6-22-94

MEMORANDUM

SUBJECT: **Chlorpyrifos.** Confined Rotational Crop Study. Reregistration Case No. 0100 Chemical No. 059101 MRID #43210801 DP Barcode D203434 CBRS #13710

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The Chlorpyrifos Registration Standard Guidance Document was issued 9/28/84 and the SRR was issued 11/18/88. In support of the reregistration of chlorpyrifos, DowElanco, the registrant, has submitted a confined rotational crop study (MRID #43210801). This study is reviewed below.

The nature of the residue in plants and animals is adequately understood. The residue of concern is chlorpyrifos per se (Federal Register, Final Rule, 4/14/93). Revised tolerances reflecting residues of chlorpyrifos per se (O,O-diethyl O-(3,5,6-trichloro-2-pyridil)phosphorothioate for the racs listed in 40 CFR §180.342 (a) and (b); and 40 CFR §185.1000 appear in the Federal Register, 4/14/93. Metabolites of chlorpyrifos (CHL) include 3,5,6-trichloro-2-pyridinol (TCP) and 3,5,6-trichloro-2-methoxypyridine (TMP).

Recommendations

When chlorpyrifos was applied to soil at less than the maximal seasonal application rate (4.8 lb ai/A, 0.8X), at the 30 day plant back interval TRR levels in all the rotational crops examined exceeded 0.010 ppm and chlorpyrifos per se was found at up to 0.009 ppm. Therefore, field rotational crop trials (Guideline 165-2) will be required to support a 30 day plant back interval. When conducting field rotational crop trials, an application rate of

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6.0 lb ai/A should be used. Samples should be analyzed for residues of chlorpyrifos per se.

If the registrant does not wish to conduct field rotational crop trials to support a 30 day plant back interval, a 132 day plant back interval would be appropriate.

Conclusions

1. The test material has been adequately described. 2,6-^{14}C-chlorpyrifos, with a specific activity of 34.3 mCi/mmol and >98% radiochemical purity was used in the study.

2. CBRS concludes that the test system was adequately described. The application rate to soil in the test plots was 4.8 lb ai/A. The desired application rate was 5.0 lb ai/A. The registrant stated this application rate reflected the maximum intended use rate for chlorpyrifos. CBRS notes that use rates for the 50 WP formulation (Lorsban 50W, EPA Reg. No. 62179-39) permit application to the Brassica crop group at up to 6.0 lb ai/A/season.

2.a. Carrot, lettuce, and wheat crops were planted at three intervals, 30 days after soil treatment (DAST), 120-DAST, and 132-DAST. The 120-DAST planting was terminated after a heater malfunction in the screenhouse caused the temperature to rise to 100 F, causing irreversible wilting of the plants. The crops were replaced by the 132-DAST planting.

2.a. Crops harvested included immature wheat (tiller stage) and carrots (one-half maturity) [for both the 30- and 132-DAST crops] and mature crop harvests of wheat, carrots, and lettuce. The mature wheat harvest occurred when wheat plants were dry and the grain was mature. Grain spikes were cut from the plant, and after separation, chaff was combined with the straw. Carrots were harvested at one-half and full maturity by removing the entire plant from the soil. Lettuce was harvested at full maturity.

3. CBRS concludes that the extraction/fractionation procedures used have been adequately described. The registrant has adequately demonstrated the recovery of ^{14}C-CHL through the extraction scheme used in this study. For all matrices examined, CHL is recovered almost exclusively (>86%) in the initial acetone:water extract. Additionally, CHL is almost completely retained by the C_{18} SPE column and is eluted by 100% MeOH.

4. TRR - Except for wheat forage, there was little difference in TRR levels between crops planted at 30 versus 132 days after soil treatment. In some cases (wheat straw, wheat grain, and carrot roots) the TRR levels in the 132-DAST samples were higher than those obtained for 30-DAST samples. For the 30-DAST samples, TRR values ranged from 0.192 ppm in carrot roots to 0.795 ppm in wheat chaff/straw. For the 132-DAST samples, TRR values ranged from 0.083 ppm in lettuce to 1.297 ppm in wheat chaff/straw.

5. Identification of Residues. For both the 30- and 132-DAST samples, radioactive residues were adequately identified/characterized.

5.a. 30-DAST Samples - The CHL metabolites TCP and/or TMP were identified in all matrices examined with the 30-DAST.

