Sack, Chris A

From: DeLancey, Siobhan
Sent: Sunday, September 17, 2017 9:24 AM
To: Sack, Chris A
Cc: Strachman-Miller, Jason; Naum, Marianna
Subject: RE: Glyphosate Webpage

Hi Chris—thanks for this. I've made some additional edits—mostly just moving some things around for flow, but I did add a question and answer about why we decided to develop a method specifically for glyphosate when we state earlier in the document that EPA says it has low toxicity. There's also one comment asking for the month in which we resumed the sampling program.

Here's the link:

(b) (5)

Can you take a look and let Jason and I know if these are good? As you know, I'm going to be on leave for the next couple weeks, so Jason will be picking this up. Marianna Naum is acting as team leader in my absence, so I am copying her as well.

Jason, I added a clearance chain at the bottom. Once Chris is good, you should accept changes and delete comments and move it on. Please note that EPA will have to see this, as we describe their role on our page. Megan or one of the other folks in OMA should be able to share through their press office to the SMEs. OCC will also have to review, but whomever does it needs to understand that this is a consumer-focused piece that shouldn't describe FIFRA or the FFD&C Act.

Thanks!

From: Sack, Chris A
Sent: Tuesday, August 22, 2017 9:29 AM
To: DeLancey, Siobhan
Subject: RE: Glyphosate Webpage

Hi Siobhan,

I have attached my edits and comments. I can't speak to the toxicity/safety issues. I did add a thought re why we had not tested glyphosate earlier. It is important for you (within FDA) to understand that FDA has worked continuously to expand the scope of pesticide coverage. The most efficient and effective way to do that was to increase the pesticides we covered using our (b) (4) (b) (4). From the mid 2000s to 2011 the number of pesticides we covered grew from about (b) (4) to over (b) (4) We knew that glyphosate would require its own selective residue method (b) (4) which is extremely costly requiring a dedicated $400K instrument along with the staff to operate the instrument and conduce the extractions. The differences in the cost per residue ratio between the (b) (4) and a (b) (4) are astronomical. Add to that, the fact that glyphosate is less acutely toxic than table salt, it was an easy decision where we would concentrate our efforts to protect the health of the U.S. consumers. It was the GAO audit that eventually pushed us to develop the glyphosate method.

Pleased to serve with you,

Chris

Ph: 240-402-2464

From: DeLancey, Siobhan
Sent: Monday, August 21, 2017 4:13 PM

To: Sack, Chris A
Subject: Glyphosate Webpage

Hi Chris,

I asked Sue Kelly to take a look at the text for the glyphosate web page and I wanted to get your thoughts first before sending to Lauren and Charlotte. Can you take a look and let me know what you think? Sue also posed an additional question: why we decided to begin testing for glyphosate in the first place.

Thanks!

Siobhan

Sack, Chris A

From: McSeveney, Megan
Sent: Monday, October 23, 2017 8:06 AM
To: Robin, Lauren (Posnick)
Cc: DeLancey, Siobhan; Strachman-Miller, Jason; Cassell, Peter; Sack, Chris A
Subject: RE: Flagging: JAMA study finds increased glyphosate in older adults

This is very helpful. As far as I know, we have not had any inquiries from mainstream or trade press about glyphosate for awhile and as a result, I don't think we have announced that we are incorporating glyphosate into routine testing. Are there any concerns about making that information public? Given the feedback from both Lauren and Chris – I think we may want to defer questions about the study to EPA in the short term and provide general background on what we are doing re: sampling and also FDA's role. For our awareness, I think it would make sense to reach out to CDC for any insights they may have too. Thoughts?

Megan McSeveney
Press Officer

Office of Media Affairs
Office of External Affairs
U.S. Food and Drug Administration
Tel: 240-402-4514/Cell:202-380-7748
Megan.McSeveney@fda.hhs.gov

From: Robin, Lauren (Posnick)
Sent: Sunday, October 22, 2017 11:36 PM
To: McSeveney, Megan
Cc: DeLancey, Siobhan; Strachman-Miller, Jason; Cassell, Peter; Sack, Chris A
Subject: RE: Flagging: JAMA study finds increased glyphosate in older adults

HI Megan

A couple of thoughts. It doesn't seem surprising that a herbicide would show up in urinary screens after its introduction, since the tolerances allow for some glyphosate residues to be present on food. In addition to contacting EPA, I would suggest contacting our colleagues at CDC/NHANES. They monitor a wide range of chemicals in urine, and should be able to provide some perspective on these numbers (are they low? Normal? High?). They may also be able to address the appropriateness of the test, methodology, and study group (e.g., is studying an elderly population the most appropriate population, given changes in kidney function?). I'm out of my league here, but CDC should be helpful. Also, they have probably considered and/or are looking into putting glyphosate testing in NHANES.

Lauren

From: Sack, Chris A
Sent: Saturday, October 21, 2017 9:20 AM
To: McSeveney, Megan <Megan.McSeveney@fda.hhs.gov>; Robin, Lauren (Posnick) <Lauren.Robin@fda.hhs.gov>
Cc: DeLancey, Siobhan <Siobhan.Delancey@fda.hhs.gov>; Strachman-Miller, Jason <Jason.Strachman-Miller@fda.hhs.gov>;

Cassell, Peter <Peter.Cassell@fda.hhs.gov>
Subject: RE: Flagging: JAMA study finds increased glyphosate in older adults

Hi Megan,

I will be out of the office this week till Thursday. Some thoughts before I go.

The glyphosate assignment concluded at the end of FY-17. We will begin analyzing the data from the assignment this fall when the records are completed and the samples are closed out. CFSAN-OC will prepare an internal report for the assignment; I am not sure if we will issue a separate report for the public.

We have now moved on to include glyphosate and acid herbicide analysis in selected human and animal food commodities during routine pesticide screening. For human foods we selected (b) (4) different raw agricultural commodities, including fresh fruits and vegetables, grains, dried legumes, olives and peanuts. For animal foods CVM designated (b) (4) different commodities consisting of plant by-products, hay and silage. These analyses started Oct 1, 2017.

Re the study, I would certainly expect the levels of glyphosate in humans to rise over time, given the steady increase of the use of glyphosate, both domestically and internationally. Re the levels, assuming the study is valid (big assumption), it certainly looks alarming that the levels have increased over 1000 % in the last 20 years. However, I don't know if sub ppb levels of glyphosate in urine is significant; a 1000 % of nothing is still nothing.
We would need to defer to EPA's expertise re the levels; although, we might consider consulting the FDA NCTR for their thoughts.

My access to email will be extremely limited while I am out. I will try to check in if I can find time to get to wifi. I will be back late Wed afternoon.

Have a wonderful weekend.

Chris

Ph: 240-402-2464

From: McSeveney, Megan
Sent: Friday, October 20, 2017 3:13 PM
To: Robin, Lauren (Posnick) <Lauren.Robin@fda.hhs.gov>; Sack, Chris A <Chris.Sack@fda.hhs.gov>
Cc: DeLancey, Siobhan <Siobhan.Delancey@fda.hhs.gov>; Strachman-Miller, Jason <Jason.Strachman-Miller@fda.hhs.gov>; Cassell, Peter <Peter.Cassell@fda.hhs.gov>
Subject: Flagging: JAMA study finds increased glyphosate in older adults

Hi Lauren and Chris,

Flagging the below JAMA press release that went out under embargo yesterday along with the attached study that found an increase in the level of glyphosate found in older adults over the last 20+ years. Luckily one of my colleagues in OMA flagged this for me. The embargo lifts Tuesday.

I am working on a reactive statement – that I'll share with you all either today. I don't think we would comment on the study, but we could mention our ongoing glyphosate special assignment. As we have done in the past, I would push questions of how glyphosate tolerances are set and how it is approved back to EPA.

I admit – I'm a bit confused by the study as it seems like these levels aren't that high – especially when I tried to compare them to the tolerances set by EPA. That being said, I am not an expert and appreciate your insights.

Thanks,
M7

Study Finds Increase of Herbicide in Older Adults

JAMA Research Letter: Excretion of the Herbicide Glyphosate in Older Adults Between 1993 and 2016

EMBARGOED FOR RELEASE: 11 A.M. (ET) TUESDAY, OCTOBER 24, 2017

JAMA

Among a sample of older adults living in Southern California, average urine levels of the herbicide glyphosate and its metabolite increased between 1993 and 2016, as did the proportion of samples with detectable levels, according to a study published by *JAMA*.

Glyphosate, the primary ingredient in a herbicide sprayed onto genetically modified crops, is found in these crops at harvest. Genetically modified crops were introduced in the United States in 1994. Environmental exposure through dietary intake of these crops has potential adverse health effects and can be assessed by measuring urinary excretion.

Paul J. Mills, Ph.D., of the University of California, San Diego, and colleagues measured excretion levels of glyphosate and its metabolite aminomethylphosphonic acid (AMPA) in participants from the Rancho Bernardo Study of Healthy Aging. Among the participants in the study, 112 had routine morning spot urinary biospecimens obtained at each of five clinic visits that took place from 1993 to 1996 and from 2014 to 2016. One hundred of these 112 were randomly selected for this study (average age in 2014-2016 was 78 years; 60 percent were women).

The researchers found that the average glyphosate level increased from 0.024 µg/L in 1993-1996 to 0.314 µg/L in 2014-2016, and reached 0.449 µg/L in 2014-2016 for the 70 participants with levels above the limits of detection (LOD). Average AMPA levels increased from 0.008 µg/L in 1993-1996 to 0.285 µg/L in 2014-2016, and reached 0.401 µg/L in 2014-2016 for the 71 participants with levels above the LOD. The prevalence rates of glyphosate samples above the LOD increased significantly over time, from 0.120 in 1993-1996 to 0.700 in 2014-2016. The prevalence of AMPA samples above the LOD increased significantly from 0.050 in 1993-1996 to 0.710 in 2014-2016.

The authors write that animal and human studies suggest that chronic exposure to glyphosate-based herbicides can induce adverse health outcomes. In July 2017, in accordance with the Safe Drinking Water and Toxic Enforcement Act of 1986, the state of California listed glyphosate as a probable carcinogen. “Future studies of the relationships between chronic glyphosate exposure and human health are needed.”

The article notes some limitations, including that the study group lived in Southern California, which might have different exposures than other states.

Sack, Chris A

From: Robin, Lauren (Posnick)
Sent: Friday, October 27, 2017 9:42 AM
To: Sack, Chris A
Subject: FW: For Review: Glyphosate Webpage

Importance: High

In case you didn't receive this. . . this is the glyphosate page to review ASAP.

Thanks
Lauren

From: DeLancey, Siobhan
Sent: Friday, October 20, 2017 1:19 PM
To: Strachman-Miller, Jason <Jason.Strachman-Miller@fda.hhs.gov>; Robin, Lauren (Posnick) <Lauren.Robin@fda.hhs.gov>; Liang, Charlotte <Charlotte.Liang@fda.hhs.gov>
Subject: RE: For Review: Glyphosate Webpage

Our plan is to put this up shortly before the pesticide report goes out, so we can point to it if we get questions. Thanks!

From: Strachman-Miller, Jason
Sent: Friday, October 20, 2017 1:00 PM
To: Robin, Lauren (Posnick); Liang, Charlotte
Cc: DeLancey, Siobhan
Subject: For Review: Glyphosate Webpage

Greetings all,

Below you'll find the proposed glyphosate webpage for your review. If you could review and clear or provide edits by COB Monday, Oct. 23rd, it would be greatly appreciated!

(b) (5)

Thanks,

-Jason

Wong, Jon

Subject: National Pesticide Call
Location: Telecon

Start: Wed 10/4/2017 11:00 AM
End: Wed 10/4/2017 12:00 PM
Show Time As: Tentative

Recurrence: (none)

Meeting Status: Not yet responded

Organizer: Sack, Chris A
Required Attendees: Adams, Neal L; Ajayi, Olusegun J; Atkinson, Krisztina Z; Benjamin, Linda; Blount, Janet; Cassias, Irene; Chamkasem, Narong; Chang, Eugene; Chu, Gabriel; Cooke, William; Coppin, Julia; Councell, Terry; Cromer, Michele; Damanti, Angelo; Day, Thomas; Drake, Connie P.; Eide, David J; Fairchild, Russell D.; Files, Darin; Gonzales, Steven A.; Graham, David F; Graham, Lori M.; Hanson, Madison; Hassan, Nazmul; Hayward, Douglas G; Hetz, Stacy C; Ingram, Shannon; Iorsh, Michael; Islam, Mohammed R; Jenkins, Roy; Johnson, Tonya R.; Jones, Jennifer M.; Katsoudas, Eugenia; Lane, Shannon; Lapainis, Theodore; Liang, Charlotte; Mabry-Smith, Ronald C; Makovi, Carolyn M; Masse, Claude; Mercer, Gregory E; Moore, Joshua; Morris, Cynthia; Nickols, Susan M; Noonan, Gregory; Olson, Melissa; Phan, Vinh T; Podhorniak, Lynda; Purnell, Standra; Ross, Mark S; Russell, Franklin N; Sagardia-Vazquez, Daniel; Satterfield, Gregory E.; Shelby, Rebecca; Si, Siv; Sram, Jacqueline; Thompson, Richard L.; Viner, Marianna; Vonderbrink, John; Waters, Lisa; Wilson, Sarah; Wong, Jon; Wong, Maxine; Yee, Sally; Zhang, Kai
Optional Attendees: Winfield, Sarah

Agenda,

1. ORA Update (Moh)
2. PesTAG Update (Mercer)
3. Analysis of glyphosate and acid herbicides (Sack/Atkinson)
4. Tolerances (Sack)

Chris Sack invites you to an online meeting using WebEx.

Meeting ID: (b) (6)
Meeting Password: (b) (6)

To join this meeting
---
1. Go to https://fda.webex.com/fda/j.php?MTID=m41e2298a6667e2f3004e634073ce84a8
2. If requested, enter your name and email address.
3. If a password is required, enter the meeting password: (b) (6)
4. Click "Join".
5. Follow the instructions that appear on your screen.

Teleconference information
---
1. Provide your number when you join the meeting to receive a call back. Alternatively, you can call one of the following numbers:
Local: (b) (6)
toll free: (b) (6)
2. Follow the instructions that you hear on the phone.
Your Cisco Unified MeetingPlace meeting ID: (b) (6)

FDARichMedia@fda.hhs.gov

Technical support:
Contact FDA Rich Media at 301-796-3333.

IMPORTANT NOTICE: This WebEx service includes a feature that allows audio and any documents and other materials exchanged or viewed during the session to be recorded. By joining this session, you automatically consent to such recordings. If you do not consent to the recording, discuss your concerns with the meeting host prior to the start of the recording or do not join the session. Please note that any such recordings may be subject to discovery in the event of litigation.

Wong, Jon

From: Mercer, Gregory E
Sent: Monday, October 23, 2017 2:21 PM
To: Sack, Chris A; Cassias, Irene; Eide, David J; Islam, Mohammed R; Katsoudas, Eugenia; Thompson, Richard L.; Wong, Jon; Noonan, Gregory; MacMahon, Shaun
Subject: FMD-81

After some sidebar discussions regarding our equipment needs, particularly with the demands on our current (b) (4) systems from the herbicide methods, I've asked ARKL if they would be willing to expand their FMD-81 request to include new (b) (4) systems for the field labs. Any thoughts? I think KC has more (b) (4) systems than the rest of us (because they received one that ARKL requested a couple years ago!) so I'm not sure they would get one if we asked (Moh?)…

As I mentioned on the call, I believe (b) (4) is the future but think adding it at this point in time is a recipe for failure – initially at least. Given our limited manpower, we would all struggle to bring a new, complex technology online and still perform all of our usual duties. I also think we are on borrowed time with our current (b) (4) instruments and workload. We could all use a backup system, and the (b) (4) equipment can be added almost seamlessly. The (b) (4) will also do a better job in negative mode, which could be handy for glyphosate (if we are willing to crap up another LC with ion pair reagents). The challenge will be getting the system we want.

The same could be said for our (b) (4) systems. We need backups, the instruments are getting a little old, and the technology has improved quite a bit. The newest (b) (4) in our lab is from 2010 and has had some problems as of late. PNL is requesting new (b) (4) systems for the field too… All I know is we don't get diddly unless we ask!

-Greg

Wong, Jon

Subject: November PesTAG call

Start: Wed 11/15/2017 12:00 PM
End: Wed 11/15/2017 1:00 PM
Show Time As: Tentative

Recurrence: (none)

Meeting Status: Not yet responded

Organizer: Mercer, Gregory E
Required Attendees Sack, Chris A; Cassias, Irene; Eide, David J; Islam, Mohammed R; Thompson, Richard L.; Wong, Jon; Liang, Charlotte; Noonan, Gregory; MacMahon, Shaun; Martin, William B.

Attached is our (b) (4) methods/standard mix spreadsheet. I was also asked to add another agenda item for Bill Martin…

It might be tough to get everyone together on the 3rd Wednesday next month so let's have a quick call tomorrow. I switched to 9:00 AM PST to accommodate most schedules…

Agenda:

(b) (5)

I will try to get a spreadsheet of our (b) (4) methods sent out. Hopefully I can highlight most of the changes being made to simplify the transition…
Greg Mercer invites you to an online meeting using WebEx.

Meeting ID: (b) (6)
Meeting Password: (b) (6)

To join this meeting
---

1. Go to https://fda.webex.com/fda/j.php?MTID=mdf3f170209415026844dc44caa6fa7eb
2. If requested, enter your name and email address.
3. If a password is required, enter the meeting password: 8u5bnK7K
4. Click "Join".
5. Follow the instructions that appear on your screen.

Teleconference information
---
1. Provide your number when you join the meeting to receive a call back. Alternatively, you can call one of the following numbers:
Local: (b) (6)
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FDARichMedia@fda.hhs.gov

Technical support:
Contact FDA Rich Media at 301-796-3333.

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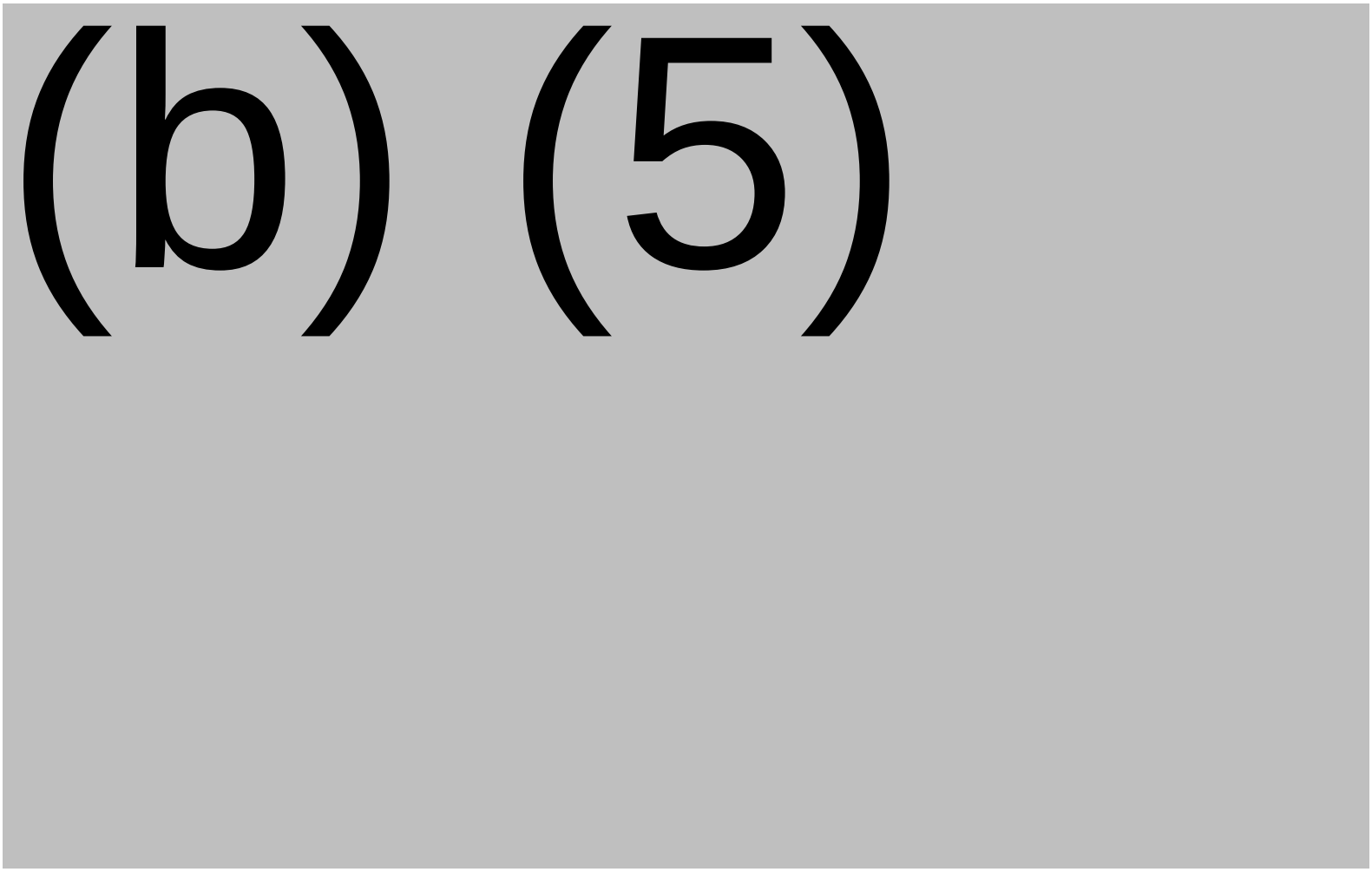
Parker, Christine

From: Parker, Christine
Sent: Friday, September 01, 2017 2:24 PM
To: Yakes, Betsy; Noonan, Gregory
Cc: MacMahon, Shaun
Subject: RE: FY2017 Accomplishments--Executive Plans

Greg,

Below are a few additions I have to Betsy's list. I included everything that came to mind, but realize many of these items may not meet the level of significance for documentation. Please let me know if additional information is required.

