

From: [Sack, Chris A](#)
To: [McSeveney, Megan](#)
Cc: [Robin, Lauren P](#); [South, Paul](#)
Subject: RE: URGENT Media Inquiry - Glyphosate - CBS Evening News- 5:00 pm today deadline
Date: Friday, January 27, 2017 4:21:00 PM

Megan, we have no updates at this time. The proposed response is still current.

Have a wonderful weekend,

Chris

Ph: 240-402-2464

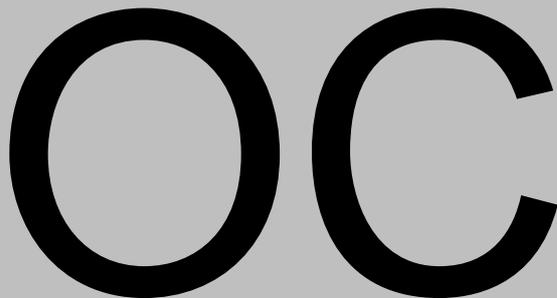
From: South, Paul
Sent: Friday, January 27, 2017 3:11 PM
To: Sack, Chris A
Cc: Robin, Lauren P
Subject: FW: URGENT Media Inquiry - Glyphosate - CBS Evening News- 5:00 pm today deadline

Hi Chris,

Can you take a quick look at the response below.

Thanks, Paul

From: McSeveney, Megan

A large, bold, black graphic consisting of the letters 'O' and 'C' side-by-side. The 'O' is a simple circle, and the 'C' is a simple curve. They are set against a solid gray rectangular background.

OC

From: [Sack, Chris A](#)
To: [Thompson, Richard L.](#); [Chang, Eugene](#)
Cc: [Mercer, Gregory E](#); [Islam, Mohammed R](#); [Cooke, William](#); [Vonderbrink, John](#); [Masse, Claude](#); [Chamkasem, Narong](#)
Subject: RE: Glyphosate Method
Date: Wednesday, January 04, 2017 2:06:00 PM

Thanks Richard. That is awesome!

Happy New Year to everyone,

Chris

Ph: 240-402-2464

From: Thompson, Richard L.

ORA

From: Sack, Chris A
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Thompson, Richard L](#); [Vonderbrink, John](#)
Cc: [Noonan, Gregory](#); [Cromer, Michele](#)
Subject: RE: Revised Minutes for PestAG meeting Dec 14, 2016
Date: Monday, January 09, 2017 1:04:00 PM

Hey everyone,

Just wanted to touch base to see how you are progressing with the glyphosate method implementation. Please let me know if your lab

1. purchased the supplies and reagents
2. attempted the LCMS method
3. generated recoveries using whole method
4. received the collaboration matrices
 - a. carrots from KAN
 - b. avocado from ARL

Thanks,

Chris

Ph: 240-402-2464

From: Sack, Chris A
Sent: Friday, December 16, 2016 11:13 AM
To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John
Cc: Noonan, Gregory; Cromer, Michele
Subject: Revised Minutes for PestAG meeting Dec 14, 2016

PestAG PMC Meeting Minutes

Date: December 14, 2016

Attendance: Greg Mercer, Bill Cooke (PNW), Eugene Chang, (PSW), Narong Chamkasem (SRL), Richard Thompson(ARL), Claude Masse (NRL), Chris Sack, (CFSAN) Moh Islam (ORA-ORS)

Agenda: Glyphosate method progress and collaboration

We really need to get this method finalized, validated and collaborated. Although there is some external pressure to resume the assignment, we are not late yet. Sack assured CFSAN-OC that the assignment would resume in early CY-17 and we can still make that timeframe.

LC-MS/MS method

Eugene updated his latest LC-MS/MS method that uses ion-pairing with reverse phase chromatography. The essential LC parameters are listed below and listed in the attached file.

LC Parameters		Gradient	
		Time	MPB
Column:	Phenomenex Luna C8(2), 150 x 4.6 mm, 5 µm, with Phenomenex KrudKatcher guard column		
MP A:	10 mM tetrabutylammonium formate + 0.1 % formic acid in water (pH 2.8±1)	0.00	5
MP B:	MeCN	1.00	5
Flow:	0.6 mL/min (4.6 mm column)	5.00	95

Inj Vol: 10 µL	6.50	95
Temp 45 °C	6.60	5
	10.00	5

MS/MS Parameters

Q1	Q3	RT	Transition	DP	EP	CE	CXP
110	63	2.5	AMPA 1	-40	-11	-30	-9
110	79	2.5	AMPA 2	-40	-11	-34	-9
112	63	2.5	AMPA IS	-60	-11	-26	-9
180	63	4.0	Glufosinate 1	-60	-11	-66	-9
180	95	4.0	Glufosinate 2	-40	-11	-24	-5
180	85	4.0	Glufosinate 3	-60	-11	-25	-9
183	63	4.0	Glufosinate IS	-60	-11	-40	-9
168	63	5.0	Glyphosate 1	-30	-11	-28	-9
168	79	5.0	Glyphosate 2	-30	-11	-56	-9
168	150	5.0	Glyphosate 3	-30	-11	-16	-9
171	63	5.0	Glyphosate IS	-30	-11	-28	-9
210	63	6.0	N-acetyl glyphosate 1	-85	-11	-40	-13
210	124	6.0	N-acetyl glyphosate 2	-85	-11	-17	-13
210	79	6.0	N-acetyl glyphosate 3	-85	-11	-50	-13

MS Parameters

CUR	25
CAD	MEDIUM
IS	-4000
GAS 1	65
GAS 2	65
TEM	700
Q1	UNIT
Q3	UNIT

Preparation of the mobile phase:

- For 1 liter mobile A: to make 10 mM TBAOH, add 25 ml of 0.4 M TBAOH solution to ~900 mL DI water containing 0.40 mL formic acid. Adjust pH to 2.8-3.2 using 0.1% formic acid solution. OR
- Add 1.0 mL formic acid (98%) and 3.01 grams TBA acetate in 1 L DI water; pH range between 2.8-3.0.

Eugen provided chromatograms using the current LCMS parameters for standard mixture, corn control and corn spiked at 10 ng/g (see attached).

Several people challenged Eugene's use of neat MeCN for the organic modifier, suggesting it should contain the same level of IP reagent and have same pH as the aqueous phase. Eugene defended his method saying it did not seem to be a problem; however as he uses the method to analyze matrices he will observe the stability of the LCMS response and retention. Richard mention he tried this latest LC method and found the glyphosate was tailing. Eugene suggested replacing all metal LC tubing with PEEK tubing between the autosampler and

injection valve (see attached pic) because glyphosate can be retained on metal surfaces.

Richard alerted everyone to be sure and order the correct glyphosate isotope. He inadvertently ordered the wrong isotope using different carbon labelling and was not getting any response until he realized the mass of the isotope he ordered was actually different than that for the method.

What about others, e.g. ethephon, quats, ...? Groan, this will have to wait till we get glyphosate assignment restarted.

Eugene provided a chromatogram of a 10 ng/g glyphosate in corn (attached).

