I have finished my peer review. It is now yours Eric.

David

From: Qian, Yaorong <qian.yaorong@epa.gov>
Sent: Thursday, February 16, 2023 9:11 AM
To: Graybill, Eric <graybill.eric@epa.gov>
Cc: Nguyen, Thuy <Nguyen.Thuy@epa.gov>; French, David <French.David@epa.gov>
Subject: B23-05b QA review

Hi Eric,

All the data files and checklist for B23-05b are on my OneDrive folder. I have sent you a link to it. After David is done, you can do a QAO review. Additional supplemental information is on SharePoint site under B23 Projects. I will move all the files to the SharePoint site after we brief the senior management.

Thanks,

I have finished my peer review. It is now yours Eric.

David

From: Qian, Yaorong <qian.yaorong@epa.gov>
Sent: Thursday, February 16, 2023 9:11 AM
To: Graybill, Eric <graybill.eric@epa.gov>
Cc: Nguyen, Thuy <Nguyen.Thuy@epa.gov>; French, David <French.David@epa.gov>
Subject: B23-05b QA review

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Thanks,

From:	Qian, Yaorong
То:	Overstreet, Anne (she/her/hers); Anderson, Neil
Cc:	<u>Nguyen, Thuy</u>
Subject:	Draft report memo on the test results of PFAS in Lasee"s pesticide product samples
Date:	Wednesday, March 8, 2023 4:12:00 PM
Attachments:	Results Memo-draft.docx

Hi Anne,

Here is the draft memo on the PFAS test results of Lasee's pesticide product samples. We also tested four pesticide products of the same brands that we purchased. All the tests show that these products are PFAS free.

Thuy have reviewed the memo.

Please review and let us know your comments and suggestions.

Thanks,

Hi Yaorong,

I believe the study report should be sufficient to show how the method was validated by using the spikes.

Thanks!

Stephen

From: Qian, Yaorong <qian.yaorong@epa.gov>
Sent: Thursday, March 9, 2023 11:13 AM
To: Giler, Stephen <Giler.Stephen@epa.gov>
Cc: Graybill, Eric <graybill.eric@epa.gov>
Subject: RE: PFOS Project

Hi Stephen,

We talked about the need for a write-up of the procedure. There is a study report (B23-05b Summary report) in the folder which has a section on the method. Will it suffice?

Thanks,

Yaoorng

From: Giler, Stephen <<u>Giler.Stephen@epa.gov</u>>
Sent: Wednesday, March 8, 2023 2:40 PM
To: Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Cc: Graybill, Eric <<u>graybill.eric@epa.gov</u>>
Subject: PFOS Project

Hi Yaorong,

I think I've gone over everything I need to and have just a couple of follow up questions for this project. Whenever you get a chance to meet, let me know and I can come by your office to discuss everything. I have also had a chance to take a look at Gideon's AAPCO as well.

Thank you.

Stephen Giler Chemist/Quality Assurance Officer (QAO) Analytical Chemistry Branch U.S. Environmental Protection Agency <u>Giler.Stephen@epa.gov</u> Office: (410) 305-2653

From:	Qian, Yaorong
То:	Nguyen, Thuy
Subject:	RE: Qian, Yaorong left a comment in "PFAS Memo for BEAD IO - Results Memo-draft"
Date:	Tuesday, April 11, 2023 1:00:00 PM
Attachments:	image001.png
	image002.png
	image003.png
	image004.png
	image005.png

Made some edits. It looks good.

From: Nguyen, Thuy <Nguyen.Thuy@epa.gov>
Sent: Tuesday, April 11, 2023 9:43 AM
To: Qian, Yaorong <qian.yaorong@epa.gov>
Subject: RE: Qian, Yaorong left a comment in "PFAS Memo for BEAD IO - Results Memo-draft"

I made some changes to the report. Please review. Will need to send it to Anne/Neil by end of day Thanks

Thuy

From: Qian, Yaorong <<u>no-reply@sharepointonline.com</u>>

Sent: Thursday, April 6, 2023 4:33 PM

To: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>

Subject: Qian, Yaorong left a comment in "PFAS Memo for BEAD IO - Results Memo-draft"

PFAS Memo for BEAD IO - Results Memo-draft.docx
,
Qian, Yaorong added a comment
Go to comment
Why am I receiving this notification from Office?

Microsoft

Privacy Statement | Notification Settings

From:	Overstreet, Anne (she/her/hers)
То:	<u>Qian, Yaorong; Nguyen, Thuy</u>
Cc:	Anderson, Neil
Subject:	Signatures Needed- PFAS Study Report
Date:	Tuesday, May 2, 2023 12:06:04 PM
Attachments:	BEAD PFAS Study Results Final 2023.docx
Importance:	High

Yaorong and Thuy, I believe this document can be finalized. I've provided final edits and removed all the track changes. I've also dated the memo for today.

If you are comfortable with the edits, please sign ASAP, make it into a PDF and return to me so I can get this moving to the IO along with the letter to the journal editors TODAY.

Thanks, Anne

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 Overstreet.anne@epa.gov

http://www.epa.gov/pesticides

From:	Overstreet, Anne (she/her/hers)
To:	Anderson, Neil
Cc:	Nguyen, Thuy; Qian, Yaorong
Subject:	Final Documents for Posting - PFAS
Date:	Thursday, May 4, 2023 8:34:46 AM
Attachments:	PFAS Journal Letter 05 2023.pdf BEAD PFAS Study Results Final 2023.pdf
Importance:	High

Neil, I've incorporated Mike's couple of suggestions into the letter, finalized and signed. I've added a cc to Dr. Steven Lasee so we need to send an electronic copy to him after we release our findings on the web and our OPP Update publishes next week.

Great work, Yaorong and Thuy. Let's move forward on getting the final method for testing pesticides containing surfactants to me and Neil at your earliest convenience. I'm certain we will get many questions related to that especially given the recent press around CBD's release. In the CLA meeting yesterday, they asked me about it again.

If you all could take a look at the web edits from Emily today and make sure the OPP Update is going smoothly through the process, that would be great. Whew, glad we're almost over the finish line on this one

Anne



Thank you Anne. We will try to get the method reviewed and finalized as quickly as possible.

Yaorong

From: Overstreet, Anne (she/her/hers) <overstreet.anne@epa.gov>
Sent: Thursday, May 4, 2023 8:35 AM
To: Anderson, Neil <Anderson.Neil@epa.gov>
Cc: Nguyen, Thuy <Nguyen.Thuy@epa.gov>; Qian, Yaorong <qian.yaorong@epa.gov>
Subject: Final Documents for Posting - PFAS
Importance: High

Neil, I've incorporated Mike's couple of suggestions into the letter, finalized and signed. I've added a cc to Dr. Steven Lasee so we need to send an electronic copy to him after we release our findings on the web and our OPP Update publishes next week.

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If you all could take a look at the web edits from Emily today and make sure the OPP Update is going smoothly through the process, that would be great. Whew, glad we're almost over the finish line on this one

Anne

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 <u>Overstreet.anne@epa.gov</u> http://www.epa.gov/pesticides

You are right, Stephen. B23-05b has been QA reviewed. You are to review B23-05C, which includes both method validation and sample analysis using the validated method. Some of the sample information is in B23-05b. The same samples were analyzed twice, once by dilution method (B23-05b) and once by the method in B23-05c.

We have some descriptions and comments in the PAC with regard to the data in B23-05c. There are confirmative analysis (qualitative) and quantitative data. Hopefully it will not be too confusing after you look at the comments.

B23-05a is not reviewed. As this time I do not think we need to review it. I think B23-05c would be a better method and would replace the B23-05a.

Thanks,

Yaorong

From: Giler, Stephen <Giler.Stephen@epa.gov>
Sent: Thursday, May 11, 2023 8:34 AM
To: Qian, Yaorong <qian.yaorong@epa.gov>
Subject: RE: PFAS method

Hi Yaorong,

I want to make sure I review the correct project folder for this one. If I remember correctly, B23-05b was QA reviewed by Eric. It looks like B23-05a contains a method validation for qualitative extraction only that has not been Peer Reviewed or QA Reviewed, so I assume I should not be reviewing that portion of the project... Am I just looking at the quantitative method validation for B23-05c along with the sample results? (To include the LOQ validation check using the SciEx instrument).

I want to make sure that I can give the project a thorough review without spending too much time looking through raw data files that are unnecessary.

Thank you!

Stephen

From: Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Sent: Wednesday, May 10, 2023 2:31 PM
To: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>; Giler, Stephen <<u>Giler.Stephen@epa.gov</u>>
Cc: French, David <<u>French.David@epa.gov</u>>

Subject: RE: PFAS method

Hi Stephen,

The data and method have been peer reviewed. David and I are going through the PAC and signing it off today. The data include the method validation (B23-05c) and Lasee sample analysis. Some information on the samples in in the folder of B23-05b.