5.a.1. CHL was found in 30-DAST carrots. Levels of CHL in carrots ranged from 0.004 ppm (2.0% TRR) in mature carrot root to 0.009 ppm in both carrot top-growth (1.4% TRR) and immature carrots (1.6% TRR).

5.a.2 Lettuce/Wheat - For the 30-DAST lettuce, wheat forage, wheat chaff/straw, and wheat

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grain samples, radioactive residues in the SPE concentrate of the acetone:water extract were ≤0.010 ppm, ranging from 0.001 ppm (0.3% TRR) in wheat grain to 0.010 ppm (1.5% TRR) in wheat forage. Because of the low levels of radioactivity, characterization of residues was not attempted.

5.b. 132-DAST Samples
5.b.1. Carrots - Although levels of radioactive residues were <0.010 ppm in the SPE concentrate of the acetone:water extract, CHL was not detected (<0.002 ppm) in mature carrot root, mature carrot root peels, mature peeled carrot root, carrot top-growth, and immature carrot. TMP was the major metabolite identified in the SPE concentrate fraction, ranging from 7% TRR (0.026 ppm) in carrot top-growth to 54.4% TRR (0.236 ppm) in carrot peels.

5.b.2 Lettuce/Wheat - For the 132-DAST lettuce, immature wheat forage, and mature wheat grain samples, radioactive residues in the SPE concentrate of the acetone:water extract were ≤0.010 ppm. For the mature wheat straw/chaff sample, radioactive residues in this fraction were 0.019 ppm, however, CHL and TMP were not detected by HPLC analysis.

6. Storage Stability - Samples were stored frozen on dry ice or at -20 C from the time of collection to the time of analysis. Most samples were oxidized within 2 weeks of harvest for TRR determination. Most samples were also extracted and chromatographically characterized within 3 weeks of harvest. Additionally, two separate extractions were performed on eight different plant tissues at least 5 months apart. The results for these two extractions were qualitatively and quantitatively similar. The chlorpyrifos SRR concluded that residues of CHL are stable for up to 15 months in/on frozen root/bulb crops and are stable for up to 27 months in/on frozen corn grain, forage, and fodder.

Detailed Considerations

The in-life portion of the study was conducted by Plant Sciences, Inc., Watsonville, CA, and the analytical phase was conducted by Hazleton Wisconsin, Inc. (HWI), Madison, WI.

Test Material

2,6-^{14}C-chlorpyrifos, with a specific activity of 34.3 mCi/mmol and >98% radiochemical purity was used in the study. One mg (1 mg) of the test material was diluted with 13.6 mg of cold chlorpyrifos, and 4 mL of acetone were added. This mixture was analyzed by HPLC immediately after mixing and then 22 hours later to determine the stability in acetone. There was no degradation over the 22 hours (HPLC chromatogram and integration data provided), indicating that the test material would be stable under the formulation conditions used during application. The specific activity of the test material was experimentally determined to be 15,522 dpm/ug.

CBRS concludes that the test material has been adequately described.

Test System

Lettuce (Royal Green), carrots (Blaze) and wheat (Azna) were used as the test crops. For each of the test crops, four study plots (3.0 ft. x 2.5 ft.) were constructed of plywood and lined with polyethylene. Each plot was filled with sandy loam soil. Plots were segregated outdoors, with control plots isolated from treated pots. Designated plots were appropriately treated (see

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below). All plots remained fallow in there respective enclosures until planting. At each planting, control and treated plots were moved into separate climate controlled screenhouses.

The test material (0.2717 g ^{14}C-chlorpyrifos and 3.16 g of "cold" chlorpyrifos, for a total of 3.43 g of test material) was received at the test site as a frozen solid and was stored frozen until preparation of the application solution. The entire dose of test material was reconstituted with 1 L acetone. Aliquots (110 mL) were volumetrically transferred to amber glass bottles. Aliquots were removed to determine concentration and homogeneity of the test solutions.

At each treatment test plot, a hand trigger sprayer was screwed onto the top of the amber bottles and the test solution was applied to the plot. After application, the bottles were rinsed with acetone, with the rinsate also being sprayed onto the test plot.

