



PUBLIC EMPLOYEES FOR ENVIRONMENTAL RESPONSIBILITY

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PEER's RESPONSE TO EPA'S APRIL 24, 2024 LETTER

May 28, 2024

EPA's comment on page 1, 2nd paragraph: "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."

PEER's response: EPA is correct that these things were true early in its interactions with Dr. Lasee. However, this transparency and cooperation ended after Dr. Lasee revealed that he spiked the samples before he sent them to EPA. EPA did not share how they did their "re-testing," or the results of this "re-testing," or the new method they developed. Dr. Lasee did *not* "agree" with EPA's findings; in fact, he told EPA to repeat their tests because they did not find his spike. Dr. Lasee also could not agree with results he never saw, and EPA did not share the results from their newly developed method.

Further, Dr. Lasee and his coauthors asked for a retraction because their original results could not be reproduced. Dr. Lasee requested a retraction of his publication in the *Journal of Hazardous Materials Letters* without seeing EPA's test results. Had he seen their results, he would have given an update on the paper including the EPA's results.

EPA's comment on page 2, 2nd paragraph: "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."

PEER's response: Dr. Yaorong Qian told Dr. Lasee in his Nov. 4, 2023 email, "Any information you provided will remain confidential and will not be revealed to outside parties *without your explicit permission*" (emphasis added). That explicit permission was never given.

The fact that Dr. Lasee "never express[ed] any reservations nor raise[d] any objections" is irrelevant; Dr. Lasee relied upon EPA's written representation that it would not reveal the names of the products without his "explicit permission," which was never given.

In addition, EPA only quoted a short phrase from Dr. Lasee's February 23rd, 2023 email. The full sentence read:

“In the letter it was mentioned that the EPA would be releasing a report in the next few weeks off of the samples I sent to you. You've showed me a great deal of courtesy throughout this process, and I felt it was appropriate that I do the same to you. I spiked several of the samples I sent to you with PFOS. There are EPA employees and contractors that know that I did this.”

By way of context, Dr. Qian had just written a letter to Senator Hettleman of Maryland on PFAS analysis. Dr. Lasee's full comment (not the partial comment quoted by EPA) was a polite way of saying that he appreciated Dr. Qian writing the letter to Senator Hettleman at his request, and Dr. Lasee was going to show him similar courtesy. The fact that Dr. Lasee spiked the insecticide samples before he sent them to Dr. Qian indicating that the EPA analysis *should* have detected PFOS, and the fact they did not indicates there was an issue with Dr. Qian's work.

EPA's comment on page 2, 3rd paragraph: “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” (emphasis added).

PEER's response: The March 16, 2023 results from the EPA's QTRAP 6500+ Low Mass (which are the results EPA used in the May 18, 2023 memo) on PFOS (page 262 – 270) showed PFOS concentrations *above* the method detection limits (and within the calibration curve) and *above* background levels (0.0128 ng/ml) in five samples:

- 1 (0.0429 ng/ml)
- 2 (0.0379 ng/ml)
- 3 (0.0613 ng/ml)
- 4 (0.0372 ng/ml) and
- 5 (0.0797 ng/ml)
- 6 (non detect, although this sample was also spiked)

It is worth noting that these concentrations should be adjusted *upwards* due to dilution factors; because we do not know what dilutions EPA used, we cannot adjust the concentrations to realistic estimates. However, these results cannot be described as “no PFOS.”

Additionally, the spectra from these samples were not integrated correctly and neglected the peaks from the PFOS isomers. thus, biasing the results toward lower concentrations. In other words, EPA ignored certain peaks that were from PFOS isomers, which artificially reduced the amount of PFOS calculated in the sample (see Figure 1, below, for PFOS isomer peaks that were ignored by EPA).

Figure 1

1	RT (Exp. RT): 15.15 (15.20) min [Calculated Conc]: 0.0429 ng/ml Area: 20386 Area Ratio: 0.08 Sample Type: (Unknown)	
2	RT (Exp. RT): 15.15 (15.20) min [Calculated Conc]: 0.0379 ng/ml Area: 18313 Area Ratio: 0.07 Sample Type: (Unknown)	
3	RT (Exp. RT): 15.15 (15.20) min [Calculated Conc]: 0.0613 ng/ml Area: 60438 Area Ratio: 0.12 Sample Type: (Unknown)	

It is important to note that the PFOS standard used by Dr. Lasee to spike these samples contained multiple PFOS isomers. Dr. Lasee spiked these samples by weight at approximately 1 ng/g for samples 1 and 2, 5 ng/g for samples 3 and 4, and 10 ng/g for samples 5 and 6. EPA's much lower results suggest poor performance of their method; to put it plainly, the results were much lower than the spike itself, therefore something may be wrong.

EPA's comment page 3, 3rd paragraph: "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."

