
From: Hinshaw, Gary - GIPSA [<mailto:Gary.D.Hinshaw@usda.gov>]
Sent: Wednesday, May 06, 2015 3:20 PM
To: Chamkasem, Narong
Subject: RE: Soybeans with incurred glyphosate

OK, I'll get you some of our highest. I'll double check prior surveys where we ran this method but I don't recall anything particularly higher. I believe the EPA tolerance is 20 ppb and the CODEX MRL is 10 ppb for soybeans.

From: Chamkasem, Narong [<mailto:Narong.Chamkasem@fda.hhs.gov>]
Sent: Wednesday, May 06, 2015 1:24 PM
To: Hinshaw, Gary - GIPSA
Subject: RE: Soybeans with incurred glyphosate

The 5 to 10 ppb is relatively low in my scale. Please give the highest concentration you have so I have no issue detecting it. I have 6500, 5500, and 4000 Q-trap and plan to use the least sensitive LC/MS (4000) for this work. I want to use it to evaluate the extraction method. I saw two extraction methods using acetonitrile with acid and acetonitrile with sodium hydroxide and acid later. Only the sample with incurred residue can be used to evaluate the extraction procedure. The fresh spike would not give much information on extraction efficiency because the analyte does not have time to be absorbed into the matrix.

Narong Chamkasem, Ph.D.
Southeast Regional Laboratory
Chemistry II/ Pesticides
Phone (404) 253 – 2302
Fax 404-253-1209
narong.chamkasem@fda.hhs.gov

From: Hinshaw, Gary - GIPSA [<mailto:Gary.D.Hinshaw@usda.gov>]
Sent: Wednesday, May 06, 2015 2:08 PM
To: Chamkasem, Narong
Subject: RE: Soybeans with incurred glyphosate

I haven't followed glyphosate controversies lately but I know there have been many. It's a pesticide I've been rather interested for many years. I was involved in selling test kits for endocrine disrupting chemicals about 12 years ago and glyphosate was the subject of some heated arguments at meetings I attended around that time.

I ran glyphosate (along with AMPA and glufosinate) in water samples at USGS and we were starting to get into air samples about the time I left that agency in 2008. We were also just beginning to try to create a method for the surfactant in Roundup, polyoxyethyleneamine (POEA).

By the way, we had a number of hits for 2,4-D in a survey completed about a year ago. They would be our freshest historical soybean samples on hand known to contain 2,4-D. The results ranged from 5 to 10 ppb. Would you like a sample on each end of our range?

Gary

From: Chamkasem, Narong [<mailto:Narong.Chamkasem@fda.hhs.gov>]
Sent: Wednesday, May 06, 2015 7:23 AM
To: Hinshaw, Gary - GIPSA
Subject: RE: Soybeans with incurred glyphosate

Thanks,

For the tolerance, it is the EPA area and will take a while before it is established. I believe we will see a lot of violation for glyphosate from now, particularly for the crops that are not in the list of 40CFR (it will be no tolerance). (b) (5), (b) (7)(E)

(b) (5), (b) (7)(E)

(b) (5), (b) (7)(E)

Narong Chamkasem, Ph.D.
Southeast Regional Laboratory
Chemistry II/ Pesticides
Phone (404) 253 – 2302
Fax 404-253-1209
narong.chamkasem@fda.hhs.gov

From: Hinshaw, Gary - GIPSA [<mailto:Gary.D.Hinshaw@usda.gov>]
Sent: Tuesday, May 05, 2015 6:03 PM
To: Chamkasem, Narong
Subject: RE: Soybeans with incurred glyphosate

Narong,

Sounds good. We'll focus on the 2,4-D soybean samples. I'll see what we have on hand tomorrow.

I think our procedures are similar for our 2,4-D method but it's been a couple of years since I ran it so I need to pull out a copy and re-familiarize myself and then I'll get back to you. We ran a modified QUECHERS method including liquid-liquid extraction as I recall. As to glyphosate, I've used FMOC in the past at another agency and TMOA/acetic acid here for derivatization. Avoiding derivatization would certainly speed up the analysis.

I've been wondering whether we'll see a change in 2,4-D levels in our surveys with the approval of the new pesticide formulation.

Gary

From: Chamkasem, Narong [<mailto:Narong.Chamkasem@fda.hhs.gov>]
Sent: Tuesday, May 05, 2015 1:59 PM
To: Hinshaw, Gary - GIPSA
Subject: RE: Soybeans with incurred glyphosate

First priority is sample with incurred 2,4 D (soybean, corn, and/or wheat) so I can evaluate my extraction method. On the internet, they recommend to add sodium hydroxide to break the binding 2,4 D from the matrix then add acid later to neutralize before phase separation. Are you using this method? The new GMO soybean developed by Dow Agro Science called Enlist Duo soybean that has resistance to glyphosate and 2,4 D (see the link below)

<http://www.ewg.org/release/half-million-people-oppose-dow-s-toxic-herbicide-mix>

Normally, glyphosate will use pre-column derivatization with FMOC to make it stick to a reversed-phase column. I have a mixed-phase mode that does not need derivatization. This way, I can shake and shoot the sample extract. The same column can be used to analyze 2,4 D as well with different mobile phase (isocratically). This way, I can separately extract glyphosate and 2,4 D from the same sample using two extraction methods and inject on to the same column with two different isocratic modes overnight to get the data the next day.

At this point, you can give me the 2,4 D samples say about 500 g each (ground or whole seed)