Extraction Procedure

1. 5 g sample + 25 ml extraction solvent
2g sample plus 10 ml extraction solvent for dry products
[Extraction solvent: 50 mM Acetic acid + 10 mM EDTA]
2. Add 10 ml PE or MeCl to fatty matrices
3. Spike with isotopes @ 200 ng/g (could be included in the extraction solvent)
4. Shake @ 1000 for 10 min
5. Centrifuge at = 3000 rpm for 5 min
6. Filter aqueous extract thru HLB SPE cartridge
7. Filter for injection (could be included with SPE step)
8. Sample concentration: 0.2 g/ml

Initial Validation (LA)

Instrument:

- Evaluate linearity and range of calibration curve from 10 to 2500 ng/ml.
- Determine instrument sensitivity @ 1, 2, and 5 ng/ml – Need S/N of 10:1 for calculation ion and 3:1 for the confirmation ion
- Determine instrument response precision during validation analyses

Method

- Matrices: carrots, avocado, and corn
- Selectivity: analyze matrix blanks
- Sensitivity: analyze = 7 replicate spikes at 10 ng/g, calculate MDL per 40 CFR 136, calculate the LOQ as 3.3 x MDL
- Linearity/Accuracy: analyze 6 replicates of each matrix spiked at 50, 200, and 500 ng/g
- Range: analyze duplicate spikes at 1000 ng/g

Collaboration

We agreed to collaborate corn, carrots and avocado using the following fortification protocol.

QC	Matrix	Level	N
Control	Corn	0	1
Linearity	Corn	50	2
Linearity	Corn	200	2
Linearity	Corn	500	2
Control	Carrot	0	1
Linearity	Carrot	50	2
Linearity	Carrot	200	2
Linearity	Carrot	500	2
Control	Avocado	0	1
Linearity	Avocado	50	2
Linearity	Avocado	200	2

Total Analyses per Lab 21

Narong has previously shipped both corn and soy with and without incurred residues to all participating ORA labs .KAN has carrots and ARL has avocados they will ship to participating labs. Each lab will receive ~ 100 g composite per matrix.

Lab	Contact	Address	Phone
PNW	Bill Cooke	22201 23rd DR SE, Bothell WA, 98021	(425) 487-5324
PSW	Eugene Chang	19701 Fairchild, Irvine, CA	(949) 608-2970
KAN	John Vonderbrink	11510 W 80th St, Lenexa, KS 66214	(913) 752-2703
ARL	Richard Thompson	3900 NCTR Road, Jefferson, AR 72079	(870) 543-4054
SRL	Narong Chamkasem	60 Eighth St NE, Atlanta, GA, 30309	(404) 253-2302
NRL	Claude Masse	158-15 Liberty Ave Jamaica NY 11433	(718) 340-7050
CFSAN	Greg Noonan	5001 Campus Drive, College Park, MD 20740	(240) 402-2250

Action items

1. Purchase reagents and supplies – all labs
 - a. Phenomenex Luna C8, 150 x 2 mm, 5 µm, Phenomenex 00F-4040-B0 or Phenomenex Luna C8(2), 100 Å, 5 µm, 150 x 4.6 mm, Phenomenex 00F-4249-E0
 - b. Phenomenex guard column KrudKatcher P/N AFO-8497
 - c. Glyphosate isotope
 - d. Glufosinate isotope
 - e. Tetrabutylammonium hydroxide titrant, 0.4 M in Water, HPLC Grade, ACROS Organics (pic attached)
 - f. Tetrabutylammonium acetate, Aldrich No. 335991-10G (pic attached)
 - g. N-acetyl-glyphosate, available from Toronto Research Chemicals (TRC No A178245), or Santa Cruz BioTech (SCBT No. sc-479500)
2. Ship collaboration matrices
 - a. KAN ships 100 g carrots
 - b. ARL ships 100 g avocados
3. LA validates method and submits validation report for review
4. All labs set up LA LC-MS/MS method on AB 5500 or 6500

Have a nice holiday,

Chris

(b)(4) Consultation

From: Sack, Chris A [mailto:Chris.Sack@fda.hhs.gov]
Sent: Tuesday, January 10, 2017 11:49 AM
To: (b) (6)
Cc: (b) (6)
Subject: RE: Glyphosate analytical method

Hi Pamela,

I hope your holidays were full of everything good.

Do you have any experience with N-acetyl-glyphosate? We are attempting incorporate it into our procedure and finding it is quite unstable, quickly degrading to glyphosate.

Thanks and happy new year,

Chris Sack

Residue Expert
Office of Food Safety
Center for Food Safety and Applied Nutrition
US Food and Drug Administration

Phone: 240-402-2464

(b)(4) Consultation

From: Sack, Chris A [<mailto:Chris.Sack@fda.hhs.gov>]
Sent: Wednesday, June 15, 2016 6:43 AM
To: (b) (6)
Subject: RE: Glyphosate analytical method

Hi Pam,

Thanks for sharing your experiences. I will forward your observations to our team of analysts who are working to improve Narong's method to make it more rugged. Interesting, that you point out the problems with the chrom. It was the first problem our analysts noted about the method also. Since then, they have come up with a "gradient" using acetonitrile and the method aqueous MB to move the peaks out to about 4-5 minutes. That seems to have fixed that problem. Re the MeCl₂, I know our analysts will not use it because it is chlorinated; we have dispensed with everything chlorinated in the last 10 years. So, I am glad you found another cleanup using the Phenomenex Strata-X Drug-N plate. We will look into that.

Thanks again for taking time to review the method. I am hoping to have a finalized method by end of summer so we can collaborate it in the fall.

If I invited you to join us for a telecon to discuss our glyphosate method development, would you be able/willing to accept?

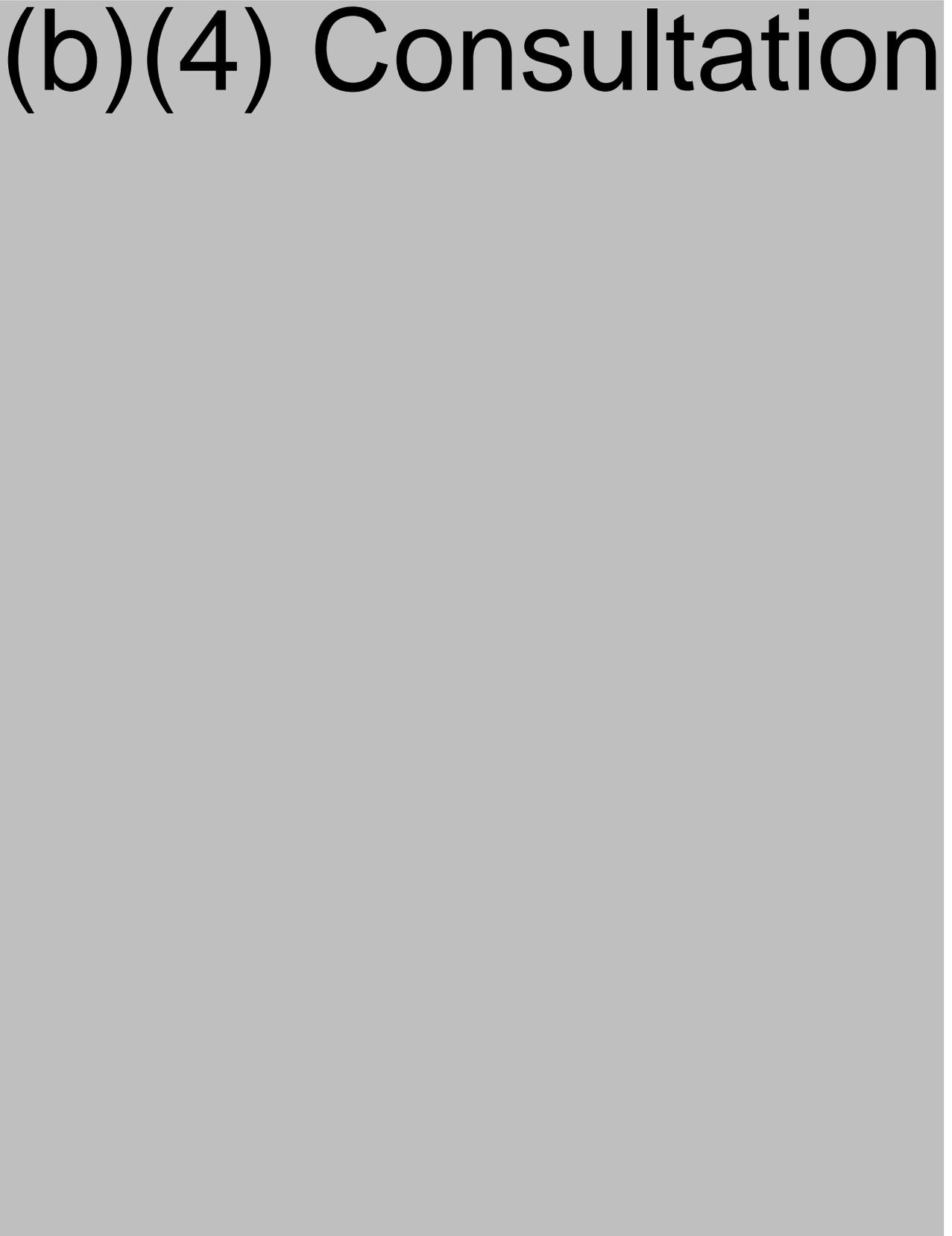
Will keep in touch.