Thanks,

Yaorong

From: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Sent: Wednesday, May 10, 2023 2:26 PM
To: Giler, Stephen <<u>Giler.Stephen@epa.gov</u>>
Cc: Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>; French, David <<u>French.David@epa.gov</u>>
Subject: PFAS method

Stephen

Have you had a chance to look at the new PFAS method? I know Yaorong completed the peer review on it yesterday

I want to send it out with the results of the Lasee's samples this Friday at the latest

Thanks

Thuy

Hi Thuy,

We spiked Lasee's samples at two different levels (we prepared three different sets of samples, with one set not spiked). The first set was spiked at about 9 ppm equivalent in the pesticide products (we spiked samples after dilution and the equivalent spiking level is back calculated to the undiluted products). The second set was spiked at 1 ppm equivalent in the pesticide products.

The recoveries of the spiked PFAS were between 60%-140% for different compounds in different samples at the 9 ppm spiking level. The recoveries of the spiked PFAS at 1 ppm were mostly between 40%-200%, with a few outliers. The 1 ppm level is near our detection limits (0.2 ppm-1 ppm are the detection limits for different compounds).

Thanks,

From:	<u>Giler, Stephen</u>
То:	French, David; Qian, Yaorong
Cc:	Nguyen, Thuy
Subject:	B23-05c PFAS PAC - Incomplete
Date:	Tuesday, May 16, 2023 2:33:57 PM
Attachments:	image001.png
	image002.png

Good Morning,

The current status for the QA Review of this project is:

INCOMPLETE

I had a few comments that I added to the PAC that should be addressed by both David and Yaorong. Responses to comments are not expected to have an effect on the final results.

Yaorong – There are a couple of spaces on the PAC that you did not initial in the Peer Review section, so those areas need to be reviewed and initialed by you as peer reviewer. The calibration standard prep calculations in the referenced notebook also need to be reviewed and initialed per our SOP and ISO guidelines. Yan has that notebook and the pages are referenced in David's notebook.

David – When everything is all fixed, let me know and I can go back and sign off on the QA review and add it to my master audit list and add this method to the master methods list. It also appears that there are a few duplicate files laying around in the project folder for validation results and the analytical method, so when you get a chance, please remove the files that are out-dated.

The project folder can be found on the SharePoint site at the following link: <u>B23-05c Method Validation and Pesticide Product Analysis ACB method</u>

B23-05c PFAS in Pesticide Products PAC.docx

Thank you!

Stephen Giler Chemist/Quality Assurance Officer (QAO) Analytical Chemistry Branch U.S. Environmental Protection Agency <u>Giler.Stephen@epa.gov</u> Office: (410) 305-2653 From:Overstreet, Anne (she/her/hers)To:Lasee Research & ConsultingCc:Anderson, Neil; Nguyen, Thuy; Qian, YaorongSubject:RE: EPA Completes Scientific Testing of Pesticide Products for PFAS - Response to Journal PublicationDate:Tuesday, May 30, 2023 4:16:38 PM

Dr. Lasee, thank you for the update. Much appreciated, Anne

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 <u>Overstreet.anne@epa.gov</u>

http://www.epa.gov/pesticides

Please note that I sent this at a time that was convenient for me without expectation for a response outside of business hours. If you receive this email outside of your normal working hours, please know that I do not expect a response until you are back at work during your normal hours.

From: Lasee Research & Consulting <hello@laseeconsulting.com>

Sent: Tuesday, May 30, 2023 4:11 PM

To: Overstreet, Anne (she/her/hers) <overstreet.anne@epa.gov>

Cc: Anderson, Neil <Anderson.Neil@epa.gov>; Nguyen, Thuy <Nguyen.Thuy@epa.gov>; Qian, Yaorong <qian.yaorong@epa.gov>

Subject: Re: EPA Completes Scientific Testing of Pesticide Products for PFAS - Response to Journal Publication

Hello Anne,

Thank you for reaching out. After Dr. Yaorong Qian informed me of his results, I contacted the lab I did the research in and that spurred their own investigation. Samples were collected and tested using 1633 by TTU and a replicate was sent to Duke to do the same. Neither labs found the PFOS we originally detected. So we contacted JHML to see what they wanted to do. That was 4 weeks ago, we have not gotten a response.

Here was our email:

"Dr. Shaily Mahendra Editor-in-Chief *Journal of Hazardous Materials Letters*

On behalf of all authors (the senior author is copied on this email), I am requesting that the article published in *Journal of Hazardous Materials Letters* (**Volume 3, November 2022, 100067**) entitled, "Targeted analysis and Total Oxidizable Precursor assay of several insecticides for PFAS" be retracted.

The article contains the results of liquid chromatography-quadrupole time of flight-mass spectrometry (LC-QTOF-MS) determinations of several insecticide formulations for perand poly-fluoroalkyl substances (PFAS). The LC-MS determinations reported in the article met all of the quality assurance requirements for identifying PFAS (in this case perfluorooctane sulphonic acid, PFOS) including calibration (R2 of 0.99 or higher), check standards (70-130% accuracy), internal standard recovery (50-150%), identification of both quantifier and qualifier ion transition masses, blanks (non-detect), and validation. Additionally, LC-QTOF-MS is a high-resolution instrument; the PFOS in the initial samples met criteria for exact mass matching (within 5 ppm). However, the results of repeated targeted analysis of those same insecticide formulations in our lab and an external academic lab have not been successful in duplicating those initial results. Data quality in the repeated analysis are of equal or better quality than the original dataset in terms of the acceptance criteria previously mentioned. As a result, the weight of evidence indicates that PFOS is not present in those insecticide formulations as we had previously reported in the article.

We stand behind the PFAS determinations in soils and vegetation reported in the article.

Please let me know if additional steps are needed in order to retract this article from JHML, or if you would like for us to provide additional details regarding the initial and final analysis.

Sincerely,

Steve Lasee, Ph.D.

On behalf of all co-authors: Kaylin McDermett Naveen Kumar Jennifer Guelfo Paxton Payton Zhao Yang Todd Anderson"

I would like to apologize to Dr. Qian, I did not intend to go dark on him, I was not a part of the investigation I mentioned and it took months to complete. I felt it was best to get a response from the journal before I respond to Dr. Qian. We intend on doing another investigation of the site due to having found several PFAS in all other samples over several sampling events at the site. Our investigation of the site was ultimately incomplete due to the COVID 19 outbreak and associated shut down and the graduation of most of the key authors. Our conversation with the journal was going to be on weather to retract the paper or complete a follow up investigation and update the paper.

There are quite a few other factors impacting our intended actions. If you would like to speak about them, I am more than happy to talk about it.

Thank you,

Steven Lasee, MS PhD Environmental Toxicologist

(920) 264.4909

hello@LaseeConsulting.com LaseeConsulting.com

----- Original Message ------

On Tuesday, May 30th, 2023 at 2:17 PM, Overstreet, Anne (she/her/hers) <<u>overstreet.anne@epa.gov</u>> wrote:

Dear Dr. Lasee, I wanted to share with you EPA's release and findings related to the publication in the Journal of Hazardous Letters in September 2022. This release and a letter conveying our results was also shared with the Journal editors. You'll note that the method used by EPA to test these pesticide formulations was released and posted to our webpages along with our study results.

I am happy to respond to any questions you may have. Best, Anne Overstreet

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 <u>Overstreet.anne@epa.gov</u> http://www.epa.gov/pesticides

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EPA Completes Scientific Testing of Pesticide Products for PFAS

Today, the U.S. Environmental Protection Agency (EPA) is taking another step in addressing concerns that per- and polyfluoroalkyl substances (PFAS) have been found in pesticide products by releasing a summary of the laboratory analysis of 10 pesticide products reported to contain PFAS residues. EPA did not find any PFAS in the tested

pesticide products, differing from the results of a published study in the Journal of Hazardous Materials. EPA is also releasing its newly developed and validated analytical methodology used in the testing process alongside the summary of its findings. EPA is confident in the results of this newly released method, which is specifically targeted to detect the presence of PFAS in pesticide products formulated with surfactants.

Since learning about potential PFAS contamination in a small number of mosquitocide products in September 2020, EPA has taken a number of steps to address this issue. This includes <u>releasing data in March 2021</u> that preliminarily determined that PFAS in those specific products was most likely formed from a chemical reaction during the container fluorination process which then leached into the pesticide product, <u>releasing another study in September 2022</u> testing the leaching potential of PFAS over a specific time into test solutions packaged in different brands of HDPE fluorinated containers, and <u>notifying manufacturers (including importers)</u>, processors, distributors, users, and those that dispose of fluorinated HDPE containers and similar plastics that the presence of PFAS formed as a byproduct in these containers may be a violation of the Toxic Substances Control Act. Following that notification, the Department of Justice, on behalf of EPA, filed a complaint against Inhance, the company that manufactured the plastic mosquitocide containers in which PFAS was found, for its failure to comply with TSCA's notice, review, and determination requirements prior to manufacture.

As a continuation of these ongoing efforts, EPA has completed its verification analysis of a study published in September 2022 in the Journal of Hazardous Materials entitled "Targeted analysis and Total Oxidizable Precursor assay of several insecticides for <u>PFAS</u>." This study reported the presence of PFOS in six of 10 pesticide products tested.

EPA evaluated the 10 pesticide products included in this study using two different test methods to detect PFAS. The first method was developed by the Agency to specifically measure PFAS in pesticide samples containing surfactants and non-volatile oils, and the second method was used in the study published in the Journal of Hazardous Materials.

