OFFICE OF CHEMICAL SAFETY AND POLLUTION PREVENTION
WASHINGTON, D.C. 20460

April 22, 2024

Mr. Tim Whitehouse, Executive Director
Public Employees for Environmental Responsibility (PEER)
962 Wayne Avenue, Suite 610
Silver Spring, MD 20910

Dear Mr. Whitehouse,

Below you will find EPA's detailed response to PEER's March 4, 2024 letter related to EPA's May 18, 2023 release of our analysis of the ten pesticide products reported by Dr. Lasee et al., to have contained PFAS residues. In that release, EPA summarized the two methods used to analyze the products as well as the results obtained during testing. The first analysis followed the dilution method used by Dr. Lasee (with some modification to lower the detection limit) and the second analysis utilized a newly developed analytical method by the EPA. Both sets of data were released alongside the new method which was designed to specifically test for PFAS in complex matrices such as pesticide products. As you are aware, EPA's analysis using both methods showed no PFAS in any of the tested pesticide products at or above the method detection limits or the levels encountered as method background (sub parts-per-billion (ppb) levels). EPA remains confident in our findings, which differ from the results published by Dr. Lasee in the Journal of Hazardous Materials Letters, which showed Perfluorooctanesulfonate (PFOS) at concentrations between 3.9 parts-per-million (ppm) and 19 ppm in six of those ten products.

EPA requested the samples from Dr. Lasee (see attached email dated 11/4/22) who provided them with the understanding that our lab would test the samples for PFAS and subsequently release the results as part of the Agency's commitment to transparency. EPA, on multiple occasions, discussed the Agency's methodology and the results with Dr. Lasee prior to release of those results. Through those discussions, follow up emails (appended to this response), and re-testing by Dr. Lasee and his team, he agreed with EPA's findings, admitted that PFAS was not present in the samples above background levels, and subsequently petitioned the Journal of Hazardous Materials Letters to retract his publication approximately four weeks prior to EPA's release of its analysis.

Below, EPA has addressed each of the points in the PEER letter and provides a response that supports the Agency's conclusion that no PFAS was present in the samples provided by Dr. Lasee. Further, the response details EPA's numerous interactions with Dr. Lasee, including specific dates and scope of those discussions. The communication reinforces that EPA was clear to Dr. Lasee regarding the intent of the Agency to test samples utilizing both the dilution method and EPA's new method as well as its intent to release those findings.

Excerpt from PEER Letter:
"On November 4, 2022, Dr. Yaorong Qian of EPA's Analytical Chemistry Branch (ACB) emailed Dr. Lasee, asking him if he could send "an aliquot of the pesticide products you tested and found PFAS." Dr. Qian also stated, "Any information you provided will remain confidential." Dr. Lasee sent the requested pesticides on or about January 17, 2023, making it clear to Dr. Qian that EPA could not release the names/brands of the pesticides. Dr. Qian assured Dr. Lasee that this information would "not be revealed to outside parties without your explicit permission." EPA received the samples on January 19, 2023."

The letter further noted that Dr. Lasee spiked the samples to "avoid damaging their equipment."

EPA Response:

EPA contacted Dr. Lasee on November 4, 2022, for aliquots of the pesticide products (hereinafter referred to as "samples") he tested. Dr. Lasee agreed to provide the samples and shipped them to EPA's Analytical Chemistry Branch which received the samples on January 19, 2023. The amount received was approximately 1 gram of each product apart from one sample which was about 0.3 g (refer to email chain dated 1/19/23). In all the communications, either via emails or on Microsoft Teams calls, Dr. Lasee never, in any circumstance, told EPA that the names of the products or that the results should not be released. In EPA's correspondence with Dr. Lasee, we assured him that we would discuss any results with him prior to release. During one of the conversations on February 15, 2023, regarding EPA's test results, EPA informed Dr. Lasee of our communication plan to release the results of our analysis to the public. In an email dated February 23, 2023, Dr. Lasee expressed appreciation to Dr. Qian for showing a "great deal of courtesy throughout this process" as he acknowledged EPA's plan to release the results. He never expressed any reservation nor raised any objections.