The desired application rate was 5.0 lb ai/A. The registrant stated that this rate reflected the maximum intended use rate for chlorpyrifos. CBRS notes that use rates for the 50 WP formulation (Lorsban 50W, EPA Reg. No. 62179-39) permit application to the Brassica crop group at up to 6.0 lb ai/A/season

The actual application rate calculated from the quantity of CHL in the test material application solution (24.0 mCi) was 4.8 lb ai/A. To verify the treatment rate, prior to application, three petri dishes were placed below the soil surface in each test plot, with the top edge of the dish flush with the surface. After application, the dishes were removed, frozen, and shipped to the analytical lab for soil analysis. The application rate as calculated from soil analysis indicated an application rate of 7.2 lb ai/A. The registrant explained that this result may be artificially high, perhaps because the applicator unknowingly focused spray solution towards the Petri dishes in the soil.

Crops were planted at three intervals, 30 days after soil treatment (DAST), 120-DAST, and 132 DAST. The 120 DAST planting was terminated after a heater malfunction in the screenhouse caused the temperature to rise to 100 F, causing irreversible wilting of the plants. The crops were replaced by the 132 DAST planting.

Immature wheat (tiller stage) and carrots (one-half maturity) were harvested for both the 30- and 132-DAST crops. Mature crop harvests of wheat, carrots, and lettuce were taken at the 30 and 132-DAST harvest intervals. During the tiller stage, harvest at one-half and at full maturity, one third of the crop was harvested by cutting the immature green plants above the soil. The mature harvest occurred when wheat plants were dry and the grain was mature. Grain spikes were cut from the plant, and after separation, chaff was combined with the straw.

Carrots were harvested at one-half and full maturity by removing the entire plant from the soil. Roots were brushed and rinsed free of soil. The one-half maturity plants remained intact as one sample, whereas with the full maturity carrots, foots were cut off from the foliage to yield two samples.

Lettuce was harvested at full maturity. Plants were cut above the soil surface, and leaves touching the soil were removed.

Samples of lettuce, carrot tops and roots, and wheat straw and chaff were processed in a commercial food processor using dry ice. Wheat grain samples were ground in a small mill. Processed samples were stored frozen in labeled glass bottles until extraction.

CBRS concludes that the test system was adequately described.

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Analytical Techniques

TRR Measurements - At the test site, duplicate control soil and plant samples and triplicate treated soil and plant samples were oxidized. The resulting $^{14}CO_2$ evolved was trapped in a scintillation vial containing Carbon-14 cocktail and counted by LSC. Samples having more than 100 dpm/g and a standard deviation from the mean of >10% were rehomogenized and reoxidized. Combustion efficiency and background values were determined at the beginning of each samples set.

In the analytical laboratory TRR determinations were made using a Packard Tri-Carb Model 1600CA, 1900TR, or 2500TR. All plant and soil samples were analyzed by LSC in triplicate for a maximum of 5 minutes or 100,000 counts. Analyses were repeated using fresh samples if relative standard deviations for sample replicates were >10%. Samples generated as a result of extraction analysis were analyzed in duplicate or triplicate for 2 to 20 minutes.

HPLC - A Perkin Elmer Series 250 or 410 HPLC equipped with a Perkin-Elmer LC95 UV/vis detector and a Perkin-Elmer LCI-100 Integrator was used for HPLC analyses. A Gilson 230 or ISCO Foxy fraction collector was used to obtain fractions for analysis by LSC. Two types of columns (Radial Pak RP-18 or Radial-Pak Amino Bond) and five different solvent systems were used. The amount of radioactivity injected onto the column was determined by counting a separate aliquot. Appropriate standards were analyzed with all samples. Representative chromatograms for standards were provided.

TLC - One dimensional TLC was used for analysis of the TCP metabolite in wheat chaff/straw. Whatman 60A, K6F plates were developed using heptane:acetone:acetic acid (77:20:3). Migration was measured with standards observed using UV light and by imaging using an AMBIS Radioanalytic Imaging System.

GC/MS - GC/MS analysis of TMP was performed using a HP 5890 GC coupled to a HP 5970 MS. GC/MS analysis of TCP was conducted using a Fisons 8065 GC and a Quattro MS.

CBRS concludes that the analytical techniques used were adequately described.

Reference Standards

The following reference standards were all supplied by DowElanco: chlorpyrifos (99.9%), 3,5,6-trichloro-2-pyridinol (TCP, 99.8%); 3,5,6-trichloro-2-methoxypyridine (TMP, 99.9%). D-glucose phenylosazone and ^{14}C-D-glucose phenylosazone (both 99%) were supplied by HWI.