PEER's response: While it is true that the PFOS found in EPA's tests were lower than those Dr. Lasee found and reported in his published paper, the important point here is that the spikes should have been detected by EPA's new method that it claims, "was 1,000x more sensitive than the dilution method used by Dr. Lasee...". EPA appears to be saying that Dr. Lasee spiked the samples with PFOS concentrations that were too low, but the fact remains that the Sciex results should have detected them. Finally, PFOS was detected in five samples, as noted in the previous comment.

EPA's comment page 3, 4th paragraph: "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. Lasee 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."

PEER's response: The exact method EPA used to spike its own samples for method validation is not described in EPA's memo. Dr. Lasee spiked the samples sent to the EPA with a PFOS standard, vortexed the samples, allowed them to dry, and periodically vortexed the sample over the course of five days before he sent them to the EPA. This form of spiking is referred to as a "Matix Spike". It is a common quality control technique utilized in numerous analytical chemistry methods used to evaluate performance and precision of an analysis.

Dr. Lasee informed Dr. Qian of his methods during a telephone call. If the EPA: 1) spiked the samples just prior to their extraction, or 2) after dissolving the pesticides in methanol, this would result in drastically different recoveries than the spiking method Dr. Lasee used. Either of these two methods would also be less true to native recoveries than the method used by Dr. Lasee. Although EPA did not reveal which spiking method they used, it appears they may have spiked it right before they used it, which could account for the discrepancy.

EPA's comment page 4, 2nd paragraph: "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."

PEER's response: Analysis results from the Sciex 6500+ LC/MSMS for the developed method, in several instances, show concentrations that *directly* contradict 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." These instances in Dr. Lasee's samples and the insecticides obtained by the EPA are noted below.

All of the following numbers are in ng/ml (ppb) and were the values prior to dilution adjustment (meaning estimated product concentrations will be higher). Additionally, several of the chromatograms showed appropriate peaks, but were not integrated. Therefore, lack of PFAS *cannot* be established in these samples.

"Ob (oberon), Ped (Pedstal), Mara (Marathon), and Avid" were all pesticide products purchased by the EPA. Numbers 1-10 are the samples provided to them by Dr. Lasee. Highlighted sample/numbers are greater than three times the background, or not correctly quantified due to significant peaks that were ignored by EPA. Any samples above 0.0250 are within the calibration curve.

- 4:2 FTS
Solvent blanks showed no contamination
Method blanks showed no contamination
Ped – 0.096
- 6:2 FTS
Solvent blanks showed no contamination
Method blanks showed contamination <0.07
"Ped" – 26.2
"Mara" – not quantified, but had a significant peak
"Avid" – 0.64
2 - not quantified, but had a significant peak
5 – 0.92
6 - not quantified, had a significant peak
7 - not quantified, had a significant peak
8 – 0.54
9 – 1.89
10 – 1.25
- 8:2 FTS
Solvent blanks showed no contamination

Method blanks showed no contamination

10 – 0.248

- N-EtFOSAA

Solvent blanks showed no contamination

Method blanks showed contamination 0.027

4 – 0.11

- PFUdA

Solvent blanks showed no contamination

Method blanks showed contamination 0.0087

10 – 0.095

- PFDA

Solvent blanks showed no contamination

Method blanks showed contamination 0.0137

9 - not quantified, had a significant peak

10 - not quantified correctly, had a significant peak

- PFOS (none of the sample peaks were correctly quantified- i.e., EPA did not quantify the isomer peaks.)

Solvent blanks showed no contamination

Method blanks showed contamination 0.013

3 – 0.061

4 – 0.037

5 – 0.080

- PFOA

Solvent blanks showed no contamination

Method blanks showed contamination 0.021

6 – 0.22

10 – 0.37

- PFBS (issues with qualifying ion; could have PFBS in many samples, also could not.)

- FOSAA

Solvent blanks showed no contamination

Method blanks showed no contamination

2 – 0.030

- HFPO-DA (issues with qualifying peaks require further investigation; could be matrix issues)

Solvent blanks showed no contamination

Method blanks showed no contamination

“Ob” - not quantified, but had a significant peak

“Ped” - not quantified, but had a significant peak

“Mara” - not quantified, but had a significant peak

“Avid” - not quantified, but had a significant peak

2 - not quantified, but had a significant peak

3 – 0.049

5 – 0.140

7 - not quantified, but had a significant peak

9 – 0.204

10 - not quantified, but had a significant peak

- NaDONA (if concentrations exist, they are very low)

Solvent blanks showed no contamination

Method blanks showed no contamination

“Ob” - not quantified, but had a significant peak

“Ped” - not quantified, but had a significant peak; odd qualifying ion

1 – very small concentration

EPA’s comment page 5, 1st paragraph: “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."