When testing the samples, EPA followed the method described in Dr. Lasee's published paper, with one important exception. As described in EPA's study conclusions dated May 18, 2023 and released May 30, 2023 (BEAD PFAS Study Results 2023.pdf (epa.gov)), the final concentrations of the pesticide samples after dilution in our lab were 10X more concentrated than those in the method used by Dr. Lasee to enhance EPA's confidence that any PFAS present in the samples would be detected. Further, the methodology used by EPA for the analysis of these samples was repeated using three separate dilution approaches as described in EPA's findings dated May 18, 2023. Results of EPA's tests on these pesticide products showed no PFOS nor any other tested PFAS analytes in any of the samples, at or above our method detection limits or our method background levels.

Once EPA determined that our results differed from those obtained by Dr. Lasee and published in the Journal of Hazardous Materials Letters, Dr. Yaorong Qian of EPA's Analytical Chemistry Lab spoke with Dr. Lasee on February 7, 2023 and February 15, 2023 and followed up via email to convey the results as well as to discuss aspects of the study design (see email chains dated 2/15/23 and 2/24/23). Specifics of those conversations and emails included:

- Dr. Qian provided specifics around the study and offered his thoughts on why they differed from Dr. Lasee's results. As a follow up to those discussions and to better understand how Dr. Lasee conducted the study, Dr. Qian asked Dr. Lasee on February 13, 2023, for all associated data generated from the instrument(s) used for his sample analysis, including quality control (QC) samples (hereafter referred as "raw data"). EPA's analytical

results were sent in their entirety, including QC sample results, to Dr. Lasee on February 24, 2023. EPA never received the requested information from Dr. Lasee (See email chain of 2/15/2023 and 2/24/2023 appended to this response).

- EPA verbally discussed with Dr. Lasee its plan to release the results to the public upon completion of the analysis. EPA also shared with Dr. Lasee that EPA would utilize the most recent method developed which would have a much lower detection limit (at least 1000x lower) than Dr. Lasse's dilution method. If PFAS were present in the products at concentrations as reported in Dr. Lasse's publication or even at 1000x lower, the new method would detect them.
- On February 23, 2023 via email, Dr. Lasee shared with EPA that he had spiked several samples that were sent to EPA for analysis with PFOS. In response, via email on February 24, 2023, EPA asked Dr. Lasee about the amount of PFOS he spiked in the samples. EPA shared the calculations for the minimum level required for the analytical method (using the dilution method used by Dr. Lasee) to detect the spiked PFOS. During that time, Dr. Qian stressed that the dilution method, as performed by the EPA, would detect PFOS if it was present at ½ of the estimated limit of detection (0.5 ppm) or higher. Dr. Lasee responded via email on February 24, 2023 that he might have spiked the samples below 0.1 ppm, a level less than ½ of the limit of detection (See email chain dated 3/23/23).

Excerpt from PEER Letter:
"This [Dr. Lasee's indication to Dr. Qian that the samples were spiked with PFOS] was prior to running their newly developed method on their Sciex 6500+ on March 16th, 2023. Dr. Lasee did not share the spiked concentration with the EPA, as the expectation is that they would tell him what the concentrations were."

EPA Response:

As noted earlier, Dr. Lasee shared with Dr. Qian that he spiked the samples with PFOS at concentrations likely below 0.1 ppm. Specific to the dilution method, if Dr. Lasee had spiked the pesticide products below 0.1 ppm, the spiked PFOS would not be detected. In the published paper, Dr. Lasee reported levels ranging from 3.9 ppm to 19.2 ppm of PFOS in six of the tested products, which is over 10X higher than EPA's method detection limit. EPA would have readily detected PFOS at those levels had PFOS been present.