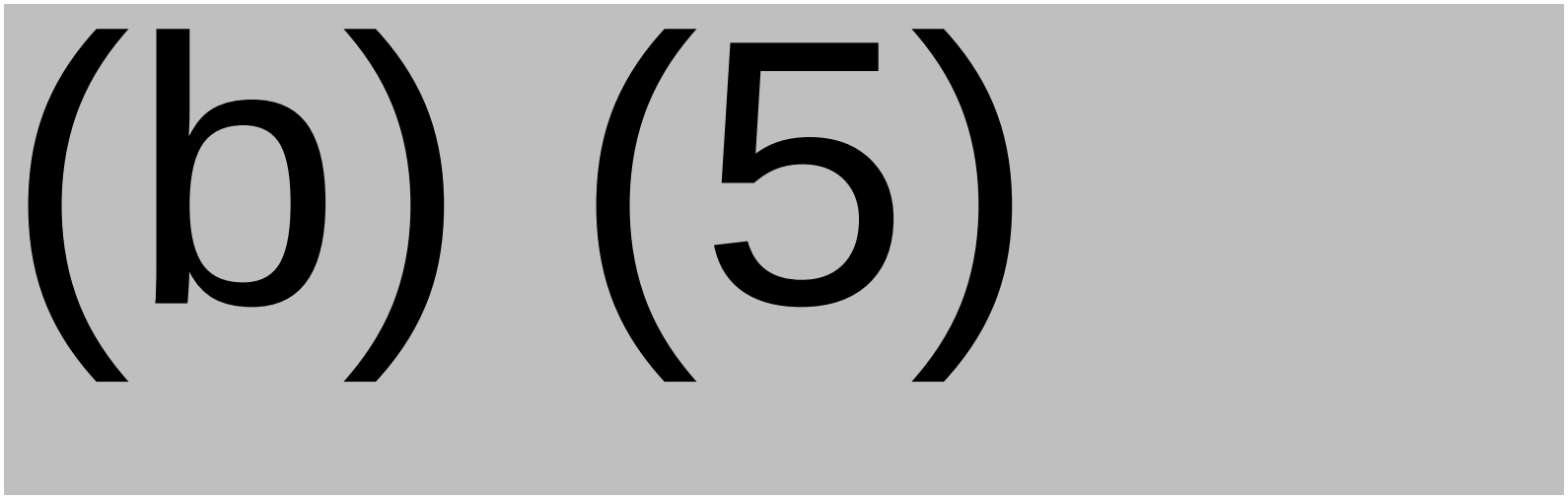


Enjoy the weekend,
Christine

From: Yakes, Betsy
Sent: Friday, September 01, 2017 7:52 AM
To: Noonan, Gregory
Cc: MacMahon, Shaun; Parker, Christine
Subject: RE: FY2017 Accomplishments--Executive Plans

Morning Greg,

I went back through my notes from June to Oct. 2016, and these potentially fit into what Kelly is requesting:



I am not sure on the status and full details of these as the year progressed nor if these are significant enough to warrant passing up the management chain. If you need me to search things out more, just let me know.

Enjoy the long weekend,
Betsy

From: Noonan, Gregory
Sent: Thursday, August 31, 2017 8:08 PM
To: MacMahon, Shaun; Parker, Christine; Yakes, Betsy
Subject: Fwd: FY2017 Accomplishments--Executive Plans
Importance: High

I didn't ask you to do this and if you don't recall that is fine. But if you have recollection of any significant accomplishments can you please send them to me. I don't need any supporting text just bullets of items you recall. I have a bunch, but I am sure I am forgetting something.
Thanks.

Greg Noonan
240-701-7415

From: Bunning, Vincent <Vincent.Bunning@fda.hhs.gov>
Date: August 31, 2017 at 6:52:36 PM EDT
To: Brown, Detra <Detra.Brown@fda.hhs.gov>, CFSAN ORS DIV DIRS <CFSANORSDIVDIRS@fda.hhs.gov>
Cc: Bunning, Vincent <Vincent.Bunning@fda.hhs.gov>
Subject: FY2017 Accomplishments--Executive Plans
Importance: High

I am asking the Divisions to report out on all significant accomplishments to date please for FY17. I have asked the DDs to keep an ongoing list of accomplishments throughout the year, as I have requested the last two years, to make data calls (budget, individual performance plans---yours and mine, CFSAN Operating Plan [Yellow Book]*etc.)* easier. I

hope you have been able to do this! The format we agreed upon in the past is the most helpful (see attached). Developing accomplishments now will also put the DDs and other OD direct reports in good shape for the FY2017 PMAP closeout and rating period. Some areas that are important for the Executive Plans (remember my plan closes out at the end of the fiscal year/end of September) that will be useful to me in the near term are listed below. I realize the thrust areas have overlap---just develop accomplishments in these areas and I will sort out the best applicability to a given item below.

Some areas of focus (those in **red** are specifically called out for FY17 by OFVM) for my Executive Plan include:

1. Implementation of OFVM and CFSAN strategic action plans, FY2017 program Alignment Objectives, Performance outcomes (HHS and FDA goals)etc.

(b) (5)

2. FDA's commitment to diversity and inclusion; employee professional development; maximizing employee potential

(b) (5)

3. Responsiveness to OM requests to administer human, financial, material, and information resources/Effective management of resources

- Budget request and process
- IT---CFSAN ITRB
- Real property/inventory (**Detra Brown** please describe process and accomplishment in a short narrative); safety; QA; select agent; radiation safety

4. Internal and external collaboration and leveraging/outreach

- Meetings with individual states or state organizations (AFDO, 50 state meeting, Delmarva)/advance IFSS principles
- Successful science and research outreach/collaborations with industry achieving an outcome
- Other examples of working with states and/or industry

5. Performance results related to strategic plan

A. Food Safety

- ORS science/research outcomes that fostered completion of FSMA/cGMP modernization rules: 3rd party; produce; PCs; foreign supplier verification
- ORS science/research outcomes that fostered completion of final risk assessments for any product/hazard
- Other examples in the vein of the above
- ORA coordination efforts---SRSC as well as our own

B. Nutrition

- ORS science/research outcomes that fostered completion/support for voluntary gluten free labeling of hydrolyzed and fermented food---studies to understand nature of gluten hydrolysis---subsequent methods development---transfer and use
- Other nutrition examples

C. Regulatory Science and Innovation

- (b) (5) ---add to/update info below in **red** from last year please
- Improved coordination with ORA micro and chem method development and validation (specific method examples; SOP development; outcomes from specific ORA collaborations)
- Other examples of strengthening FDA's regulatory lab capacity

D. Globalization

- Expanding WGS to international partners
- ISO efforts and outcomes
- CODEX efforts and outcome
- Other international

Thanks for all your help and hard work!

This FY2017 accomplishment data call is due to me by **COB Friday September 15, 2017**---I have to submit my narrative accomplishments for the Executive Plan by COB Tuesday September 22, 2017.

Kelly

V. Kelly Bunning, Ph.D.

Director

FDA/CFSAN/Office of Regulatory Science (HFS-700)

5001 Campus Drive

College Park, MD 20740-3835

(PH) 240-402-2404

(PH-Direct) 240-402-1908

(iPhone) 240-704-5067

(FAX) 301-436-2332

(E-mail) Vincent.Bunning@fda.hhs.gov

Public-private partnerships for ORS in FY2016

(b) (5)

(b) (5)

Parker, Christine

From: Noonan, Gregory
Sent: Monday, November 27, 2017 2:54 PM
To: Parker, Christine
Subject: RE: Wish List
Attachments: NonCARTS Projects June 2017.docx; Instrument Wish List for 2018.xlsx

Here is the wish list and the NonCARTS (which should be up to date). The FY18 budget should contain any purchase as either primary or additional needs. The budget is on the ORS Sharepoint
Greg

From: Parker, Christine
Sent: Monday, November 27, 2017 2:16 PM
To: Noonan, Gregory <Gregory.Noonan@fda.hhs.gov>
Subject: Wish List

Hi Greg,

Do you have the FY2018 DBC Equipment list? I may have missed it, but I was unable to locate the list on the DBC SharePoint site. Alternatively, please let me know if there are any updates to the FY18_ORS_projected_6_1_2017 spreadsheet (ORS Shared Documents)

Christine

Christine H. Parker, Ph.D.
Research Chemist

Center for Food Safety and Applied Nutrition
Office of Regulatory Science
U.S. Food and Drug Administration
Tel: 240-402-2019
Christine.Parker@fda.hhs.gov

From: Pawar, Rahul
To: Noonan, Gregory
Subject: RE: glyphosate
Date: Friday, September 22, 2017 2:15:00 PM

Greg,
Yes, I agree. I will look into this next week.
Rahul

From: Noonan, Gregory
Sent: Friday, September 22, 2017 1:15 PM
To: Pawar, Rahul <Rahul.Pawar@fda.hhs.gov>
Subject: glyphosate

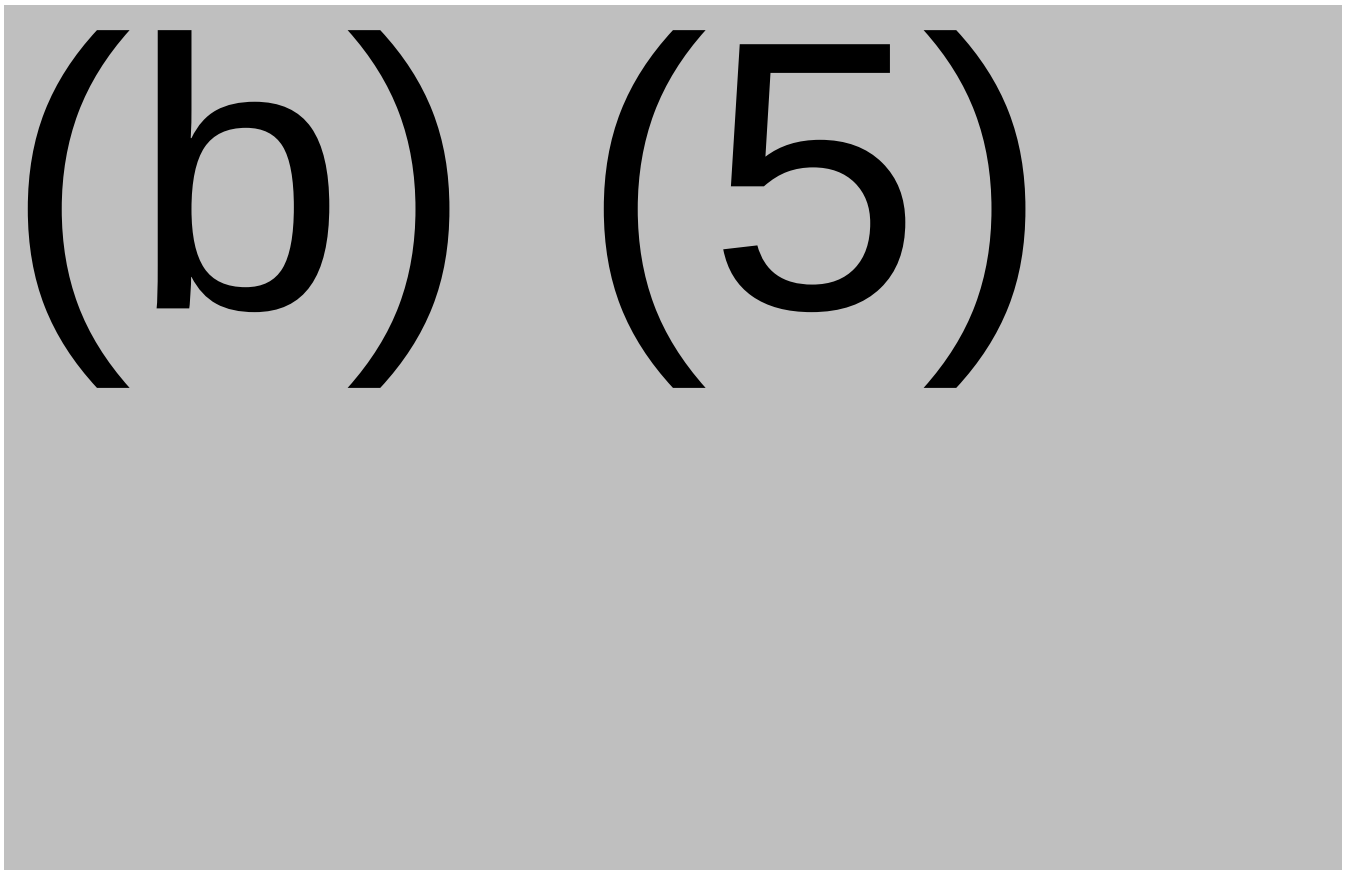
Rahul,
Can you get some details on the glyphosate work that is being done. I don't really want Jimmy spending much time on this, I prefer he work on his aminoglycosides and the vet residues. If Jon wants to spend time and is working on a project it needs to get written up for CARTS or at least captured in some manner.
Greg

Gregory O. Noonan, PhD
Director, Division of Bioanalytical Chemistry
Food and Drug Administration
5001 Campus Drive, HFS 715
College Park, MD 20740

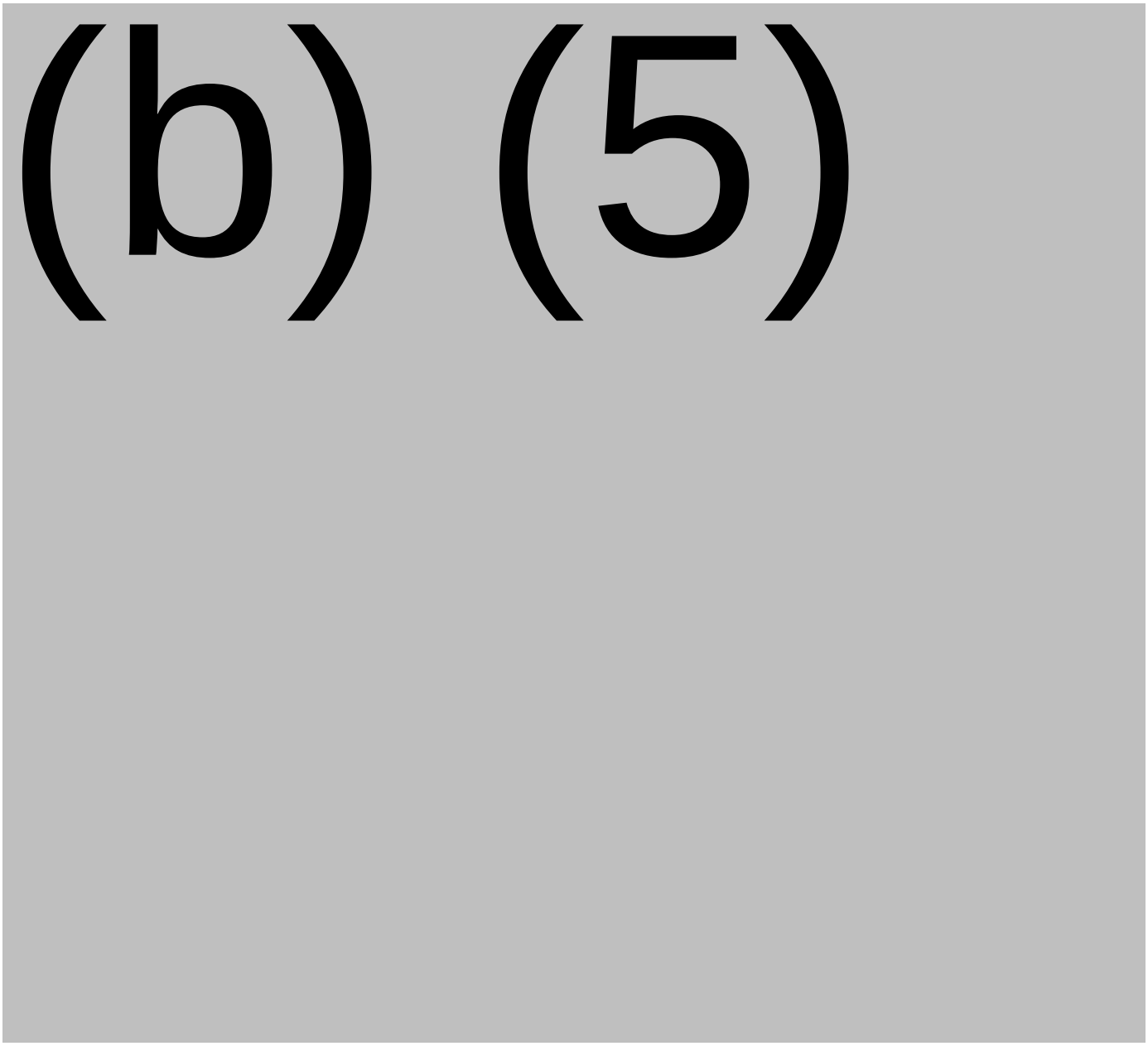
PH: 240-402-2250
FAX: 301-436-2634
Mobile: 240-701-7415
Gregory.Noonan@fda.hhs.gov

CRCG Agenda: 11/16/2017 Meeting

1. Research Coordination
    a. Reorganization of existing projects under Research Outcomes: see Word document (Research Outcomes and projects 11_16_2017.docx) (also spreadsheet under CRCG Sharepoint site)
        i. John Callahan to update how these will be handled in CARTS
            1. CARTS Administrator will connect project with RO under profile (admin to consult with manager or RCG)
            2. Managers will supply quarterly update through CARTS (reminders will be sent- 1000 character limitation)
            3. Updates will be pulled from CARTS automatically using business object tool
            4. CRCG can request that projects be put under an RO
        ii. Do we want to add a technology/instrumentation evaluation RO?
        iii. Specific questions
            1. Line 16: CVM IV01033- Zilpaterol in liver- cross-cutting?
            2. Line 17: CVM beta-lactams in milk; will there be a project?
            3. Line 21: CVM IV01305- residues in sheep fat; cross-cutting?
            4. Line 30: CVM 00323: residue incursion in fish; cross-cutting?
    b. Carts Bi-Weekly updates; projects of note- see Bi-weekly CARTS summaries 11_16_2017.docx
2. CMVS Updates- Shaun MacMahon (last month's report in red)



4. Methods Compendium Update
    a. Methods under consideration or in process



(b) (5)

My proposal for method numbering is this:

(b) (5)

Would it be possible for you to bring this up at the CRCG meeting on Thursday?