Extraction and Fractionation

Acetone:water was added to samples, which were then homogenized and centrifuged. This procedure was repeated two additional times and all extracts were pooled. The remaining solids were then extracted in a similar manner with water. Therefore, three fractions were produced: acetone:water extract; water extract; and PES. The water extract was analyzed by LSC and HPLC.

An aliquot of the acetone:water extract was removed for LSC. This extract was then passed through a C_{18} SPE column. The SPE flow through fraction (containing polar compounds) was concentrated and analyzed by HPLC. The column was eluted with 100% MeOH and the eluate

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was analyzed by HPLC. The flow through fraction was further fractionated as follows: after concentration, NaOH was added until the sample was approximately 1.0 M NaOH, the sample was then refluxed, cooled, neutralized by addition of HCl, and extracted with EtOAc (3-4 times). The EtOAc extracts were combined and analyzed by LSC and HPLC. The remaining aqueous extract was also analyzed by LSC and HPLC.

An aliquot of the PES was analyzed by combustion analysis and the remainder was subjected to base hydrolysis. The PES were hydrolyzed with 1.0 M NaOH at reflux for 2-4 hours, the sample was then cooled, neutralized by addition of HCl, extracted with EtOAc (3-4 times), and centrifuged. The EtOAc extracts were combined and analyzed by LSC and HPLC. The remaining pellet was extracted with water (1-2 times) and this extract was combined with the aqueous extract remaining from the EtOAc extraction. The combined aqueous extract was analyzed by LSC and HPLC. The remaining PES were dried and an aliquot was removed for combustion analysis. For some samples, the PES were further analyzed for cellulose [Van Soest Method ($KMnO_4$/t-butyl alcohol used to oxidize/dissolve non-cellulose components, oxalic acid/HCl in ethanol used to demineralize and wash cellulose fibers), followed by final ethanol (80%) rinse)] and lignin [Honeycutt/Adler Method (PES sample autoclaved and sonicated with 2.5 M NaOH, filtered, filter cake rinsed with hot 2.5 M NaOH, filtered rinsed with water - the three supernatants were put into separate flasks, concentrated HCl was added, and lignin precipitated). Starch was isolated by high speed blending of PES with DMSO:water (9:1), the sample was allowed to stand overnight with occasional mixing. Anhydrous EtOH was added to cloud the supranatant. The sample was centrifuged and the supranatant decanted, and starch precipitation was repeated additional times. Cellulose and lignin were also isolated from various matrices/extracts using a 2 M HCl:1,4-Dioxane (1:9) extraction procedure. After mixing with HCl:dioxane, samples were placed in a 70 C water bath for about 5 hours. The sample was filtered hot and the supernatant removed. The precipitate (cellulose) was washed with hot HCl:dioxane, followed by three washes with water, dried, and oxidized. To isolate lignin, the supranatant and HCl:dioxane rinses were concentrated, followed by addition of anhydrous ethyl ether and vigorous shaking in a 39 C water bath. Samples were then centrifuged and the supranatant removed. Fresh anhydrous ethyl ether was added to the precipitated lignin and the extraction procedure was repeated.

Preparation of Osazones - Osazones were prepared from cellulose and starch samples as follows. Cellulose was broken down into individual sugar moieties by treatment with chilled 70% H_2SO_4. Following digestion, samples were neutralized to pH 6 with 12 M NaOH. Starch was broken down to monomers by reflux in 0.4 N H_2SO_4, followed by adjustment to pH 6 with 1.2 M NaOH. Monomerized solutions were derivatized with phenylhydrazine, dried, brought up in MeOH, and analyzed by HPLC. For some samples, osazones were recrystallized prior to HPLC and MS analysis.

CBRS concludes that the extraction/fractionation procedures used have been adequately described.

Results

Validation of Analytical Procedures for Recovery of CHL

Validation of Extraction Method

Control plant tissues were fortified with ^{14}C-CHL and extracted as described above. Table 1 summarizes the results for recoveries of the radiolabeled material in the various fractions. The vast majority of the radiolabeled material was found in the acetone:water extract. A

representative HPLC radiohistogram was provided for carrot root and demonstrated that only CHL was present.

Table 1. Recovery of ^{14}C-CHL from various matrices. Homogenized samples were fortified prior to extraction as described above.