EPA applied the newly developed method in February and March 2023 to the prepared samples. The new method has a limit of detection of about 0.2 ppb of each PFAS analyte in the pesticide products, which is over 1000X more sensitive than the dilution method used by Dr. Lasee that EPA initially followed. As part of EPA's quality control of sample analysis utilizing either the new method developed by EPA or the earlier dilution method, aliquots of pesticide product samples we obtained from Dr. Lasse were spiked with a suite of 29 PFAS (including PFOS) at 2 ppb level (for the new method) and 5 ppm (for Dr. Lasee's dilution method). The spiked compounds were successfully recovered at greater than 50% of the amount spiked, demonstrating that both methods used by EPA were able to properly detect and quantify the PFAS analytes if they were present at or above the detection limits of the two methods used. These quality control procedures further demonstrate that unless Dr. Lasee's samples were spiked at a concentration lower than the detection limit of 0.0002 ppm (0.2 ppb), PFOS would have been detected with EPA's new method. Furthermore, in the published paper, Dr. Lasee reported PFOS ranging from 3.9 ppm to 19.2 ppm in six of the tested products, which

is 10,000X higher than the new method detection limit. EPA would have readily detected PFOS at those levels had PFOS been present.

Excerpt from PEER Letter:
"EPA stated in their memo that "none of the 29 PFAS compounds…was detected in any of the samples above the instrument's background levels." This is patently untrue. EPA's Sciex 6500+ LC/MSMS test found evidence of 14 PFAS, including PFOS, in the pesticides."

EPA Response:

EPA's statement that "none of the 29 PFAS compounds was detected in any of the samples [received from Dr. Lasee] above the instrument's background levels" is true and accurate. EPA's Sciex 6500+ LC/MSMS is a very sensitive instrument in detecting PFAS. When blanks, such as solvents, are processed through the method and analyzed by this instrument, trace amounts of PFAS can be detected and are likely attributed to solvents, materials and supplies used during the preparation process. These trace levels of PFAS detected are treated as instrument and method background levels. If similar levels of PFAS were detected in samples, including blanks, the detected PFAS would also be treated as background. Since the samples are pesticide products, false identification of a target analyte is common, due to high level of matrix interference, attributed to the particular formulation of the products. Although four PFAS analytes (PFPeA, PFHpS, 6:2 FTS, and HFPO-DA) were identified by EPA in the pesticide samples with the dilution method, the levels were similar to those found in the blanks tested and the detects were clearly identified as laboratory background.

Results from the newer EPA method developed and used in February and March 2023 to analyze the pesticide samples received from Dr. Lasee suggested that the levels of the PFAS detected on Sciex 6500+ LC/MSMS in the pesticide product samples were equivalent to levels found in the blanks. Because of the low limit of detection of the new method (about 0.2 ppb), more trace levels of PFAS were detected than by the dilution method. At this low level of detection, it became critical to closely evaluate the possible detects near the detection limits. The occasional detects of the PFAS in some pesticide product samples were carefully examined and were determined to be background levels that were not attributable to the pesticide samples.

The reported PFOS levels by Dr. Lasee in these pesticide products were 3.9 ppm to 19.2 ppm, over 10,000x higher than the limit of detection of 0.2 ppb and background levels of the new method. Therefore, if PFOS was present at the levels reported by Dr. Lasee, it would have been detected by either the dilution method or EPA's newly developed method.

Excerpt from PEER Letter:
""Pesticide sample analysis shows that all the detected peaks in some samples are near the background levels as in blanks and control blanks (generally <2X of that in blanks). Therefore, all the peaks detected are all false positives and will not be reported." This is a false statement by the EPA's own data. PFAS concentrations significantly higher than < 2X were found in both the pesticide sample Dr. Lasee sent to the EPA and the products they purchased for 4:2 FTS, 6:2 FTS, 8:2 FTS, N-EtFOSAA, PFUdA, PFDA, PFOS, PFOA, and FOSAA."