All the best,

Chris Sack
Residue Expert
Office of Food Safety
Center for Food Safety and Applied Nutrition
US Food and Drug Administration

Phone: 240-402-2464

(b)(4) Consultation



(b)(4) Consultation

From: Sack, Chris A [<mailto:Chris.Sack@fda.hhs.gov>]
Sent: Wednesday, June 08, 2016 7:09 AM
To: (b) (6)
Cc: (b) (6)
Subject: RE: Glyphosate analytical method

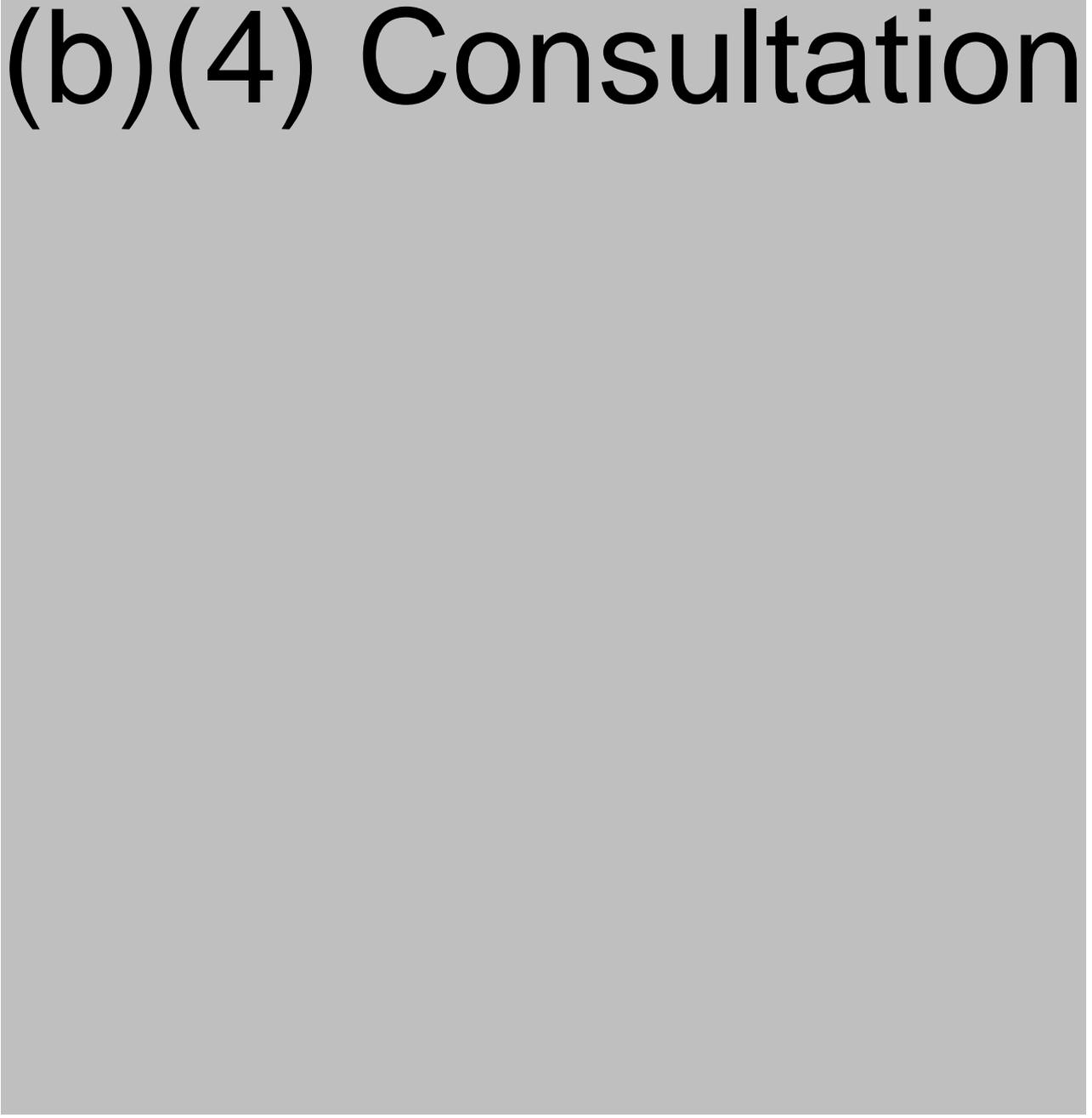
Thanks Marion!

Chris

Ph: 240-402-2464

(b)(4) Consultation

(b)(4) Consultation



(b)(4) Consultation



From: [Chang, Eugene](#)

ORA

From:

[Chang, Eugene](#)

ORA

From:

[Chang, Eugene](#)

ORA

From: [Chang, Eugene](#)

ORA

From: Chang, Eugene

ORA

From: Sack, Chris A

Sent: Tuesday, January 10, 2017 7:10 AM

To: Chang, Eugene

Subject: RE: Clarify the part number of column: Luna 5 μ C8(2) 150X4.6mm, 00F-4249-E0

Hi Eugene,

Some labs are hesitant to buy this column because they want to make sure it is the column we will use for the collaboration. I just want to verify with you that the Luna 5 μ C8(2) 150X4.6mm, part no. 00F-4249-E0 is final?

Thanks,
Chris

Ph: 240-402-2464

From: Chang, Eugene

A large, bold, black sans-serif font spelling 'ORA' is centered on a solid gray rectangular background.

From: Sack, Chris A

Sent: Thursday, December 22, 2016 9:23 AM

To: Chang, Eugene

Subject: RE: Clarify the part number of column: Luna 5 μ C8(2) 150X4.6mm, 00F-4249-E0

Hi Eugene,

Just checking back with you to see if the preferred column is still : Luna 5 μ C8(2) 150X4.6mm, 00F-4249-E0? Is this final?

Thanks,

Chris

Ph: 240-402-2464

From: Chang, Eugene

A large, bold, black sans-serif font spelling 'ORA' is centered on a solid gray rectangular background.

ORA

From: Sack, Chris A

Sent: Friday, December 16, 2016 8:37 AM

To: Chang, Eugene

Subject: RE: Clarify the part number of column: Luna 5 μ C8(2) 150X4.6mm, 00F-4249-E0

Hi Eugene,

Just to be clear the pic you sent yesterday lists a different part number: 00F-4040-E0 – see attached pic. Which is correct?

Thanks,

Chris

Ph: 240-402-2464

From: Chang, Eugene

ORA

From: [Chang, Eugene](#)

ORA

From: Chang, Eugene

ORA

From: [Sack, Chris A](#)
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed B](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Thompson, Richard L](#); [Vonderbrink, John](#); [Noonan, Gregory](#)
Subject: Glyphosate method update
Start: Friday, January 13, 2017 12:00:00 PM
End: Friday, January 13, 2017 1:00:00 PM
Location: Telecon

Good Morning,

Let's have a quick meeting to update our progress with the glyphosate method implementation. If you can't make it send me a brief update via email.