Miscellaneous Chemicals/Toxins

(b) (5)

(b) (5)

PesTAG Meeting Minutes

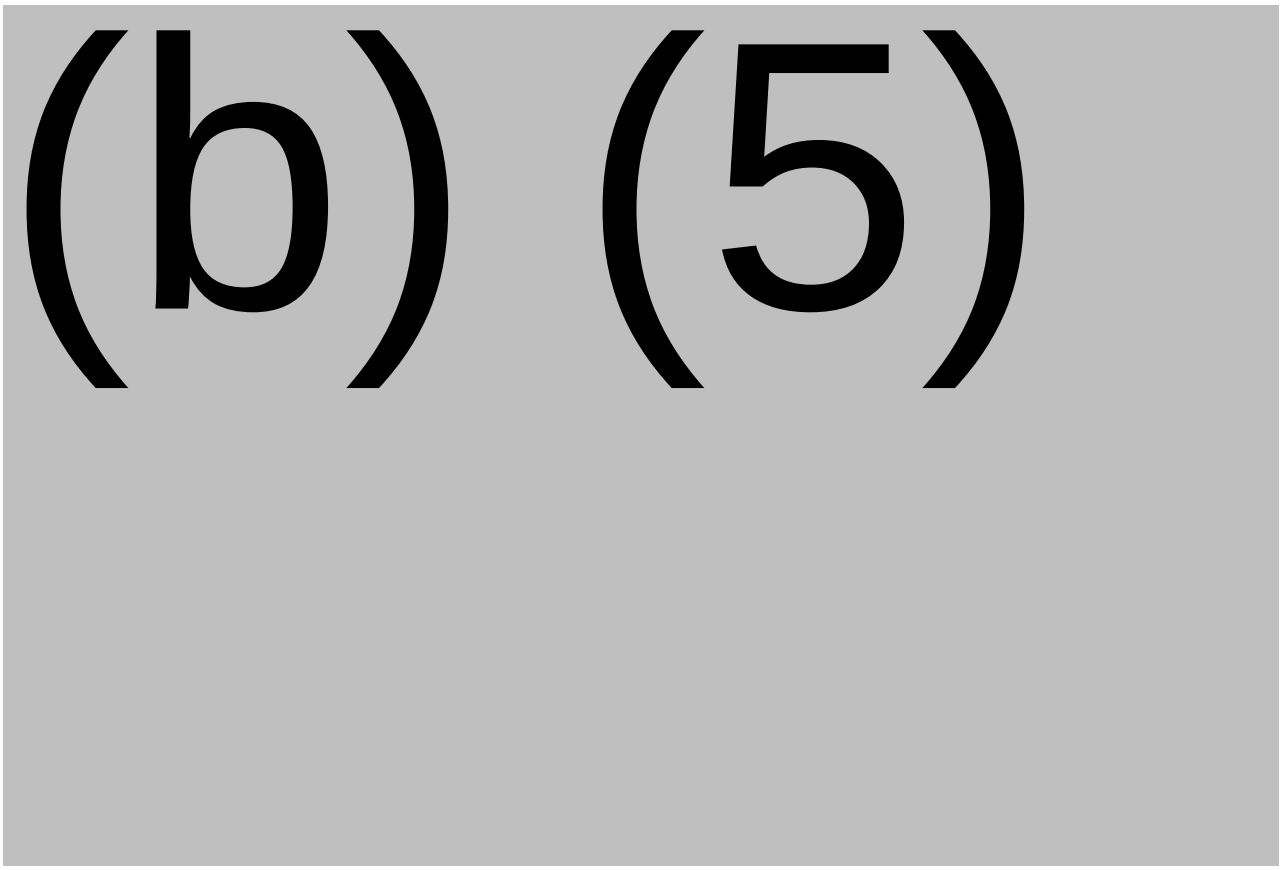
Date: October 18, 2017 Conference Call
Time: 1:00 PM EDT

Attendance: PNL, PSFFL, KCL, ARKL, NFFL CFSAN, ORA-ORS

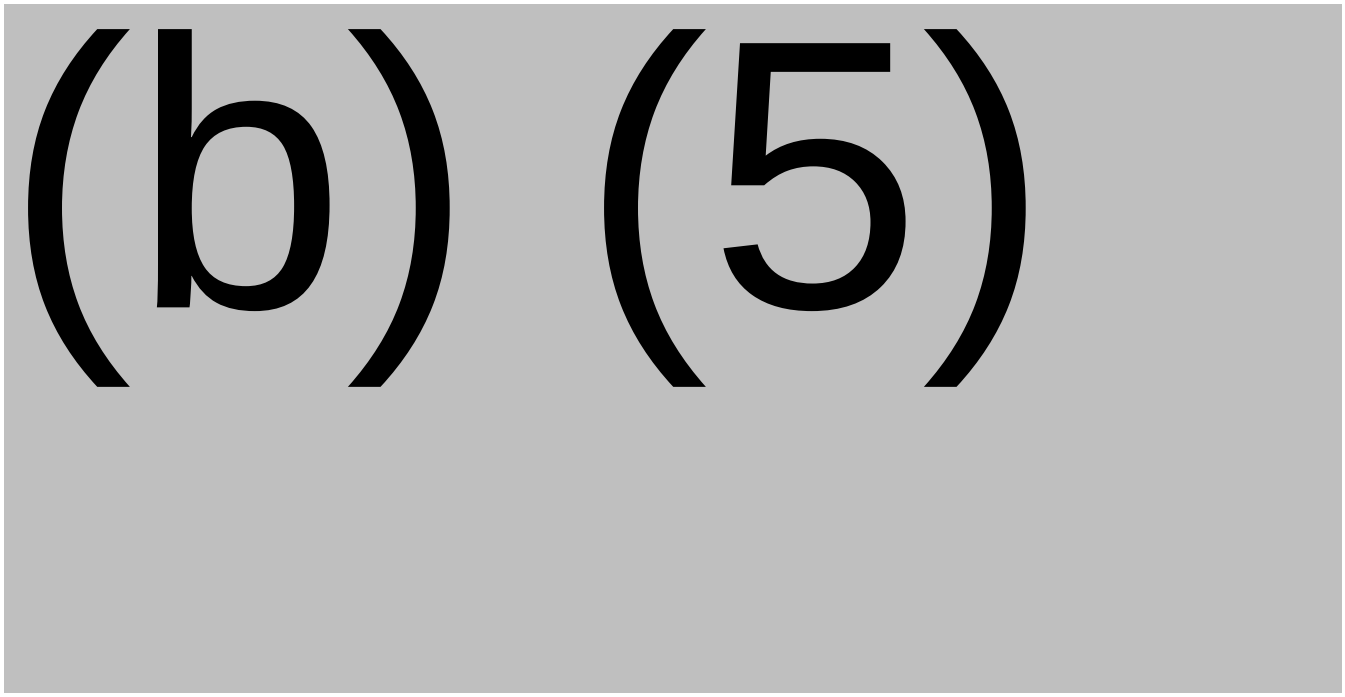
Agenda Items:

1) **Around the horn – discuss AH and glyphosate progress/headaches**
2) **Finalize ORA-LAB.010**
3) **New standard mixes – plans for purchasing**
4) **Charter – Moh/CFSAN update?**

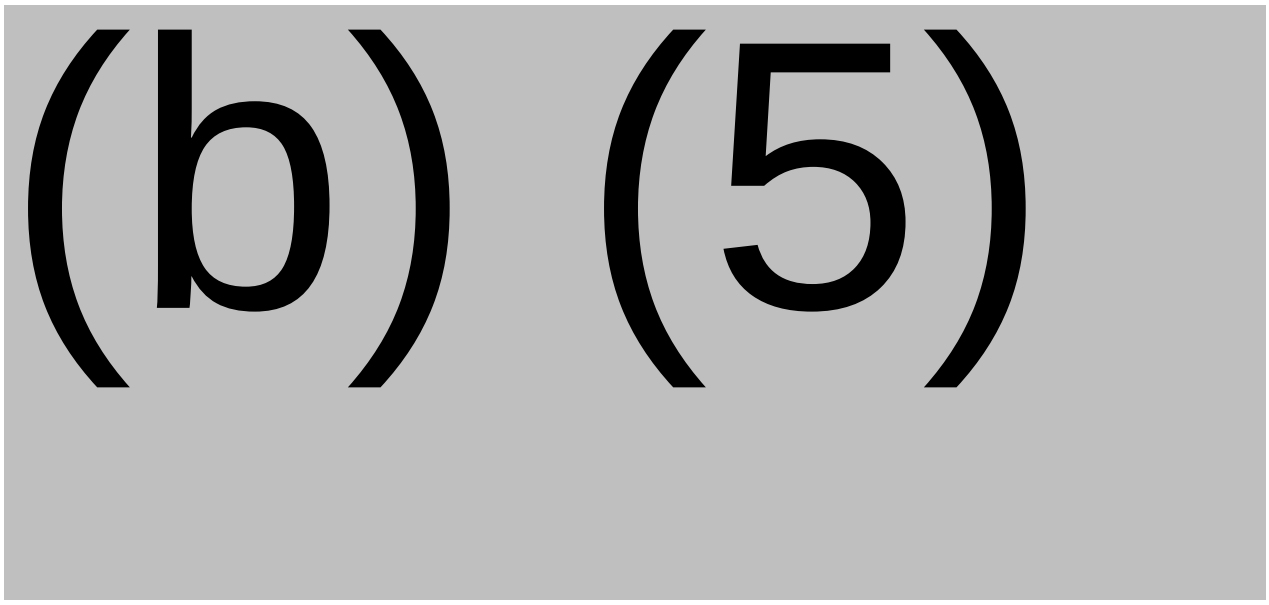
1) Acid Herbicides and Glyphosate progress/headaches



2) Finalize ORA-LAB.010



3) New standard mixes – plans for purchasing



4) Charter – Moh/CFSAN update?

Moh said he forwarded the draft charter to Michael A. McLaughlin, Director- OFFLO (ORS) on 8-28-2017. He will check the progress with ORCE and ORS management and share the outcome with group.

5) Other notes

Greg asked for suggestions for the next meeting. He said we need to prepare for the FMD-81 cycle. Few, if any, purchases have been made recently and the instruments will soon need to be replaced or upgraded.
Moh asked to submit your FY18 FMD-81 requests for major equipment as soon as possible if they haven't already been submitted.

Glyphosate Method Collaboration Report

Introduction

A multi-laboratory validation (collaboration) was conducted of a method for the determination of residue levels of glyphosate, glufosinate, and two degradants of glyphosate N-acetylglyphosate and AMPA. Single laboratory validation of the method was conducted at PSW prior to the collaboration. Seven FDA pesticide laboratories plan to participate in the collaboration eventually. Data from three laboratories (ARL, PNW, and PSW) have been received at this time; this preliminary collaboration report summarizes data submitted from those three laboratories only. A final report encompassing all participating laboratories will be issued after all data from all laboratories has been submitted.

In addition to the collaboration summary of data from all three laboratories, an abbreviated report for the single laboratory validation and each collaborating laboratory are included as attachments to the collaboration report.

Conclusion

The collaboration data indicates the method is suitable for the purpose of quantitative determination for residues of glyphosate, glufosinate and N-acetylglyphosate and semi quantitative determination of AMPA residues in the three primary matrix types analyzed in the FDA pesticide program, i.e., high moisture, low moisture, and high fat items. The collaboration meets all the requirements of a level three multi-laboratory validation as per the "Guidelines for Validation of Chemical Methods for the FDA FVM Program, 2nd Edition.

Protocols and Procedure

Commodities were selected to represent the three major food commodity types analyzed in the FDA pesticide program, i.e. grain corn for dry products, carrots for high moisture products, and avocados for high lipid commodities. Composites of each of these three study matrices were prepared, composited, and distributed to the participating laboratories (PNW, PSW, KAN, ARL, SRL, NRL and CFSAN). Note: avocados were prepared without the outer peel. Each lab analyzed all matrices fortified with each analyte at the fortification levels in replicate as listed below:

i none: 2x
ii 0.050 ppm: 2x
iii 0.250 ppm: 2x
iv 0.500: 2x

Each lab was additionally sent two samples previously found to contain incurred glyphosate residues when analyzed at SRL using the method described in LIB 4596, i.e., ground grain corn in which 0.04 ppm was found and ground soy beans in which 4.5 ppm was found.

Glyphosate Method Collaboration Report

A detailed protocol is provided in attachment A and the method is provided in attachment B.

PSW conducted a single laboratory validation (SLV) of the procedure using the same procedure and collaboration protocol. The SLV results and protocols are reported in the C attachments.

Prior to conducting multiple laboratory method validation each participating laboratory was required to demonstrate proficiency with the procedure. Instrument proficiency was demonstrated conducting system suitability tests that included determination of accuracy, precision, linearity and LOQ by preparing and injecting standards. Results of the system suitability testing are reported with the attached individual laboratory reports (attachments C, F, G and H).

The concentrations and spike recoveries were calculated by single level calibration using average responses of matrix matched standards bracketing the samples and prepared at the same concentration as the spiked sample. For glyphosate and glufosinate residue levels were calculated using corresponding isotopic internal standards added to the extraction solvent prior to analysis. AMPA residues were calculated against the glyphosate isotopic internal standard. Residues of N-acetylglyphosate were calculated using external standard calibration.

The mean recoveries for all three spike levels (50, 250, and 500 ng/g) were calculated by matrix for each laboratory. The overall mean, RSD and method uncertainty (MU) of all three laboratories was calculated for each matrix. The linearity coefficient of determination (R^2) was calculated from the concentrations found at each level for each matrix and laboratory by squaring the Excel correlation function (Correl); the average R^2 of the three laboratories is reported in Table 1. Method specificity was evaluated by the analysis of control matrices. Acceptable validation specifications for the collaboration study are listed below.

Specificity: No residues found in blank control matrices

Recovery: 70-120 % RSD: 15% MU: 30% R^2: 0.990

Results and Discussion

The method collaboration results in this report were provided by three of the participating laboratories: ARL, PNW and PSW. Table 1 contains the summary statistical analysis of all collaboration analyses; results that did not meet specifications are highlighted in red font. Scatter plots of the recoveries are provided in attachment D. No residues were found in the control samples analyzed for each matrix. All results for glyphosate, glufosinate, and N-acetylglyphosate were within the validation specifications. The linearity of the AMPA results did not meet the specification of R2 = 0.99 in any of the three matrices studied, however all were above 0.95. One lab reported low recoveries (48.6 % and 61.3 %) of AMPA in avocado and carrot, respectively.

Glyphosate Method Collaboration Report

Table 1. Summary data includes the average spike recovery for each lab, overall average recovery, RSD, method uncertainty (MU) of the spike recoveries and the average coefficient of determination (R^2) of the spike concentrations.

Matrix	ARL	PNW	PSW	Mean	RSD	MU	R^2
Glyphosate					(b) (5)	(b) (5)	
Avocado	85.3	87.2	96.6	89.7			0.9990
Carrot	80.0	85.9	83.7	83.2			0.9995
Corn	91.4	95.1	101.8	96.1			0.9995
Glufosinate					(b) (5)	(b) (5)	
Avocado	82.9	87.0	94.4	88.1			0.9970
Carrot	81.0	90.4	84.6	85.3			0.9991
Corn	98.4	101.4	102.0	100.6			0.9994
N-acetylglyphosate				(b) (5)	(b) (5)	(b) (5)	
Avocado	(b) (5)	90.3	106.3				0.9941
Carrot	79.7	86.7	97.7	88.0			0.9965
Corn	93.1	94.4	117.9	101.8			0.9979
AMPA	(b) (5)				(b) (5)	(b) (5)	(b) (5)
Avocado		87.3	85.9	74.0			
Carrot		83.4	90.9	78.5			
Corn		76.5	90.3	87.5			

The matrix effect for each analyte/matrix combination was evaluated by calculating residue concentrations using both matrix matched standards and standards prepared in solvent and comparing the slopes of the corresponding linearity charts. Results of the matrix study are tabulated in Table 2 and linearity charts for each analyte/matrix combination are provided in attachment E. Results indicate none of the matrices in the study had much effect on the determination of glyphosate, glufosinate and N-acetylglyphosate. However, all three matrices had a significant impact on residues of AMPA with matrix effects of 391 % in avocado, 327 % in carrot, and 455 % in corn. These results also reflect the advantage of using isotopically labelled internal standards.

Table 2. Matrix effects as percentages of slope ratios of residues calculated for the three spike levels using standards prepared in solvent vs matrix extracts

Compound	Avocado	Carrot	Corn
Glyphosate	91.1	102.2	100.7
Glufosinate	89.4	90.5	103.3
N-acetylglyphosate	108.1	103.1	101.3
AMPA	391	327	455

Each laboratory analyzed a corn sample and a soy sample previously analyzed and found to contain incurred residue of glyphosate. Results of the incurred residue analysis, tabulated in Table 3, are in excellent agreement.

Table 3. Incurred residues (ppb) in corn and soy samples.

Matrix	Original	ARL	PNW	PSW	Mean	RSD
Corn	40	36	35	46	39.3	(12.7)
Soy	4500	4290	4610	4620	4510	(3.4)

For the method collaboration study spike recoveries were calculated based upon a single level calibration at the same concentration as the spike level, i.e., the 50 ng/g spikes were calculated based upon calibration at 50 ng/g equivalence, or 10 ng/ml. Once implemented for routine analysis calibration will be conducted at a single level equivalent to 250 ng/g in the sample. In Table 4 the relative percent difference (RPD) of spike recoveries from the collaboration and the same spike recoveries calculated using a single level standard at concentration equivalent to 250 ng/g. Very low RPDs demonstrate the linearity of the method and accuracy of residue levels calculated from a single level calibration.

Table 4. Relative Percent Difference (RPD) of average recoveries for all levels and laboratories calculated based upon a single level calibration at 250 ng/g vs. calibration per each individual spike level.

Matrix	Single Level	Per Level	RPD	Single Level	Per Level	RPD
	Glyphosate			*Glufosinate*		
Avocado	90.1	89.7	0.4	87.6	88.1	0.6
Carrot	84.7	83.2	1.7	86.8	85.3	1.7
Corn	98.4	96.1	2.4	101.2	100.6	0.6
	N-acetylglyphosate			*AMPA*		
Avocado	87.6	96.9	10.1	65.9	74	11.5
Carrot	86.8	88	1.4	76.9	78.5	2.0
Corn	101.2	101.8	0.6	90.6	87.5	3.4

Glyphosate Method Collaboration Report

Attachments

A. Collaboration Protocol
B. Analytical Method
C. Single Laboratory Validation
    C_1 SLV Method Recovery Charts
    C_2 SLV Method Linearity Charts
D. Method Collaboration Recovery Charts
E. Method Collaboration Matrix Effects Charts
F. PSW Collaboration Data and System Suitability
    F_1 PSW Recovery Charts
    F_2 PSW Linearity Charts
G. PNW Collaboration Data and System Suitability
    G_1 PNW Recovery Charts
    G_2 PNW Linearity Charts
H. ARL Collaboration Data and System Suitability
    H_1 ARL Recovery Charts
    H_2 ARL Linearity Charts

Collaboration Protocol

Matrices: corn (dry), carrot (high moisture), avocado (high fat)

Analyses:

Recovery Study

Level	N*
Control	2
Spike 50	2
Spike 250	2
Spike 500	2

** replicates per matrix*

Incurred Residues

Matrix	Level
Corn	~40 ng/g
Soybean	~4.5 µg/g

Preparation of Standards: Prepare calibration/fortification standards in both solvent and in matrix extracts and listed below.

Calibration Standards in Solvent				Matrix Calibration Standards			
Std Conc (ng/ml)	Spk Std[1] Conc (µg/ml)	Spk Std Volume Added (µl)	Dilution[2] Volume (ml)	Std Conc (ng/ml)	Spk Std[1] Conc (µg/ml)	Spk Std Volume Added (µl)	Dilution[3] Volume (ml)
	corn (2 g sample)						
10	1	100	10	10	1	50	5
50	5	100	10	50	5	50	5
100	5	200	10	100	5	100	5
	carrot/avocado (5 g sample)						
10	5	50	25	10	1	100	10
50	5	250	25	50	5	100	10
100	50	50	25	100	50	20	10

[1] *Prepare mixed native standards as directed in method step C.4*

[2] *Dilute with 50 ng/ml IS fortified extraction solvent*

[3] *Dilute with control sample matrix*

Fortification Procedure:

Spike Level (ng/g)	Spk Std Conc (µg/ml)	Volume Added (µl)
corn (2 g/sample)		
50	1	100
250	5	100
500	5	200

Collaboration Protocol

carrot/avocado (5 g/sample)		
50	5	50
250	5	250
500	50	50

Extraction Cleanup for Avocado:

Follow method as written. Re the cleanup option for avocadoes; i.e. dichloromethane (DCM) vs petroleum ether (PE) three ORA labs agreed to use DCM and the remaining three ORA labs agreed to use PE. CFSAN can choose either.

DCM	PE
ARL	PNW
SRL	PSW
KAN	NRL

LCMS Transition Names:

AMPA[110-63] 1
AMPA[110-79] 2
AMPA[110-81] 3
Glu[180-63] 1
Glu[180-95] 2
Glu[180-85] 3
Glu[183-63] IS
Gly[168-63] 1
Gly[168-79] 2
Gly[168-150] 3
Gly[171-63] IS
N-acetyl[210-150] 1
N-acetyl[210-63] 2
N-acetyl[210-168] 3

LCMS Calibration: Calibrate using single level calibration for each spike level. Assign the internal standards as below.

Analyte	Internal Standard
Glyphosate:	Glyphosate-^{13}C
N-acetylglyphosate:	Glyphosate-^{13}C
AMPA:	Glyphosate-^{13}C
Glufosinate:	Glufosinate-D^{3}

Collaboration Protocol

Inj Sequence: Group by spike level. Assign Sample Name to Sample description and the Sample Types and Actual Concentrations listed in the table below.