EPA Response:

EPA stands by its analysis and conclusion. The Analytical Chemistry Branch (ACB) laboratory is ISO-17025 accredited. Our Standard Operating Procedures (SOPs) related to the reporting of results specifies that only values above the limit of quantitation are reliable and therefore reportable. Values above the limit of detection but below the limit of quantitation are not reliable. Detects below the limit of detection are likely false positive and are to be treated as background levels that were not attributable to the pesticide samples. EPA detected some PFAS in the pesticide samples at similar levels as those found in procedural blanks (i.e., water or solvents) and control matrix blanks (generally below 1 ppb, including PFOS). Across all samples tested (both purchased products and those received from Dr. Lasee), the detected levels (below 1 ppb) are more than 3 orders of magnitude lower than those reported in Dr. Lasee's findings.

Because PFAS are widely present in the environment and are commonly detected in procedural and control blanks, those levels are treated as background levels. Due to the high uncertainty in identifying PFAS near the background levels, a range of 3X of the background level is generally applied. The few detects of PFAS in some pesticide samples tested are those commonly seen at background levels, and the calculated values are below 3X. Given that these values are less than the 3X background level, those detects are not attributable to the pesticide samples with high confidence, per ACB's SOPs.

Excerpt from PEER Letter:

"EPA concluded that "Furthermore, since low amounts of PFAS are readily observed in the environment, incorrectly interpreted background data could be multiplied by a large dilution factor (if dilution was used as sample preparation), resulting in reporting of an overexaggerated concentration of a background PFAS or a false-positive identification. These large dilution factors utilized by Lasee et al. could have contributed to the high results obtained in that study." Had EPA reported Dr. Lasee's method blank instead of removing it, it would have showed that Dr. Lasee did *not* have background contamination. Removing the method blank from the data they presented is a serious scientific integrity violation."

EPA Response:

The statement that EPA removed Dr. Lasee's method blank data is incorrect. EPA asked Dr. Lasee repeatedly for his raw data, including the blank(s), samples, and other quality control data to understand his results given that EPA could not confirm his findings. EPA did not receive any of the requested data (see email chain dated 3/23/23). EPA has no knowledge of Dr. Lasee's method blank data nor any other quality control data. Based on the observations in EPA's analyses and experience in chemical residue analyses in general, EPA offered a likely explanation for Dr. Lasee's reported results. Importantly, after many back-and-forth discussions, in an email on May 30, 2023, Dr. Lasee stated that he and his two collaborating labs re-analyzed the same pesticide products and did not detect any PFAS in those same pesticide products and he requested the Journal to retract his paper (see email chain dated 5/30/23).

Excerpt from PEER Letter:

"Aliquots sent by Dr. Lasee were about 1 mL in volume, meaning EPA would not have been able to complete the extractions they claimed to have done."

EPA Response:

Dr. Lasee sent EPA about 1 g of each sample, except for one sample which was about 0.3 g. This is an adequate amount of sample to run a series of tests. EPA used 0.02 g to 0.1 g of each sample for the dilution method (Dr. Lasee's method). EPA's findings dated May 18, 2023 (BEAD PFAS Study Results 2023.pdf (epa.gov)) described three separate analyses performed using the dilution method. No PFAS was found in any of the samples, at or above our method background levels.

The newer method utilized for the analysis was validated by EPA for up to 4 grams using pesticide products purchased on the open market by EPA. These products were the same EPA registration numbers as Dr. Lasee's samples. Lesser amounts of samples (less than 4 g) can be used with this method. Except for one sample, which was not of sufficient sample size (0.01 g) for an accurate analysis using this new EPA method, all other samples (ranging in size from 0.2 g to 0.8 g) were successfully analyzed. Using less than 4 g (0.2 g to 0.8 g in this case) of samples without altering other steps of the method resulted in less matrix and cleaner target compound chromatographic peaks on the analytical instrument, with only a minimal (about 10x) dilution factor.