Talk soon,

Chris

Chris Sack invites you to an online meeting using WebEx.

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

To join this meeting

-
1. Go to <https://fda.webex.com/fda/j.php?MTID=m071356f56d5e219a8aff810eafa9367f>
 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.

From: [Chang, Eugene](#)

ORA

-----Original Appointment-----

From: Sack, Chris A

Sent: Friday, January 13, 2017 5:24 AM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John; Noonan, Gregory

Subject: Glyphosate method update

When: Friday, January 13, 2017 12:00 PM-1:00 PM (UTC-06:00) Central America.

Where: Telecon

Good Morning,

Let's have a quick meeting to update our progress with the glyphosate method implementation. If you can't make it send me a brief update via email.

Talk soon,

Chris

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Meeting ID: (b) (6)

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From: [Sack, Chris A](#)
To: [Chamkasem, Narong](#); [Chang, Eugene](#)
Subject: N-acetyl-glyphosate
Date: Friday, January 13, 2017 10:24:00 AM
Attachments: [Glyphosate RAR 07 Volume 3CA-CP B-5 2013-12-18 san.pdf](#)
[Glyphosate RAR 09 Volume 3CA-CP B-7 2013-12-18.pdf](#)

Hi Eugene and Narong,

I asked Monsanto about the stability of N-acetyl-glyphosate and they sent me the attached documents. I looked briefly through of the 07 document and I don't see any indication that N-acetyl-glyphosate is unstable.

Chris

From: [Chamkasem, Narong](#)

ORA

-----Original Appointment-----

From: Sack, Chris A

Sent: Friday, January 13, 2017 8:24 AM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John; Noonan, Gregory

Subject: Glyphosate method update

When: Friday, January 13, 2017 12:00 PM-1:00 PM (UTC-06:00) Central America.

Where: Telecon

Good Morning,

Let's have a quick meeting to update our progress with the glyphosate method implementation. If you can't make it send me a brief update via email.

Talk soon,

Chris

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Meeting ID: (b) (6)

Meeting Password: (b) (6)

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From: Sack, Chris A
To: (b) (6)
Subject: RE: Glyphosate_RAR_09_Volume_3CA-CP_B-7_2013-12-18
Date: Friday, January 13, 2017 12:56:00 PM

Hi Marian,

We would like to include N-acetylglyphosate in our glyphosate method but we seem to be having some problem with the standard. At first we thought maybe the compound was unstable, but the literature doesn't suggest that. Now we think we may have gotten a bad standard. We don't see a lot of vendors for the compound. I was wondering you could recommend (b) (5) or perhaps (b) (4) has the reference material and would be willing to provide some to the FDA for this method development project.

Thanks and have wonderful weekend,

Chris

Ph: 240-402-2464

(b)(4) Consultation

From: [Thompson, Richard L.](#)

ORA

From: [Mercer, Gregory E](#)

ORA

From: [Chang, Eugene](#)

ORA

From: Mercer, Gregory E

ORA

From: [Chang, Eugene](#)

ORA

From: Chang, Eugene

ORA

From: Mercer, Gregory E

ORA

From: [Chamkasem, Narong](#)

ORA

From: Sack, Chris A
Sent: Monday, January 16, 2017 9:47 AM
To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John
Cc: Noonan, Gregory; Viner, Marianna
Subject: Draft Minutes for PMC meeting Jan 13, 2017

*****DRAFT*****

Thanks for meeting on short notice. Please review the minutes, but more importantly, I need you to review the attached method to ensure it is correct. Note I highlighted the standard preparation section for special attention. I will finalize the minutes early this week.

Stay warm,
Chris

PesTAG PMC Meeting Minutes

Date: January 13, 2017

(b) (5)

(b) (5)

Happy New Year,

Chris

From: Chamkasem.Narong

ORA

From: Eric.Eichhorn@cvuas.bwl.de [mailto:Eric.Eichhorn@cvuas.bwl.de]

Sent: Monday, January 16, 2017 2:55 AM

To: Chamkasem, Narong

ORA

Von: Chamkasem, Narong [mailto:Narong.Chamkasem@fda.hhs.gov]

ORA

From: Sack, Chris A

Sent: Friday, January 13, 2017 11:24 AM

To: Chamkasem, Narong; Chang, Eugene

Subject: N-acetyl-glyphphosate

Hi Eugene and Narong,

I asked (b) (4) about the stability of N-acetyl-glyphosate and they sent me the attached documents. I looked briefly through of the 07 document and I don't see any indication that N-acetyl-glyphosate is unstable.

Chris

From: Sack, Chris A
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Thompson, Richard L](#); [Vonderbrink, John](#)
Cc: [Noonan, Gregory](#); [Viner, Marianna](#)
Subject: Status update
Date: Monday, January 23, 2017 11:48:00 AM

Hi Everyone,

Barring a few tweaks we might find necessary during implementation the glyphosate method is about finalized. At this time all labs are expected to have ordered the requisite supplies and reagents and implemented the LC-MS/MS method on a 5500 or 6500. All labs need to evaluate the linearity, range and precision on their instrument. I recommend you start by determining if your instrument has the necessary sensitivity for the analysis; i.e. to achieve an LOQ of 10 ng/g for the method glyphosate needs to be quantifiable at 2 ng/ml (10 ng/g * 0.2 g/ml). [Note: Only Eugene needs to determine the MDL and associated LOQ based upon the multiple iterations of the extraction procedure as per 40 CFR 136]. To demonstrate instrument sensitivity I propose injecting a low level sensitivity curve at 1, 2, 5 and 10 ng/ml. Determine the LOQ for each level as per ORA-LAB.10; i.e. we just need to know the concentration at 10 x of the S/N for the quant ion assuming the S/N of qualifier ion = 3.

For the high end of the range I would recommend you include standards at concentrations exceeding the range we have agreed to collaborate (500 ng/g = 100 ng/ml solution concentration). Narong's standard curve went up to 250 ng/ml. For precision, I would recommend multiple injections at our calibration level which we have not discussed – Narong and Eugene do you have a recommendation?

If you have different ideas about demonstrating instrumental determination suitability let me know. Please send me a status update. I will plan a phone call later this week at our normal time of 11 AM EST. When you send me your status let me know if you prefer Wed or Thurs.

Thanks

Chris

Ph: 240-402-2464

From: Sack, Chris A
Sent: Monday, January 23, 2017 10:31 AM
To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John
Cc: Noonan, Gregory; Viner, Marianna
Subject: Minutes for PMC meeting Jan 13, 2017

PesTAG PMC Meeting Minutes

(b) (5)

(b) (5)

Happy New Year,

Chris

From: [Islam, Mohammed R](#)

ORA

From: Sack, Chris A

Sent: Tuesday, January 24, 2017 11:52 AM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John

Subject: FW: Glyphosate LOQ

Wow Richard! Results are amazing. If this sensitivity is duplicated around the country we might consider (b) (5) [REDACTED]

Chris

Ph: 240-402-2464

From: Thompson, Richard L.

ORA

ORA

From: [Vonderbrink, John](#)

ORA

From: Sack, Chris A
Sent: Tuesday, January 24, 2017 11:01 AM
To: Vonderbrink, John
Subject: Carrots

Hi John,

Hope all is well. It was good to see you and Jeannie. I hope I didn't seem to smug about current job situation. I can't tell you how many days I would get home from lab frustrated and/or furious – it would take me about 10-15 miles into a bike ride to bring BP down. Jeannie was right, it is much easier now that I don't report to the lab.