Description	Sample Name	Sample Type	Actual Conc
50 ng/g spike level			
10 ng/ml calibration std in solvent	CalStd10	Standard	50
10 ng/ml calibration std in solvent	CalStd10	Standard	50
10 ng/ml corn matrix calibration std	MatStd10 Corn	QC	50
Corn control	Control Corn	Unknown	
Corn spike 50 #1	Spk50-1 Corn	QC	50
Corn spike 50 #2	Spk50-2 Corn	QC	50
Corn incurred residue	Corn Incur	Unknown	
10 ng/ml corn matrix calibration std	MatStd10 Corn	QC	50
10 ng/ml calibration std in solvent	CalStd10	Standard	50
10 ng/ml carrot matrix calibration std	MatStd10 Carrot	QC	50
Carrot control	Control Carrot	Unknown	
Carrot spike 50 #1	Spk50-1 Carrot	QC	50
Carrot spike 50 #2	Spk50-2 Carrot	QC	50
10 ng/ml carrot matrix calibration std	MatStd10 Carrot	QC	50
10 ng/ml calibration std in solvent	CalStd10	Standard	50
10 ng/ml avocado matrix calibration std	MatStd10 Avocado	QC	50
Avocado control	Control Avocado	Unknown	
Avocado spike 50 #1	Spk50-1 Avocado	QC	50
Avocado spike 50 #2	Spk50-2 Avocado	QC	50
10 ng/ml avocado matrix calibration std	MatStd10 Avocado	QC	50
10 ng/ml calibration std in solvent	CalStd10	Standard	50
250 ng/g spike level			
50 ng/ml calibration std in solvent	CalStd50	Standard	250
50 ng/ml calibration std in solvent	CalStd50	Standard	250
50 ng/ml corn matrix calibration std	MatStd50 Corn	QC	250
Corn spike 250 #1	Spk250-1 Corn	QC	250
Corn spike 250 #2	Spk250-2 Corn	QC	250
50 ng/ml corn matrix calibration std	MatStd50 Corn	QC	250
50 ng/ml calibration std in solvent	CalStd50	Standard	250
50 ng/ml carrot matrix calibration std	MatStd50 Carrot	QC	250
Carrot spike 250 #1	Spk250-1 Carrot	QC	250
Carrot spike 250 #2	Spk250-2 Carrot	QC	250
50 ng/ml carrot matrix calibration std	MatStd50 Carrot	QC	250
50 ng/ml calibration std in solvent	CalStd50	Standard	250
50 ng/ml avocado matrix calibration std	MatStd50 Avocado	QC	250
Avocado spike 250 #1	Spk250-1 Avocado	QC	250
Avocado spike 250 #2	Spk250-2 Avocado	QC	250
50 ng/ml avocado matrix calibration std	MatStd50 Avocado	QC	250
50 ng/ml calibration std in solvent	CalStd50	Standard	250

Collaboration Protocol

500 ng/g spike level

100 ng/ml calibration std in solvent	CalStd100	Standard	500
100 ng/ml calibration std in solvent	CalStd100	Standard	500
100 ng/ml corn matrix calibration std	MatStd100 Corn	QC	500
Corn spike 500 #1	Spk250-1 Corn	QC	500
Corn spike 500 #2	Spk250-2 Corn	QC	500
100 ng/ml corn matrix calibration std	MatStd100 Corn	QC	500
100 ng/ml calibration std in solvent	CalStd100	Standard	500
100 ng/ml carrot matrix calibration std	MatStd100 Carrot	QC	500
Carrot spike 500 #1	Spk250-1 Carrot	QC	500
Carrot spike 500 #2	Spk250-2 Carrot	QC	500
100 ng/ml carrot matrix calibration std	MatStd100 Carrot	QC	500
100 ng/ml calibration std in solvent	CalStd100	Standard	500
100 ng/ml avocado matrix calibration std	MatStd100 Avocado	QC	500
Avocado spike 500 #1	Spk250-1 Avocado	QC	500
Avocado spike 500 #2	Spk250-2 Avocado	QC	500
100 ng/ml avocado matrix calibration std	MatStd100 Avocado	QC	500
100 ng/ml calibration std in solvent	CalStd100	Standard	500
100 ng/ml soy matrix calibration std	MatStd100 Soy	QC	500
Soy control	Control Corn	Unknown	
Soy incurred residue	Soy Incur	Unknown	
Soy incurred residue Dil 1-10	Soy Incur (1-10)	Unknown	
100 ng/ml soy matrix calibration std	MatStd100 Soy	QC	500
100 ng/ml calibration std in solvent	CalStd100	Standard	500

Data: Provide the following data fields when reporting results

Index
Sample Name
Sample Type
Dilution Factor
Peak Name (Transition Name)
Peak Area
IS Peak Area
Retention Time (RT)
Actual Concentration (Spk level or Std conc)
Calculated concentration

Glyphosate Method Collaboration Report Attachment B

Analytical Method

A. Reagents and Supplies

1. Acetonitrile, HPLC grade
2. Petroleum ether
3. Methylene chloride
4. Water, HPLC grade
5. Formic acid, 98% solution
6. Acetic acid
7. Ammonium formate
8. Ethylenediaminetetraacetic acid disodium salt (Na_2EDTA)
9. Tetrabutylammonium hydroxide (TBAOH) titrant, 0.4 M in Water, HPLC Grade, ACROS Organics
10. Tetrabutylammonium acetate (TBuAA), Aldrich No. 335991-10G (optional)
11. Tetrabutylammonium acetate 1 M (TBuAA 1M), Aldrich No. 401803 – 50 ML (optional)
12. 50-mL plastic centrifuge tubes
13. Filter, 0.2 µm, 25 mm, nylon
14. Waters Oasis HLB SPE, 60 mg, 3cc, 30 µm
15. Extraction solvent (50 mM acetic acid/10 mM Na_2EDTA): mix 2.9 mL acetic acid and 3.7 g Na_2EDTA in 1000-mL of purified water.
16. 50 ng/ml IS fortified extraction solvent: dilute IS 20 µg/ml mixed isotope internal standard, prepared in step C.2.a, 1:400 using extraction solvent, prepared in step A.15, e.g. 2.5 ml (IS 20 µg/ml) to 1000 ml extraction solvent
17. Mobile phase A (4 mM tetrabutylammonium formate)
    a. Add 10.0 ml of 0.4 M TBAOH to ~900 mL HPLC water, and adjust the pH to 2.8±0.05 using formic acid (~ 3 ml). OR
    b. Add 1.20 g TBA acetate in 1 L HPLC water; and adjust the pH to 2.8±0.05 using formic acid (~2 mL). OR
    c. 4 ml 1M TBuAA in 1 L HPLC water; and adjust the pH to 2.8±0.05 using formic acid (~2 mL).

B. Standard Reference Materials

1. Glyphosate
2. Glufosinate
3. AMPA
4. N-acetyl-glyphosate, available from EPA and Toronto Research Chemicals (TRC No A178245)
5. Glyphosate-^{I3}C
6. Glufosinate-D^3

C. Standard Solutions

1. General instructions
    a. Unless otherwise indicated prepare standards in DI water
    b. Store standard solutions in plastic containers because glass can leach standard reference material from solution. Use of glass volumetric flasks for standard preparation is OK if solution is removed from the glassware after preparation.
    c. Store standard solutions in a refrigerator. Do not store standards prepared with water or aqueous media in the freezer.
2. Stock standards 1 mg/ml

a. Includes all native and isotopic standards listed in Section B
b. Prepare individual stock standard for each compound

3. Isotopic working solutions
   a. IS 20 µg/ml mixed isotope internal standard
      i) Combine isotopes Glyphosate-^{13}C and Glufosinate-D^3 (step B.5 & 6)
      ii) Dilute 1 mg/ml stock isotope internal standards, prepared in step C.2, 1:50
4. Intermediate mixed standards
   a. 50 µg/ml mixed native standard
      i) Combine native 1 mg/ml stock standards, prepared in step C.2
      ii) Include glyphosate, glufosinate, AMPA, and N-acetyl-glyphosate (Step B.1-4)
      iii) Dilute 1:20
   b. 5.0 µg/ml mixed native standard
      i) Dilute 50 µg/ml mixed standard, prepared in step C.4.a, 1:10
   c. 1.0 µg/ml mixed native standard
      i) Dilute50 µg/ml mixed standard, prepared in step C.4.a, 1:50
5. LC-MS/MS calibration standard 50 ng/ml
   a. Dilute 5.0 µg/ml mixed native standard, prepared in step C.4.b, 1:100, using 50 ng/ml IS fortified extraction solvent (A.16)

D. **Equipment and Instrumentation**

1. Genogrinder
2. Centrifuge
3. Pipettes
4. LC-MS/MS
   a. Shimadzu HPLC system: two LC-20AD pumps, Sil-20AC autosampler, CTO-20AC column oven
      NOTE: Replace all metal LC tubing with PEEK tubing between the autosampler and injection valve because glyphosate can be retained on metal surfaces.
   b. AB model 5500, or 6500, Q-TRAP mass spectrometer
   c. HPLC columns: Phenomenex Luna C8(2), 100 Å, 5 µm, 150 x 4.6 mm, Phenomenex No. 00F-4249-E0; Or Phenomenex Luna C8, 100 Å, 5 µm, 150 x 2 mm, Phenomenex No. 00F-4040-B0
   d. HPLC guard column: Phenomenex guard column KrudKatcher P/N AFO-8497

      NOTE: Install peek tubing between the autosampler and column because metal can affect glyphosate and glufosinate chromatography

E. **Extraction Procedure**

1. 5 g sample + 25 ml 50 ng/ml IS fortified extraction solvent prepared in step A.15
   For dry products containing less than 50 % moisture: 2 g sample plus 10 ml 50 ng/ml IS fortified extraction solvent prepared in step A.15 for dry products
2. Add 10 ml PE, or $MeCl_2$, for matrices containing more than 3 % fat.
3. Shake @ 1000 shakes per min for 10 min
4. Centrifuge at ≥ 3000 rpm for 5 min NOTE: When using PE to remove lipid co-extractants high fat matrices, the PE will be the top layer. When using $MeCl_2$, the $MeCl_2$ will be the bottom layer in the centrifuge tube.

Analytical Method

5. Filter aqueous extract thru HLB SPE cartridge, limit filter volume to less than 2 mls.
6. Filter for injection (could be included with SPE step)
7. Sample concentration: 0.2 g/ml

F. **LC-MS/MS method**

	LC Parameters	Gradient	
Column:	Phenomenex Luna C8(2), 150 x 4.6 mm, 5 µm OR Phenomenex Luna C8, 150 x 2 mm, 5 µm Guard Column: Phenomenex KrudKatcher	**Time**	**MPB**
MP A:	4 mM tetrabutlyammonium formate + 0.1 % formic acid in water (pH 2.8±0.05)	0.00	5
MP B:	MeCN	1.00	5
Flow:	0.45 mL/min (4.6 mm column)	5.00	90
	0.3 mL/min (2.0 mm column)	7.00	90
Inj Vol:	10 µL	8.00	5
Temp	40 °C	14.00	5
Divert Valve	Divert flow from mass spectrometer about 30 seconds before the first analyte and 60 seconds after the last analyte elutes		

MS/MS Parameters (5500 & 6500)

Q1	Q3	RT	Transition	DP*	EP	CE	CXP
110	63	1.3	AMPA 1	-40	-11	-30	-9
110	79	1.3	AMPA 2	-40	-11	-34	-9
110	81	1.3	AMPA 3	-40	-11	-34	-9
112	63	2.5	AMPA IS	-60	-11	-26	-9
180	63	3.0	Glufosinate 1	-60	-11	-66	-9
180	95	3.0	Glufosinate 2	-60	-11	-19	-5
180	85	3.0	Glufosinate 3	-60	-11	-25	-9
183	63	3.0	Glufosinate IS	-60	-11	-40	-9
168	63	4.4	Glyphosate 1	-30	-11	-28	-9
168	79	4.4	Glyphosate 2	-30	-11	-56	-9
168	150	4.4	Glyphosate 3	-30	-11	-16	-9
171	63	4.4	Glyphosate IS	-30	-11	-28	-9
210	150	5.3	N-acetyl glyphosate 1	-20 (-40)	-11	-20	-13
210	63	5.3	N-acetyl glyphosate 2	-20 (-40)	-11	-40	-13
210	168	5.3	N-acetyl glyphosate 3	-20 (-40)	-11	-18	-13

**DP: if more than one DP is provided the first is optimized for the 6500 and the DP in () is optimized for the 5500*

Analytical Method

MS Parameters

Ionization:	Ionspray in negative ionization mode		
CUR:	35	TEM:	450 °C (6500)
CAD:	medium		650 °C (5500)
IS:	-4000	Q1:	unit
GAS 1 & 2:	65	Q3:	unit

G. **Quantitation of Residues**

1. Calibrate instrument using single level calibration standard at 50 ng/ml
2. Calibrate using internal standard calibration for glyphosate, glufosinate and AMPA
   a. Assign internal standard calibration standards
      i) Glyphosate: Glyphosate-^{13}C
      ii) Glufosinate: Glufosinate-D_3
      iii) AMPA: Glyphosate-^{13}C
3. Calibrate using external calibration for N-acetylglyphosate
4. Reportable residues must meet the identification criteria provided in Appendix A "Identification of Residues" in ORA-LAB.10
5. Quantitate residues per instructions in Appendix B "Quantitation of Residues" in ORA-LAB.10. Give preference to quantitation using the primary MS/MS transition, e.g. "Glyphosate 1", however, use of secondary transitions for quantitation may be advisable if/when matrix coextractants interfere with the primary transition response.

Single Laboratory Validation

The PSW laboratory conducted single laboratory validation (SLV) for the procedure "Analysis of Glyphosate in Food by HPLC-MS/MS" (Att. B). Standards were prepared as per glyphosate procedure (Att. B) at 1, 2, 10, 50, 100, 200, 250, 350, 400 and 500 ng/ml in extraction solvent fortified at 50 ng/ml with isotopic internal standards. The matrices studied were the collaboration samples of corn, carrot and avocado. Recovery studies were conducted using the calibration protocols and analysis sequences prescribed in the collaboration protocol (Att. A). Each matrix was analyzed as an unfortified control and fortified in duplicate at three different levels: 50, 250, and 500 ng/g; i.e. six analyses per matrix, 21 analyses altogether. For the MDL study each of the three matrices was fortified at 20 ng/ml and seven replicates were analyzed per the instructions of 40 CFR 136 Appendix B.

Prior to starting the collaboration, instrument system suitability (SS) was demonstrated. Standards were injected at concentrations of 10, 50, 100, 200, 350, and 500 ng/ml to determine accuracy and linearity. Five replicates of the 50 ng/ml standard were injected to determine precision. The instrument LOQ was determined as per ORA-LAB.10 by injecting a 2 ng/ml standard in solvent and determining the S/N of the quantifier and qualifier ions. The LOQ was calculated as the lowest level where the S/N of the quantifier ion ≥ 10 and the S/N of the qualifier ion ≥ 3. Results for the instrument system suitability study are listed in the table below.

SS Factor	Glyphosate		Glufosinate		AMPA		N-acetylglyphosate	
LOQ (ng/mL)	0.3		0.3		0.5		0.2	
Precision (RSD)	99.1	(1.4)	99.8	(2.3)	97.7	(2.1)	102.3	(1.2)
Accuracy (R^2)	100.4	(0.9997)	104.4	(0.9996)	96.1	(0.9998)	96.6	(0.9998)

For the recovery study the average recovery, RSD, method uncertainty (MU), and the coefficient of determination (R^2) for all levels was determined for each matrix and overall. MU at the 95 % confidence level was calculated as 2 * the RSD as prescribed in ORA-LAB.5.4.6. Linearity (R^2) was calculated by squaring the Excel correlation function (Correl) of the spike level and calculated concentrations of the spiked samples. The method LOQ was determined by multiplying the standard deviation of the concentrations of seven replicate 20 ng/ml spikes per matrix by 10. For the overall method LOQ the standard deviation was calculated by adding the variances and degrees of freedom of the individual matrix concentrations taking the square root. Specificity was determined by the analysis of the control samples. Acceptable method validation specifications for each method performance metric are listed below.

Recovery: 70-120 % RSD: 15% MU: 30%
R^2: 0.990 LOQ: ≤ 10 ng/g

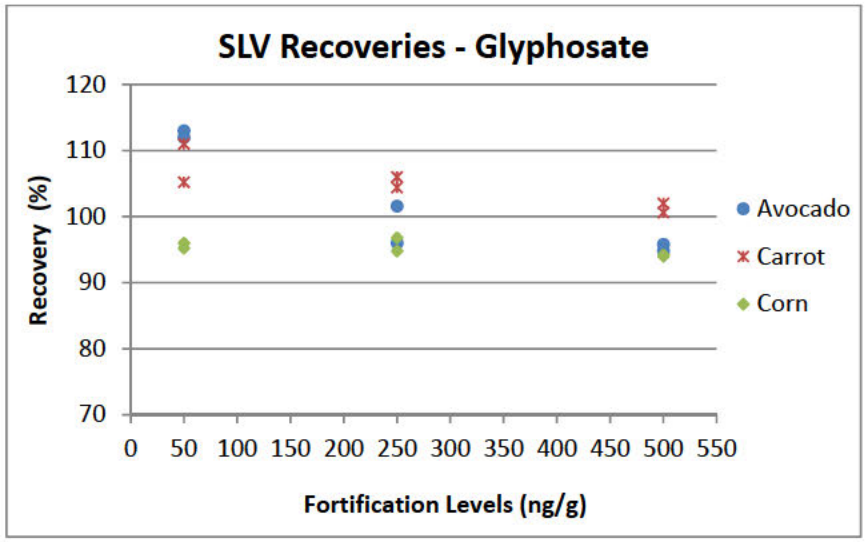
Results of the SLV are summarized in the Table C1 below; results that were not within validation specifications are indicated in red font. Scatter plots of recoveries and linearity charts for each analyte are provided in attachments C_1 and C_2. Results for both of the pesticides, glyphosate and glufosinate met all validation performance specifications and results for the glyphosate degradant N-acetylglyphosate met all specifications with the exception of the R^2 of

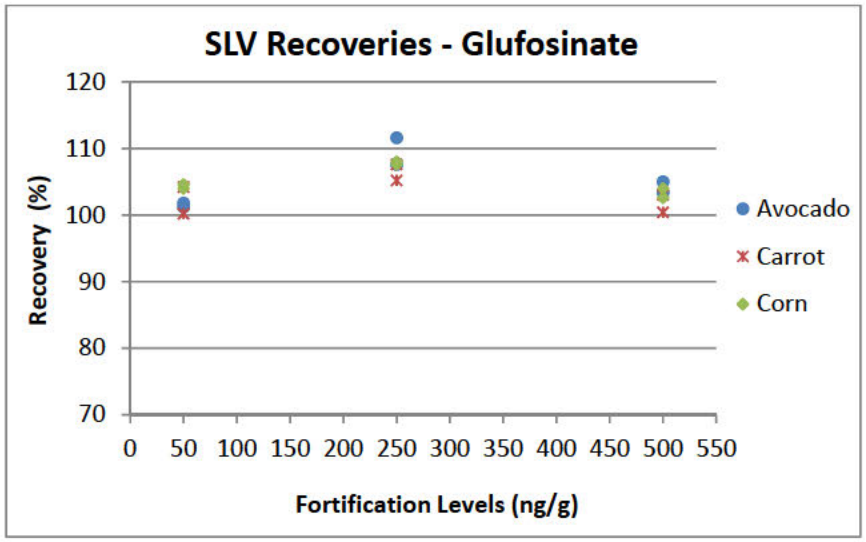
0.9871 for avocado recoveries was just below the specification of 0.99. Recoveries of the glyphosate degradant AMPA were very low, averaging 19.8 %, however it did meet most of the other specifications. AMPA will be considered qualitative and will not be reported for routine analyses

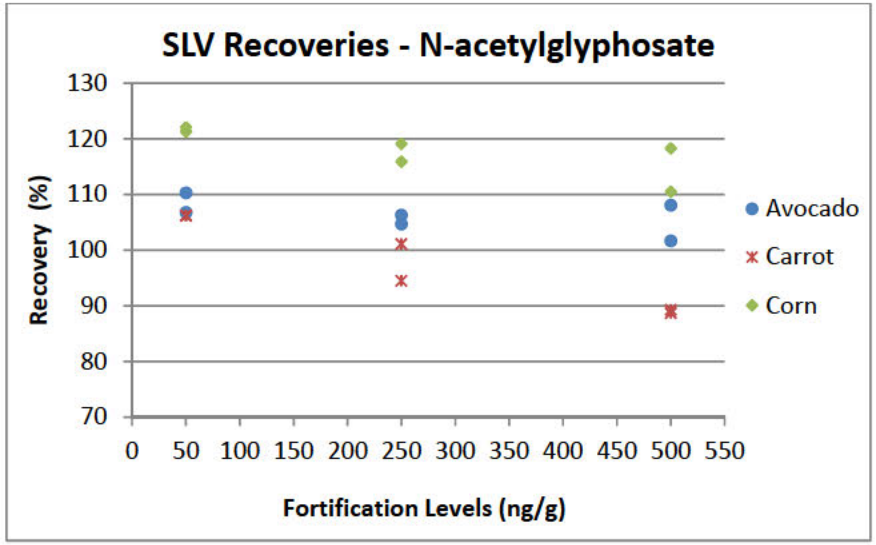
Table 1. Summary data includes the average, RSD, method uncertainty (MU) and coefficient of determination (R^2) from the recovery study and method limit of quantitation (LOQ) from the LOQ study.

