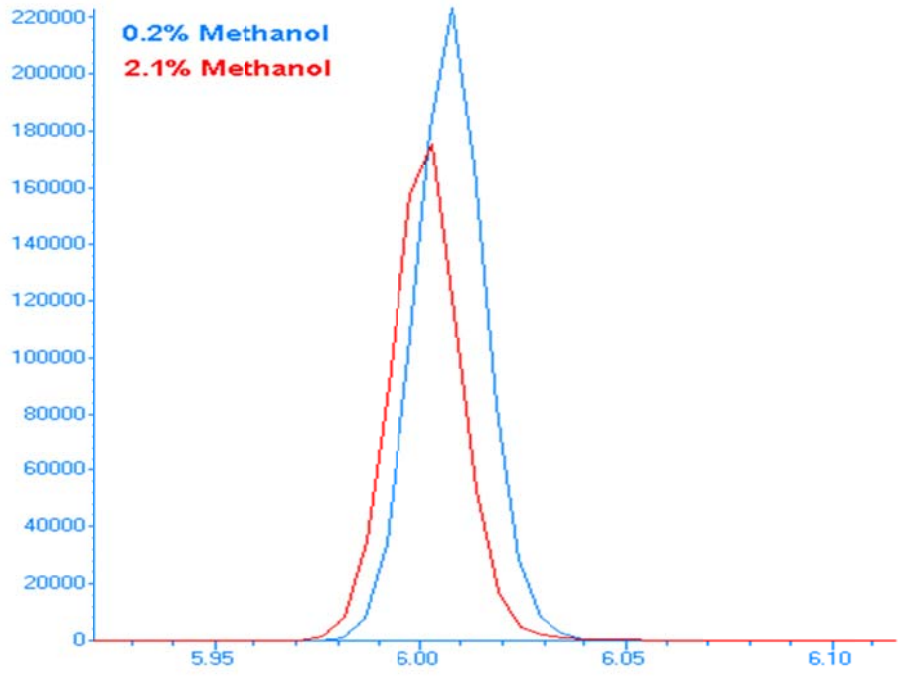


Figure 1: Overlay of Bromoform Response Ion 173 (Blue plot 0.2% Methanol, Red plot 2.1% Methanol)