Do you have any glyphosate results yet? Can you send me a status update? Also, did you ship the carrots?

Thanks,

Chris

From: [Sack, Chris A](#)
To: [Robin, Lauren P](#)
Subject: RE: followup
Date: Tuesday, January 24, 2017 1:36:00 PM
Attachments: [image001.png](#)

Hi Lauren,

I requested an update from all labs yesterday. Two ORA labs are ready to run the method. Two more ORA labs should be ready by end of week. One ORA lab has been diverted, one ORA lab is setting up. CFSAN has ordered supplies. All labs will have the collab samples by end of this week. I will have a meeting with them all this Thursday AM.

Chris

Ph: 240-402-2464

From: Robin, Lauren P
Sent: Tuesday, January 24, 2017 10:41 AM
To: Sack, Chris A
Subject: followup

Hi Chris

Did you get a chance to put together a summary of the glyphosate methodology status?

Lauren

Lauren Posnick Robin, Sc.D.
Chief, Plant Products Branch
DPPB/OFS/CFSAN
U.S. Food and Drug Administration
HFS-317
5001 Campus Drive
College Park, MD 20740
240-402-1639
lauren.robins@fda.hhs.gov



From: [Sack, Chris A](#)
To: [Robin, Lauren P](#)
Subject: RE: followup
Date: Tuesday, January 24, 2017 1:53:00 PM
Attachments: [image001.png](#)

The two labs that have done the sensitivity testing on the instruments are reporting sub 10 ppb level equivalence. By the end of the week I hope to know if sensitivity can be achieved in two more labs. The validating lab (LA) tells me they should have a validation report by end of week. I assume the results are satisfactory, otherwise they would have notified me. Hopefully, next week two-three more labs will have some recovery data. If I can get three labs demonstrating good sensitivity and recovery data by end of next week I hope to get the collab started the week after (Feb 6). Assuming all that goes well, it will take a few weeks to work up the data. So we are looking at the end of Feb. That would be my best guesstimate right now.

Chris

Ph: 240-402-2464

From: Robin, Lauren P
Sent: Tuesday, January 24, 2017 1:38 PM
To: Sack, Chris A
Subject: RE: followup

What LOQ?

What is the timing on when the assignment can restart (earliest)?

From: Sack, Chris A
Sent: Tuesday, January 24, 2017 2:36 PM
To: Robin, Lauren P
Subject: RE: followup

Hi Lauren,

I requested an update from all labs yesterday. Two ORA labs are ready to run the method. Two more ORA labs should be ready by end of week. One ORA lab has been diverted, one ORA lab is setting up. CFSAN has ordered supplies. All labs will have the collab samples by end of this week. I will have a meeting with them all this Thursday AM.

Chris

Ph: 240-402-2464

From: Robin, Lauren P
Sent: Tuesday, January 24, 2017 10:41 AM
To: Sack, Chris A
Subject: followup

Hi Chris

Did you get a chance to put together a summary of the glyphosate methodology status?

Lauren

Lauren Posnick Robin, Sc.D.
Chief, Plant Products Branch
DPPB/OFS/CFSAN
U.S. Food and Drug Administration
HFS-317
5001 Campus Drive
College Park, MD 20740
240-402-1639
lauren.robin@fda.hhs.gov



From: [Chamkasem, Narong](#)

ORA

From: Sack, Chris A
Sent: Wednesday, January 25, 2017 8:12 AM
To: Chamkasem, Narong; Chang, Eugene
Subject: Glyphosate cleanup

Eugene and Narong,

FYI. Our UCT sales rep shared this with me yesterday.

Chris

Ph: 240-402-2464

(b)(4) Consultation

(b)(4) Consultation

From:

[Chang, Eugene](#)

ORA

From: Sack, Chris A
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Thompson, Richard L](#); [Vonderbrink, John](#)
Subject: Some initial validation results from LA
Date: Thursday, January 26, 2017 8:30:00 AM

Hi everyone,

Just wanted you to see some initial validation results I received from LA yesterday.

Eugene provided spiked carrots, corn and avocado each, 7 replicates @ 20 ng/g, and 3 replicates each at 50, 200, and 500 ng/g. In the table below MDLs and LOQs were calculated based upon the 20 ng/g spikes. Recovery, RSD and Linearity were calculated based upon all recoveries. Note AMPA stats based upon external standard calibration, all others based upon isotopic internal standard calibration.

	Carrot	Corn		
	Glyphosate	Glyphosate	Glufosinate	AMPA
MDL (ng/g)	3.5	2.1	2.4	2.0
LOQ (ng/g)	11.4	7.0	8.0	6.5
Recovery	105	101	101	43
RSD	4.1	4.5	3.2	18.9
Linearity	0.9996	0.9995	0.9997	0.9327

Excellent work Eugene and LA crew! For our meeting today I need everyone to provide an update on the progress made to implement the glyphosate method in their labs.

Talk soon,

Chris

From:

[Cooke, William](#)

ORA

From:

[Chang, Eugene](#)

ORA

From: [Chang Eugene](#)

ORA

From: Sack, Chris A
Sent: Thursday, January 26, 2017 12:48 PM
To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John
Cc: Drake, Connie P.
Subject: Minutes for PestAG PMC meeting January 26, 2017

*****DRAFT*****

Please review the minutes and send me correction by first of next week.

Thanks,

Chris

.....

PestAG PMC Meeting Minutes

Date: January 26, 2017

(b) (5)

(b) (5)

From: [Noonan, Gregory](#)
To: [Sack, Chris A](#)
Cc: [Wong, Jon](#); [Parker, Christine](#)
Subject: Glyphosate
Date: Friday, January 27, 2017 7:51:55 AM
Attachments: [Glyphosate Collaboration Details.docx](#)

Chris,

Attached is what I have as the collaboration protocol (I have the method in a separate document). I am sending this to answer some questions Jon had, but I still have a few questions, and Jon, who will be working on the samples, may have a few more.

1. I have received avocado and carrot is on the way, but haven't gotten any corn, should I just contact Narong directly?
2. I don't see a list of the calibration standards you want prepared for the method. Are you leaving the exact number, concentration, and range to each lab or did I miss it?
3. Can you add Jon to the list of collaborators. It was unclear who from CFSAN would be participating, but with some recent changes, Jon has agreed to coordinate the lab work.

I have also included Christine Parker on this email. She is the acting Branch Chief of the Bioanalytical Methods Branch. Basically, she is busy trying to clean up the mess Greg Mercer left behind☺.

Thanks,
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

From: Sack, Chris A
To: [Chamkasem, Narong](#)
Cc: [Noonan, Gregory](#); [Wong, Jon](#)
Subject: Collab samples for CFSAN
Date: Friday, January 27, 2017 8:47:00 AM

Hi Narong,

CFSAN is setting up to participate in the glyphosate collaboration. Would you mind shipping them the collab samples, including the corn and soy samples containing incurred residues, you sent everyone else? You can ship them to Greg Noonan at the address below.

	5001 Campus Drive, College Park, MD	
Greg Noonan	20740	(240) 402-2250

Thanks and have a wonderful weekend,

Chris

From: [Vonderbrink, John](#)

ORA

From: Sack, Chris A
Sent: Tuesday, January 31, 2017 10:02 AM
To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Thompson, Richard L.; Vonderbrink, John; Wong, Jon; Noonan, Gregory
Subject: Quick update

Hi everyone,

Just wanted to give you a quick update of some data that is coming in.

Eugene's validation data is summarized below. Data looks amazing Eugene!