Excerpt from PEER Letter:

"The memo states that ACB's method "involves a more intense extraction and clean up procedure to isolate PFAS compounds from the sample matrix before instrumental analysis, thus reducing matrix interference which results in better/more accurate detection limits" – in other words, that ACB's methods were better than Dr. Lasee's. What EPA did not report is that their methods had substantial contamination. The results from the Sciex 6500+ LC/MSMS instrument, the instrument the EPA used to quantify their new method, showed background contamination for most PFAS analyzed."

EPA Response:

EPA's description of its method is correct. Dr. Lasee's method, the dilution method, is useful for analyzing compounds at high concentrations that do not have matrix interference. Formulated pesticide products typically contain high levels (percent) of active ingredients (AIs) and complex inert ingredients. A large degree of dilution must be used to dilute the AIs and inert ingredients for the analysis of PFAS in the pesticide products. PFAS, if present, would be expected to be at substantially lower concentrations than the AIs and inert ingredients. However, additional dilutions also further dilute the PFAS which are at very low concentrations to start with (if present in the first place), while the AIs and inert ingredients are still present and could interfere with the analysis of PFAS in the diluted samples. The presence of these complex matrices in the diluted samples would still complicate the analysis of PFAS which would be at low concentrations (if present in the first place).

EPA's new method removes most of the AIs and the complex inert ingredients from the samples. The resulting "cleaner" sample extracts have much less interference and ensures a more reliable and accurate analysis of the targeted PFAS analytes. The limits of detection of PFAS are also much lower because the samples (and PFAS) are concentrated instead of diluted as in the method used by Dr. Lasee and described above, ensuring the enriched analytes will be readily detected using the new method. The estimated limit of detection of PFAS utilizing EPA's method is about 0.2 ppb. In comparison, the limits of detection of the dilution method used by Dr. Lasee can vary from 0.2 ppm to 1 ppm for different PFAS, more than 1000X higher than EPA's newer method.

As mentioned above, the dilution method that Dr. Lasee employed is useful for analyzing samples with analytes at high concentrations, and without any matrix interference. However, with complex sample matrices, such as those of pesticide products, the instrument noise and/or a false identification of a target analyte could be misinterpreted and therefore must be examined closely. Furthermore, since low amounts of PFAS are readily observed in the environment and in the laboratory reagents and supplies, if background data is misinterpreted and then multiplied by a large dilution factor (as necessitated by the dilution method), the results could overly exaggerate the concentration of a background PFAS.

Excerpt from PEER Letter:
"EPA cited a table with all the product names, claiming it was from Dr. Lasee's paper. It was not, and in fact, Dr. Lasee had told EPA representative in a conversation that the names of the products could not be released, and EPA had assured him they would not release this information without Dr. Lasee's explicit permission."

EPA Response:

The statement that "Dr. Lasee had told EPA representative…. that the names of the products could not be released" is not true. Dr. Lasee never, in any circumstance, told EPA that the names of the products could not be released. It was EPA that offered to Dr. Lasee that we would speak to him before releasing any results and information. EPA communicated with Dr. Lasee numerous times throughout the testing period via emails and verbal conversations (Microsoft Teams meetings). During one of these communications, after sharing the test results with Dr. Lasee, Dr. Qian told Dr. Lasee that EPA planned to release the test results and all the information associated with the analysis to the public as part of the Agency's commitment to scientific transparency. Dr. Lasee never raised any objections to EPA releasing the information. EPA informed Dr. Lasee on May 30, 2023, about the release of the test results. Dr. Lasee thanked Dr. Qian and informed EPA that he enlisted two laboratories (Texas Tech University and Duke University) to repeat the tests on the same pesticide products. Dr. Lasee noted that those two laboratories, like EPA, did not find any PFOS. He also informed EPA that he had contacted the JHML (Journal of Hazardous Materials Letters) where the paper was published to retract his publication and he was waiting for a response from the Journal.

