Minimizing Carryover During Dioxin Analysis Using the Xevo TQ-S with APGC

Adam Ladak, Waters Corporation, Beverly, MA, USA
Kari Lynn Organtini and Frank L Dorman, Pennsylvania State University, University Park, PA, USA

GOAL

To eliminate carryover from sample to sample to ensure accurate quantification of unknown dioxin and furan containing samples.

BACKGROUND

Dioxins and furans are produced when organic compounds are incinerated in the presence of chlorine for example during PVC production, paper bleaching, and from natural sources such as volcanoes. Dioxins are extremely toxic and readily bioaccumulate in many animal species due to their lipophilic properties. They are also suspected mutagens and carcinogens. They are also suspected mutagens and carcinogens.

The Xevo TQ-S with Atmospheric Pressure GC (APGC) has provided a very sensitive detection system for the accurate determination of dioxins and furans at regulatory levels. During the analysis of samples of an unknown concentration, extremely high levels of these compounds may be observed. Therefore there is the potential for carryover of target compounds into the following sample injection. The consequence of this would be a falsely elevated quantitative result.

The carryover was investigated on an APGC fitted with a split/splitless injector coupled with a Waters® Xevo TQ-S. A single goose-neck splitless liner was installed into the injector. Five nonane washes were performed pre and post injection. A 1 in 10 dilution of the CS 5

Carryover was significantly reduced to 0.007% which is a 20-fold decrease compared to the original values. This virtually eliminated the chance of a false positive result.
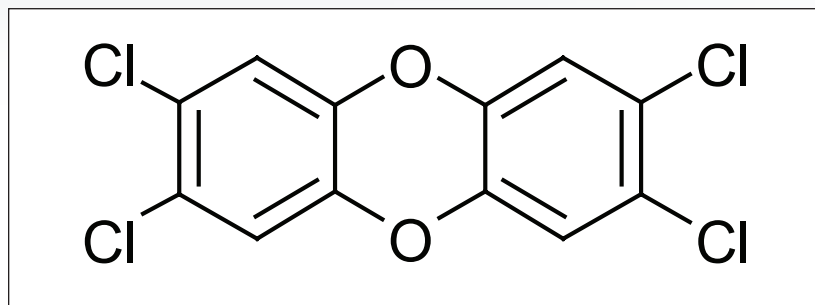

Figure 1. 2,3,7,8-Tetrachlorodibenzodioxin (TCDD).

standard from Wellington Laboratories (Guelph, ON) was injected followed by several nonane blanks.

The initial carryover, calculated using peak height, was 0.15% (Figure 2). This level of carryover could cause inaccurate quantification in samples analyzed immediately following this in a batch analysis. Experiments were performed in order to reduce the carryover observed to an acceptable level.

THE SOLUTION

A number of possible approaches to reduce carryover were investigated:

- Reducing the number draw/purge cycles during wash
- Placing glass wool in the liner
- Changing the type of autosampler syringe
- Changing solvent washes
- Using different liners

Waters
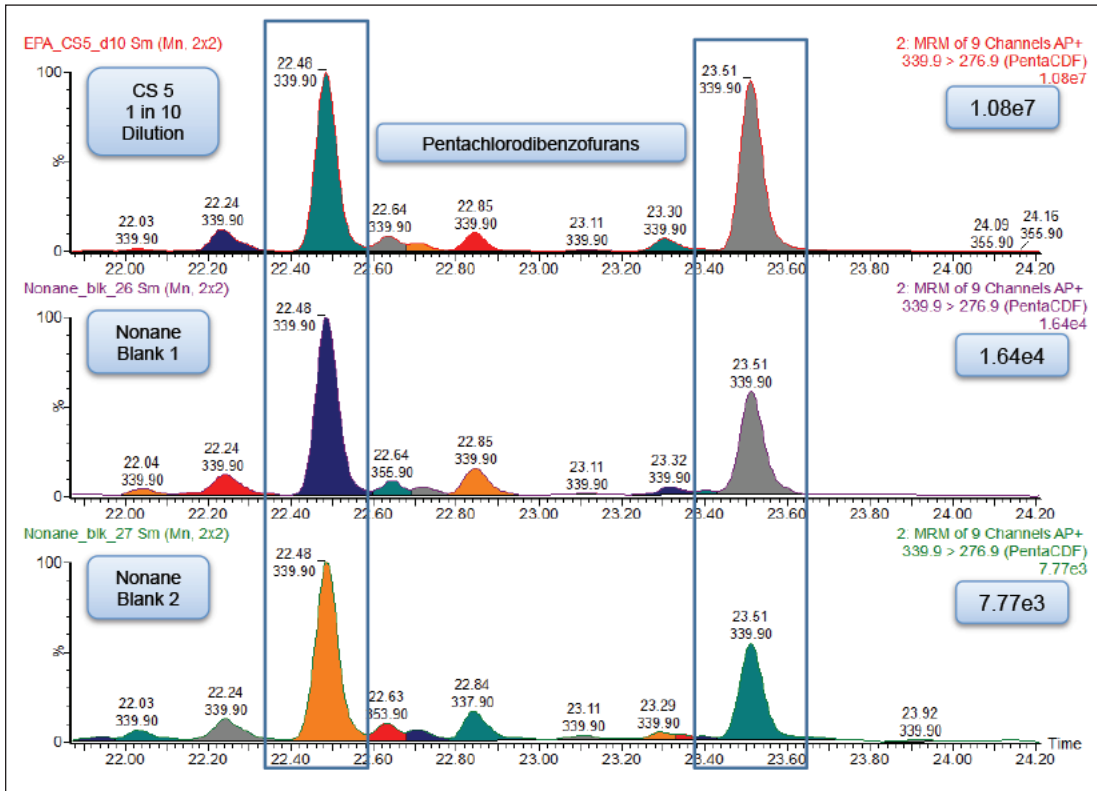THE SCIENCE OF WHAT'S POSSIBLE.®

Figure 2. Extracted ion chromatograms for pentachlorodibenzofurans displaying ~0.15% carryover prior to the implementation of optimized injection cycle parameters.