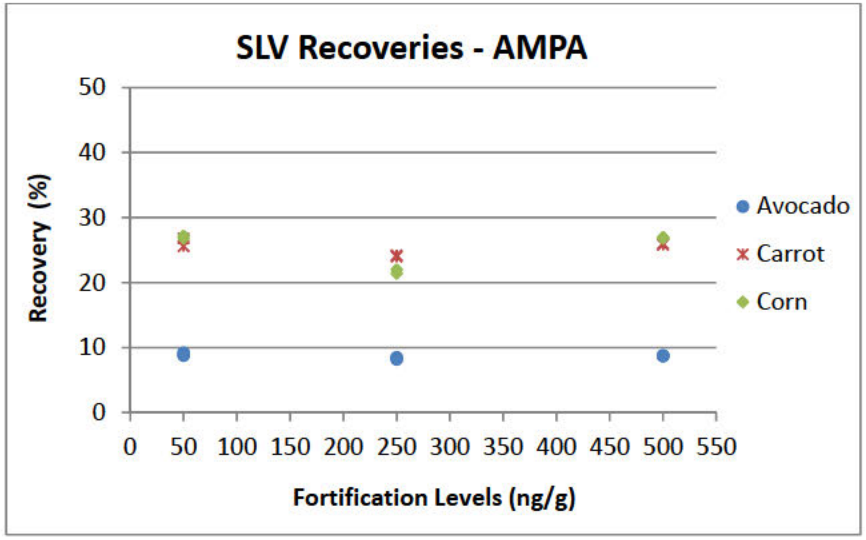
	Recoveries				Limits
Matrix	**Average**	**RSD**	**MU**	**R^2**	**LOQ**
Glyphosate			(b) (5)		
Avocado	102.2	8.2		0.9993	3.5
Carrot	104.9	3.5		0.9994	7.5
Corn	95.2	1.1		0.9998	5.2
Overall	100.7	6.5		0.9957	5.7
Glufosinate			(b) (5)		
Avocado	105.1	3.7		0.9984	7.4
Carrot	103.4	2.8		0.9986	8.8
Corn	105.1	2.1		0.9991	10
Overall	104.6	2.9		0.9984	8.8
N-acetylglyphosate	(b) (5)	(b) (5)	(b) (5)	(b) (5)	
Avocado					8.4
Carrot					4.4
Corn					7.6
Overall					7.0
AMPA	(b) (5)		(b) (5)		
Avocado		3.8		0.9986	6.1
Carrot		4.3		0.9978	9.9
Corn		10.8		(b) (5)	3.9
Overall		(b) (5)		(b) (5)	7.1