EPA obtained samples of the specific pesticide products from the study author and purchased additional products with the same EPA registration numbers on the open market to conduct analyses. EPA tested all samples using both methods and did not detect the presence of PFOS, nor any of 28 additional PFAS it screened for, above the lowest level that our lab instruments can detect (0.2 parts per billion) in any of the pesticide products using either method of detection. The equipment and methodology used by EPA would have shown PFAS detections if present in those pesticide products given that our level of detection (LOD) is 2,500 times more sensitive than the LOD reported by the equipment used by the study author. EPA requested additional information, including raw data from the study author, but did not receive any beyond the published results. EPA's study <u>report</u> contains additional scientific details regarding how the two methods differ and the significance of using the Agency's new method when testing these specific formulations.

One of the most important differences between the two methods is that EPA's <u>method</u> ensures accurate measuring of PFAS by eliminating interference from the oils and surfactants present in these formulations which can result in false positive detections.

EPA's <u>PFAS Strategic Roadmap</u> renewed the Agency's commitment to using sound science and investing in research to proactively stop PFAS chemicals from entering the environment. This latest action is an important step in EPA's ongoing efforts to better understand and manage, when necessary, pesticide formulations that contain

PFAS to ensure enduring and protective solutions. As part of our continuing efforts, EPA will continue to invest in scientific research to fill gaps in understanding of PFAS, to identify which PFAS may pose human health and ecological risks at which exposure levels and develop methods to better test and measure them.

Read the report containing the summary of EPA's study and learn more about the Agency's work on PFAS in pesticide containers.

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This email was sent to <u>overstreet anne@epa.gov</u> using GovDelivery Communications Cloud on behalf of: U.S. EPA Office of Chemical Safety and Pollution Prevention · 707 17th St, Suite 4000 Denver, CO 80202 · 1-800-439-1420



Yes, it was.

From: Nguyen, Thuy <Nguyen.Thuy@epa.gov>
Sent: Wednesday, September 13, 2023 11:35 AM
To: Qian, Yaorong <qian.yaorong@epa.gov>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Was the method on the Sciex validated?

From: Qian, Yaorong <qian.yaorong@epa.gov>
Sent: Wednesday, September 13, 2023 11:03 AM
To: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Yes, the whole data package. An explanation should clear their question.

From: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Sent: Wednesday, September 13, 2023 11:02 AM
To: Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Did we send them the blanks?

From: Qian, Yaorong <qian.yaorong@epa.gov>
Sent: Wednesday, September 13, 2023 11:00 AM
To: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Actually we did send them all the Sciex data on Lasee samples, in addition to Agilent and Thermo HRMS. Reading of the email request I think they are looking for the explanation of why the data from Sciex were not used, specifically for the compounds we have a hit on Sciex. We excluded those hits because they are laboratory contamination/high background levels (PFPeA, 6:2 FTS). We do not have clear written explanations for excluding those compounds in the final report, although we have some limited notes in the some files saying they are similar to background levels or we have laboratory contamination for these specific compounds.

I think we can write an explanation or rationale for the decision of excluding those compounds.

Yaorong

From: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>

Sent: Tuesday, September 12, 2023 9:36 PM
To: Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Subject: Fwd: EPA-2023-006302 (PEER PFAS Verification) FOIA

Thuy

Begin forwarded message:

From: "Taylor, Jeffrey A." <<u>Taylor.Jeffrey@epa.gov</u>>
Date: September 12, 2023 at 8:09:08 PM EDT
To: "Overstreet, Anne" <<u>overstreet.anne@epa.gov</u>>, "Nguyen, Thuy"
<<u>Nguyen.Thuy@epa.gov</u>>
Cc: "Ingram, Earl" <<u>Ingram.Earl@epa.gov</u>>
Subject: EPA-2023-006302 (PEER PFAS Verification) FOIA

Hi Anne and Thuy,

We received the following EPA-2023-006302 (PEER PFAS Verification) FOIA from PEER's Kyla Bennett:

- Please provide all documents, including but not limited to emails, memos, notes, electronic chats on Microsoft Teams and other platforms, calendars, and letters, from January 1, 2023 until the present, regarding EPA's May 18, 2023 memo entitled, "Verification Analysis for PFAS in Pesticide Products (ACB Project B23-05b)." We are specifically interested in all documents discussing EPA's test looking for PFOS in pesticides using the instrument known as "ACB's Sciex QTRAP 6500+ Low Mass," and any documents related to the results from this instrument.
- 2. By way of explanation, EPA included results in the May 18, 2023 memo from two other instruments: 1) the Agilent liquid chromatography/tandem mass spectrometry (LC/MS/MS); and 2) the Thermo Scientific liquid chromatography/high resolution accurate mass spectrometry (LC/HRAMS). The results from ACB's Sciex QTRAP 6500+ Low Mass were not included. Therefore, we seek all documents related to the discussion of the results from all three instruments, and the decision to include two but not the third in the memo. Please note that PEER already has the May 18, 2023 memo, and all the lab results from the three instruments. We are only seeking documents related to the intra-agency discussion of these three tests, and any interagency documents or documents with parties outside the government.

Please let me know what you think regarding providing responsive documents to us in terms of:

 Are the documents isolated enough wherein you can secure them yourself, or will a more time-consuming eDiscovery email/attachment communications search and Relativity records review be necessary? If an eDiscovery search is necessary, then we would begin by submitting search criteria to our IT team regarding the date range, search terms, and custodians (and we would need you to identify custodians).

2. How long do you estimate it might take to either provide responsive materials or the custodian information to us?

Thanks, Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From:	<u>Nguyen, Thuy</u>
То:	Overstreet, Anne; Qian, Yaorong
Cc:	Anderson, Neil
Subject:	RE: EPA-2023-006302 (PEER PFAS Verification) FOIA
Date:	Wednesday, September 27, 2023 8:20:01 PM
Attachments:	ACB FOIA 092023.docx
	B23-05c PEAS in Pesticide Products PAC ndf

Anne

Attached are our responses to the FOIA request and the additional document (B23-05c PFAS pesticide product PAC.pdf), which documents our discussion/interpretation of the data generated from the 3 instruments. Specifically, we discussed that PFAS peaks detected in the tested pesticide samples were near the background levels as found in blanks and control blanks (~10 parts-per-trillion), and were not reported as positive hits in the samples. None of the peaks were at the ppm (part-per-million) level as reported by Lasee et al.

Below is an excerpt of the conclusion from our May 18, 2023 memo:

BEAD's Analytical Chemistry Branch could not confirm the presence of PFOS as reported in Lasee's publication (3.9 ppm to 19.2 ppm), nor detect any PFAS above the method detection limits (0.2 ppb) in those pesticide products. Some background levels of PFAS were seen at less than 10 ppt (based on instrument response only, and not taking into consideration any dilution factor or sample preparation factor).

Thuy

From: Overstreet, Anne <overstreet.anne@epa.gov>
Sent: Wednesday, September 13, 2023 9:16 AM
To: Taylor, Jeffrey A. <Taylor.Jeffrey@epa.gov>; Nguyen, Thuy <Nguyen.Thuy@epa.gov>
Cc: Ingram, Earl <Ingram.Earl@epa.gov>; Anderson, Neil <Anderson.Neil@epa.gov>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Thanks, Jeff. We have an internal meeting set and will loop back.

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 Overstreet.anne@epa.gov http://www.epa.gov/pesticides Please note that I sent this at a time that was convenient for me without expectation for a response outside of business hours. If you receive this email outside of your normal working hours, please know that I do not expect a response until you are back at work during your normal hours.

From: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>
Sent: Wednesday, September 13, 2023 8:44 AM
To: Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>; Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Cc: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>; Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Hi Anne,

Yes, I'll extend the FOIA due date in the meantime as we wait to receive your feedback.

Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From: Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>
Sent: Wednesday, September 13, 2023 8:14 AM
To: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>; Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Cc: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>; Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Hi Jeff. Thuy and I will meet and discuss which route of collection would be best. We'll get back with you the last week of September given that I'm in training for the next two weeks. Does that work? Anne

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 Overstreet.anne@epa.gov

http://www.epa.gov/pesticides

From: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>
Sent: Tuesday, September 12, 2023 8:09 PM
To: Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>; Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Cc: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>
Subject: EPA-2023-006302 (PEER PFAS Verification) FOIA

Hi Anne and Thuy,

We received the following EPA-2023-006302 (PEER PFAS Verification) FOIA from PEER's Kyla Bennett:

- Please provide all documents, including but not limited to emails, memos, notes, electronic chats on Microsoft Teams and other platforms, calendars, and letters, from January 1, 2023 until the present, regarding EPA's May 18, 2023 memo entitled, "Verification Analysis for PFAS in Pesticide Products (ACB Project B23-05b)." We are specifically interested in all documents discussing EPA's test looking for PFOS in pesticides using the instrument known as "ACB's Sciex QTRAP 6500+ Low Mass," and any documents related to the results from this instrument.
- By way of explanation, EPA included results in the May 18, 2023 memo from two other instruments: 1) the Agilent liquid chromatography/tandem mass spectrometry (LC/MS/MS); and 2) the Thermo Scientific liquid chromatography/high resolution accurate mass spectrometry (LC/HRAMS). The results from ACB's Sciex QTRAP 6500+ Low Mass were not included. Therefore, we seek all documents related to the discussion of the results from all three instruments, and the decision to include two but not the third in the memo. Please note that PEER already has the May 18, 2023 memo, and all the lab results from the three instruments. We are only seeking documents related to the intra-agency discussion of these three tests, and any interagency documents or documents with parties outside the government.