	<u>Corn</u>	<u>Carrot</u>	<u>Avocado</u>	<u>Avg</u>
	<u><i>Glyphosate</i></u>			
MDL (ng/g)	2.1	1.9	1.7	1.9
LOQ (ng/g)	7.0	6.2	5.7	6.3
Recovery	101	104	101	102.2
RSD	4.5	4.4	5.2	4.7
Linearity	0.9995	0.9998	0.9998	0.9997

	<u>Glufosinate</u>			
MDL (ng/g)	2.4	2.3	2.3	2.3
LOQ (ng/g)	8.0	7.5	7.5	7.7
Recovery	101	102	101	101.4
RSD	3.2	3.4	2.8	3.1
Linearity	0.9997	0.9996	0.9999	0.9997

	<u>AMPA</u>			
MDL (ng/g)	2.0	1.1	1.4	1.7
LOQ (ng/g)	6.5	3.7	4.5	5.5
Recovery	43	15.4	19.0	31.2
RSD	18.9	23.9	16.0	17.5
Linearity	0.9327	0.9990	0.9997	0.9662

ARL provided their instrument proficiency data. I summarized in the table below. Great job Richard!

	<u>Glyphosate</u>	<u>Glufosinate</u>	<u>AMPA</u>
Lq (ng/ml)	0.2	0.3	0.2
Accuracy	100.3	99.8	100.5
Precision	6.3	1.9	11.9
Linearity	0.9970	0.9999	0.9985

Richard injected standards at 1, 2, 5, and 10 ng/ml for the Lq data. The Lq for each level was fairly consistent. The Lq was calculated per the ORA-LAB.10 instructions; i.e. the lowest level that meets the S/N requirements of 10*S/N of the quantitation ion OR 3*S/N of the confirmation ion. Accuracy and precision were calculated from 8 replicates on the injection of a 50 ng/ml standard. And linearity correlation coefficient was calculated from standards injected at 1-200 ng/ml.

From: [Sack, Chris A](#)
To: [Cromer, Michele](#)
Subject: RE: columns for glyphosate
Date: Wednesday, February 01, 2017 3:09:00 PM
Attachments: [Glyphosate method 1-31-17.docx](#)

Hi Michele,

Hope all is well with you and your family. Both of the columns are listed in the attached method.

Have a great day,

Chris

Ph: 240-402-2464

From: Cromer, Michele

A large, bold, black sans-serif font spelling 'ORA' is centered on a light gray rectangular background.

From: Cromer, Michele

A large, bold, black sans-serif font spelling 'ORA' is centered on a light gray rectangular background.

From: [Sack, Chris A](#)
To: [Mercer, Gregory E](#)
Subject: Spiking protocol for collab
Date: Thursday, February 02, 2017 1:24:00 PM
Attachments: [Collab-Glyphosate 2-3-16.xlsx](#)
[Glyphosate method 1-31-17.docx](#)

Hi Greg,

Would you mind reviewing the attached spiking protocol and let me know what you think? It is just a rude draft and I want to run it by you first, then take it to Eugene for his thoughts. Would love to discuss if/when you have a minute. I have also attached the latest version of the method which includes my standard prep for routine analysis.

I am sure you will have questions,

Chris

From: [Mercer, Gregory E](#)

ORA

From: Mercer, Gregory E

ORA

From: Sack, Chris A
Sent: Thursday, February 02, 2017 11:24 AM
To: Mercer, Gregory E
Subject: Spiking protocol for collab

Hi Greg,

Would you mind reviewing the attached spiking protocol and let me know what you think? It is just a rude draft and I want to run it by you first, then take it to Eugene for his thoughts. Would love to discuss if/when you have a minute. I have also attached the latest version of the method which includes my standard prep for routine analysis.

I am sure you will have questions,

Chris

From: Sack, Chris A
To: [Masse, Claude](#)
Subject: FW: Glyphosate Update
Date: Wednesday, February 08, 2017 7:10:00 AM

Hi Claude,

Can you send me your LCMS proficiency results in a xls file?

Thanks,

Chris

Ph: 240-402-2464

From: Sack, Chris A
Sent: Monday, February 06, 2017 10:43 AM
To: Masse, Claude
Cc: Viner, Marianna; Islam, Mohammed R
Subject: RE: Glyphosate Update

Hi Claude,

Chroms look pretty good. When you are finished with the instrument proficiency download the results into a xls file and send it to me.

Thanks,

Chris

Ph: 240-402-2464

From: Masse, Claude

The image shows the letters 'ORA' in a large, bold, black, sans-serif font. The letters are set against a solid grey rectangular background. The 'O' is a simple circle, the 'R' has a vertical stem and a curved top, and the 'A' is a simple triangle with a horizontal bar. The overall appearance is clean and professional.

From: Sack, Chris A
Sent: Monday, February 06, 2017 11:17 AM
To: Masse, Claude
Subject: RE: Glyphosate Update

Hi Claude,

Did you have an attachment?

Chris

Ph: 240-402-2464

From: Cooke, William

ORA

From: Sack, Chris A

Sent: Wednesday, February 08, 2017 5:26 AM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Noonan, Gregory; Thompson, Richard L.; Vonderbrink, John; Wong, Jon

Subject: Glyphosate method and collab

Hi everyone,

Re the method and collab I heard back from CFSAN, ARL and LA, and the attached method and collab protocol has been updated. The collab protocol is fairly prescriptive, so let me know your thoughts.

Re the LCMS proficiency, I have data from ARL and PNW only. I have not received any method proficiency data from anyone. We cannot begin the collab until we have acceptable LCMS and method proficiency data from at least 3 labs.

Thanks,

Chris

From: [Sumter, Jeffery](#)
To: [Sack, Chris A](#)
Subject: RE: Herbicides Assignment
Date: Wednesday, February 08, 2017 8:30:58 AM

Thanks for the update, sounds like it all working out.

Happy New Year.

From: Sack, Chris A
Sent: Wednesday, February 08, 2017 9:12 AM
To: Sumter, Jeffery
Cc: Islam, Mohammed R
Subject: RE: Herbicides Assignment

Hi Jeffery,

If all goes well I think we can start up the first of March. We are hoping to start the national method collaboration soon. I should have enough data to prepare a partial collaboration report before the end of Feb. We will need to revise the acid herbicide to change the analytical labs where the glyphosate samples are shipped and analyzed. That is an ORA decision, I am working with them to sort that out.

Happy new year,

Chris

Ph: 240-402-2464

From: Sumter, Jeffery
Sent: Wednesday, February 08, 2017 8:02 AM
To: Sack, Chris A
Subject: RE: Herbicides Assignment

Hello Chris,

How are things going with the Herbicides Assignment?

From: Sack, Chris A
Sent: Thursday, September 22, 2016 12:28 PM
To: Sumter, Jeffery; Islam, Mohammed R
Subject: RE: Herbicides Assignment

Hi Jeffery,

It was good to visit with you. What if you change January 2017 to "calendar year 2017". And add a statement indicating that (b) (5) [REDACTED] (b) (5) [REDACTED] It's just a suggestion but it allows us some wiggle room for completion and approval of the collaboration.

Thanks,

Chris

Ph: 240-402-2464

From: Sumter, Jeffery
Sent: Thursday, September 22, 2016 11:09 AM
To: Sack, Chris A; Islam, Mohammed R
Subject: Herbicides Assignment

Hello,

A decision has been made to hold the Herbicides Assignment in abeyance until January 2017. CFSAN will draft a memo that explains the reason for the abeyance (i.e., updated multi-lab methodology), effective date (i.e., October 1), and a NLT date for resuming the assignment.

Is there any additional information you would like to add? Are you okay with resuming the assignment NLT January 15? (or suggest another date).