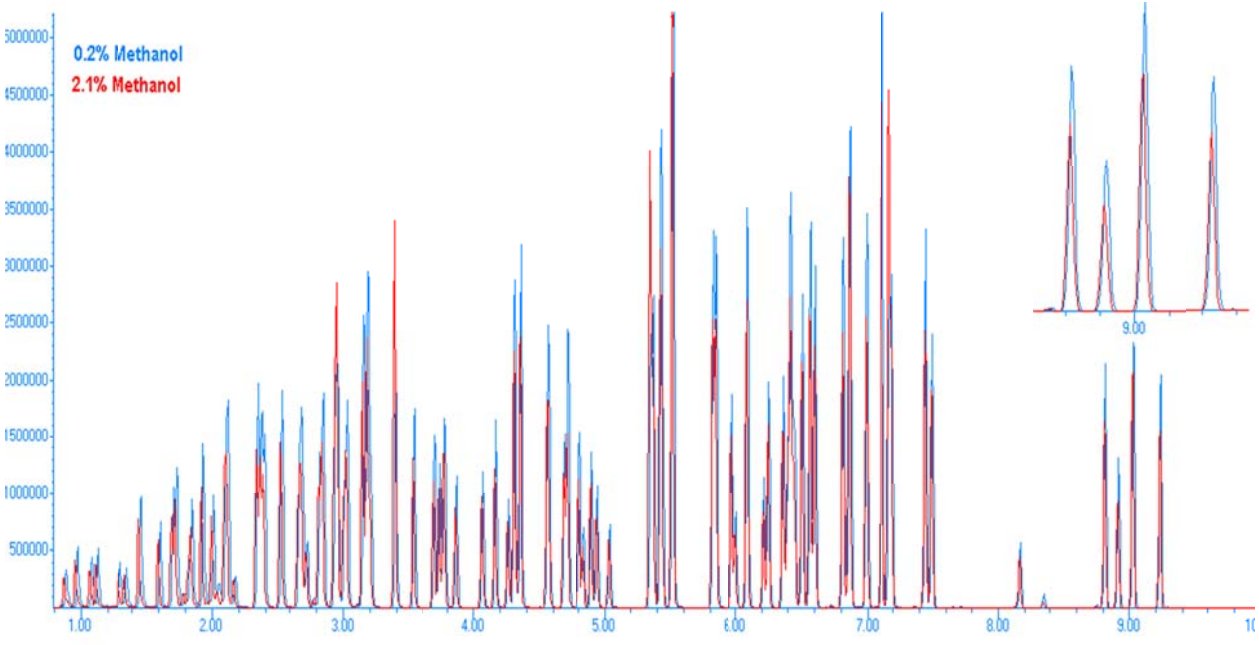


Figure 2: Overlay of 50ppb Standard Chromatograms with 0.2% (Blue) and 2.1% (Red) Methanol

Conclusion:

The Encon Evolution proved to be an excellent system for handling large amounts of methanol. Compound responses passed for all of the SPCC compounds up to 2.1% methanol, but the Bromoform response decreased dramatically with the increased percent of methanol in the standards. Curve linearity was consistent through the study up to 2.1% methanol; however at 5.1% methanol linearity could not be achieved. Overall, 5.1% methanol in the standards was too much for the system to handle and the recommendation would be to introduce less than 2% methanol for better results. It should be noted that the lower the methanol volume in the calibration standards produced the optimum compound responses. (For a standard preparation illustration with 0.2% methanol refer to Appendix A.)

Appendix A

Standard and Curve Preparation Illustration for 0.2% Methanol

To Make an 8260 Standard at 200ppm Diluted in P&T Methanol

| Amount | Restek Part # | Standard | Concentration | Final Vol. |
|--------|---------------|------------------|---------------|------------|
| 200µl | 30265 | 2-Cleve | 2.0mg/ml | 2.0ml |
| 200µl | 30633 | Cal Mix #1 | 2.0mg/ml | 2.0ml |
| 200µl | 30042 | 502.2 Cal Mix #1 | 2.0mg/ml | 2.0ml |
| 200µl | 30489 | Acetates | 2.0mg/ml | 2.0ml |
| 200µl | 30465 | Cal Oxy | 2.0-10.0mg/ml | 2.0ml |
| 200µl | 30287 | 1,4-Dioxane | 2.0mg/ml | 2.0ml |
| 160µl | 30073 | Surr. Mix | 2.5mg/ml | 2.0ml |
| 80µl | 30006 | VOA Cal Mix #1 | 5.0mg/ml | 2.0ml |

Use 2ml volumetric flask and dilute standards to 2.0ml in purge and trap methanol

To Make a 10x Dilution of the 8260 Standard (From 200ppm to 20ppm) Diluted in P&T Methanol

200µl of 200ppm standard diluted to 2.0ml in purge and trap methanol

To Make an 8260 Internal Standard at 50ppm Diluted in P&T Methanol

| Amount | Restek Part # | Standard | Concentration | Final Vol. |
|--------|---------------|----------|---------------|------------|
| 125µl | 30074 | 8260IS | 2.0mg/ml | 5.0ml |

Use 5ml volumetric flask and dilute standards to 5.0ml in purge and trap methanol

Note: Restek Standards used for this Standard Preparation example. Standard choices and concentrations are dependent on Customer needs.

To Prepare an 8260 Curve Diluted in De-ionized/UV Treated Water

| Concentration | Standard | Standard Amount | Final Vol. |
|---------------|----------|-----------------|------------|
| 0.5ppb | 20ppm | 2.5µl | 100ml |
| 1ppb | 20ppm | 5µl | 100ml |
| 2ppb | 20ppm | 10µl | 100ml |
| 5ppb | 20ppm | 25µl | 100ml |
| 10ppb | 20ppm | 50µl | 100ml |
| 20ppb | 200ppm | 10µl | 100ml |
| 50ppb | 200ppm | 25µl | 100ml |
| 100ppb | 200ppm | 50µl | 100ml |
| 200ppb | 200ppm | 100µl | 100ml |

Water Standards

Fill 40ml Vial with final standard leaving no headspace in the vial.

Soil Standards

Add 5ml of final standard to a 40ml vial.