Please let me know what you think regarding providing responsive documents to us in terms of:

- Are the documents isolated enough wherein you can secure them yourself, or will a more time-consuming eDiscovery email/attachment communications search and Relativity records review be necessary? If an eDiscovery search is necessary, then we would begin by submitting search criteria to our IT team regarding the date range, search terms, and custodians (and we would need you to identify custodians).
- How long do you estimate it might take to either provide responsive materials or the custodian information to us?

Thanks, Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From:	Taylor, Jeffrey A.
To:	<u>Nguyen, Thuy</u>
Cc:	Ingram, Earl; Overstreet, Anne; Anderson, Neil; Qian, Yaorong
Subject:	RE: EPA-2023-006302 (PEER PFAS Verification) FOIA
Date:	Friday, September 29, 2023 1:14:27 PM

Great, thanks, Thuy – I'll submit the eDiscovery search criteria to our IT team today.

Have a nice weekend, Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From: Nguyen, Thuy <Nguyen.Thuy@epa.gov>
Sent: Friday, September 29, 2023 1:00 PM
To: Taylor, Jeffrey A. <Taylor.Jeffrey@epa.gov>
Cc: Ingram, Earl <Ingram.Earl@epa.gov>; Overstreet, Anne <overstreet.anne@epa.gov>; Anderson, Neil <Anderson.Neil@epa.gov>; Qian, Yaorong <qian.yaorong@epa.gov>
Subject: Re: EPA-2023-006302 (PEER PFAS Verification) FOIA

Hi Jeffrey Yes 'AND' for the 3 terms for the e-discovery in response to this FOIA

Thank you for your help Thuy

On Sep 29, 2023, at 12:20 PM, Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>> wrote:

Hi Thuy,

Thanks for the thorough information. I'll provide your responses (with QA checklist document) to the requester in the short-term. For the more time-consuming eDiscovery search with Relativity review, I believe you're stating that the search term criteria would appear as follows:

1. "PFAS" AND "pesticide products" AND "Sciex QTRAP"

... so that any responsive emails or attachments would need to contain each of those three terms, but please let me know if the "AND" connectors would be too restrictive and then I could offer some suggestions.

Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From: Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Sent: Friday, September 29, 2023 11:23 AM
To: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>; Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>;
Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>; Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Cc: Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>; Qian, Yaorong <<u>qian.yaorong@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Good morning Jeffrey

Below are our responses to the FOIA questions. Please let us know if additional information is needed

> Please provide all documents, including but not limited to emails, memos, notes, electronic chats on Microsoft Teams and other platforms, calendars, and letters, from January 1, 2023 until the present, regarding EPA's May 18, 2023 memo entitled, "Verification Analysis for PFAS in Pesticide Products (ACB Project B23-05b)." We are specifically interested in all documents discussing EPA's test looking for PFOS in pesticides using the instrument known as "ACB's Sciex QTRAP 6500+ Low Mass," and any documents related to the results from this instrument.
> ACB Response: We would like for an e-Discovery to be performed. Our suggested search terms are PFAS, pesticide products, and

Our suggested search terms are PFAS, pesticide products, and Sciex QTRAP; and custodian, all BEAD staff included in this e-mail. Thank you

2. By way of explanation, EPA included results in the May 18, 2023 memo from two other instruments: 1) the Agilent liquid chromatography/tandem mass spectrometry (LC/MS/MS); and 2) the Thermo Scientific liquid chromatography/high resolution accurate mass spectrometry (LC/HRAMS). The results from ACB's Sciex QTRAP 6500+ Low Mass were not included. Therefore, we seek all documents related to the discussion of the results from all three instruments, and the decision to include two but not the third in the memo. Please note that PEER already has the May 18, 2023 memo, and all the lab results from the three instruments. We are only seeking documents related to the intra-agency discussion of these three tests, and any interagency documents or documents with parties outside the government.

ACB Responses:

- We disagree with the statement made in regard to the instruments used to generate the data for our May 18, 2023 memo. Our study was based two different types of instrumentation platforms– LC-MS/MS and LC/HRAMS, which included three different instruments: the Agilent 6470 and the Sciex QTRAP 6500+ instruments categorized as LC-MS/MS, and the Thermo Q Exactive as LC/HRAMS.
- 2. As for "The results from ACB's Sciex QTRAP 6500+ Low Mass were not included", we also disagree. We have provided PEER instrument data from all three different instruments (Agilent LC/MSMS, Sciex Qtrap LC/MSMS, and Thermo LC/HRAMS). We are now providing PEER our internal QA review document (Project Audit Checklist), where we documented our discussion, interpretation and conclusions on the data generated from these three instruments, including data from the Sciex QTRAP.
- 3. There was no discussion of the test data and results outside of ACB, prior to the release of the May 18, 2023.

From: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>
Sent: Wednesday, September 13, 2023 9:48 AM
To: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>; Overstreet, Anne
<<u>overstreet.anne@epa.gov</u>>; Nguyen, Thuy <<u>Nguyen.Thuy@epa.gov</u>>
Cc: Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Sounds great, Anne, and thanks for the heads-up.

Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

From: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>
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Cc: Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Thanks Anne,

We appreciate you and your staff's assistance as always.

Earl

From: Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>
Sent: Wednesday, September 13, 2023 9:16 AM
To: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>; Nguyen, Thuy
<<u>Nguyen.Thuy@epa.gov</u>>
Cc: Ingram, Earl <<u>Ingram.Earl@epa.gov</u>>; Anderson, Neil <<u>Anderson.Neil@epa.gov</u>>
Subject: RE: EPA-2023-006302 (PEER PFAS Verification) FOIA

Thanks, Jeff. We have an internal meeting set and will loop back.

<image001.jpg>

Anne Overstreet, Director Biological and Economic Analysis Division Office of Pesticide Programs U.S. Environmental Protection Agency (202) 566-2425 <u>Overstreet.anne@epa.gov</u> <u>http://www.epa.gov/pesticides</u>

Please note that I sent this at a time that was convenient for me without expectation for a response outside of business hours. If you receive this email outside of your normal working hours, please know that I do not expect a response until you are back at work during your normal hours.

From: Taylor, Jeffrey A. <<u>Taylor.Jeffrey@epa.gov</u>>
Sent: Wednesday, September 13, 2023 8:44 AM
To: Overstreet, Anne <<u>overstreet.anne@epa.gov</u>>; Nguyen, Thuy
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Anne

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- **2.** How long do you estimate it might take to either provide responsive materials or the custodian information to us?

Thanks, Jeff

Jeffrey Taylor Public Information & Records Integrity Branch Communications Services and Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention U.S. Environmental Protection Agency (202)-565-2317

<B23-05c PFAS in Pesticide Products PAC.pdf>



OFFICE OF CHEMICAL SAFETY AND POLLUTION PREVENTION

May 14, 2024

Kyla Bennett P.O. Box 574 North Easton, MA 02356

Re: Freedom of Information Act Request No. 2024-EPA-03080

Dear Kyla Bennett:

This letter concerns the above-referenced Freedom of Information Act (FOIA) request, submitted to U.S. Environmental Protection Agency (EPA) on March 15, 2024, in which you stated:

- Pursuant to the Freedom of Information Act, 5 U.S.C. 552, as amended ("FOIA"), Public Employees for Environmental Responsibility ("PEER") requests certain documents from the U.S. Environmental Protection Agency ("EPA") relating to the testing of per- and polyfluoroalkyl substances ("PFAS") in pesticides prior to the issuance of its May 18, 2023 memo (see https://www.epa.gov/system/files/documents/2023-05/BEAD%20PFAS%20Study%20Results%202023.pdf) and associated press release.
- This request applies to all records obtained or created by EPA between January 19, 2023 and the present.
- Specifically, we request:
 - All documents, including but not limited to emails, memos, texts, letters, Microsoft Teams chats, transcriptions of phone calls, and meeting notes, relating to the results from the Sciex 6500+ LC/MSMS (note that we do not need the results of the test itself);
 - All documents relating to the decision not to include the results of the Sciex 6500+ LC/MSMS in the May 18, 2023 memo and associated press release; and
 - All documents relating to directing the preparation of the above-referenced memo and accompanying press release, including records reflecting which officials authorized, reviewed, and gave the order to publicly issue the memo and release, as well as the distribution list for same; and
 - All documents related to the decision not to include in the press release or memo the information that Dr. Steven Lasee had spiked the samples he provided to EPA.
- Possible custodians of these records include, but are not limited to, Dr. Yaorong Qian, Ed Messina, and Thuy Nguyen.

EPA's Office of Chemical Safety and Pollution Prevention (OCSPP) provides a full grant of 16 documents that respond to your request.