Single Laboratory Validation – Method Recovery Charts

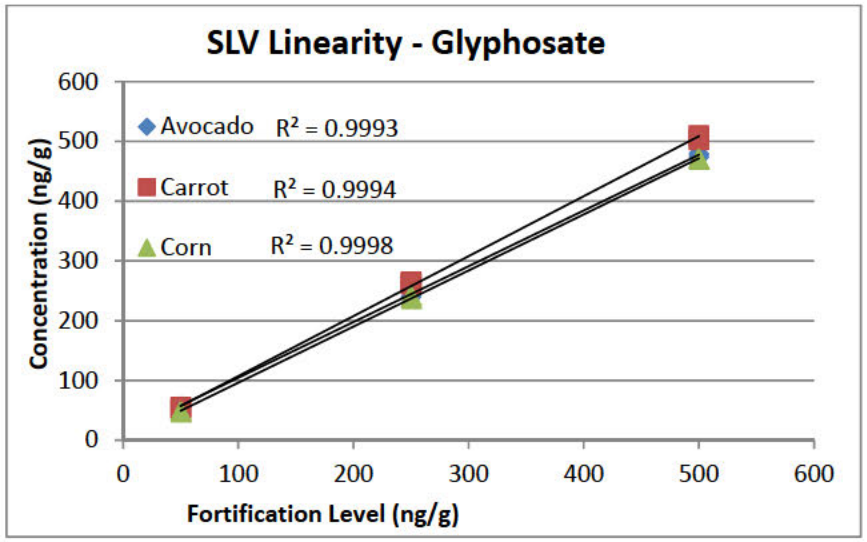


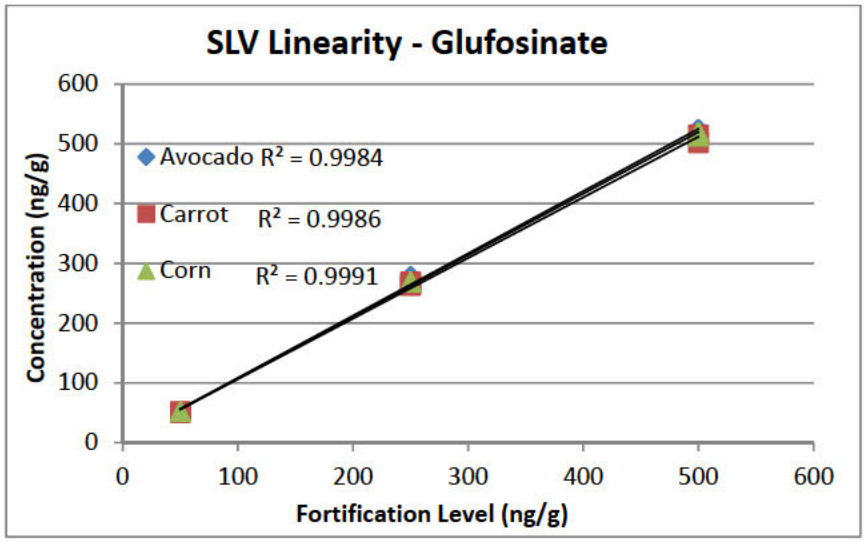


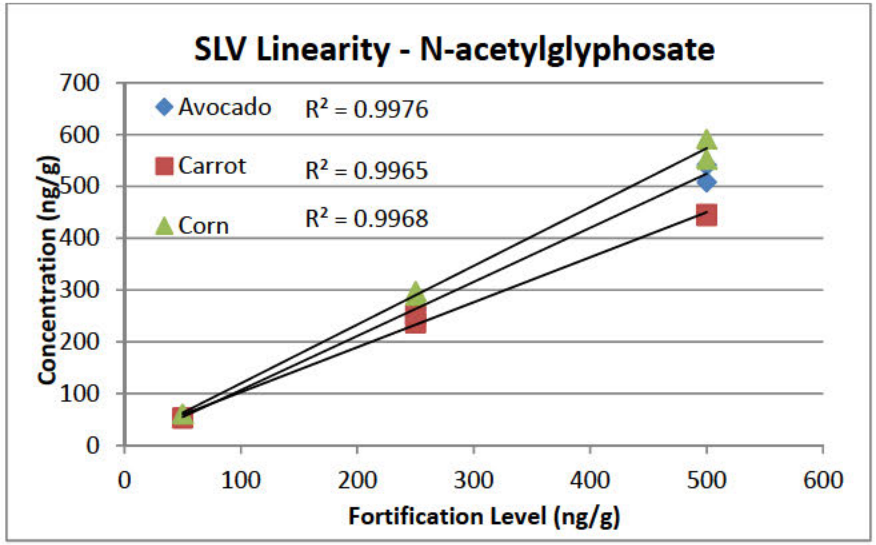


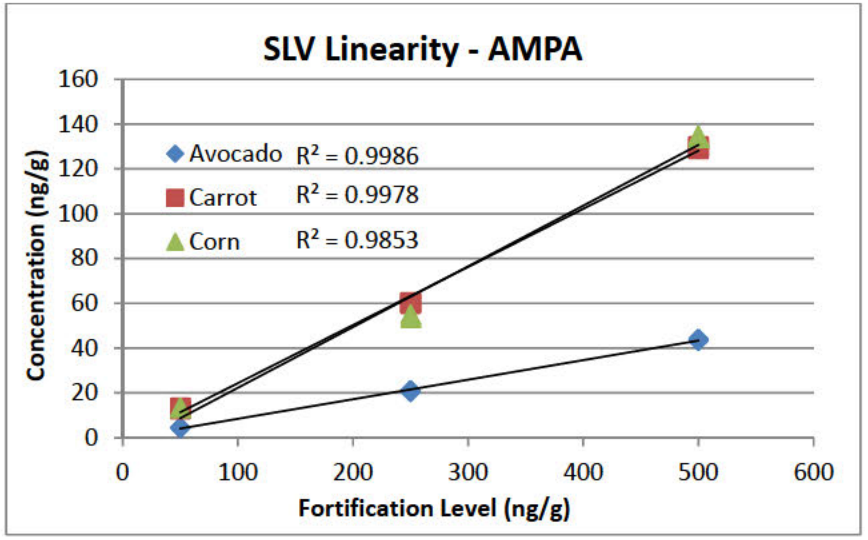


Single Laboratory Validation – Method Linearity Charts

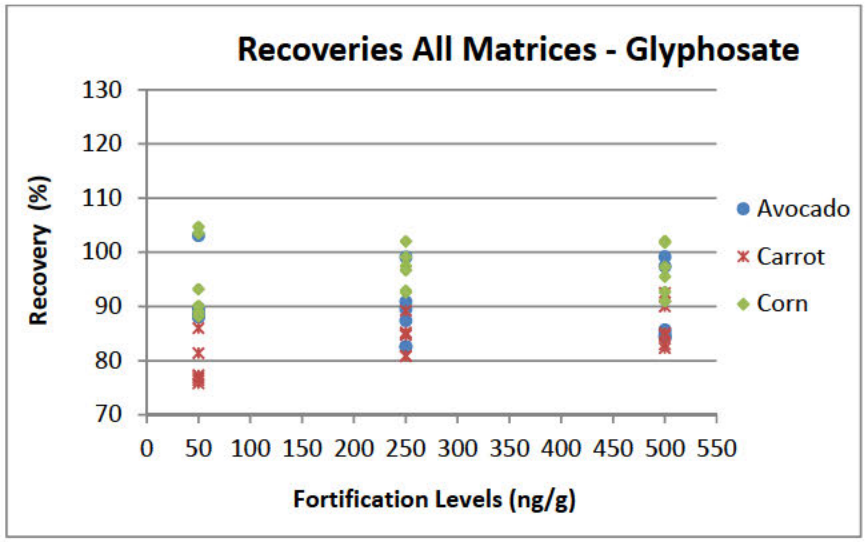


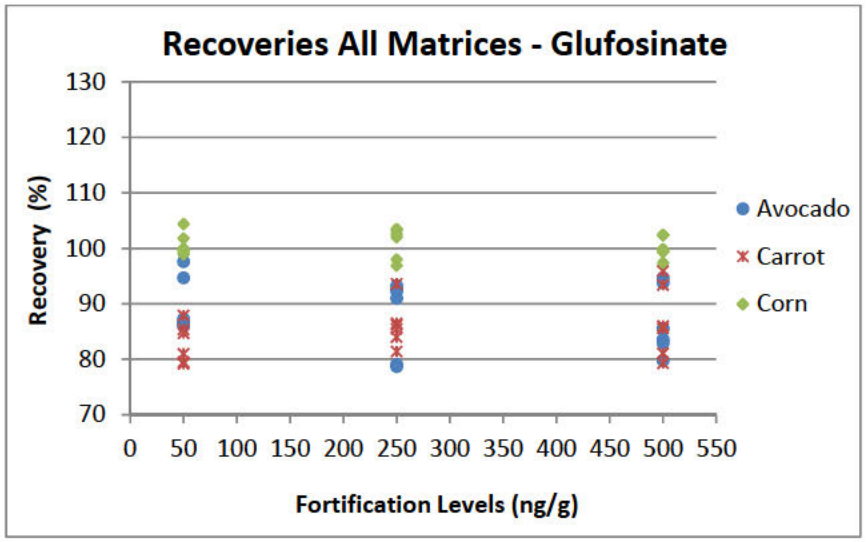


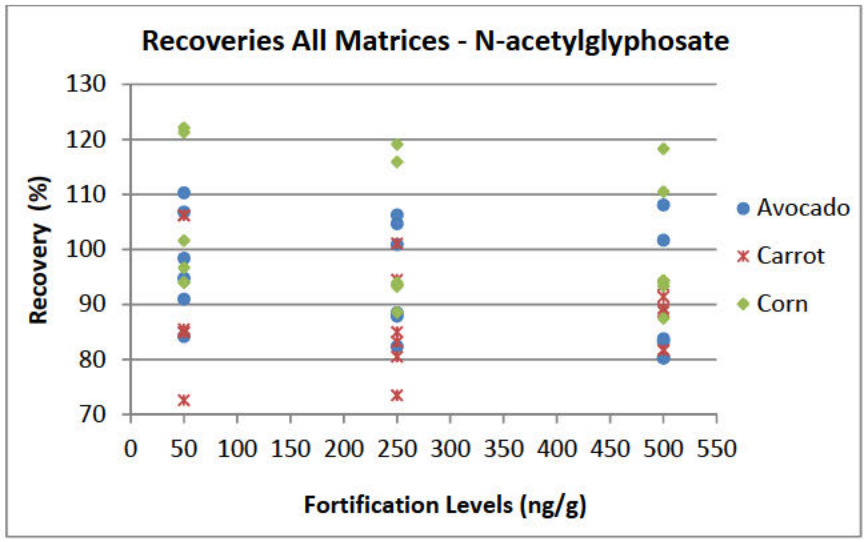


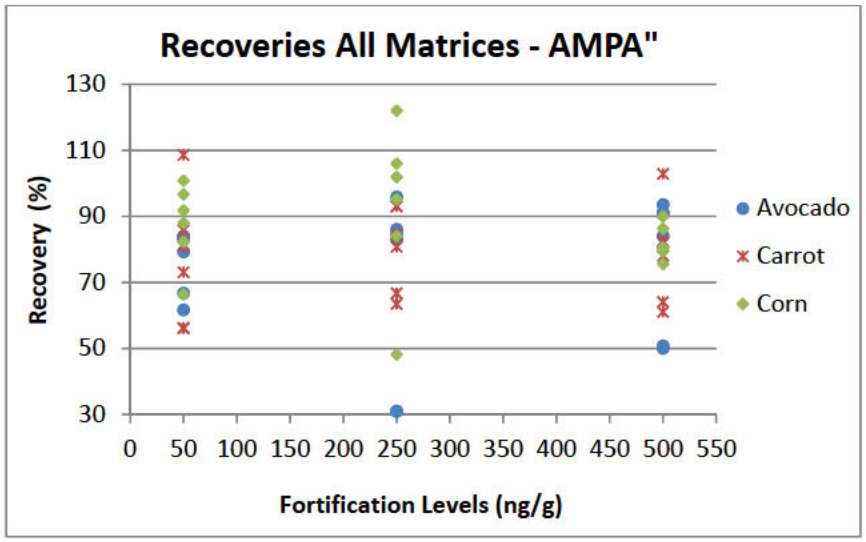


Method Collaboration Recovery Charts

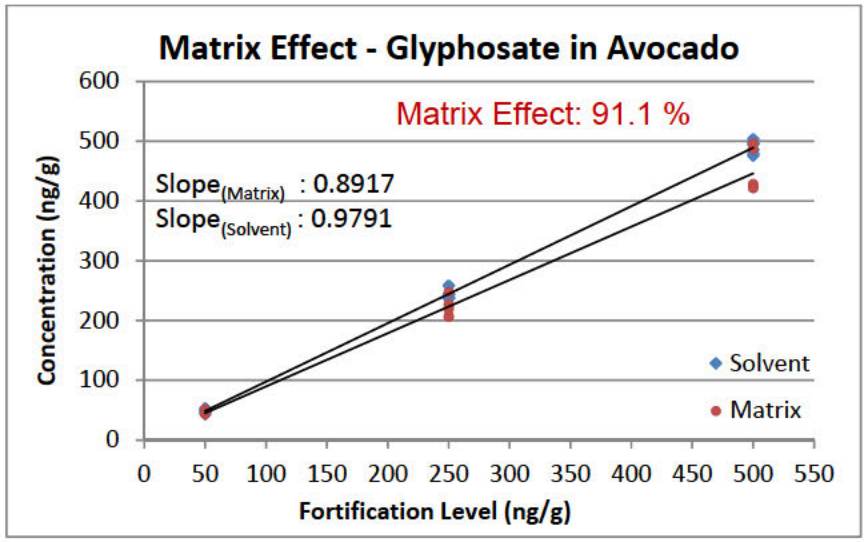


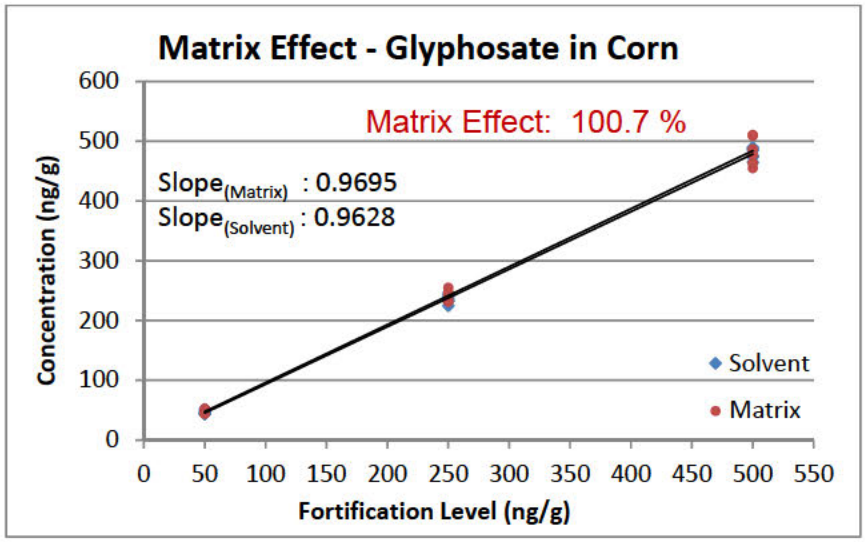


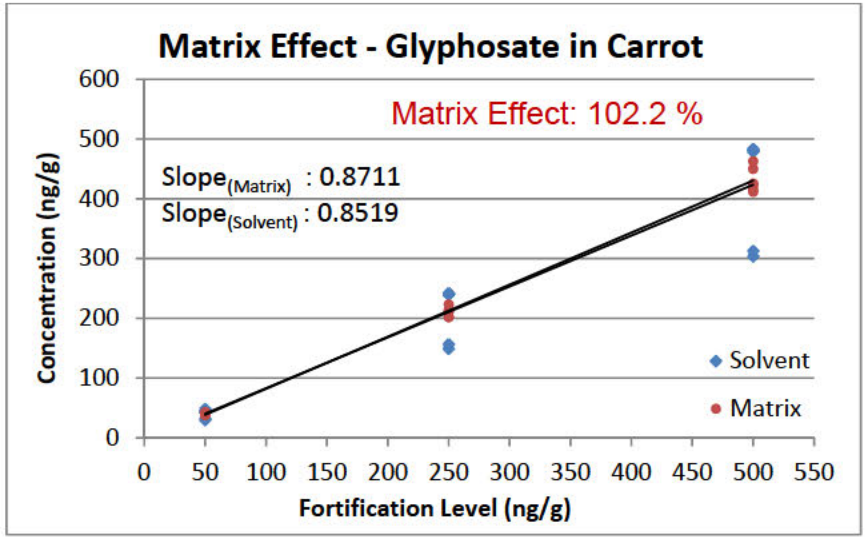


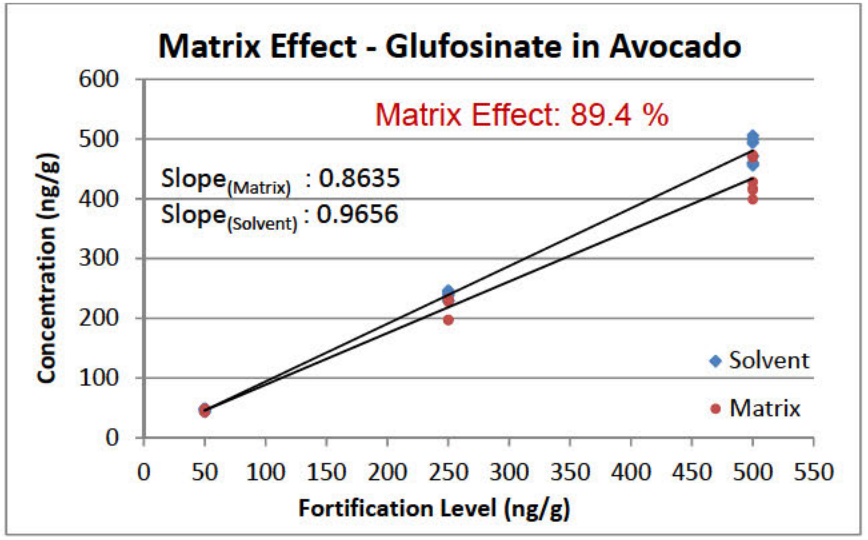


Method Collaboration Matrix Effects Charts

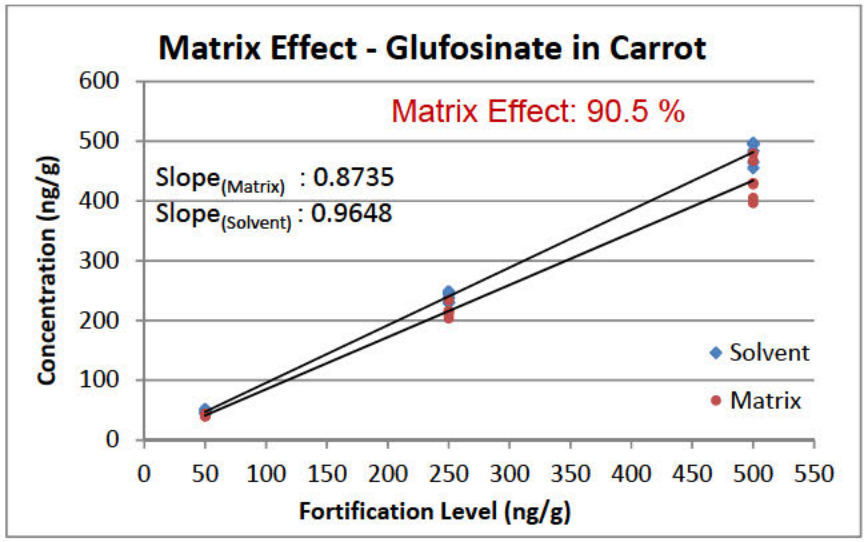


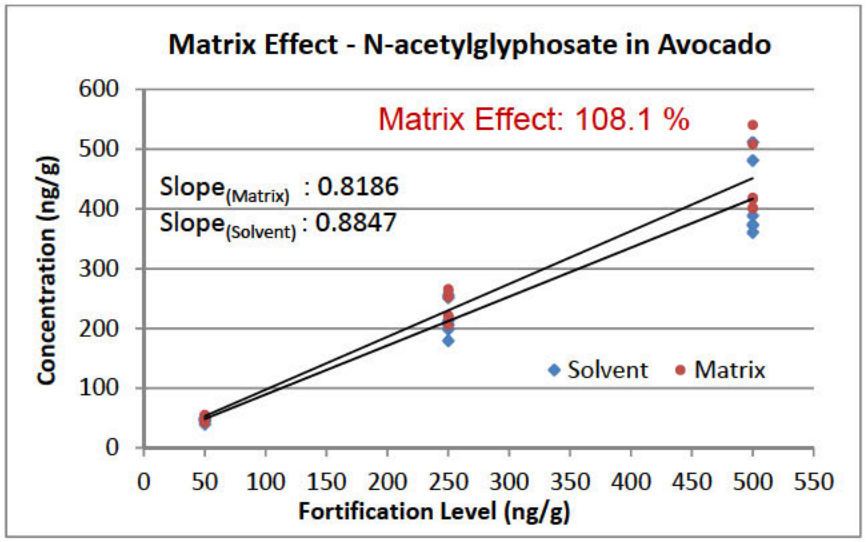


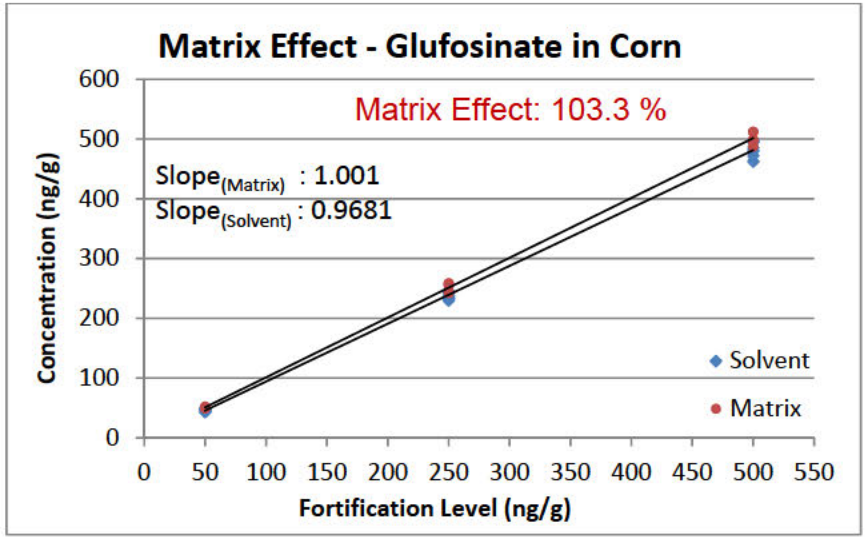


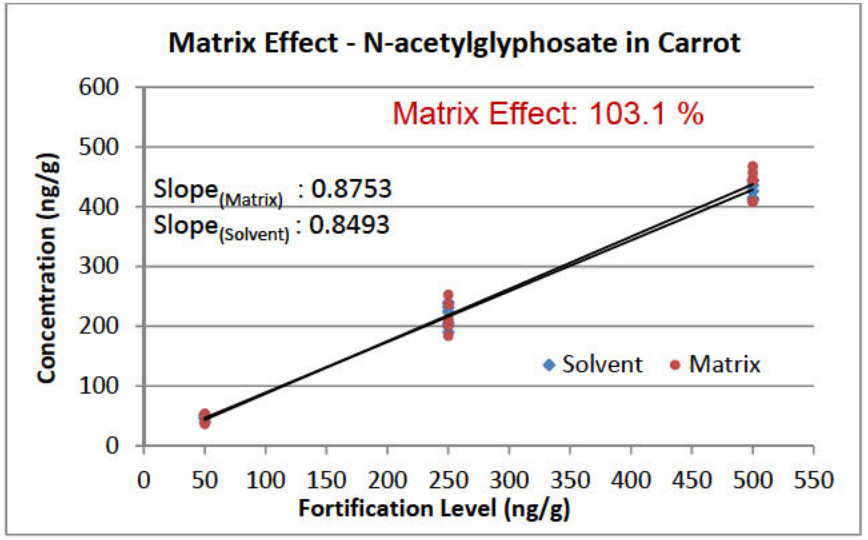


Method Collaboration Matrix Effects Charts

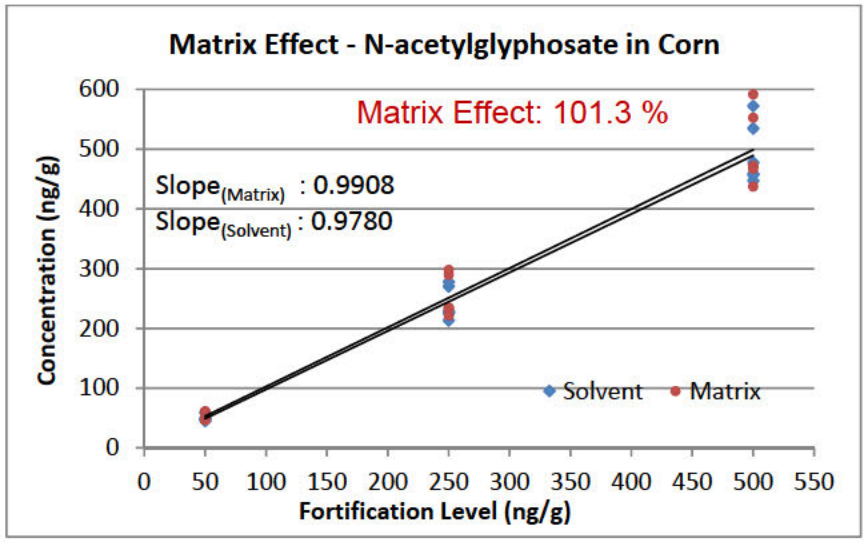


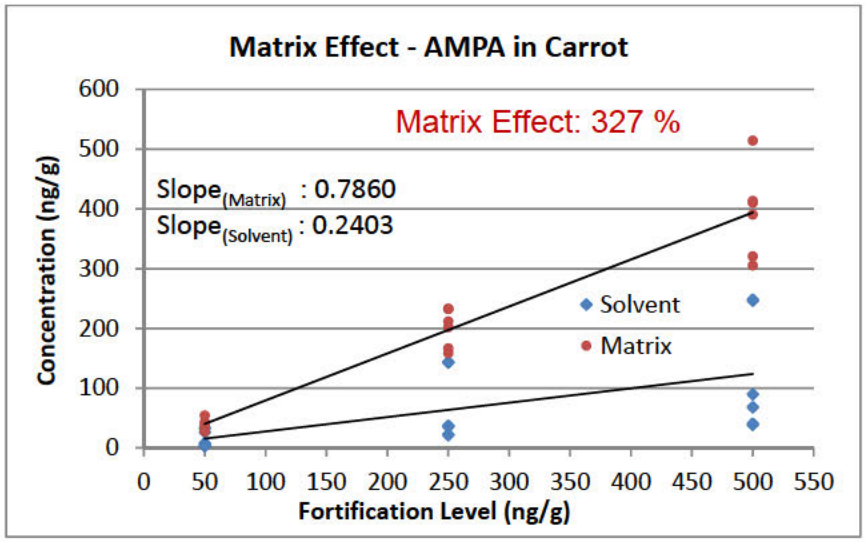


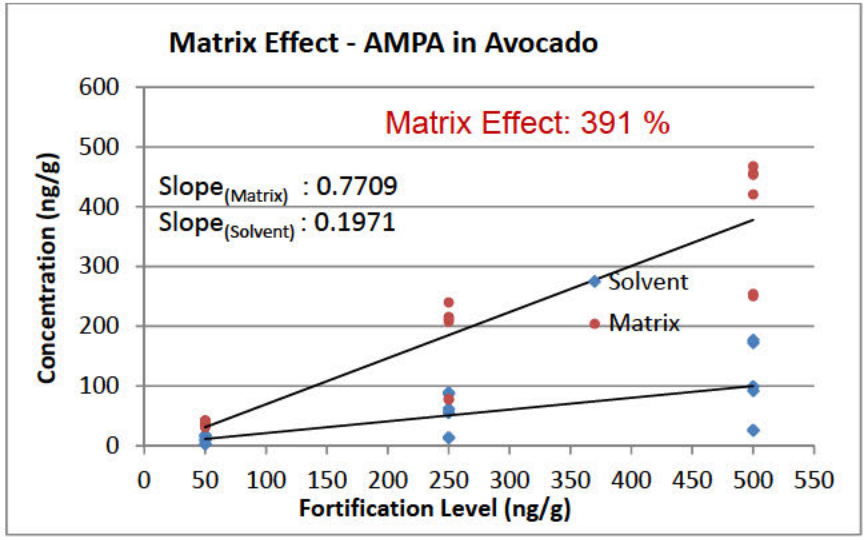


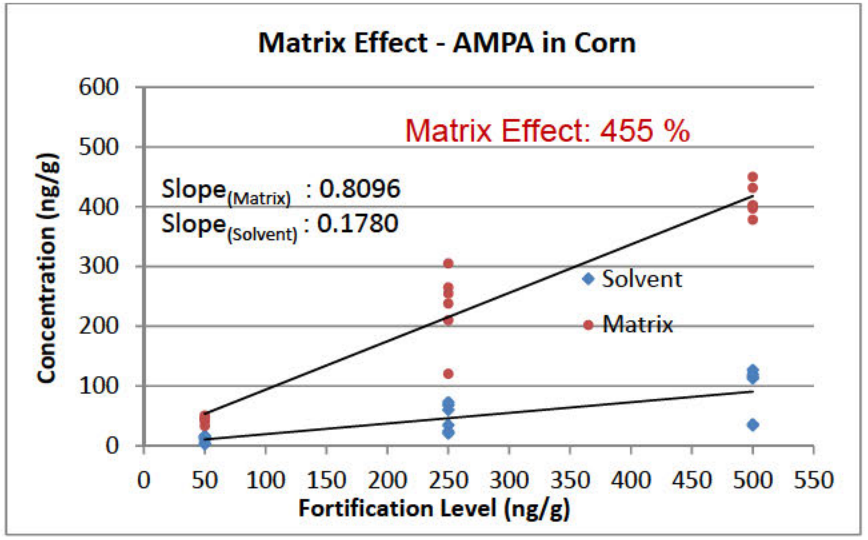


Method Collaboration Matrix Effects Charts









PSW Collaboration Data and System Suitability

All data and derived statistics in this attachment are from the method collaboration analyses conducted at PSW only. Results of the instrument system suitability and method collaboration verify that PSW is able to the method proficiently.

Instrument System Suitability

Prior to starting the collaboration instrument system suitability (SS) was demonstrated. Standards were injected at concentrations of 10, 50, 100, 200, 350, and 500 ng/ml to determine accuracy and linearity. Five replicates of the 50 ng/ml standard were injected to determine precision. The instrument LOQ was determined as per ORA-LAB.10 by injecting a 2 ng/ml standard and determining the S/N of the quantifier and qualifier ions. The LOQ was calculated as the lowest level where the S/N of the quantifier ion ≥ 10 and the S/N of the qualifier ion ≥ 3. Results for the instrument system suitability study are listed in the table below. Criteria for instrument system suitability are tabulated below.

LOQ (ng/ml)	Precision (RSD)	Accuracy (%)	Linearity (R^2)
≤ 2	≤ 10	90 - 110	0.995

Results for the instrument system suitability study, listed in the table below, are all within acceptable criteria.

SS Factor	Glyphosate		Glufosinate		AMPA		N-acetylglyphosate	
LOQ (ng/mL)	0.3		0.3		0.5		0.2	
Precision (RSD)	99.1	(1.4)	99.8	(2.3)	97.7	(2.1)	102.3	(1.2)
Accuracy (R^2)	100.4	(0.9997)	104.4	(0.9996)	96.1	(0.9998)	96.6	(0.9998)

Method Collaboration

The method and collaboration protocol are described in attachments A and B, respectively. The mean, RSD, method uncertainty (MU) of the recoveries for all three spike levels (50, 250, and 500 ng/g) were determined by matrix and overall. The linearity coefficient of determination (R^2) was calculated from the concentrations found at each level for each matrix by squaring the Excel correlation function (Correl). Statistics for all matrices were calculated from the whole set of data without correction for matrix bias. Acceptable method validation specifications for the collaboration study are listed below.

Recovery: 70-120 % RSD: 15% MU: 30% R^2: 0.990

Method collaboration results contributed by PSW are summarized in the Table F1 below; results that did not meet specifications are highlighted in red font. Scatter plots of the recoveries and linearity charts are provided in attachments F_1 and F_2, respectively. All results were within the validation specifications, with the exception of the R^2 for AMPA in corn of 0.9721 was just below the 0.99 specification.

PSW Collaboration Data and System Suitability

Table F1. Summary data includes the mean, RSD, method uncertainty (MU) of spike recoveries and coefficient of determination (R^2) of the three spike levels for each matrix.

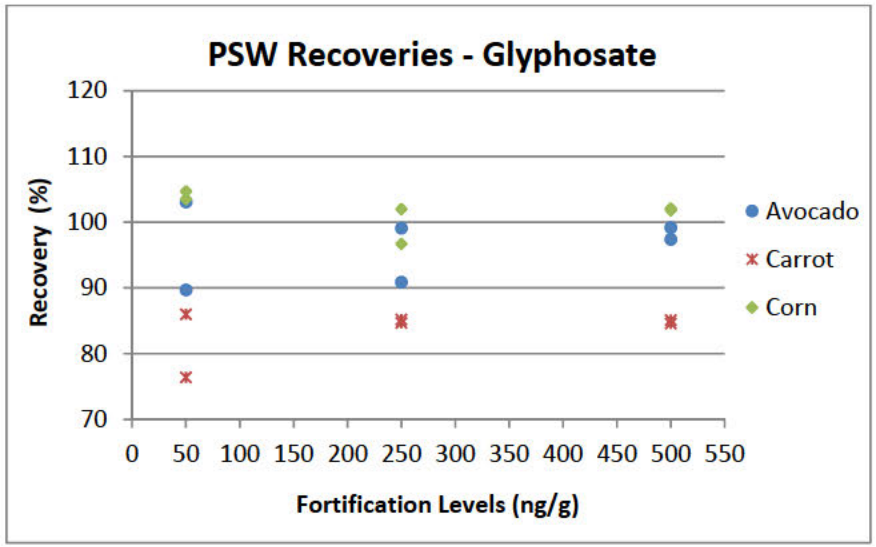
Matrix	N	Mean	RSD	MU	R^2
Glyphosate				(b) (5)	
Avocado	6	96.6	5.4		0.9982
Carrot	6	83.7	4.3		0.9999
Corn	6	101.8	2.7		0.9993
Glufosinate				(b) (5)	
Avocado	6	94.4	1.8		0.9998
Carrot	6	84.6	3.0		0.9999
Corn	6	102.0	1.9		0.9995
N-acetylglyphosate		(b) (5)	(b) (5)	(b) (5)	
Avocado	6				0.9976
Carrot	6				0.9965
Corn	6				0.9968
AMPA				(b) (5)	
Avocado	6	85.9	6.3		0.9971
Carrot	6	90.9	10.9		0.9943
Corn	6	90.3	11.2		(b) (5)

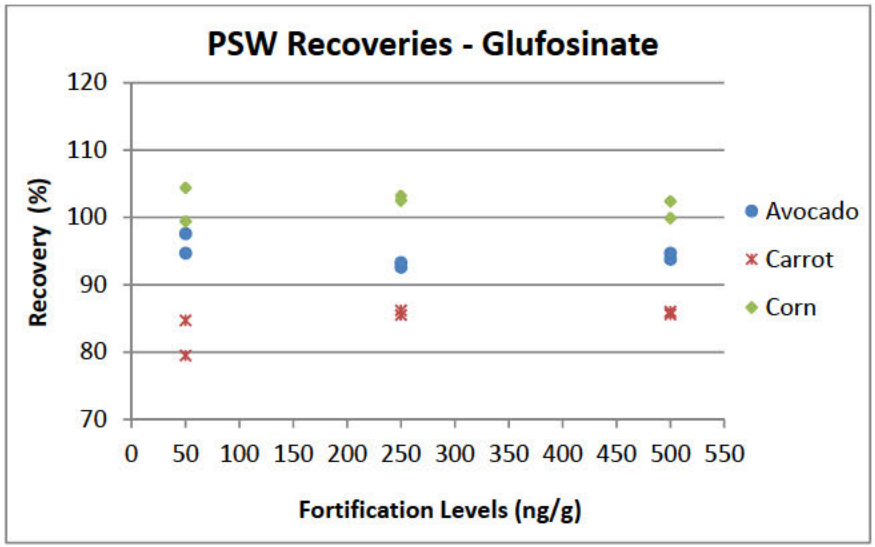
Analysis of Incurred Residues

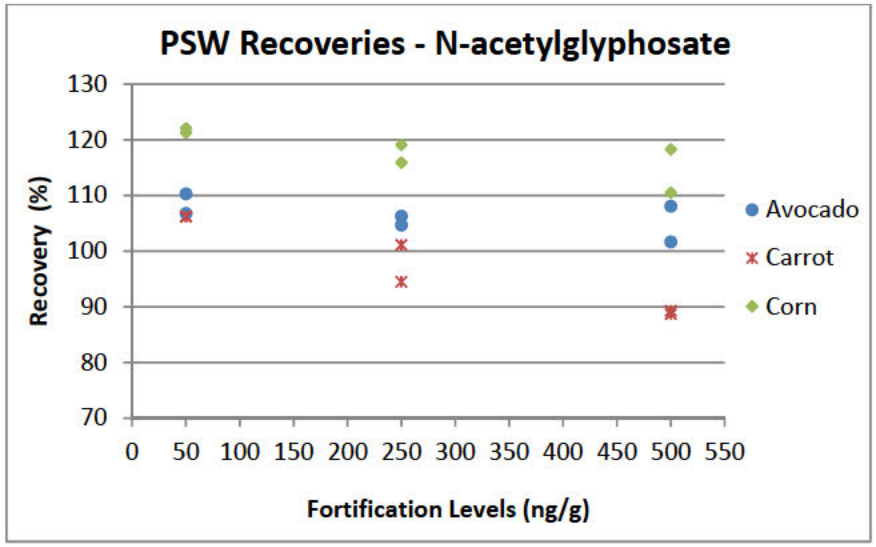
Results of the analysis of corn and soy containing incurred glyphosate residues are tabulated below. PSW findings are consistent with the range of residues levels reported from four different laboratories.

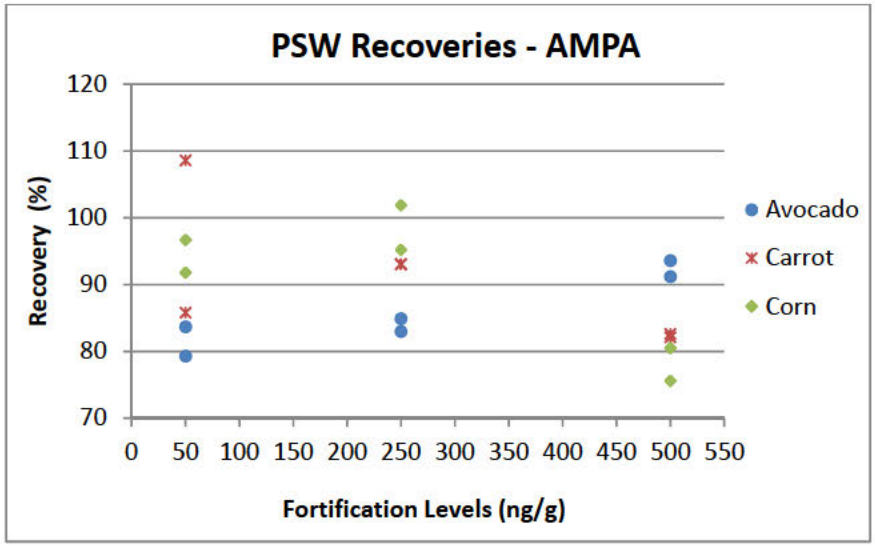
Matrix	Range	PSW
Corn	35-46	46
Soy	4290-4620	4620