Excerpt from PEER Letter:
"Dr. Lasee used mass labeled internal standards for quantification, but the memo implied that he did not. These actions violated EPA rules embodied in its Scientific Integrity Policy which posits an "expectation" that the agency's "scientific work is of the highest quality, free from political interference or personal motivations." These actions also fly in the face of EPA's own Information Quality Act guidelines which purport to "to ensure and maximize the quality, including objectivity, utility and integrity, of disseminated information" by the agency. They also arguably constitute false official statements within the meaning of 18 U.S.C.§ 1001, a felony."

EPA Response:

EPA did not state nor imply that Dr. Lasee did not use mass labeled internal standards for quantification. Additionally, whether or not Dr. Lasee used mass labeled internal standards for quantification is irrelevant to the determination of whether PFAS were present in these samples.

EPA's released findings described the procedures that the Agency's laboratory used to analyze and verify if there was any PFAS in these pesticide samples. EPA used mass labeled PFAS standards and non-mass labeled PFAS standard in different procedures to demonstrate that the methods EPA used would have detected the PFAS in those samples if they were present. EPA's Analytical Chemistry Branch (ACB) maintained scientific integrity and is in compliance with established good laboratory practices.

If you have any questions or would like to speak with me directly, feel free to contact me at overstreet.anne@epa.gov.

Regards,

Anne Overstreet

Anne Overstreet, Director
Biological and Economic Analysis Division

Enclosures:

Timeline of Notable Correspondences Between OPP/BEAD's Dr. Qian and Dr. Lasee

11/4/2022	Contacted Dr. Lasee via email and asked if he could send aliquots of the products he tested. EPA received a response on the same day with the list of products tested. Dr. Lasee noted that he would contact USDA in Lubbock, TX to get the products.
1/19/23	EPA's Analytical Chemistry Branch received aliquots of ten pesticide products, plus one solvent blank.
2/7/23	Spoke with Dr. Lasee (Microsoft Teams meeting) about the samples and the results using his method (dilute and shoot) and not finding any PFAS.
2/13/23	Followed up with an email asking for the raw data of Dr. Lasee's analysis to figure out the possible explanations or different ways to confirm the results.
2/15/23	Spoke with Dr. Lasee (Microsoft Teams meeting) reiterated the request for specific data and material from his analysis. Dr. Lasee said he would contact Texas Tech for the information.
2/24/23	Sent Dr. Lasee EPA's test results using the dilution method via email.
3/8/23	Talked with Dr. Lasee (Microsoft Teams meeting) about EPA's test results and reiterated the need to see his raw analytical data.
3/23/23	Contacted Dr. Lasee via email and informed him that EPA had completed the analysis of the samples he provided using our newly developed method. No PFAS was found. Dr. Lasee responded that he was talking with the group from Texas Tech and would get back to Dr. Qian. No additional communication on the test results.
5/30/23	Informed Dr. Lasee via email that EPA was releasing the study results. Dr. Lasee responded via email that he had asked two laboratories to conduct the tests on the same samples for PFAS and those two laboratories did not find any PFAS in those

samples. Dr. Lasee asked the JHML to retract his publication approximately 4 weeks prior to EPA's release.

Attached Notable Correspondences between BEAD's Analytical Chemistry Branch and Dr. Lasee

11/4/22	Pesticide products you analyzed for PFOS
11/4/22	PFAS in pesticides
1/19/23	RE: Happy holidays
2/6/23	RE: Extraction and analysis of pesticides for PFAS
2/15/23	RE: Additional information
2/24/23	RE: Thank you and other things
3/23/23	RE: Thank you and other things
5/30/23	RE: EPA completes scientific testing of pesticide products for PFAS – Response to Journal publication