Thank you,

Jeffery

From: [Sack, Chris A](#)
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Noonan, Gregory](#); [Thompson, Richard L.](#); [Vonderbrink, John](#); [Wong, Jon](#)
Subject: FW: Glyphosate method and collab
Date: Thursday, February 09, 2017 9:31:00 AM
Attachments: [image004.png](#)
[AMPA IS filter.PNG](#)
[Glufosinate IS filter.PNG](#)
[Glyphosate Filter.PNG](#)
[Glyphosate IS filter.PNG](#)

FYI. Good work, Bill. Evidently the GD/X and nylon filters retain glyphosate. Is anybody using PVDF filters? I believe most labs use PTFE for pesticides, correct? If that is the case, it looks like the results from the PTFE filters are OK.

Chris

Ph: 240-402-2464

From: Cooke, William

A large, bold, black sans-serif font spelling out the letters 'O', 'R', and 'A' in sequence. The letters are set against a solid gray rectangular background that occupies the central portion of the email body.

From: Sack, Chris A
Sent: Wednesday, February 08, 2017 7:34 AM
To: Cooke, William
Subject: RE: Glyphosate method and collab

Hi Bill,

I need to see some spike recovery data from PNW before you start the collab. One spike from each collab matrix would be fine. You can use the solvent standard calibration at the same level as the spike. Just need evidence that you can run the method and get satisfactory results.

Thanks,

Chris

Ph: 240-402-2464

From: Cooke, William

The image shows the word "ORRA" in a large, bold, black, sans-serif font. The letters are set against a solid gray rectangular background. The 'O' is a simple circle, the 'R' has a thick vertical stem and a curved top, and the 'A' is a simple triangle with a horizontal bar. The overall appearance is clean and professional.

From: Sack, Chris A

Sent: Wednesday, February 08, 2017 5:26 AM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Noonan, Gregory; Thompson, Richard L.; Vonderbrink, John; Wong, Jon

Subject: Glyphosate method and collab

Hi everyone,

Re the method and collab I heard back from CFSAN, ARL and LA, and the attached method and collab protocol has been updated. The collab protocol is fairly prescriptive, so let me know your thoughts.

Re the LCMS proficiency, I have data from ARL and PNW only. I have not received any method proficiency data from anyone. We cannot begin the collab until we have acceptable LCMS and method proficiency data from at least 3 labs.

From: [Chamkasem, Narong](#)

ORA

From: Sack, Chris A

Sent: Thursday, February 09, 2017 3:56 PM

To: Chamkasem, Narong; Chang, Eugene; Cooke, William; Islam, Mohammed R; Masse, Claude; Mercer, Gregory E; Noonan, Gregory; Thompson, Richard L.; Vonderbrink, John; Wong, Jon

Subject: RE: Glyphosate method and collab

Narong filtered 2 ml at 100ng/ml vs Bill filtered 300ul at 25 ng/ml. Bill, I assume you prepared the standard in the extraction solvent? Maybe the affect can only be seen at lower levels? That's fairly common.

Chris

Ph: 240-402-2464

From: Chamkasem, Narong

ORA

ORA

From: Chamkasem, Narong

ORA

From:

[Cooke, William](#)

ORA

From: Sack, Chris A
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Noonan, Gregory](#); [Thompson, Richard L](#); [Vonderbrink, John](#); [Wong, Jon](#)
Subject: Glyphosate proficiency
Date: Friday, February 10, 2017 2:08:00 PM

Hi Everyone,

Just a quick update about the progress of the glyphosate method lab proficiency results. To date I have received complete instrument proficiency data from ARL and PNW and partial data from NRL. The only method proficiency I have received came from PNW – see below. PNW calculated the AMPA with and without the IS, and the N-acetyl glyphosate with and without the glyphosate IS – see data below. Good Job, Bill! The AMPA obviously improves significantly with IS. The N-acetyl glyphosate improved slightly. Do we want to consider using the AMPA IS? Does everyone have the AMPA IS? Tell me what you think. It looks like carrot recoveries were slightly lower; it will be interesting to see if this is typical for all the labs.

We need to get the collab started. At this time LA and PNW are the only labs ready to collab. We need at least one more lab to provide both instrument and method proficiency data before we can begin.

Thanks and have a nice weekend,

Chris

	<u>Glyphosate</u>	<u>ISTD</u>	<u>ESTD</u>
Carrot		79.4	
Corn		98.0	
Avocado		93.3	
	<u>Glufosinate</u>		
Carrot		88.5	
Corn		97.5	
Avocado		91.7	
	<u>AMPA</u>		
Carrot		80.6	15.8
Corn		106.4	26.5
Avocado		96.1	17.4
	<u>N</u>		
	<u>acetylglphosate</u>		
Carrot		91.9	72.6
Corn		100.5	90.0
Avocado		83.5	64.9

From: [Sack, Chris A](#)
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Noonan, Gregory](#); [Thompson, Richard L.](#); [Vonderbrink, John](#); [Wong, Jon](#)
Subject: Update of instrument and method proficiency
Date: Tuesday, February 14, 2017 3:22:00 PM
Attachments: [Collab-Glyphosate 2-7-16.xlsx](#)

Hi Everyone,

Just want to give you a quick update of our progress with proficiency demonstration at each lab. I have received full instrument proficiency from 3 labs: ARL, PNW, and KAN. I have received partial instrument proficiency from NRL. Except for slightly elevated Lq for glufosinate at PNW, the instrument proficiency data indicates the LCMS method works exceptionally.

	ARL	PNW	NRL	KAN
<u><i>Glyphosate</i></u>				
Lq (ng/ml)	0.2	0.4	0.2	0.5
Accuracy	100.3	98.4		100.3
Precision	6.3	2.8		1.2
Linearity	0.9970	0.9999		0.9999
<u><i>Glufosinate</i></u>				
Lq (ng/ml)	0.3	4	0.1	0.6
Accuracy	99.8	96.2		100.2
Precision	1.9	0.7		0.6
Linearity	0.9999	0.9999		0.99999
<u><i>AMPA</i></u>				
Lq (ng/ml)	0.2	2	0.3	0.3
Accuracy	100.5	96.4		100.2
Precision	11.9	3.3		1.6
Linearity	0.9985	0.9999		0.9999
<u><i>N acetylglphosate</i></u>				
Lq (ng/ml)		6	0.3	
Accuracy		97.2		
Precision		6.7		
Linearity		0.9999		

I have received method proficiency data from only two labs: PNW and KAN. Both indicate the method works great.

	PNW*	PNW	KAN
<u><i>Glyphosate</i></u>			
Carrot		79.4	104.8
Corn		98.0	101.5
Avocado		93.3	106.1

<u>Glufosinate</u>			
Carrot		88.5	105.8
Corn		97.5	98.4
Avocado		91.7	100.6

<u>AMPA</u>				
Carrot		80.6	15.8	52.3
Corn		106.4	26.5	29.3
Avocado		96.1	17.4	62.2

<u>N</u> <u>acetylphosphate</u>			
Carrot		91.9	72.6
Corn		100.5	90.0
Avocado		83.5	64.9

* IS used for AMPA and N-acetyl

Since we have 3 labs that have demonstrated both method and instrument proficiency, we can begin the collaboration. Before we start I would like everyone to review the attached draft collaboration protocol and let me know if we can finalize the protocol. If you are OK with the protocol, please send me an email indicating you recommend no changes. Otherwise, let me know any changes you recommend.

I hope we can start the collab in three labs this week.

Thanks,

Chris

From: Sack, Chris A
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Noonan, Gregory](#); [Thompson, Richard L](#); [Vonderbrink, John](#); [Wong, Jon](#)
Subject: Glyphosate collaboration
Date: Friday, February 17, 2017 8:04:00 AM

Just a quick update. LA provided instrument proficiency data – see below.