Matrix	Fortification Level (ppm)	Acetone: Water Extract (% recovery)	Water Extract (% recovery)	PES (% recovery)	Total Percent Recovery
Carrot Root	0.023	97.5	0.2	0.7	98.4
Carrot Top	0.047	87.3	0.4	1.7	89.4
Immature Carrot	0.038	88.9	0.6	9.1	98.6
Lettuce	0.019	94.4	1.0	2.5	97.9
Wheat Grain	0.022	91.4	0.8	4.1	96.3
Wheat Forage	0.047	86.0	1.7	7.7	95.4
Wheat Chaff/Straw	0.022	94.8	0.8	2.0	97.6

Validation of SPE Method

The recovery of ^{14}C-CHL during the SPE concentration step was demonstrated by passing the acetone:water extract described in the preceding paragraph through a equilibrated SPE cartridge. The SPE column was eluted with 100% MeOH and recovery results are summarized in Table 2. Representative HPLC radiohistograms were provided and demonstrated that only parent compound was present.

Table 2. Recovery of ^{14}C-CHL following SPE concentration step. An aliquot of the acetone:water extract was applied to an equilibrated SPE cartridge and then eluted with 100% MeOH.

Matrix	Fortification Level (dpm)[a]	SPE Flow Through % Recovery	MeOH Eluate % Recovery	Total Percent Recovery
Carrot Root	13,550	1.1	99.9	101.0
Carrot Top	24,500	0.6	101.8	102.4
Immature Carrot	49,350	1.5	98.1	99.6
Lettuce	25,950	1.5	94.0	95.5
Wheat Grain	12,270	0.8	98.3	99.1
Wheat Chaff/Straw	14,200	0.7	102.0	102.7

[a] Acetone:water extracts from validation of extraction method (see Table 2 above). Dpm represents activity in a 50 mL aliquot of extract.

Validation of Base Hydrolysis Method

The recovery of CHL equivalents after base hydrolysis was demonstrated after hydrolyzing refortified control plant non-extractable residues with 1.0 M NaOH under reflux conditions for 2-4 hours and extracting the hydrolysate with EtOAc. CHL was hydrolyzed to TCP following exposure to the alkaline conditions and heat. The results for the hydrolysis and subsequent extraction are summarized in Table 3. Representative HPLC radiohistograms were provided and demonstrated that only TCP was present, no CHL was detectable.

Table 3. Recovery of CHL equivalents after base hydrolysis. All ^{14}C-CHL was hydrolyzed to TCP.

Matrix	Fortification Level (dpm)[a]	EtOAc Extraction % Recovery	Aqueous Fraction % Recovery	Total Percent Recovery
Carrot Top	60,188	94.7	0.2	94.9
Immature Carrot	60,562	94.4	0.5	94.9
Wheat Grain	59,928	93.9	0.2	94.1
Wheat Forage	63,127	94.3	1.9	96.2
Wheat Chaff/Straw	59,871	88.4	0.0	88.4

[a] Control samples were extracted and resulting PES pellets were fortified with indicated amounts of ^{14}C-CHL prior to hydrolysis/extraction.

CBRS concludes that the registrant has adequately demonstrated the recovery of ^{14}C-CHL through the extraction scheme used in this study. CHL is recovered almost exclusively in the initial acetone:water extract, is retained on the SPE column and eluted with 100% MeOH.

TRR Determinations

The results for the TRR determinations for samples are summarized in Table 4. For the 30-DAST samples, TRR values ranged from 0.192 ppm in carrot roots to 0.795 ppm in wheat chaff/straw. For the 132-DAST samples, TRR values ranged from 0.083 ppm in lettuce to 1.297 ppm in wheat chaff/straw.

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Table 4. Results of TRR analyses for plant samples at indicated plant back intervals. Soil was treated with ^{14}C-chlorpyrifos at 4.8 lb ai/A.

^{14}C Residues in Crops Planted 30 Days After Soil Treatment (DAST) Of ^{14}C-chlorpyrifos to Soil			
Crop Fraction	Harvest Date	Days from Treatment to Harvest	TRR (ppm)
Lettuce	09/22/92	75	0.232
Immature Carrot	09/25/92	77	0.537
Carrot roots	11/25/92	138	0.192
Carrot Tops	11/25/92	138	0.610
Wheat Forage	09/08/92	60	0.659
Wheat Grain	11/25/92	138	0.295
Wheat Straw/Chaff	11/25/92	138	0.795
^{14}C Residues in Crops Planted 132 Days After Soil Treatment (DAST) Of ^{14}C-chlorpyrifos to Soil			
Crop Fraction	Harvest Date	Days from Treatment to Harvest	TRR (ppm)
Lettuce	01/12/93	187	0.083
Immature Carrot	02/03/93	209	0.375
Carrot Roots	03/25/93	259	0.279
Carrot Root Peel	03/25/93	259	0.434
Peeled Carrot	03/25/93	259	0.171
Carrot Tops	03/25/93	259	0.375
Wheat Forage	12/28/92	172	0.262
Wheat Grain	04/05/93	270	0.433
Wheat Straw/Chaff	04/05/93	270	1.297