PSW Collaboration Data and System Suitability – Recovery Charts

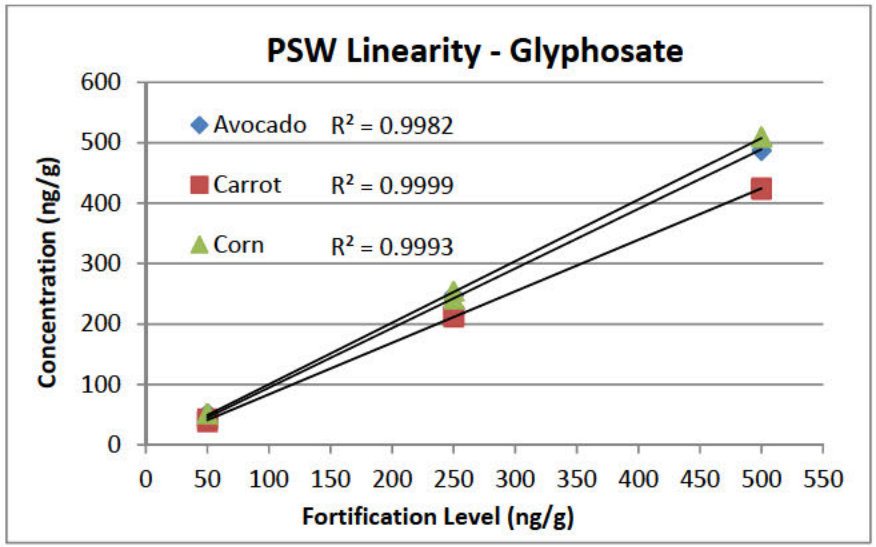


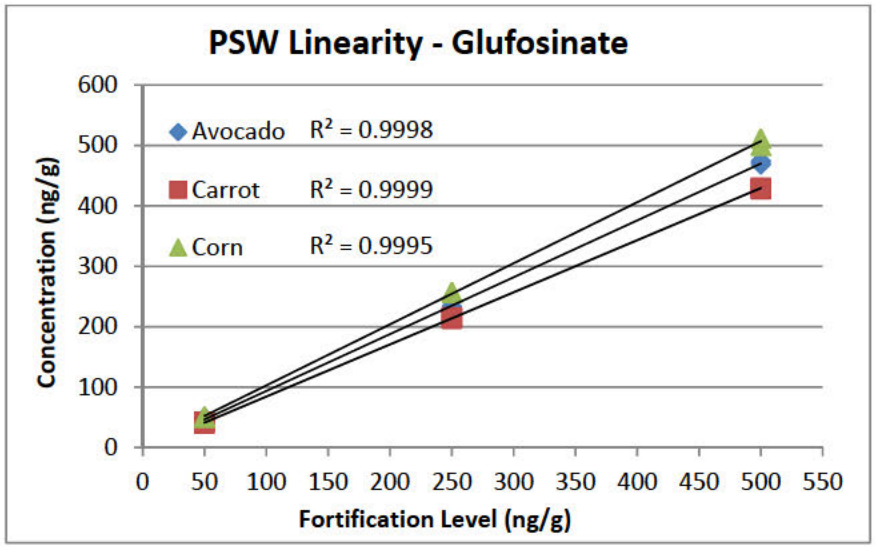


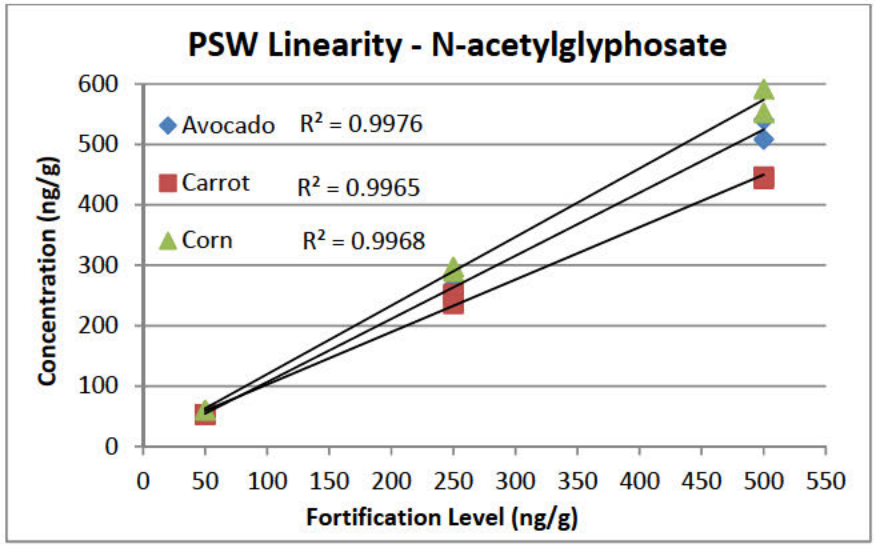


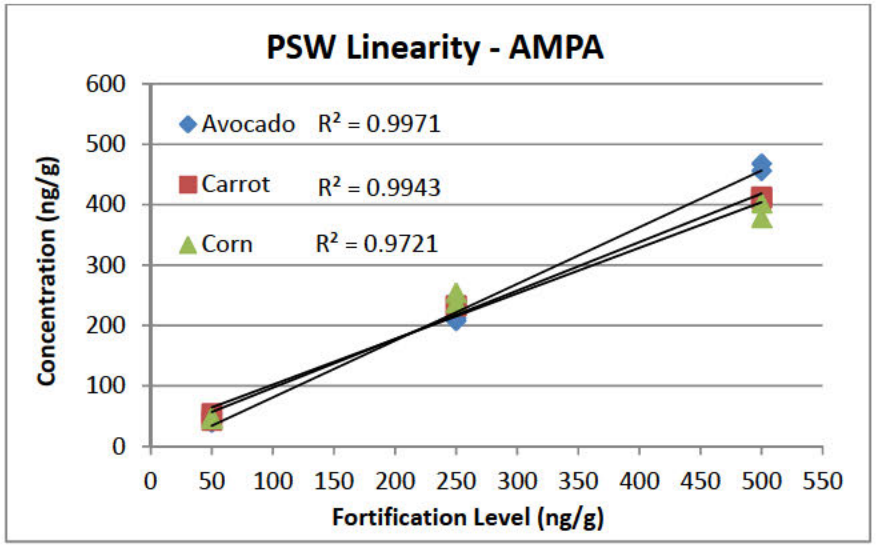


PSW Collaboration Data and System Suitability – Linearity Charts









PNW Collaboration Data and System Suitability

All data and derived statistics in this attachment are from the method collaboration analyses conducted at PNW only. Results of the instrument system suitability and method collaboration verify that PNW is able to the method proficiently.

Instrument System Suitability

Prior to starting the collaboration instrument system suitability (SS) was demonstrated. Standards were prepared and injected at concentrations of 1, 2, 5 10, 25, 50, 100, 200, 500, and 1000 ng/ml to determine accuracy and linearity; the standards at concentrations of 500 and 1000 ng/ml were not included in the accuracy and linearity calculations. Seven replicates of the 50 ng/ml standard were injected to determine precision. The instrument LOQ was determined as per ORA-LAB.10 by injecting standards at concentrations of 1, 2, 5, and 50 ng/ml and determining the S/N of the quantifier and qualifier ions. The LOQ was calculated as the lowest level where the S/N of the quantifier ion ≥ 10 and the S/N of the qualifier ion ≥ 3. Criteria for instrument system suitability are tabulated below.

LOQ (ng/ml)	Precision (RSD)	Accuracy (%)	Linearity (R^2)
≤ 2	≤ 10	90 - 110	0.995

Results for the instrument system suitability study, listed in the table below, are all within acceptable criteria with the exception of the LOQ for N-acetylglphosate at 6 ng/ml exceeded the maximum acceptable level of 2 ng/ml.

SS Factor	Glyphosate		Glufosinate		AMPA		N-acetylglyphosate	
LOQ (ng/mL)	0.4		1.4		2		6	
Precision (RSD)	98.4	(2.8)	96.2	(0.7)	96.4	(3.3)	97.2	(6.7)
Accuracy (R^2)	101	(0.9998)	99.4	(0.9999)	98.9	(0.9999)	101.1	(0.9998)

Method Collaboration

The method and collaboration protocol are described in attachments A and B, respectively. The mean, RSD, method uncertainty (MU) of the recoveries for all three spike levels (50, 250, and 500 ng/g) were determined by matrix and overall. The linearity coefficient of determination (R^2) was calculated from the concentrations found at each level for each matrix by squaring the Excel correlation function (Correl). Statistics for all matrices were calculated from the whole set of data without correction for matrix bias. Acceptable method validation specifications for the collaboration study are listed below.

Recovery: 70-120 % RSD: 15% MU: 30% R^2: 0.990

Method collaboration results contributed by PNW are summarized in the Table G1 below; results that did not meet specifications are highlighted in red font. Scatter plots of recoveries and

linearity charts for each analyte are provided in attachments G_1 and G_2. All results were within the validation specifications, with the exception of the R^2 of 0.9871 for N-acetylglyphosate, the R^2 of 0.9556 and 0.9571 for AMPA in carrot and corn, respectively, were just below the 0.99 specification. The precision and MU for AMPA in corn, 23.2 and 46.4 % also did not meet specifications of 15 and 30 %, respectively.

Table G1. Summary data includes the mean, RSD, method uncertainty (MU) of spike recoveries and coefficient of determination (R^2) of the three spike levels for each matrix.

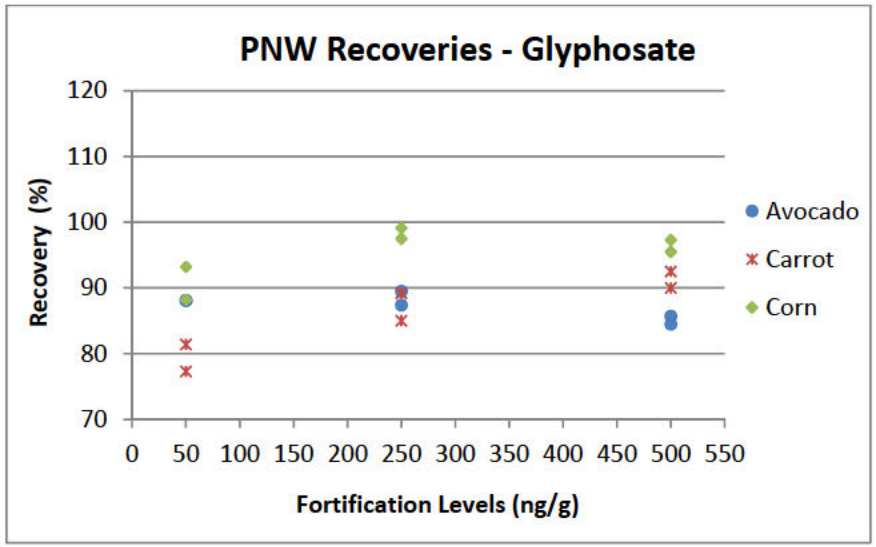
Matrix	N	Mean	RSD	MU	R^2
Glyphosate					
Avocado	6	87.2	2.1	(b) (5)	0.9992
Carrot	6	85.9	6.7		0.9988
Corn	6	95.1	4.2		0.9994
Glufosinate					
Avocado	6	87.0	5.1		0.9925
Carrot	6	90.4	4.8		0.9981
Corn	6	101.4	1.6		0.9993
N-acetylglyphosate					
Avocado	6	90.3	9.0		(b) (5)
Carrot	6	86.7	5.5		0.9957
Corn	6	94.4	1.3		1.0000
AMPA					
Avocado	6	87.3	5.7		0.9938
Carrot	6	83.4	12.3		(b) (5)
Corn	6	76.5	(b) (5)		(b) (5)

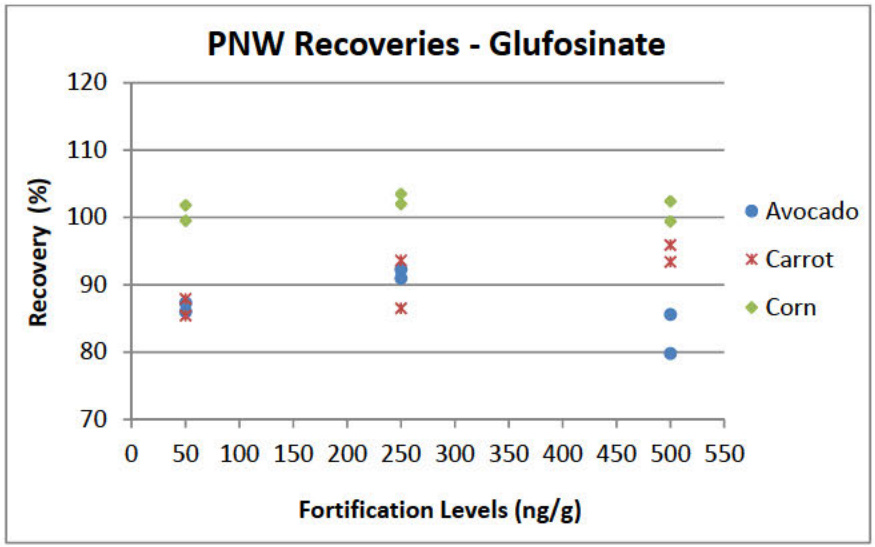
Analysis of Incurred Residues

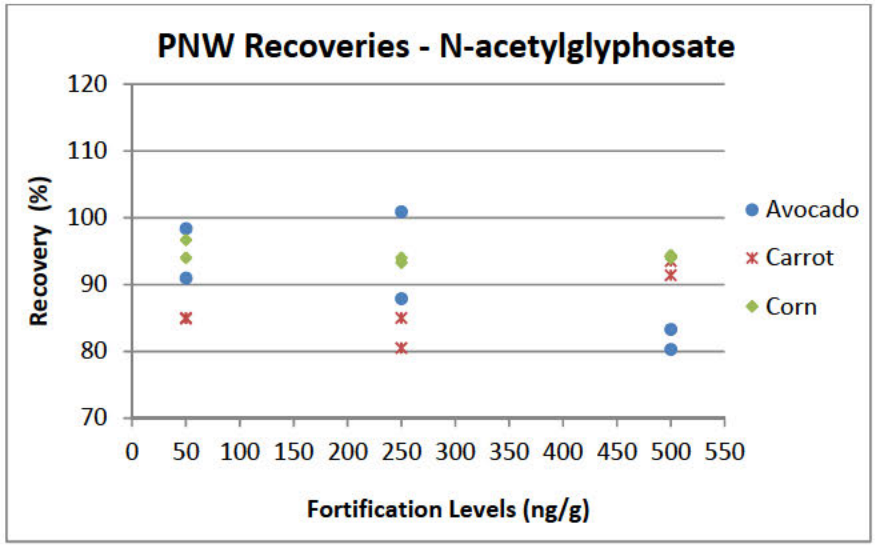
Results of the analysis of corn and soy containing incurred glyphosate residues are tabulated below. PNW findings are consistent with the range of residues levels reported from four different laboratories.

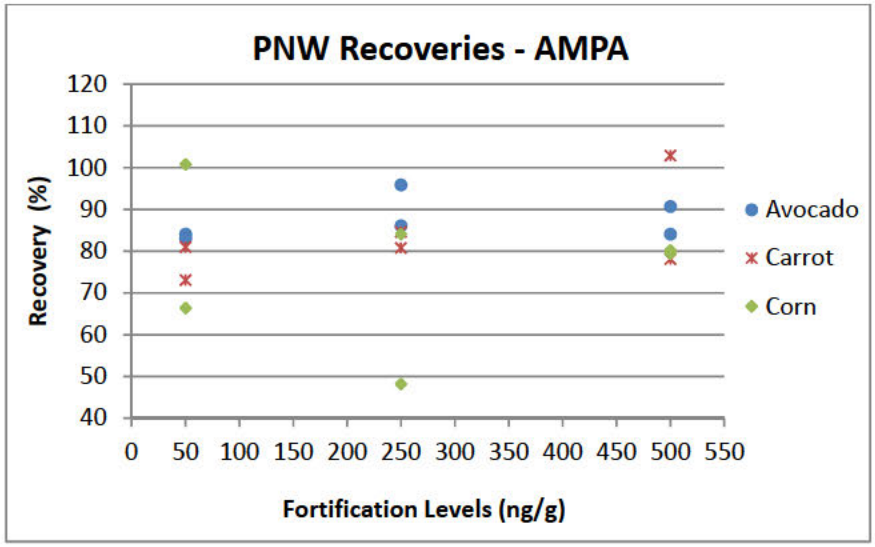
Matrix	Range	PNW
Corn	35-46	35
Soy	4290-4620	4610

PNW Collaboration Data and System Suitability – Recovery Charts

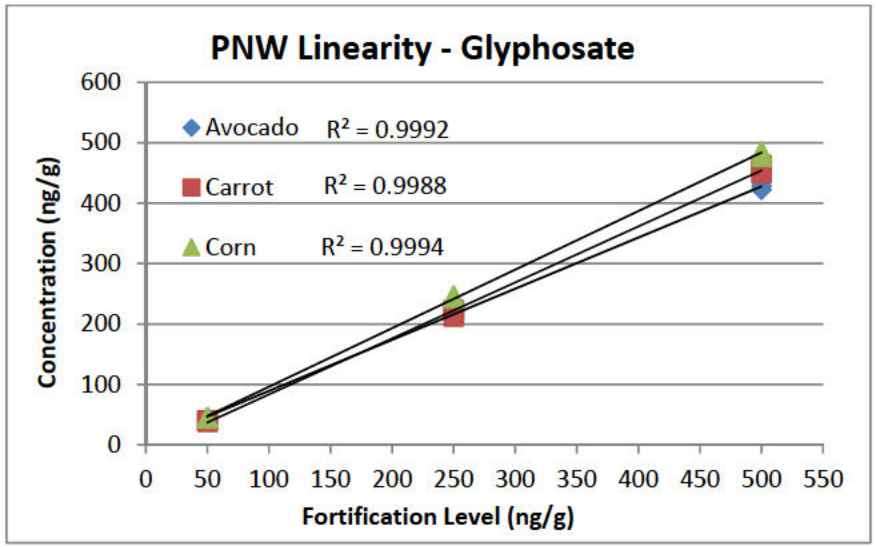


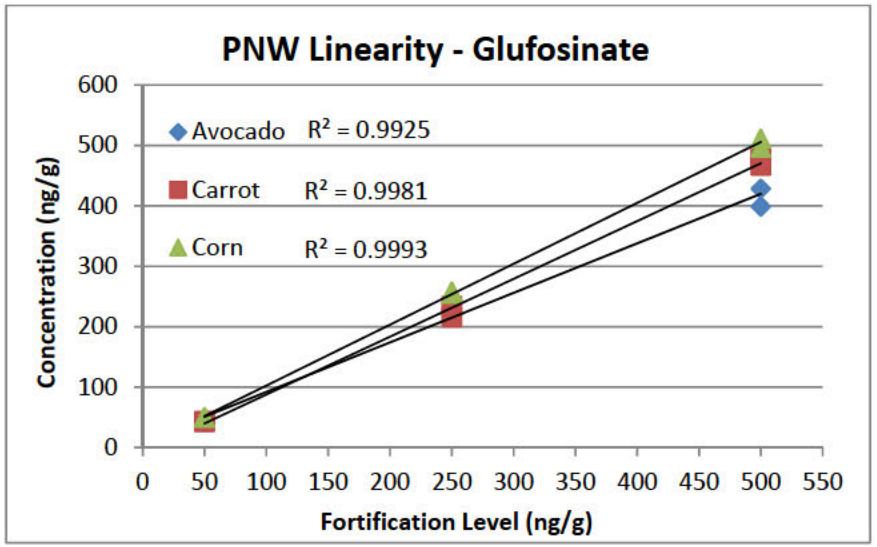


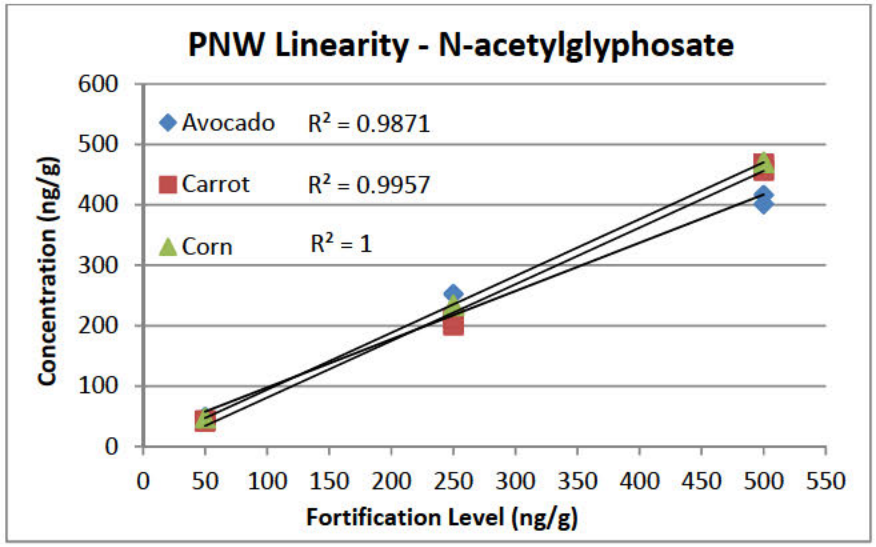


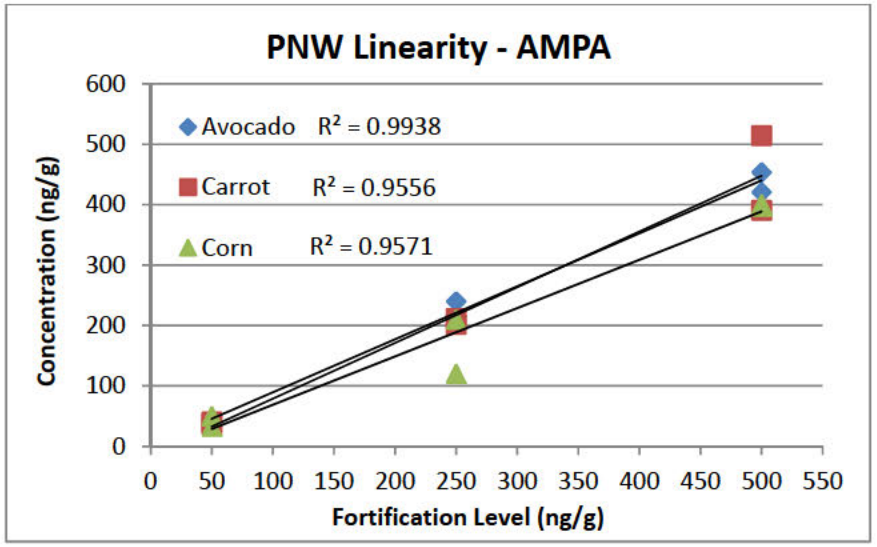


PNW Collaboration Data and System Suitability – Linearity Charts









ARL Collaboration Data and System Suitability

All data and derived statistics in this attachment are from the method collaboration analyses conducted at ARL only. Results of the instrument system suitability and method collaboration verify that ARL is able to the method proficiently.

Instrument System Suitability

Prior to starting the collaboration instrument system suitability (SS) was demonstrated. Standards were prepared and injected (b) (4) and (b) (4) ng/ml to determine accuracy and linearity. (b) (4) replicates of the (b) (4) ng/ml standard were injected to determine precision. The instrument LOQ was determined as per ORA-LAB.10 by injecting standards (b) (4) and (b) (4) ng/ml and determining the S/N of the quantifier and qualifier ions. The LOQ was calculated as the lowest level where the S/N of the quantifier ion ≥ 10 and the S/N of the qualifier ion ≥ 3. Results for the instrument system suitability study are listed in the table below. Criteria for instrument system suitability are tabulated below.

LOQ (ng/ml)	Precision (RSD)	Accuracy (%)	Linearity (R^2)
≤ 2	≤ 10	90 - 110	0.995

Results for the instrument system suitability study, listed in the table below, are all within acceptable criteria.

