Corrective Action Summary Form

Date: 3/7/2022 12:02:58 PM **Tracking No:** AMSOP_372022_640

CA Title: Phthalate Contamination in 525 Analysis

Department: Organic Prep **Originator:** Jason Savoie

Responsible Party: Jason Savoie **Date Completed:** 3/7/2022

Description:

B2EHP is a common laboratory contaminant and used as a plasticizer in many plastic materials, including tubing commonly used by laboratories. B2EHP contamination of laboratory extraction equipment and glassware surfaces is a common cause of false positive sample results in semi-volatile methods such as EPA 525.2

The pattern of exceedance results occurs at a single laboratory (SGS-Wheat Ridge) and within a relatively narrow window of time (all laboratory extractions between 02/01/22 and 02/05/22, except for one on 01/17/22). Eight out of ten exceedance results are associated with preparatory batches having B2EHP detections in the method blanks (MB). In six of those eight cases the MB result is more than 40% of the sample result for B2EHP.

Although three of the ten exceedance results are from Trip Blanks, many of the associated field samples collected and shipped together did not contain detectable B2EHP, indicating that the field sampling procedures or containers themselves are an unlikely source of the contamination.

An investigation of the SGS-Wheat Ridge 525.2 QC results for all Red Hill samples confirmed that 23% of the MB records in EDMS contained reported concentrations of B2EHP ranging from 0.58 to 17.3 ug/L. Many of the associated matrix spikes in these batches exceeded control limits for B2EHP by up to 800% indicating sporadic cases of B2EHP contamination in all QC samples.

During a review of the laboratory raw data it was noted that all of the highest concentration B2EHP detections are associated with bis (2-ethylhexyl) adipate (B2EHA) detections at concentrations ~ 3% of the B2EHP. B2EHA is another common plasticizer and sometimes used as a replacement for phthalates such as B2EHP. The pattern of B2EHP + B2EHA association in samples from very different field locations is another indicator that the contamination has a common source and is from inside the laboratory, not from the drinking water samples.

The overall pattern of erratic detections in in a single laboratory over a narrow window of time indicates that intermittent laboratory contamination explains all of the reported B2EHP exceedances, including those results where the associated method blank appeared to be clean or the MB is < 10X the sample result.

The weight of evidence suggests are all the exceedance results are false positives attributable to laboratory contamination.

Root Cause:

The laboratory has done some investigating but has been unable to idenitfy the source of the contaminant. Due to the random nature of the occurrence throughout the analysis we do not suspect a contaminated solvent, surrogate, or other reagent which were all confirmed by screening on the GCMS but expect some sort of surface contact with the contaminant.

Immediate Fix:

Hold on 525 Analysis and Investigation – The laboratory immediately put a temporary hold on the analysis for 525 when identifying that the B2EHP contamination was a problem. All solvents, surrogates, and reagents used for analysis were screened by GCMS. The laboratory also screened our SPE cartridges prior to our conditioning procedure and although B2EHP was detected, results were within acceptable levels for analysis. The nitrile gloves as well as pipette bulbs were allowed to come in brief contact with solvent and analyzed by GCMS which also were within acceptable levels. After confirming that solvent and reagents were acceptable for analysis, the laboratory performed extraction and analysis on six method blanks before continuing with the 525 analysis. All six method blanks were less than the MDL for B2EHP. None of the laboratory screens pointed to a definitive source for the B2EHP contamination.

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Retraining of Staff – Although the B2EHP contamination has been sporadic, there was a period in late January into early February where the contamination began to show more frequently than previously observed. During this period the laboratory had brought on additional staffing to support the ongoing project and individual technique or lack of awareness to phthalates may have contributed to the increase in contamination. The phthalate contamination was communicated to staff performing analysis and contact with all plastic and rubber materials were minimized and/or eliminated where possible.

Glass Bottle Top Dispensers – All solvents were moved to enclosed glass bottle top dispensers that are compatible with the solvents they contain. Solvents from these bottles are routinely screened by GCMS to confirm that they are suitable for the 525 analysis.

Glass Luer Lock Syringes – The laboratory is taking measures to minimize the use of rubber transfer pipette bulbs as the sample can come in contact with the inside of the bulb if not handled correctly. Glass luer lock syringes will be substituted as a means to retrieving the sample from the concentration vessel and bringing to a 1ml final volume.

Bottle Custody Seals – The laboratory observed that the adhesive from the custody seals on the sample bottle leaves behind a residue when removed. During the analysis the bottle is inverted into a collection funnel and this portion of the bottle can come in contact with the sample. The laboratory does have a procedure to remove the residue from the bottle prior to transferring to the collection funnel but has taken greater measures to remove the residue and clean the surface of the bottle prior to analysis. Although the lab has not confirmed the adhesive from the labels as a source of the phthalate contamination we have requested that future bottles be prepared without the custody seal.

Confirmation Analysis – Client samples with results greater than the project screening limits have been re-extracted for confirmation despite acceptable QC.

Comments:

Although the laboratory has been unable to identify a single source for the B2EHP contamination we continue to monitor this analyte in our laboratory QC as well as client samples. Since the above corrective actions were put in place the laboratory has observed a significant decrease in the occurrence of B2EHP at or greater than the RL in the 525 analysis.

Followup:

Not applicable.