

TRACE LEVELS OF BTEX IN WATER USING HAND-PORTABLE HPLC

INTRODUCTION

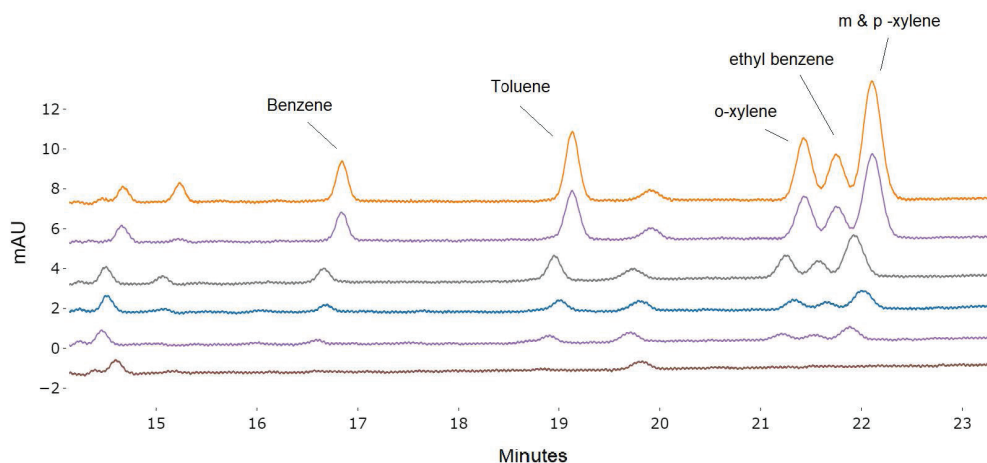
With on-column UV detection in packed capillary HPLC the analysis of low levels of analytes is challenging because of the short path length of the detector. Columns installed in the Axcend Focus LC cartridge have internal diameters in the 0.15 to 0.25 mm range for “on-column” detection, much shorter than the 1 to 10 mm path lengths in most HPLC systems. One way to compensate for this is to load a larger sample amount onto the column.

This application note shows how low levels of benzene, toluene, ethyl benzene, and xylenes (BTEX) can be determined in water with the use of relatively large injection volumes. The standard injection valve typically delivers a 40 nL injection volume. Substituting this small internal sample loop valve with a larger external loop enables injections of a wide range of sample volumes from 200 nL and higher.

OVERVIEW

This brief application note demonstrates the use of hand-portable capillary HPLC by the Axcend Focus LC for BTEX compounds in water—specifically, with a 5 μ L external injection loop and UV-absorption detection at 255 nm.

CHROMATOGRAPHIC OVERLAY



Successive runs of BTEX in a water sample. Concentrations are from top to bottom 6, 4, 2, 1, 0.5, and 0 ppm for each component.

APPLICATION DETAILS

In order to use a relatively large injection volume in any chromatographic system, it is necessary for the sample to be in a matrix that has low elution strength compared to the mobile phase at the start of the chromatographic run. With perfect trapping, detection limits are expected to decrease inversely proportional to the volume of the sample that can be loaded onto the column. Therefore, with a 5 μ L loop, the detection limits should be 125 times lower than with a 40 nL loop.

