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June 18, 2024

Mr. Phil E. Richard
Hydrogeologist
Wisconsin Department of Natural Resources
Park Falls Service Center
875 4th Avenue South
Park Falls, WI 54554-1130

Ms. Erin Endsley
Hydrogeologist Program Coordinator
Wisconsin Department of Natural Resources
1701 N. 4th St
Superior, WI 54880

Subject: Response to WDNR Comments Re: Laboratory Certification
Former DuPont Barksdale Works
72315 State Highway 13
Town of Barksdale, Bayfield County, Wisconsin
FID No. 804009140
BRRTS No. 02-04-000156

Dear Mr. Richard and Ms. Endsley:

Thank you for meeting with representatives of The Chemours Company FC, LLC (Chemours) and AECOM Technical Services Inc. (AECOM) on May 7, 2024, to discuss the Former Barksdale Works site located in the Town of Barksdale, Wisconsin. This letter summarizes Wisconsin Department of Natural Resources' (WDNR's) comments related to the laboratory certification for nitroaromatic and nitramine organic compounds (NNOCs) analysis, presents our associated follow-up discussions with the analytical laboratory, and proposes a path forward.

In 2000, the laboratory analytical method for the standard list of NNOCs at the time was switched from SW-846 8330 to SW-846 8321A to obtain lower detection limits for the target compounds, reduce matrix interferences, and improve analyte separation. The SW-846 8321A method was implemented at the Eurofins Denver (formerly known as TestAmerica Denver) laboratory.

Dinitroxylene (DNX) isomers were added as a separate analysis starting in 2006. It was a gradual process as finding analytical standards for the individual compounds were very difficult. Based on Eurofins Denver method development work, they recommended a modified SW-846 8270 for these compounds.

The additional dinitrotoluene (DNT) isomers (3,4-DNT, 2,5-DNT, 3,5-DNT) and 2,4,6-Trinitro-3-xylene (TNX) were added to the SW-846 8321A analysis in 2008. Eurofins Denver was able to obtain a good separation of these additional analytes using the SW-846 8321A method.

Eurofins Denver has recently revisited the SW-846 8321B method and has been able to include the DNX isomers, which were historically analyzed using the SW-846 8270 method. This allows for a single method to be analyzed per sample and will improve efficiencies. Now that the DNX isomers can be included in the SW-846 8321B method, Eurofins Denver has repurposed the instrument and no longer offers the modified SW-846 8270 method.

SW-846 8321B, using liquid chromatography coupled with mass spectrometry (LC/MS), was chosen as the preferred method for analysis of NNOCs because it was found to provide better separation of the DNT isomers, include more target analytes, overcome

matrix interferences, and provide consistently lower reporting limits for comparison with screening criteria.

WDNR does not appear to provide a certification for the analysis of NNOCs using LC/MS. Switching to a WDNR certified method for NNOCs (SW-846 8330B using HPLC) would undermine our ability to separate, detect, and/or quantify many of the site-specific constituents present in groundwater.

In an email dated February 5, 2024, WDNR provided the following comment:

> "*Additionally, the Eurofins Denver lab is not approved for the method being utilized for groundwater analysis. The lab is analyzing Nitroaromatics and Nitramines via method 8321B using LC/MS. The Denver lab currently only has I lab certification for this method using LC, not LC/MS. Please ensure lab analysis is conducted using a lab with WI certification for the methods being used.*"

In our May 7, 2024 meeting, you stated that Eurofins could get certified and they have chosen not to do so.

As a follow up to these comments, AECOM met with representatives of the Eurofins Denver laboratory on May 17, 2024, to discuss the status of their WDNR certification. It is our understanding from Eurofins (copied in this letter) that WDNR indicated in 2023 that WDNR was not able to approve LC/MS as WDNR did not have resources available to assess a study (see attached email from WDNR dated June 5, 2023). This comment was related to herbicides analysis and modifications to the prep portion of the method, but it is mentioned in this letter as it applies to the audit and approval process for LC/MS.