This letter concludes our response to your request. You may appeal this determination in writing within 90 calendar days from the date of this letter by one of the following methods:

- 1. Visit EPA's FOIA submission website (https://foiapublicaccessportal.epa.gov/), sign into your account by clicking Sign-In, and select Submit Appeal;
- 2. U.S. Mail sent to the following address: National FOIA Office, U.S. Environmental Protection Agency, 1200 Pennsylvania Avenue NW (2310A), Washington, DC 20460; or
- 3. Overnight delivery service to National FOIA Office, U.S. Environmental Protection Agency, 1200 Pennsylvania NW, Room 7309C, Washington, DC 20460.

The Agency will not consider appeals received after the 90-calendar-day limit. Appeals received after 5:00 p.m. EST will be considered received on the next business day. The appeal should include the FOIA tracking number listed above. For quickest possible handling, the appeal letter, and its envelope, if applicable, should be marked "Freedom of Information Act Appeal." Additionally, you may seek assistance from EPA's FOIA Public Liaison at hq.foia@epa.gov or (202) 566-1667, or from the Office of Government Information Services (OGIS). You may contact OGIS in any of the following ways: by mail, Office of Government Information Services, National Archives and Records Administration, Room 2510, 8610 Adelphi Road, College Park, MD 20740-6001; email, ogis@nara.gov; telephone, (301) 837-1996 or (877) 684-6448; or fax, (301) 837-0348.

If you have any questions or need additional information, then please contact me directly at taylor.jeffrey@epa.gov. Refer to FOIA number 2024-EPA-03080 when contacting the office about these requests.

Sincerely,

Jeffrey a. Jayle

Jeffrey Taylor Public Information & Records Integrity Branch Communication Services & Information Division Office of Program Support Office of Chemical Safety and Pollution Prevention

Reference: SOP No. ACB-004 R 3.1 Attachment 1: EXAMPLE – Project Audit Checklist (Residue)

QUALITY ASSURANCE PROJECT AUDIT CHECKLIST (PAC) FOR RESIDUE PROJECTS ANALYTICAL CHEMISTRY BRANCH US EPA, ENVIRONMENTAL SCIENCE CENTER

PROJECT: B23-05b – PFAS in Pesticide Product Analysis

AUDIT: (VALIDATION) (INTERIM) (FINAL) (circle one) DATE: 2/6/2023 Analyst Senior Review* Staff/QAO ERG A. Request for Laboratory Services [ISO-17025:2017 7.1] Note 1 B. Notebook Sheets/Worksheets: 1) initialed, dated, project number, method cited [SOP 007/3.7.1-5] ERG YQ 2) complete (in enough detail that processes might be reconstructed later ERG YQ without ambiguity), filled out chronologically [SOP 007/3.0] ERG 3) unique IDs for all analytical instruments (e.g. GC, LC), lab equipment (e.g. Note B3 Turbovaps, rotovaps, N-Evaps, Syncore evaporators, pipetters, centrifuges, pH UQ meters, titrimeters, accelerated solvent extractors, Geno/Grinders) balances, refrigerators, freezers used during the project [SOP 007/3.7.6] 4) instrument software (name/vendor/version) used for quantitation (if not ERG YQ. recorded in the instrument logbook) [SOP 007/3.7.13] 5) source, lot number, purity, expiration date of standards, reagents, solvents ERG YQ documented [SOP 007/3.7.7] 6) ID and source of any control materials (commodities) [SOP 007/3.7.8] N/A N/A 7) Sample ID and descriptions [SOP 007/3.7.10] UQ ERG ERG 8) disposition documented for controls, standards, solutions, extracts [SOP Hold till 004/3.2.4] the end of Note B8 project

Reference: SOP No. ACB-004 R 3.1 Attachment 1: EXAMPLE – Project Audit Checklist (Residue)

C. Instruments:		
 QA log book filled out while in use, and/or when modified, repaired or maintained [SOP 006B/3.1] 	YQ	ERG
2) date, analyst, project, analyte(s), column [SOP 006B/3.1A]	YQ	ERG
3) system operation entries during project (column installation date, tuning, performance) [SOP 006/3.1B]	УQ	ERG
D. Balances/Pipetters:		-
 Balance QA Log Book filled out: weight set cited, checked at least monthly, at least 4 weights used [SOP 006A/3.2.1] and on the day of each quantitative use with a minimum of 1 weight in a similar range to the mass(es) to be weighed [Balance USE Report 191021, SOP 006A/3.2.2] 	YC	ERG
2) balance acceptance criteria met for calibration masses checked [SOP 006A/3.2.1]	YQ	ERG
3) pipetter calibration check successfully verified and documented on the day of use and recorded in pipette log records [SOP 029/3.3]	Yæ	ERG Note D3
E. Analytical Method:		
1) copy in the file [ISO17025:2017 Sec.7.2.1.2]	<i>ິ່</i> y ແ Note	ERG
2) deviations recorded in notebook and/or final report [SOP 007/3.7.5]	N/A	N/A
3) Is the initial method authorized and on the ACB Master List of Methods [ACB	n/a	N/A

Reference: SOP No. ACB-004 R 3.1 Attachment 1: EXAMPLE – Project Audit Checklist (Residue)

	Analyst Review*	Senior Staff/QAO
F. Labels: All papers in file labeled with project number(s) [SOP 004/3.2.4]	уа	ERG
G. Report:		
1) draft report prepared	YQ	ERG
2) all information agreed with the customer provided, [ISO17025:2017 Sec 7.8.1.2]	N/A	N/A
H. Data:		
 Was Uncertainty of measurement estimated and records included in the project file [SOP 025/3.4] 	N/A	N/A
I. Quality Assurance Project Plan/SOP (if applicable):		
 written and approved before lab work began [ACB QMP Planning section] 	N/A	N/A
 if not, formal permission granted to initiate work while QAPP is being prepared [ACB QMP Planning section] 	N/A	N/A
 hand amendments (<i>if any</i>) to the original method are initialed and dated [QAPP requirement; TN flowchart for authorized methods] 	N/A	N/A
J. Sample Sheets:		Note J
1) History of Official Sample Sheets: (if any) completed [SOP 017/3.0]	N/A	ERG
 Sample Chain of Custody maintained: arrival date, physical state, name or initials of person receiving, etc. [SOP 030/3.1.1] 	N/A	ERG
3) Any not suitable for analysis samples reported to customer [SOP 030/3.1.1]	N/A	ERG

	Analyst Review*	Senior Staff/QAO
K. Lessons Learned (optional): Detailed in "Comments" section		
1) anything new or unique used in this project (<i>any preventive action or improvements that should be captured and shared in USE reports</i>) [SOP 004/3.2.4]	N/A	N/A
2) Nonconformance: have any potential problems/issues been identified that may need preventive action. Were USE reports generated. [SOP 022/3.2]	N/A	N/A
L. Feedback:		
1) Any feedback/complaint received and documented [SOP 024/3.3.1]	N/A	N/A
2) Did the report request any feedback from the customer [SOP 024/3.2]	N/A	N/A
M. ADDITIONAL ITEMS TO MEET QAPP OR SOP METHOD VALIDATION REQUIREMENTS: (QAO and Project Lead add lines for specific requirements) [SOP 004/3.1.]		
1) LOD/LOQ: estimated for each analyte [SOP 030/3.3.2]	N/A	ERG Note M1, M2, M3
2) LOD verified by fortification of each matrix in triplicate and processed through the entire method to meet acceptance criteria [SOP 030/3.3.3.1]	N/A	ERG Note M1, M2, M3
 LOQ verified by fortification of each matrix in 5 replicates and processed through the entire method to meet acceptance criteria [SOP 030/3.3.3.1] 	N/A	ERG Note M1, M2, M3
4) Were samples homogenized as per SOP 030/3.1.2?		
5) Refrigerators/freezers used for samples, extracts and standards monitored and recorded by an automated environmental monitoring system [SOP 009/2.0, 030/3.5.1.3]	N/A	N/A
6) Samples and extracts stored separate from standards [SOP 030/3.5.1.3]	YQ	ERG

	Analyst Review*	Senior Staff/QAO
 Were expiration dates assigned for all reference standards, solvents, chemicals and reagents and used within those expiration dated? [SOP 030/3.5.1.4, 3.5.1.5] 	YQ	ERG
8) Any extension of standard expiration dates required? [SOP 030/3.5.1.4]	N/A	N/A
 Appropriate files, including all chromatographic data, copied to electronic media and included with the project files. [SOP 030/3.8.4] 	YQ	ERG