SS Factor	Glyphosate		Glufosinate		AMPA		N-acetylglyphosate	
LOQ (ng/mL)	0.2		0.3		0.2		1.8	
Precision (RSD)	100.0	(1.0)	100.0	(1.0)	100.0	(1.8)	100.0	(1.7)
Accuracy (R^2)	102.8	(0.9998)	99.3	(0.9999)	106.7	(0.9996)	99.8	(0.9998)

Method Collaboration

The method and collaboration protocol are described in attachments A and B, respectively. Results from the analysis of spiked avocado, carrot, and corn matrices are summarized in Table E1. The mean, RSD, method uncertainty (MU) of the recoveries for all three spike levels (50, 250, and 500 ng/g) were determined by matrix and overall. The linearity coefficient of determination (R^2) was calculated from the concentrations found at each level for each matrix by squaring the Excel correlation function (Correl). Statistics for all matrices were calculated from the whole set of data without correction for matrix bias. Acceptable method validation specifications for the collaboration study are listed below.

Recovery: 70-120 % RSD: 15% MU: 30% R^2: 0.990

ARL Collaboration Data and System Suitability

Method collaboration results contributed by ARL are summarized in the Table H1 below; results that did not meet specifications are highlighted in red font. Scatter plots of individual recoveries and linearity charts for each matrix are provided in attachments H_1 and H_2, respectively. All results were within the validation specifications for glyphosate, glufosinate and the N-acetylglyphosate. Almost all results for AMPA failed validation specifications.

Table H1. Summary data includes the mean, RSD, method uncertainty (MU) of spike recoveries and coefficient of determination (R^2) of the three spike levels for each matrix.

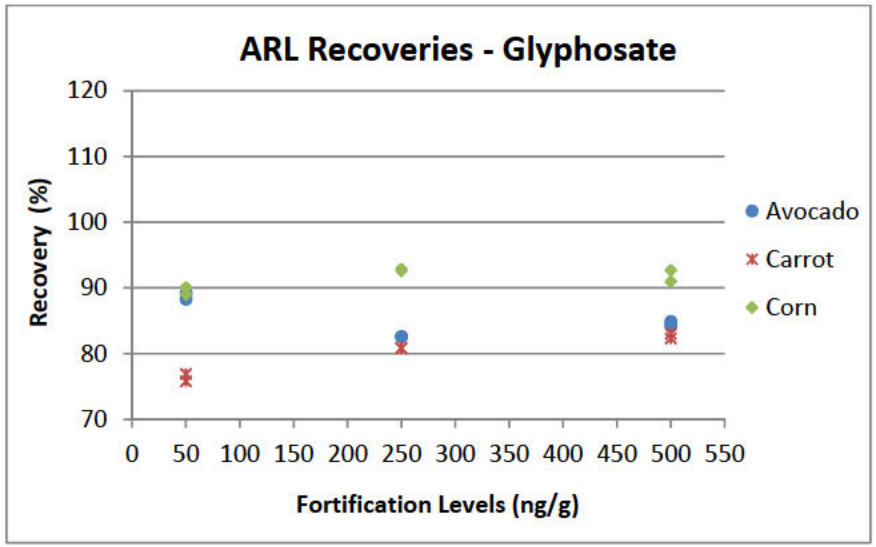
Matrix	N	Mean	RSD	MU	R^2
Glyphosate				(b) (5)	
Avocado	6	85.3	3.3		0.9996
Carrot	6	80.0	3.7		0.9999
Corn	6	91.4	1.8		0.9997
Glufosinate				(b) (5)	
Avocado	6	82.9	4.2		0.9987
Carrot	6	81.0	2.2		0.9991
Corn	6	98.4	1.2		0.9997
N-acetylglyphosate				(b) (5)	
Avocado	6	85.7	6.1		0.9975
Carrot	6	79.7	6.7		0.9972
Corn	6	93.1	5.4		0.9968
AMPA				(b) (5)	
Avocado	6	(b) (5)	(b) (5)		(b) (5)
Carrot	6	(b) (5)	7.1		0.9972
Corn	6	95.8	(b) (5)		(b) (5)

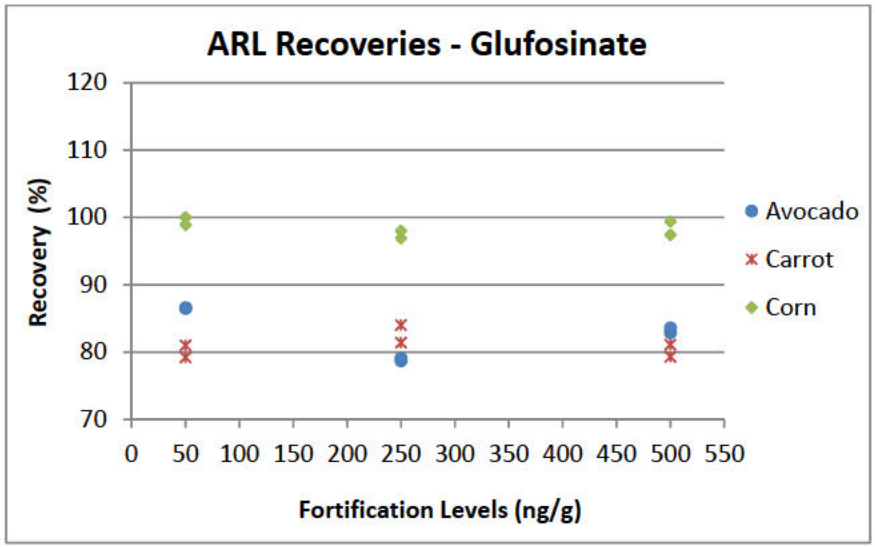
Analysis of Incurred Residues

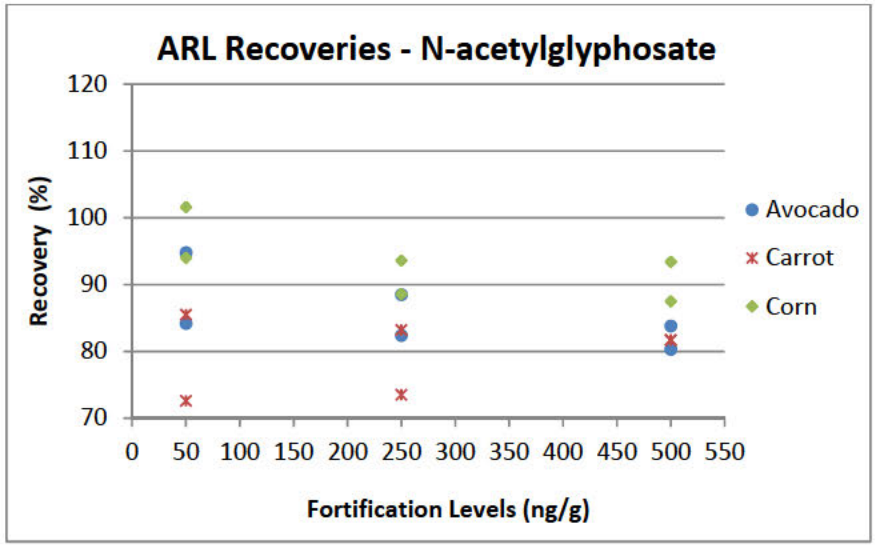
Results of the analysis of corn and soy containing incurred glyphosate residues are tabulated below. ARL findings are consistent with the range of residues levels reported from four different laboratories.

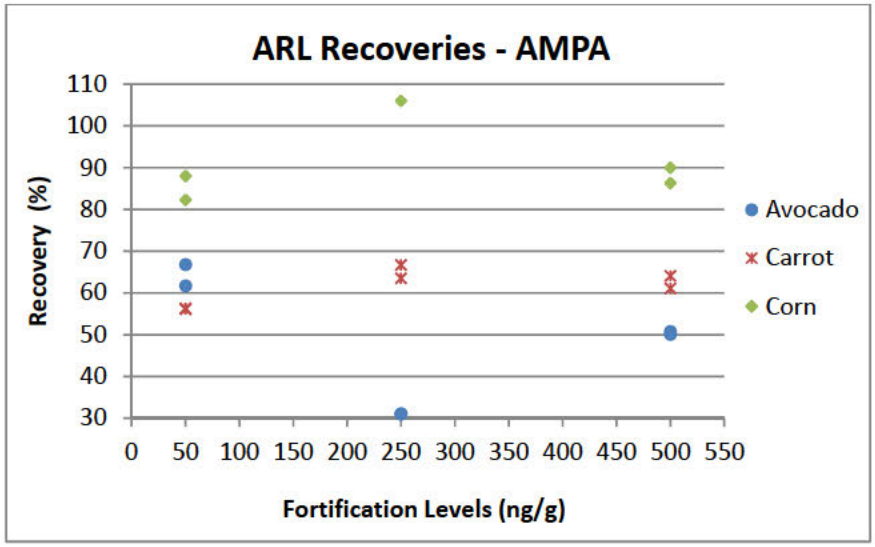
Matrix	Range	ARL
Corn	35-46	36
Soy	4290-4620	4290

ARL Collaboration Data and System Suitability – Recovery Charts

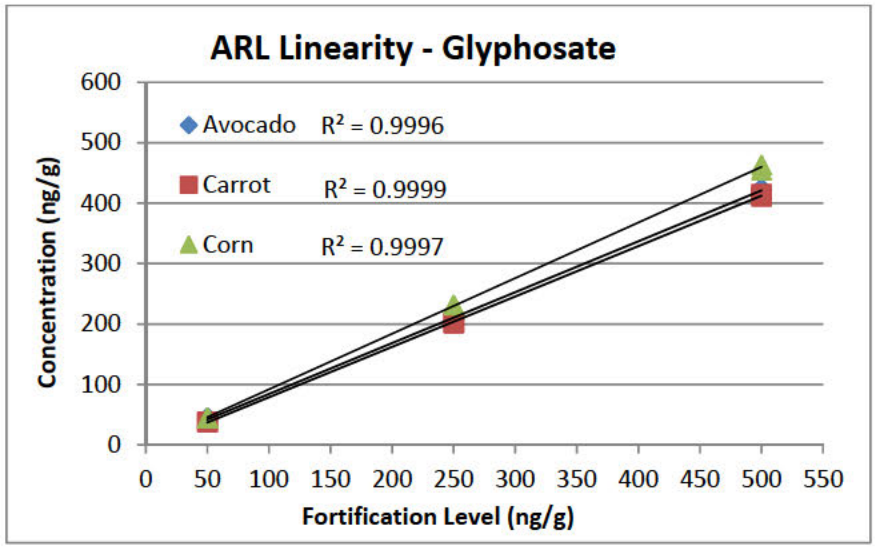


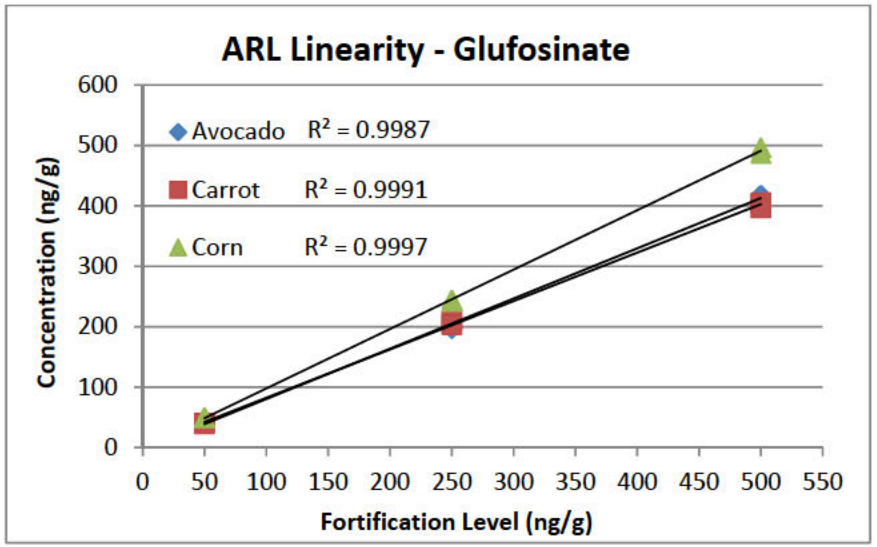


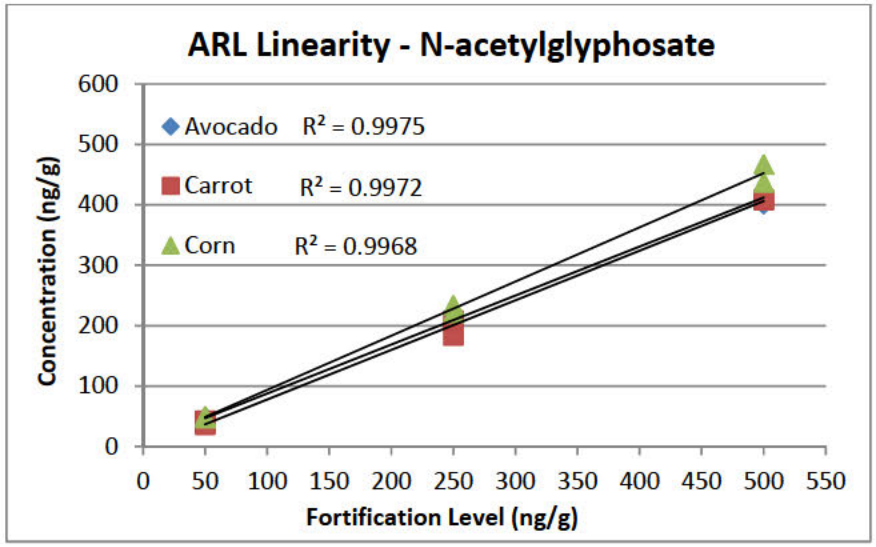


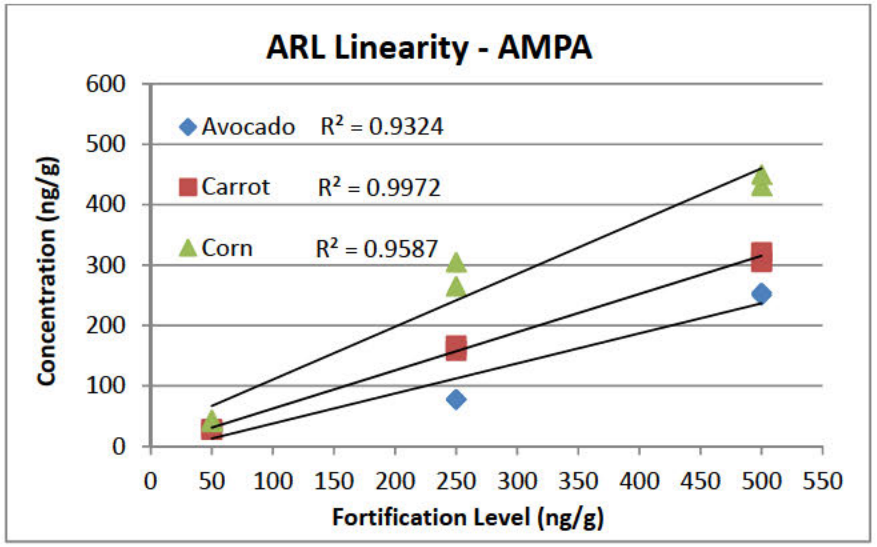


ARL Collaboration Data and System Suitability – Linearity Charts









Noonan, Gregory

From: Sack, Chris A
Sent: Monday, November 6, 2017 10:47 AM
To: Adams, Neal L; Ajayi, Olusegun J; Atkinson, Krisztina Z; Benjamin, Linda; Blount, Janet; Cassias, Irene; Chang, Eugene; Chu, Gabriel; Cooke, William; Coppin, Julia; Councell, Terry; Cromer, Michele; Damanti, Angelo; Day, Thomas; Drake, Connie P.; Eide, David J; Fairchild, Russell D.; Files, Darin; Gonzales, Steven A.; Graham, David F; Graham, Lori M.; Hanson, Madison; Hassan, Nazmul; Hayward, Douglas G; Hetz, Stacy C; Ingram, Shannon; Iorsh, Michael; Islam, Mohammed R; Jenkins, Roy; Johnson, Tonya R.; Jones, Jennifer M.; Katsoudas, Eugenia; Lane, Shannon; Lapainis, Theodore; Liang, Charlotte; Mabry-Smith, Ronald C; Makovi, Carolyn M; Masse, Claude; Mercer, Gregory E; Moore, Joshua; Nickols, Susan M; Noonan, Gregory; Olson, Melissa; Phan, Vinh T; Podhorniak, Lynda; Purnell, Standra; Ross, Mark S; Russell, Franklin N; Sack, Chris A; Sagardia-Vazquez, Daniel; Satterfield, Gregory E.; Shelby, Rebecca; Si, Siv; Thompson, Richard L.; Viner, Marianna; Vonderbrink, John; Wilson, Sarah; Wong, Jon; Wong, Maxine; Yee, Sally; Zhang, Kai
Subject: Minutes for National Pesticide phone call Nov. 1, 2017

Minutes for Pesticide Phone Call

Date: November 1, 2017

Attendance: PNL, PSFFL, KCL, ARKL, NFFL, CFSAN, CVM, ORA-ORS

Agenda

1. PesTAG Update (Mercer)
2. FMD-81
3. Pesticide Sample Collection Issues (Sack)
4. LST Dashboard Issues (Gonzales)

Around the Horn

PSFFL found phorate sulfone in dried radish tops. They are not finding glyphosate in RACs, however they are seeing low levels in soybeans. PNL is shut down right now for about 3 more weeks. They found about (b) (4) ppb glyphosate on dried white beans, but none in RACs. Ron put together a purchase order for 5 Agilent (b) (4) . KCL has gotten a few EU honey samples. They are gearing up for the revised TDS program with monthly collections; should be an interesting adjustment. The first collection was finished in October. Ron Sisk retired last Friday. ARKL is getting EU eggs and milk. They are finishing up the validation of the AcH method. They put together the purchase order for (b) (4) plus (b) (4) NFFL reported quite a few violative samples, e.g. fresh basil. Standra stated that the only "herb" to be collected is cilantro. NFFL was encouraged to refuse basil. They also had two over tolerance domestic violations: acephate/methamidophos in stringbeans and chlorothalonil in kale. They are planning to shut down the end of November for lab modifications.

ORA-ORS Update (Moh)

- They received all FMD-81 requests
- On October 27 there was fire in WV near a honey producer. ORA may be requested to conduct some non pesticide chemical testing of the honey exposed to fire.

LST Dashboard Issues (Gonzales)

- All regulatory pesticide labs (PNL, PSFFL, ARKL, and NFFL) reported they are getting slammed with samples on Thursdays and Fridays. The problem seems to be that each lab sets their individual weekly capacity; however if samples are not collected early in the week, the full weeks allotment is collected late in the week. The LST is updates daily based upon all samples pending and in-process. Additionally, any changes to the lab capacities in the LST must be justified and approved.
- With the NSD you could adjust in real time; whereas the LST cannot be adjusted in real time. Moh doesn't think the NSD will be revived.
- Connie Drake mentioned that other program areas are also reporting problems. ORA-ORS is gathering information to address the issues. Report problems with the LST to Moh.
- Some suggestions/thoughts:
  - The LST is set up for weekly; can it be changed to daily?
  - Can the capacity be different for each day of the week. For example, (b) (5) (b) (5)
- Naz mentioned the import industries purposely bring samples into port late in the week because they know the collections are reduced Friday and no samples are collected over the weekend.
- Moh mentioned that two labs are currently not operational. Do we adjust the LST for the remaining two labs? All agreed this is not feasible because the agency would have to approve over time; and, even if they did nobody wants to work weekends.

PesTAG Update (Mercer)

(b) (5)

FMD-81

- AB 6500 plus LC-MS/MS (4): One for each lab conducting SRM analyses for regulatory pesticide samples; i.e. PSFFL, PNL, ARKL, and NFFL. ARKL developed and submitted a purchase request for four systems; although (b) (5) may be needed. ORA-ORS will need to make that decision.
- Agilent GC-QQQ (5): One for each lab to replace the original GC-QQQs purchased in 2009-2011. PNL submitted purchase order for 5 instruments.
- What about adding (b) (5) NFFL submitted a purchase order.
- PSFFL mentioned they have access to an (b) (4) in the lab and they might evaluate it for pesticide analysis.

Pesticide Sample Collection Issues (Sack)

- Problems with pesticide sample collections persist. From April thru August one laboratory refused 25 different samples: 14 multiple ingredient, 6 dried peppers, 2 dietary supplements, 1 spice, 1 coffee and 1 non-food feed additive. One mis-collection was averted because the collector actually contacted the lab to find out if the ginger/honey tea crystals they planned to collect were appropriate for pesticide analysis.
- In addition to ad hoc mis-collections collectors are actually being assigned to collect inappropriate samples by compliance officers, because the firm has been flagged by PREDICT.

- Sack has prepared a simple guidance for pesticide sample collectors and management (attached). The guidance addresses three collection issues: appropriate and inappropriate commodities and sample scheduling.
- The guidance will provide a basis for a discussion with the two HAF directors and DIOP this month. Hopefully, we can get some directives issued from the upper management of the collectors. We also hope to establish a communication channel with upper management for any future issues, or even to provide occasional reminders.
- Janet has ppt about inappropriate samples. She agreed to send it to Chris.
- DIO has an import phone call. Standra will send Chris a POC.

Closeout
Lynda mentioned that the AOAC pesticide meeting was pushing (b) (5) This is just what FDA is planning to do, however, (b) (5) needs careful navigation. Greg sent some samples to Sciex who analyzed by their Q-TOF. They are planning a webex to present the results; Greg will forward the webex invitation to everyone.

Unless otherwise notified our next national pesticide call is scheduled for the first Wednesday in December (6th) at 11:00 AM EST.

Always a pleasure working with you,

Chris