Eurofins Denver believes a study is not warranted. SW-846 8321B Section 1.3 states "*This method may be applicable to the analysis of other nonvolatile or semivolatile compounds that are solvent-extractable, are amenable to HPLC, and can be ionized under thermospray introduction for mass spectrometric detection or can be determined by a UV detector.*" Barksdale's target NNOC list of constituents fits these criteria.

In addition, both the SW-846 8321B and SW-846 8330B methods allow aqueous samples to be extracted by SW-846 3535A. SW-846 3535A Section 1.2 states "*This technique may also be applicable to other semivolatile or extractable compounds. It may also be used for the extraction of additional target analytes or may employ other solid-phase media and extraction solvents, provided that the analyst demonstrates adequate performance (e.g., recovery of 70 - 130%, or at levels that meet project-specific recovery criteria) using spiked sample matrices and an appropriate determinative method of the type included as an 8000 series method in this manual.*"

Eurofins Denver has historically demonstrated adequate performance recovery for Barksdale's target NNOC list of constituents by SW-846 8321B with the SW-846 3535A prep method for aqueous samples.

WDNR has audited Eurofins Denver on SW-846 8321B for herbicides before with the only issue noted being the aforementioned modification to the prep procedure. There were no issues noted with the analytical portion of the SW-846 8321 procedure. WDNR has also audited and approved the SW-846 3535A prep method that is tied to the SW-846 8330B analysis.

Based on this information, Chemours is requesting that WDNR reaffirm historical concurrence to using SW-846 8321B (LC/MS) for NNOCs analysis for Barksdale groundwater samples.

If you have comments or questions that you would like to discuss, please contact me via email at Bradley.S.Nave@Chemours.com or by phone at (812) 406-7117.

Sincerely,

Bradley S. Nave
Principal Project Manager
Chemours Corporate Remediation Group

Attachments: Email from WDNR dated June 5, 2023

cc: Cary Pooler, AECOM
Eric Schmidt, AECOM

Sent: Monday, June 5, 2023 9:51 AM
To: Herrera, Amy <Amy.Herrera@et.eurofinsus.com>
Cc: Baker-Muhich, Brandy A - DNR <Brandy.BakerMuhich@wisconsin.gov>; Trainor, Tom J - DNR <Tom.Trainor@wisconsin.gov>
Subject: update on WDNR application
Importance: High

Hi Amy,

I hope you are well and the weather there is as beautiful as it is here.

A couple of items for the application:

If the LCS gets analyzed when WI samples has detections of PCBs (a standard that is assessed to the LCS limits), that is all that would be needed to add PCBs to your scope for A (aqueous) and S (solid) matrices. **Please update me by the end of June on this one remaining item for PCBs. If we don't hear back, the application time frame of one year (and the extra time after the staffing change) will expire.**

Unfortunately, at this time, we are not able to grant Pesticides for either the Aqueous or Solid matrices using the current methods available. I emailed the EPA/ORCR some questions for their input on the lab's extraction differences, and several points of concern were brought up. While, ideally, it could be possible for the lab to do a study (perhaps with Pesticide analogs) our department does not have the resources to determine the study parameters and then assess them. In addition, that may be outside our scope at this time.

> We think the EPA/ORCR should be encouraged to approve another method. This was a comment in their response to me on the issue of an LC/MS/MS method: "Yes, we see the need to revise the method or publish a new general liquid chromatography/tandem mass spectrometry-based determinative method in the SW-846 Compendium. The thermospray ionization source that is listed in this method is no longer manufactured, and it should be replaced with electrospray ionization, which is commonly used in contemporary commercial LC/MS instruments. We've gotten similar requests from states and commercial laboratories, but we do not yet have a defined timeframe for publishing a new or revised method."

Thank you and of course, if you have any additional comments or concerns about the Pesticide application, please feel free to reach out. Tom is available as well.

Respectfully,