	Analyst Review*	Peer Review*	Senior Staff/QAO
N. Specific PAC Items Requiring Peer Review:			
Notebook Sheets/Worksheets:			
1) Raw data such as weights, volumes, etc. [SOP 007/3.7.9]	YQ	DNF	ERG
2) Details of standard solution preparation with concentrations and calculations [SOP 007/3.7.11]	YQ	DNF	ERG
3) Amount and ID of standards used in procedure [SOP 007/3.7.12]	YQ	DNF	ERG
Calculations that impact reported results:			
4) Logic and accuracy checked, initialed, dated [SOP 003/3.1]	YQ	DNF	ERG
5) if more than 50 reported results: at least 10% of results checked with less than 3 errors. [SOP 003/3.2] Non-detect in samples. Recovery data for method performance verification	YQ	DNF	ERG
6) Spreadsheets: (if used) cell formulas checked and initialed [SOP 003/3.1]	YQ	DNF	ERG
7) instrument software (<i>if used</i>) operator-entered values, transcriptions, algorithms checked [SOP 003/3.3]	YQ	DNF	ERG
Calibration Plot and Regression Analysis:			
8) Curve will be established for all analytes on instruments used prior to analysis of samples [SOP 030/3.5.2.3]	Note 1	DNF ok	ERG Note N8,9,10,1 1,12,13
9) minimum of four concentration levels (linear) or five levels (quadratic) [SOP 030/3.5.2.3]	Note 1	DNF ok	ERG Note N8,9,10,1 1,12,13
10) sample results within 10% of range [SOP 030/3.5.2.3]	N/A	N/A	ERG Note N8,9,10,1 1,12,13
11) Correlation coefficient (r2) for each quantified analyte > 0.98 [SOP 030/3.5.2.3]	N/A	N/A	ERG Note N8,9,10,1 1,12,13
12) any unused data points explained [SOP 011/3.0a]	<i>Yୁ</i> ଥ Note 2	DNF ok	ERG Note N8,9,10,1 1,12,13

	Analyst Review*	Peer Review*	Senior Staff/QAO
13) calibration curve traceable from generation through sample analysis [SOP 003/3.3]	YQ Note 3	ok	ERG Note N8,9,10,1 1,12,13
Instrument Conditions:			
14) instrument conditions (methods, sequence tables) documented for each sample set [ISO-17025:2017 6.4.13]	уа	DNF N.14	ERG
Sequence: (typically includes)			
15) Calibration standards at beginning [SOP 030/3.5.2.3]	<i>ମୁ</i> ଥ Note 3	DNF ok	ERG
16) Generally contains a method (procedural) blank, a matrix blank (control), and a matrix spike [SOP 030/3.5.2.1]	<i>ମ୍ବା</i> ଥ Note 4	DNF ok	ERG
17) Matrix blanks free of target analytes at the LOD [SOP 030/3.5.2.2]	YQ	DNF	ERG
18) Matrix spikes should be between 60% - 120 % ideally [SOP 030/3.5.2.5]	່ມຜ Note 1	DNF ok	ERG
Continuing Calibration Check			
19) At least one analyzed at a minimum rate of 1 every 10 samples [SOP 030/3.5.2.4]	уa	DNF	ERG
20) < 20% RPD or % difference between cal. curve and calibration check standard [SOP 030/3.5.2.4]	N/A	N/A	N/A
Compound Identification:			
21) RT criteria: retention time match between analytical standard and sample [SOP 030/3.6]	УQ	DNF	ERG
Confirmation of analytes:			
22) Confirmation of identity using two specific or selective detectors [SOP 030/3.6.1]	YQ	DNF	ERG

	Analyst Review*	Peer Review*	Senior Staff/QAO
 23) If single stage MS: at least three different structurally significant ions monitored, ratios of monitored ions ±20% of reference standards [SOP 030/3.6.2] 	N/A	N/A	N/A
24) If triple stage MS: at least two different precursor/product ions, ratios of monitored ions ±30% of reference standards [SOP 030/3.6.3]	YQ	DNF	ERG
25) If High Resolution Accurate Mass (HRAM): At least one protonated, de-protonated, or adduct precursor ion with mass accuracy ≤ 5 ppm and one additional indicator as confirmation (can include MS fragment(s) or naturally occurring isotope patterns [SOP 030/3.6.4]	YQ	DNF	ERG
Chromatograms:			
26) Instrument logbook contains date and results of calibrations [SOP 006B/3.2]	N/A	N/A	N/A
27) any manual integrations are clearly labeled [SOP 020/3.3.1] Manual integration occasionally used for poor peak shapes. Not indicated on chromatograms.	YQ	DNF	ERG
Report:			
28) transcriptions to report checked	YQ	DNF	ERG
Data:			
29) Unused Data are marked and distinguishable from reported data and documented with reason for disuse [SOP 011/3.0a]	YQ	DNF	ERG
30) Rejected Data are clearly labeled and documented with reason for rejection [SOP 011/3.0b]	YQ	DNF	ERG
31) Typically, only residues ≥LOQ are reported as numeric values [SOP 030/3.7.1]	N/A	N/A	N/A
32) If tandem MS analysis with acceptable confirmation criteria, residues between LOD and LOQ may be reported if necessary and flagged [SOP 030/3.7.1]	N/A	N/A	N/A
33) Reported results conform to ID and confirmation criteria [SOP 030/3.7.2] unless exceptions.	N/A	N/A	N/A

	Analyst Review*	Peer Review*	Senior Staff/QAO
34) No data released without the approval of the ACB BC. [SOP 030/3.8.5]	YQ	DNF	ERG
35) Changes in data entries will indicate reason for change, dated,	N/A	N/A	ERG
initialed. [SOP 020/3.3.2]		11/71	Note N35
Method Validation of a previously used method: (if required)			
36) Method blank (procedural) and matrix blank (control) processed through the entire method along with fortified samples [SOP 030/3.3.1.2]	N/A	N/A	ERG Note M1, M2, M3
37) Fortifications: fortified with all target analytes at least in triplicate and a minimum of three fortification levels [SOP 030/3.3.1.1]	N/A	N/A	ERG Note M1, M2, M3
38) Fortifications: additional levels needed if samples show residues more than highest validated level [SOP 030/3.3.1.1]	N/A	N/A	ERG Note M1, M2, M3
39) Average Recoveries: target 60 – 120 % for all analytes with %RSD ≤ 20%, report achievable performance if not met [SOP 030/3.3.1.4]	N/A	N/A	ERG Note M1, M2, M3

* Initial when verified; add comments or explanations (as needed)

ACB SOPs Referenced: 003, 004, 006, 007, 009, 011, 017, 020, 022, 024, 025, 029, 030. **COMMENTS & FINDINGS:** (project lead analyst will follow up on any open items with the QAO [SOP 004/3.2.4])

Notes:

This portion of the project is to analyze several pesticide products and verify if there is any PFAS present. The method used is a simple dilution procedure following that described in the published paper (Lasee et al., 2022). This is to verify if the reported presence of PFAS is true. This test is semi-quantitative. The diluted products were fortified and Page 9 of 11 revised November 5, 2020

the identities and responses of individual PFAS were compared with the standard solution. A bracket standard quantitation (average response factor) was used for the semi-quantitation as indication of qualitative recovery.

2. One batch of samples were re-aliquoted to vials and re-analyzed, due to poor chromatography on LC (likely caused by a change in new mobile phase). The initial run was not used.

3. The last batch of samples (10x lower spiked samples) were run on LC/MSMS with a calibration curve, even though the accurate quantitation is not necessary. Running a calibration curve is to show the responses at low levels (the lowest calibration point).

4. A procedural blank is used. All samples were spiked with extraction standards and internal standards. Two batches of samples were also spiked with native PFAS. No additional spikes are necessary.

DNF Notes

N.14 - Added a copy of the instrument method to your folder.

ERG Notes:

Correct or further explain: B3 – sonciator serial number, centrifuge serial number, shaker serial number (unless by hand), only 1 pipette???, 40 – 200 uL, also see 1 mL and 20 uL additions. YQ: Added the sonicator, centrifuge, pipette information in the notebook.

Correct or further explain: B8 – The requirement as I have been told is to write "All samples and solutions will be disposed in accordance with ESC guidelines" in the notebook at the very end. YQ: the project is ongoing. All samples and dilutions are stored in the lab (noted in the notebook now).

D3 – pipettor checked in lab notebook. YQ: Done

Correct or further explain: J – samples were received and tested from Lasee. While it seems likely no CoC is present I do believe a sample history form could be generated and included. YQ: A copy of internal sample receipt/history form prepared

M1, M2, M3 - semiquantitative only

N8, N9, N10, N11, N12, N13 - semiquantitative only, single point calibration

Correct or further explain: N35 - Some cross outs not dated, reason given, and initialed in notebook. YQ: done

Audited by:		<u>Initials</u>	Date	
Analyst(s):	Yaorong Qian	<i>Y</i> @	2/7/2023	
Page 10 of 11	revised	November 5, 2020		

- Peer Review(s): _	But mul	DNF	2/16/23
- - Senior Staff:	Eic) j:U ERG	3/7/23
- QA Officer:			

QUALITY ASSURANCE PROJECT AUDIT CHECKLIST (PAC) FOR RESIDUE PROJECTS ANALYTICAL CHEMISTRY BRANCH US EPA, ENVIRONMENTAL SCIENCE CENTER

PROJECT B23-05c – PFAS in Pesticide Products-Method Validation and Sample Analysis

AUDIT: (VALIDATION) (INTERIM) (FINAL) (circle one)