Identification/Characterization of Radioactive Residues

30-DAST Samples

CHL was only found in 30-DAST carrots, at levels indicated in Table 5. Two extractions were performed for each carrot matrix and both the extractions yielded the similar results. For the 30-DAST lettuce, immature wheat forage, mature wheat straw/chaff, and mature wheat grain, levels of radioactive residues in the SPE concentrate of the acetone:water extract were $\leq$0.010 ppm. Adequate identification/characterization of radioactive residues was achieved for all matrices examined.

Table 5. Levels of CHL found in 30-DAST carrots.

30-DAST Carrots	ppm CHL found	% TRR

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Mature Carrot Root	0.004, 0.004	2.0, 2.0
Carrot Top-growth	0.007, 0.009	1.1, 1.4
Immature Carrot	0.006, 0.009	1.2, 1.6

TCP and/or TMP were identified in all matrices examined with the 30-DAST. The other matrices examined did not contain detectable levels of CHL. Table 6 summarizes results for TCP and TMP in the 30-DAST samples.

Table 6. Levels of TCP and TMP found in 30-DAST samples.

Matrix	TCP				TMP			
	Extraction #1		Extraction #2		Extraction #1		Extraction #2	
	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm
Mature Carrot Root	10.9	0.021	10.4	0.020	26.6	0.051	25.1	0.048
Carrot Top-growth	6.4	0.039	6.4	0.039	2.7	0.017	3.9	0.024
Immature Carrot	6.6	0.035	11.2	0.061	21.1	0.113	21.4	0.115
Lettuce	5.3	0.012	ND	ND	ND	ND	ND	ND
Wheat Forage	9.4	0.062	13.3	0.091	ND	ND	ND	ND
Wheat Chaff/straw	6.0	0.048	9.2	0.066	ND	ND	ND	ND
Wheat Grain	ND	ND	0.3	0.001	ND	ND	ND	ND

Cellulose/Lignin Characterization

The PES of wheat straw/chaff were analyzed for incorporation of radioactivity into cellulose and lignin. For one extraction of 30-DAST wheat straw/chaff sample, the PES contained 48.2% TRR (0.383 ppm), of this amount, 0.106 ppm (13.3% TRR) was identified as cellulose and 0.138 ppm (17.4% TRR) was identified as lignin. For a second sample, the PES contained 46.5% TRR (0.370 ppm), of this amount, 0.148 ppm (18.6% TRR) was identified as cellulose and 0.058 ppm (7.3% TRR) was identified as lignin.

132-DAST Samples

For the 132-DAST samples, although levels of radioactive residues were <0.010 ppm in the SPE concentrate of the acetone:water extract, CHL was not detected (<0.002 ppm) in mature carrot root, mature carrot root peels, mature peeled carrot root, carrot top-growth, and immature carrot. TMP was the major metabolite identified in the SPE concentrate fraction, ranging from 7% TRR (0.026 ppm) in carrot top-growth to 54.4% TRR (0.236 ppm) in carrot peels.

For the 132-DAST lettuce, immature wheat forage, and mature wheat grain samples, radioactive residues in the SPE concentrate of the acetone:water extract were ≤ 0.010 ppm. For the mature wheat straw/chaff sample, radioactive residues in this fraction were 0.019 ppm - CHL and TMP were not detected by HPLC analysis. Adequate identification/characterization of radioactive residues was achieved for all matrices examined.

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Storage Stability

Samples were stored frozen on dry ice or at -20 C from the time of collection to the time of analysis. Most samples were oxidized within 2 weeks of harvest for TRR determination. Most samples were also extracted and chromatographically characterized within 3 weeks of harvest. Additionally, two separate extractions were performed on eight different plant tissues at least 5 months apart. The results for these two extractions were qualitatively and quantitatively similar. The chlorpyrifos SRR concluded that residues of CHL are stable for up to 15 months in/on frozen root/bulb crops and are stable for up to 27 months in/on frozen corn grain, forage, and fodder.

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