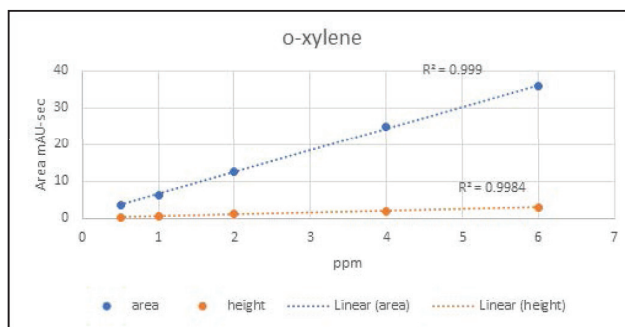
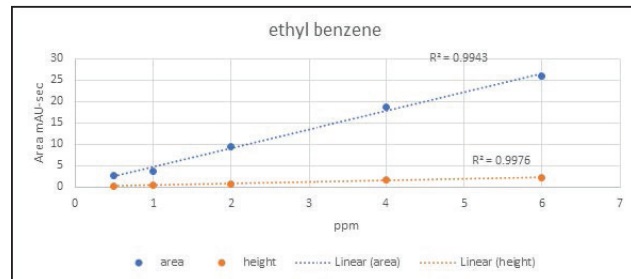
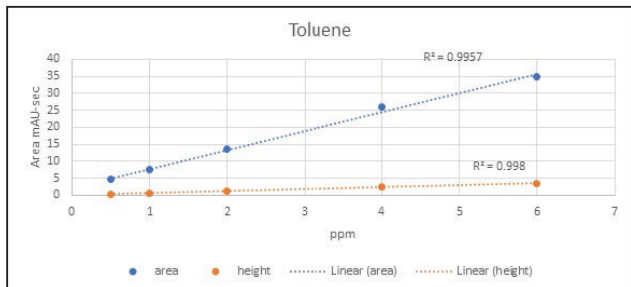
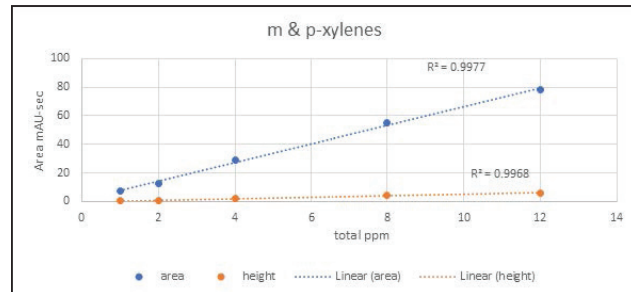
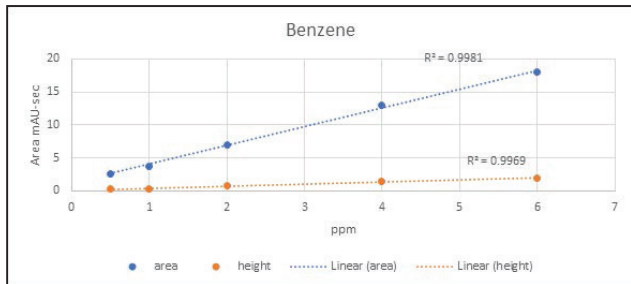
There are two important consequences of having a large injection loop. First is the method for analysis must allow for sufficient time to load the sample in the sample loop onto the column. The mobile phase, sample, and analytes can all have different viscosities, which result in different lengths of time required for each of these to migrate through the column. During this period, different sample viscosities cause the gradient to reach the column at different times, causing variations in retention time. The second is that large injection volumes contribute to the dwell time, which also has an impact on the chromatography because it takes a longer time for gradients to reach the column when the injection loop is in-line.

To mediate these concerns, an initial isocratic equilibration time must be long enough to allow the previous sample to completely elute from the column and the initial mobile phase composition to be reestablished in the column. Switching the sample loop out of the mobile phase flow path after sufficient time has passed to remove the sample from the loop will reduce the dwell volume in the system. This will allow the gradient to reach the column faster. Operating under pressure control also helps since the chromatographic conditions stabilize to a reproducible state more quickly than under flow control. Taking the sample loop out of the flow path before starting the gradient also assures that the sample loop will always be filled with a weak solvent before the next run. When the loop is left filled with Acetonitrile at the end of a run there is the potential of precipitating salts or other components soluble only in water when the next sample fills the loop. These could then be washed to the column and cause temporary or permanent plugging. Therefore, it is always important to filter samples before analysis to prevent plugging.

SUMMARY

The quantitative data obtained from these runs are close to the detection limits. Nevertheless, they exhibited good reproducibility and linearity. Run-to-run data for the compounds showing linearity for both peak area and peak height of benzene, toluene, ethylbenzene, and the xylenes are shown in the plots above. These results prove the use of hand-portable capillary HPLC by the Axcend Focus LC for BTEX compounds in water is viable.

LINEARITY PLOTS



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