DATE: 04/05/23

	Analyst Review*	Senior Staff/QAO
A. Request for Laboratory Services [ISO-17025:2017 7.1]	DNF	SAG
B. Notebook Sheets/Worksheets:DNF-22-I		
1) initialed, dated, project number, method cited [SOP 007/3.7.1-5]	DNF Note B.1	SAG
2) complete (<i>in enough detail that processes might be reconstructed later without ambiguity</i>), filled out chronologically [SOP 007/3.0]	DNF	SAG
3) unique IDs for all analytical instruments (<i>e.g. GC, LC</i>), lab equipment (<i>e.g. Turbovaps, rotovaps, N-Evaps, Syncore evaporators, pipetters, centrifuges, pH meters, titrimeters, accelerated solvent extractors, Geno/Grinders</i>) balances, refrigerators, freezers used during the project [SOP 007/3.7.6]	DNF	SAG Note B3
4) instrument software (<i>name/vendor/version</i>) used for quantitation (if not recorded in the instrument logbook) [SOP 007/3.7.13]	DNF	SAG
5) source, lot number, purity, expiration date of standards, reagents, solvents documented [SOP 007/3.7.7]	DNF	SAG Note B5
6) ID and source of any control materials (commodities) [SOP 007/3.7.8]	DNF	SAG
7) Sample ID and descriptions [SOP 007/3.7.10]	DNF	SAG
8) disposition documented for controls, standards, solutions, extracts [SOP 004/3.2.4]	DNF	SAG

	Analyst Review*	Senior Staff/QAO
C. Instruments:		
1) QA log book filled out while in use, and/or when modified, repaired or maintained [SOP 006B/3.1]	DNF	SAG
2) date, analyst, project, analyte(s), column [SOP 006B/3.1A]	DNF	SAG Note C2
3) system operation entries during project (column installation date, tuning, performance) [SOP 006/3.1B]	DNF	SAG
D. Balances/Pipetters:		
1) Balance QA Log Book filled out: weight set cited, checked at least monthly, at least 4 weights used [SOP 006A/3.2.1] and on the day of each quantitative use with a minimum of 1 weight in a similar range to the mass(es) to be weighed [Balance USE Report 191021, SOP 006A/3.2.2]	DNF	SAG
2) balance acceptance criteria met for calibration masses checked [SOP 006A/3.2.1]	DNF	SAG
3) pipetter calibration check successfully verified and documented on the day of use and recorded in pipette log records [SOP 029/3.3]	DNF	SAG
E. Analytical Method:		
1) copy in the file [ISO17025:2017 Sec.7.2.1.2]	DNF	SAG
2) deviations recorded in notebook and/or final report [SOP 007/3.7.5]	N/A	SAG
3) Is the initial method authorized and on the ACB Master List of Methods [ACB flowchart for authorizing methods]	DNF Note B.1	SAG

	Analyst Review*	Senior Staff/QAO
F. Labels: All papers in file labeled with project number(s) [SOP 004/3.2.4]	DNF	SAG
G. Report:		
1) draft report prepared	DNF	SAG
2) all information agreed with the customer provided, [ISO17025:2017 Sec 7.8.1.2]	DNF	SAG
H. Data:		
1) Was Uncertainty of measurement estimated and records included in the project file [SOP 025/3.4]	DNF	SAG Note H1
I. Quality Assurance Project Plan/SOP (if applicable):		N/A
1) written and approved before lab work began [ACB QMP Planning section]	N/A	
2) if not, formal permission granted to initiate work while QAPP is being prepared [ACB QMP Planning section]	N/A	
3) hand amendments (<i>if any</i>) to the original method are initialed and dated [QAPP requirement; TN flowchart for authorized methods]	N/A	
J. Sample Sheets:		
1) History of Official Sample Sheets: (if any) completed [SOP 017/3.0]	N/A	SAG Note J
2) Sample Chain of Custody maintained: arrival date, physical state, name or initials of person receiving, etc. [SOP 030/3.1.1]	N/A	SAG Note J
3) Any not suitable for analysis samples reported to customer [SOP 030/3.1.1]	N/A	N/A

	Analyst Review*	Senior Staff/QAO
K. Lessons Learned (optional): Detailed in "Comments" section		
1) anything new or unique used in this project (<i>any preventive action or improvements that should be captured and shared in USE reports</i>) [SOP 004/3.2.4]	DNF Note K.1	SAG
2) Nonconformance: have any potential problems/issues been identified that may need preventive action. Were USE reports generated. [SOP 022/3.2]	N/A	
L. Feedback:		
1) Any feedback/complaint received and documented [SOP 024/3.3.1] Feedback will be requested upon completion of the project	DNF	SAG
2) Did the report request any feedback from the customer [SOP 024/3.2]	N/A	SAG
M. ADDITIONAL ITEMS TO MEET QAPP OR SOP METHOD VALIDATION REQUIREMENTS: (QAO and Project Lead add lines for specific requirements) [SOP 004/3.1.]		
1) LOD/LOQ: estimated for each analyte [SOP 030/3.3.2]	DNF	SAG
2) LOD verified by fortification of each matrix in triplicate and processed through the entire method to meet acceptance criteria [SOP 030/3.3.3.1]	DNF	SAG
 LOQ verified by fortification of each matrix in 5 replicates and processed through the entire method to meet acceptance criteria [SOP 030/3.3.3.1] 	DNF	SAG Note M3
4) Were samples homogenized as per SOP 030/3.1.2?	DNF	SAG
5) Refrigerators/freezers used for samples, extracts and standards monitored and recorded by an automated environmental monitoring system [SOP 009/2.0, 030/3.5.1.3]	DNF	SAG

		Analyst Review*	Senior Staff/QAO
6)	Samples and extracts stored separate from standards [SOP 030/3.5.1.3]	DNF	SAG
7)	Were expiration dates assigned for all reference standards, solvents, chemicals and reagents and used within those expiration dated? [SOP 030/3.5.1.4, 3.5.1.5]	DNF	SAG Note M7
8)	Any extension of standard expiration dates required? [SOP 030/3.5.1.4]	N/A	N/A
9)	Appropriate files, including all chromatographic data, copied to electronic media and included with the project files. [SOP 030/3.8.4]	DNF	SAG

	Analyst Review*	Peer Review*	Senior Staff/QAO
N. Specific PAC Items Requiring Peer Review:			
Notebook Sheets/Worksheets:			
1) Raw data such as weights, volumes, etc. [SOP 007/3.7.9]	DNF	у¢	SAG Note N1
2) Details of standard solution preparation with concentrations and calculations [SOP 007/3.7.11]	DNF	уæ	Note N2
3) Amount and ID of standards used in procedure [SOP 007/3.7.12]	DNF	YQ	SAG
Calculations that impact reported results:			
4) Logic and accuracy checked, initialed, dated [SOP 003/3.1]	DNF	Уæ	SAG Note N4
5) if more than 50 reported results: at least 10% of results checked with less than 3 errors. [SOP 003/3.2]	N/A	УQ	SAG
6) Spreadsheets: (if used) cell formulas checked and initialed [SOP 003/3.1]	DNF	YQ	SAG
7) instrument software (<i>if used</i>) operator-entered values, transcriptions, algorithms checked [SOP 003/3.3]	DNF	YQ	SAG
Calibration Plot and Regression Analysis:			
8) Curve will be established for all analytes on instruments used prior to analysis of samples [SOP 030/3.5.2.3]	DNF	у¢	SAG
9) minimum of four concentration levels (linear) or five levels (quadratic) [SOP 030/3.5.2.3]	DNF	уæ	SAG Note N9
10) sample results within 10% of range [SOP 030/3.5.2.3]	DNF	Ye	SAG
11) Correlation coefficient (r2) for each quantified analyte > 0.98 [SOP 030/3.5.2.3]	DNF	YQ	SAG Note N9
12) any unused data points explained [SOP 011/3.0a]	DNF Note N.12	Ya	SAG

	Analyst Review*	Peer Review*	Senior Staff/QAO
13) calibration curve traceable from generation through sample		YQ	SAG
analysis [SOP 003/3.3]	DNF		Note N9
Instrument Conditions:			
14) instrument conditions (methods, sequence tables) documented		YQ	SAG
for each sample set [ISO-17025:2017 6.4.13]	DNF	0	Note N14
Sequence: (typically includes)			
15) Calibration standards at beginning [SOP 030/3.5.2.3]	DNF	YQ	SAG
16) Generally contains a method (procedural) blank, a matrix blank (control), and a matrix spike [SOP 030/3.5.2.1]	DNF	у¢	SAG
17) Matrix blanks free of target analytes at the LOD [SOP	DNF	YQ	SAG
030/3.5.2.2]	Note N.17		SAU
18) Matrix spikes should be between 60% - 120 % ideally [SOP 030/3.5.2.5]	DNF	YQ	SAC
030/3.3.2.3]	Note N.18		SAG
Continuing Calibration Check			
19) At least one analyzed at a minimum rate of 1 every 10 samples [SOP 030/3.5.2.4]	DNF	уæ	SAG
20) < 20% RPD or % difference between cal. curve and calibration	DNF	YQ	SAG
check standard [SOP 030/3.5.2.4] Some compounds outside the acceptable range	Note N.20		Note N20
Compound Identification:			
21) RT criteria: retention time match between analytical standard and	DNF	YQ	SAC
sample [SOP 030/3.6]	Note N.21		SAG
Confirmation of analytes:			
22) Confirmation of identity using two specific or selective detectors [SOP 030/3.6.1]	N/A		
23) If single stage MS: at least three different structurally significant ions monitored, ratios of monitored ions $\pm 20\%$ of reference standards [SOP 030/3.6.2]	N/A		