I would like to recommend a change to the glyphosate method calibration. We had agreed to use IS calibration for the parent compounds glyphosate and glufosinate, and external standard calibration for the degradants AMPA and N-acetylglyphosate. Because we are not accounting for the final volume of the extraction I think we should use (b) (5) for all analytes. Rather than messing with the IS isotopes for the degradants, I suggest we use the (b) (5) for them. Although the glyphosate IS will not account exactly for the recovery of the degradants, it will eliminate the volume of the extract as a variable. Recovery data from SEA using both ESTD and ISTD calibration of the degradants demonstrated a significant improvement for both when the ISTD calibration was used. This will also simplify the calibration method so that all analytes are calibrated using IS calibration. Let me know what you think and I will update the collaboration to reflect this.

Thanks,

Chris

	ARL	PNW	NRL	KAN	SRL	PSW	Avg
<u>Glyphosate</u>							
Lq (ng/ml)	0.2	0.4	0.2	0.5	0.2	0.3	0.3
Accuracy	100.3	98.4	101.4	100.3	99.3	99.1	99.9
Precision	6.3	2.8	1.6	1.2	0.5	1.4	2.5
Linearity	0.9970	0.9999	0.9999	0.9999	0.9997	0.9997	0.9993
<u>Glufosinate</u>							
Lq (ng/ml)	0.3	4	0.1	0.6	1.5	0.3	1.3
Accuracy	99.8	96.2	101.4	100.2	98.9	99.8	99.3
Precision	1.9	0.7	4.7	0.6	1.0	2.3	1.8
Linearity	0.9999	0.9999	0.9996	0.99999	0.9995	0.9996	0.9998
<u>AMPA</u>							
Lq (ng/ml)	0.2	2	0.3	0.3	0.3	0.5	0.62
Accuracy	100.5	96.4	105.1	100.2	98.8	97.7	100.2
Precision	11.9	3.3	2.2	1.6	1.0	2.1	4.0
Linearity	0.9985	0.9999	0.9988	0.9999	0.9991	0.9998	0.9992
<u>N</u>							
<u>acetylglyphosate</u>							
Lq (ng/ml)		6	0.3				3.2
Accuracy		97.2	102.1				99.7
Precision		6.7	5.5				6.1
Linearity		0.9999	0.9999				0.9999

From: [MacMahon, Shaun](#)
To: [Sack, Chris A](#)
Cc: [Noonan, Gregory](#)
Subject: RE: Glyphosate collab
Date: Friday, February 17, 2017 12:00:16 PM
Attachments: [Sulfite SOP.docx](#)
[Multi-laboratory Validation Plan Sulfites.docx](#)
[MLV Proposal Review SUBMISSION FORM v1.0 JNB.pdf](#)

Hey Chris,

Attached are examples of a method SOP, MLV plan, and a completed proposal for your reference. Your spiking regime hits the requirement of at least 3 spiking concentrations in at least 3 matrices. Just a few questions: Why just the high spike in soy? Is it a recovery issue? Also, wasn't the method going to be applied to eggs and wheat? I suppose wheat and soy are similar enough, but eggs is a unique enough matrix that I could see including some spikes for that.

Happy to discuss any of this with you further.

Shaun

Shaun MacMahon, PhD
Phone: 240-402-1998

From: Sack, Chris A
Sent: Wednesday, February 15, 2017 12:46 PM
To: MacMahon, Shaun
Cc: Noonan, Gregory
Subject: Glyphosate collab

Hi Shaun,

We are almost ready for the glyphosate collab. The attached draft protocol has not been finalized by the PestAG but it is close. I just wanted to run it by to see if you have any major concerns before we finalize. It is essentially the same protocol we discussed last August, just fleshed out a little. Also, I have the MLV application form you sent me. I will need some help with that.

Thanks,

Chris

From:

[Cooke, William](#)

ORA

From: [Sack, Chris A](#)
To: [Chamkasem, Narong](#); [Chang, Eugene](#); [Cooke, William](#); [Islam, Mohammed R](#); [Masse, Claude](#); [Mercer, Gregory E](#); [Noonan, Gregory](#); [Thompson, Richard L](#); [Yonderbrink, John](#); [Wong, Jon](#)
Cc: [Cassias, Irene](#); [Eide, David J](#); [Katsoudas, Eugenia](#); [MacMahon, Shaun](#); [Sack, Chris A](#); [Podhorniak, Lynda](#)
Subject: Glyphosate collaboration
Date: Monday, February 20, 2017 12:15:00 PM
Attachments: [Collab-Glyphosate Final.xlsx](#)
[SEA Layout 2-17-17.PNG](#)
[SEA Collaboration layout 2-17-17.cset](#)
[Glyphosate method Collab Final.docx](#)
[SEA N-acetyl 2 2-17-17.docx](#)

Hi Everyone,

Bill Cooke did some work with N-acetyl glyphosate on the 6500 and found two new transitions that work better than those in the method.

Q1	Q2	RT	Transition	DP	EP	CE	CXP
210	150	4.4	N-acetyl glyphosate 1	-20	-11	-20	-13
210	63	4.4	N-acetyl glyphosate 2	-20	-11	-40	-13
210	168	4.4	N-acetyl glyphosate 3	-20	-11	-18	-13

The data and chromatograms he provided (see attached file "SEA N-acetyl 2 2-17-17.docx") clearly demonstrate the advantages of changes to the LC-MS/MS parameters. I have inserted these changes in the final method and collaboration protocol that are attached. Note I highlighted the changes in red. Note also, that I changed the transition names in the calibration method for the collab protocol – those changes are in red also. I would like everyone to try these parameters and verify they work for your instrument. Please note the DP voltage for the 5500 might be optimized at much larger levels.

Bill also analyzed some spikes using various IS calibrants for AMPA and N-acetyl glyphosate. The results tabulated below clearly indicate the benefit of using the glyphosate-13C IS for the calibration N-acetyl glyphosate. For AMPA Bill compared all three available IS isotopes. Obviously, the AMPA isotope works best, but we have already decided we will not be quantitating AMPA. The glyphosate IS appears to work satisfactorily to compensate the sample volume differences between matrices. I updated the collab protocol to use glyphosate-13C as an IS for glyphosate, AMPA, and N-acetyl glyphosate and glufosinate-D3 for glufosinate. These changes are in red also.

IS	AMPA Spike 200				N-acetyl glyphosate Spike 200	
	AMPA	Glyphosate	Glufosinate	None	Glyphosate	None
Avocado	96	22	52	17	84	65
Carrot	81	20	29	16	92	73
Corn	106	30	32	26	100	90

When I was with Bill last week, I asked him to provide me a results file formatted as directed in the collab protocol. He provided a screen shot "SEA Layout 2-17-17.png" – see attached. In his example Bill has provided all the data fields listed in the protocol along with a few extras, including Height, Ion Ratio, Accuracy, Mass Info and Area Ratio. This format is fine with me. As long as the transition masses are correct in the transition name, the Mass Info data is redundant. The other

extra fields could prove useful but are not necessary.

Collab protocol

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

SEA example

Index
Sample Name
Sample Type
Dilution Factor
Component Name
Area
IS Area
Retention Time
Actual Concentration
Calculated concentration

Some notes and observations:

(b) (5)

Thanks everyone,

Chris

From: Sack, Chris A
To: [Thompson, Richard L.](#); [Chang, Eugene](#); [Cooke, William](#)
Cc: [Mercer, Gregory E](#)
Subject: PE vs no PE
Date: Tuesday, February 21, 2017 8:17:00 AM

Hi Eugene, Richard, and Bill,

In the method I have indicated the PE cleanup is optional for fatty or dirty matrices. I forgot to include instructions in the collab protocol. What do you guys think? I was assuming everyone would use the PE cleanup for the avocado. Should I include analyses with and without PE cleanup for corn and carrot? I don't want some QA guy questioning the option down the road. If it was up to me I would add the PE cleanup to all analyses for the sake of simplicity and the extra cleanup probably wouldn't hurt recoveries of such polar analytes.

What do you think?

Chris