Each of these approaches were tested to see how they affected the carryover from the 1 in 10 dilution of the CS 5 Standard to the response seen from the nonane blank injected immediately afterwards. The largest reduction in carryover was obtained by changing the wash solvents to toluene and nonane combined with the use of a Restek Uniliner (Table 1). By combining both of these changes, the carryover was significantly reduced to 0.007% which was a 20-fold decrease compared to the original values (Figure 3). This virtually eliminated the chance of a false positive result.

Parameter	Standard CS 5; 1:10 dilution (peak height)	Nonane blank (peak height)
No sample draw/purge cycles	31,500,000	23,900
No glass wool in inlet	8,250,000	2170
Vendor 1 syringe	9,040,000	920
Vendor 1 gas-tight syringe	8,190,000	1160
Vendor 2 gas-tight syringe	7,800,000	1320
Toluene/Nonane wash 1	2,760,000	528
Toluene/Nonane wash 2	8,580,000	838
Use of Uniliner 1.0 µl	4,170,000	126
Use of Uniliner 0.5 µl	2,420,000	166

Table 1. The effects of different injection cycle parameters (applied in sequence) upon the observed carryover for pentachlorodibenzofurans.
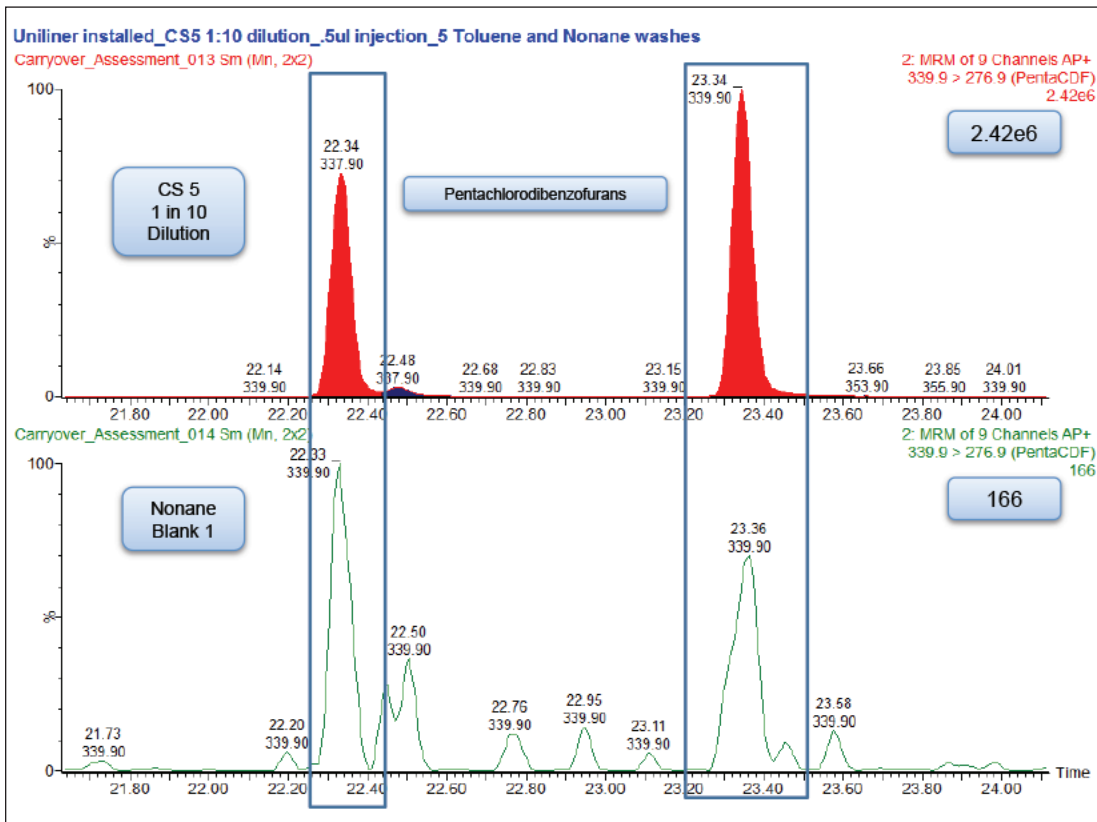
Figure 3. Extracted ion chromatograms for pentachlorodibenzofurans displaying greatly reduced carryover (0.007%) after the implementation of optimized injection cycle parameters.

SUMMARY

In order to ensure accurate quantification during dioxins and furans analysis on the Xevo TQ-S with APGC, the correct solvent washes and inlet liner should be used. Five washes of toluene followed by five washes of the sample diluent pre- and post-injection should be performed. Also a Uniliner should be installed into the inlet. The Sky 4.0 mm ID Drilled Uniliner Inlet Liner with Hole near Top from RESTEK (Part number 23311.1) was used successfully in the tests described here. The Uniliner design minimizes active sites in the sample pathway and reduces injection port discrimination. The analytical column connects directly to the bottom of the Uniliner via a press-fit seal, eliminating sample contact with any part of the injector below the column inlet and thus minimizing carryover.

By performing the analysis with this recommended configuration, confident and accurate analysis of dioxins and furans can be performed at low detection limits.

Waters

THE SCIENCE OF WHAT'S POSSIBLE.®