	Analyst Review*	Peer Review*	Senior Staff/QAO
24) If triple stage MS: at least two different precursor/product ions, ratios of monitored ions ±30% of reference standards [SOP 030/3.6.3]	DNF	Yæ	SAG
25) If High Resolution Accurate Mass (HRAM): At least one protonated, de-protonated, or adduct precursor ion with mass accuracy ≤ 5 ppm and one additional indicator as confirmation (can include MS fragment(s) or naturally occurring isotope patterns [SOP 030/3.6.4] Only for pesticide samples	DNF	YQ.	SAG
Chromatograms:			
26) Instrument logbook contains date and results of calibrations [SOP 006B/3.2]	DNF	YQ	
27) any manual integrations are clearly labeled [SOP 020/3.3.1]	DNF	YQ	SAG
Report:			
28) transcriptions to report checked	DNF	YQ	SAG Note N28
Data:			
29) Unused Data are marked and distinguishable from reported data and documented with reason for disuse [SOP 011/3.0a]	DNF	YQ	SAG
30) Rejected Data are clearly labeled and documented with reason for rejection [SOP 011/3.0b]	N/A		
31) Typically, only residues ≥LOQ are reported as numeric values [SOP 030/3.7.1]	DNF	YQ	SAG
32) If tandem MS analysis with acceptable confirmation criteria, residues between LOD and LOQ may be reported if necessary and flagged [SOP 030/3.7.1]	DNF	Ya	SAG
33) Reported results conform to ID and confirmation criteria [SOP 030/3.7.2] unless exceptions.	DNF	YQ	SAG
34) No data released without the approval of the ACB BC. [SOP 030/3.8.5]	DNF	YQ	SAG
35) Changes in data entries will indicate reason for change, dated, initialed. [SOP 020/3.3.2]	DNF	YQ	SAG

	Analyst Review*	Peer Review*	Senior Staff/QAO
Method Validation of a previously used method: (if required)			
36) Method blank (procedural) and matrix blank (control) processed through the entire method along with fortified samples [SOP 030/3.3.1.2]	DNF	YQ	SAG
37) Fortifications: fortified with all target analytes at least in triplicate and a minimum of three fortification levels [SOP 030/3.3.1.1]	DNF	YQ	SAG
38) Fortifications: additional levels needed if samples show residues more than highest validated level [SOP 030/3.3.1.1]	N/A		
 39) Average Recoveries: target 60 – 120 % for all analytes with %RSD ≤ 20%, report achievable performance if not met [SOP 030/3.3.1.4] A spreadsheet of marginally performing analytes will be added to the project folder upon completion of QA Review 	DNF Note N.18	YA	SAG

* Initial when verified; add comments or explanations (as needed)

ACB SOPs Referenced: 003, 004, 006, 007, 009, 011, 017, 020, 022, 024, 025, 029, 030.

COMMENTS & FINDINGS: (project lead analyst will follow up on any open items with the QAO [SOP 004/3.2.4])

DNF Notes:

General Notes:

1 – Some PFAS (PFBA, PFPeA, PFTeDA, PFTrDA, PFHxDA, PFODA, 4:2 FTS, and 6:2 FTS) do not perform well on Agilent 6470. The extracts of the LOQ fortification samples were rerun on 5-9-23 on the Sciex 6500+. Adequate recoveries were obtained for most of the compounds listed above. It is assumed the higher fortifications will respond accordingly, though were not reanalyzed. - A list of marginally performing analytes was added to the project folder by QAO. This can be used in addition to the final authorized method - SAG

PAC Notes:

B.1 – Method is new. None to cite. - New method has been validated according to SOP guidelines. Method was validated for some analytes at the LOQ level on the Sciex instrument - SAG

K.1 - A separate lessons learned file will be uploaded as time allows

N.12 - The highest calibration in some analytes was omitted to give better curve fits for the lower range. No samples or fortifications approached high calibration. - OK SAG

N.17 - Some contamination was present for 6:2 FTS, PFBA, and PFPeA, especially on the Agilent 6470 LC/MSMS. It is unknown if this comes from the instrument or the method. LOQ will need to be raised for these compounds in future method optimizations – OK SAG

N.18 - Most PFAS were recovered within SOP range. Those that were not have been designated as marginally performing in report or method. Due to the complexity of the matrices involved, this is expected. - See general note 1 QAO Comment - SAG

N.20 - Some calibration checks were slightly out of range. No peaks were detected in any of the actual samples. - Analytes that had trouble with recovery in CCV's have been listed with recovery % in the marginally performing standards excel spreadsheet - SAG

N.21 - For the LOQ Rerun on 5/9/23 on the Sciex, the sample data was added to the original run quant batch on 3/16/23. Some instrument maintenance was performed in between, and RT of the compounds shifted slightly. A Calibration check was run before and after to confirm the RT shift. OK SAG

General Notes:

- This package includes data for method validation and pesticide sample analysis. Method validation data were generated from Agilent 6470 LC/MSMS. Sciex 6500+ LC/MSMS was used for verification of several compounds with no/low recoveries on Agilent 6470 LC/MSMS for method validation samples. Pesticide samples processed with the developed method were analyzed on Sciex 6500+ LC/MSMS (quantitation) and Thermo Orbitrap LC/HRAMS for additional qualitative (semi-quantitative) confirmation.
- 2. There are high background levels for PFBA, PFPeA, and 6:2 FTS in most of the sample matrices during method validation and sample analysis. Recoveries of these compounds are invalid at 0.4 ppb. The LOQs need to be raised to next level of fortification (2 ppb). Additional higher level of spike may need to be performed in the future.
- 3. The validation data were generated on Agilent 6470 LC/MSMS. There were low or no recoveries for PFTrDA (C13), PFTeDA (C14), PFHxDA (C16), PFODA (C18), and PFPO-DA. Re-run of the same extracts (0.4 ppb fortification on Sciex 6500+ LC/MSMS, however, showed good recoveries for PFTrDA, PFTeDA (>80%), and low recoveries for PFHxDA and PFODA (20-40%). The low or no recoveries from Agilent instrument is, therefore, due to the limitation of this instrument, not the performance of the method.
- 4. PFPO-DA is not recovered from sample matrix, but recovered from blank spike, indicating matrix issue for this compound.
- 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.
- 6. The calculated PFAS concentrations in some samples were artificially exaggerated because of the small sample sizes. The available sample weight for some samples is less than 0.1 g. As a result, the calculated concentrations of any detected analytes were artificially exaggerated even if they were just at background levels.

Note B3 - Unique ID's of equipment used other than Agilent LC/TQMS and Sciex QTrap not listed in notebook, to include Orbitrap. DNF – Corrected – OK SAG

Note B5 - Source, Lot, Exp of reagents not listed in notebook in project folder. Standards found on pages 59-63 of notebook YLL-21-01 - Need CoA's for expiration dates and purities. DNF – Added CoA from B23-07 to "Other Documents" folder – OK SAG

Note C2 - Sciex logbook missing analyte information, project column information, and information for 5/9/23 sequence. Q-Exactive logbook missing initials on 3/22/23. DNF – Samples were included at end of YLL run beginning on 5/8/23. Note made in logbook – OK SAG

Note H1 - Uncertainty of Measurement for spike levels has been calculated, Lasee Samples do not contain analytes above background and process blank so no UoM needs to be applied to any results.

Note J - No indication of sample receipt date, condition, storage location, etc for samples found in laboratory notebook (assuming samples were not received under official seal - SOP 028. DNF – Sample History form and sample info added to Other Documents folder – OK SAG

M3 - Marginally Performing Analytes noted in spreadsheet located in the project folder along with the analytical method.

Note M7 - Cannot confirm expiration dates or standard purities, no CoA's in project folder. DNF – See above – OK SAG

Note N1 - Final weights for Lasee Samples number 5 and 9 not listed in notebook. DNF – Corrected – OK SAG

Note N2 - Were the standards for the calibration curve made on 1/26/2023 in YLL Notebook? Calibration curve preparation not signed off/checked by Peer Reviewer DNF – Peer review in process of B23-07 – Calculations checked by DNF for B23-07 project and by YQ for B23-05c usage – OK SAG

Note N4 - Calculation excel worksheets not signed off by peer reviewer. Were the calculations reviewed?

Note N14 - Instrument methods and sequences are not present in the project folder. DNF – Corrected – OK SAG

Note N28 - Lasee Sample results spreadsheet may need to be updated based on possible typo found in calculations spreadsheet. This should result in a decrease in analyte concentration (does not impact the final result) DNF – Corrected typo and updated final results table. Correction reduces apparent PFAS presence observed. - OK SAG

Reference: SOP No. ACB-004 R 3.1

Audited by:		<u>Initials</u>	<u>Date</u>
Analyst(s):	Dul ml	DNF 5/12/23	
Peer Review(s):	Yaorong Qian	Y&	
Senior Staff:	Thuy Nguyen (BC)		
QA Officer:	Stephen a Dilm_	SAG	6/21/2023