PEER's response: As discussed previously, several detections by the ACB's Sciex 6500+ LC/MSMS from its March 16, 2023 analysis were both above detection and quantification (i.e. with-in the calibration curve), making them both reportable and reliable by EPA's own measure. Additionally, many PFAS in numerous samples showed values greater than *three times* the background level. EPA simply ignored these results in its May 18, 2023 memo. EPA discusses external ISO 17025 accreditation yet does not appear to have followed its own SOPs on reporting results.

EPA's comment page 5, last full paragraph: "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)."

PEER's response: The retraction request was because collaborating labs were unable to replicate the original results, and *not* because they did not find PFAS.

In an email sent to Dr. Yaorogn Qian on November 4, 2022 from Dr. Lasee's former EPA email (lasee.steven@epa.gov) under the subject of "PFAS in insecticides," there is an attached excel sheet name "insecticides.xlsx" that was provided by Dr. Lasee to Dr. Qian. The data contained in this document were the sample ID, formulation type, active ingredient, PFOS (mg/kg), brand, manufacturer, EPA registration number, EPA establishment number and lot number of the products tested by Dr. Lasee. This table was *not* included in Dr. Lasee's publication. Only the information from the sample ID, formulation type, active Ingredient, and PFOS (mg/kg) were in the paper. The additional information on product identification was not included in the interest of protecting the manufactures of the products and Dr. Lasee from retribution, a point shared with Dr. Qian by Dr. Lasee in their first Microsoft Teams meeting.

Table 1 from ACB's May 5, 2023 memo contains all the information from the shared excel document, in the same order, except ACB had rearranged the columns, removed sample ID, formulation type, EPA registration number, EPA establishment number lot number, and, most

importantly, *removed the “blank” from the table*. Therefore, it is correct to state that EPA removed Dr. Lasee’s method blank data.

The raw data is clearly a separate issue being linked together with the removal of the blank. There were method blank and quality control data presented in Dr. Lasee’s study, and its Supplementary material. Dr. Lasee made it clear to Dr. Qian during conversations that he did not have access to the instrument data after changing positions. Unbeknownst to Dr. Qian, the lab in possession of the requested data ultimately chose not to share the data due to ACB’s reputation, and disrespectful comments made towards Dr. Lasee by Dr. Qian during conversations.

EPA’s comment page 6, paragraph 4: “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.”

PEER’s response: The instrument used in Dr. Lasee’s study was a LC/QTOF fitted with a large injection loop (which allows for 0.5 mL injections). The typical injection is between 1 and 5 microliters (0.001 – 0.005 mL). We do not know what EPA’s injection size was, but the large one used in Dr. Lasee’s study is uncommon. The larger injection loop can achieve lower detection limits. To say that the ACB replicated Dr. Lasee’s method with the instrumentation they used is largely an inaccuracy. The functionality of the two instruments are too different.

ACB's results presented in the May 5, 2023 memo were derived from their Sciex 6500+ LC/MSMS instrument on March 16, 2023. These results show method/procedure blank contamination that is within or near their calibration curve (0.025 ng/ml – 20 ng/ml) for:

- PFPeA
- PFBA
- 6:2 FTS
- N-EtFOSAA
- PFDA
- PFOS
- PFNA
- PFOA
- PFHpA
- PFHxA and
- PFODA

This contamination was *not* noted in the ACB's May 5, 2023 memo. Contamination this frequent and this high should not be ignored and indicates poor quality results. Additionally, the poor (or lack of) peak integration of several samples from the Sciex 6500+ LC/MSMS instrument in the March 16, 2023 data indicates the ACB did not examine their data closely.

EPA's comment page 7, paragraph 3: "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."

PEER's response: In Dr. Yaorong Qian's November 4, 2022 email to Dr. Lasee he stated, "Any information you provided will remain confidential and will not be revealed to outside parties without your explicit permission." Dr. Lasee never gave any representative of the EPA "explicit permission" or any permission at all to share any information given to them.

In their first conversation, Dr. Lasee told Dr. Qian the reasons for not including the product identifying information in his study; specifically, that the products he tested were old and may not be reflective of current products, and that it could put a target on his back. Dr. Qian coerced

Dr. Lasee into cooperation with promises of confidentiality only to later betray that trust and exploit his concerns of retribution to keep him silent.

Dr. Lasee objected to releasing any results when he informed Dr. Qian that his method did not detect the PFOS spike. While trying to be polite, Dr. Lasee pointing out a method's inability to detect a matrix spike in this circumstance cannot be interpreted as anything other than objecting to the result of the ACB's results.

Additionally, after Dr. Lasee revealed he spiked the samples prior to sending them to Dr. Qian, EPA largely stopped communicating with him, no longer shared their results with him, and showed no transparency with their plans. Had Dr. Lasee been shown the Sciex 6500+ LC/MSMS results, he would have objected to EPA's claim that no PFAS were found, as the results of this test clearly showed PFAS and showed a great deal of contamination.

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