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Walter J. Krol, Ph.D., Brian D. Eitzer Ph.D., Terri Arsenault, and Jason C. White, Ph.D.
Department of Analytical Chemistry (CAES)

John Fontana, Ph.D. (HCLD) ABB, Stacey Kinney
Connecticut Department of Public Health (DPH)

Ellen Sloan
Connecticut Department of Consumer Protection (DCP)

Lora Ann Lin, Ph.D., Sara Robinson, Ph.D.
Forensic Chemistry Center (FCC), Food and Drug Administration, 6751 Steger Drive, Cincinnati, OH 45237

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Introduction

The Department of Analytical Chemistry at the Connecticut Agricultural Experiment Station (CAES), along with the Connecticut (CT) Department of Consumer Protection (DCP), has collaboratively conducted an annual market basket survey of produce sold in Connecticut for pesticide residues, and published the findings, at least in part, since 1963 (Krol *et al.*, 2006, 2011). The goals of this program continue to ensure that: 1) pesticides are used in accordance with their label and 2) the public is protected from the deliberate or accidental misuse of pesticides.

Our program has gone through significant enhancements and improvements since its inception (Krol *et al.*, 2006). The most notable improvements to the program occurred in 2006 when new methodology for the extraction of pesticide residues from produce (Anastassiades, 2003; AOAC, 2007) and liquid chromatography with mass spectrometry (LC/MS) were introduced for the analysis of these extracts. In 2010, CAES began an ongoing collaboration with the Connecticut Department of Public Health (DPH) in which samples undergoing pesticide residue analysis were also screened for potential microbial contamination (Krol *et al.*, 2011).

Initially in 2011, as part of this joint screening effort with the DPH, fourteen samples of fresh herbs were tested. All but one sample, including the one organic sample tested, were found to contain at least one pesticide residue. Notably, 74 different pesticide residues found on these fourteen samples, only 26 of the residues were permitted by the EPA tolerances (*e*-CFR, 2012). In other words, only 35% of the residues found were permissible under current US law. Of the fourteen samples tested, eleven (78.6%) contained violative pesticide residues. Based upon these findings, we chose to undertake a targeted survey of the 45 samples of fresh and dried herb commodities included in this report.

During the course of this work, the Food and Drug Administration (FDA) Forensic Chemistry Center (FCC) joined the study. Thirty-one samples in this report were split and tested concurrently at the CAES and at the FDA/FCC for pesticide residues. The FDA/FCC was interested in comparing the sensitivity of their Direct Analysis in Real Time (DART) mass spectrometer (MS) to our conventional LC/MS results.

Methods:

A) Sample Collection – Department of Consumer Protection (CT DCP)

Samples of fresh and dried herbs included in this survey were grown in the United States (US) and elsewhere in the world and collected at twelve different locations in Connecticut by an inspector from the DCP. The samples were brought to our laboratory in New Haven for pesticide residue testing. Fourteen of the forty-five samples in the current report were split upon collection by the DCP inspector. In these cases the inspector delivered half of the sample to the DPH labs in Hartford for microbial testing, and the other half to the CAES laboratory in New Haven for pesticide residue analysis. The remaining thirty one samples were delivered by the DCP inspector to our New Haven laboratory, where the samples were divided. One half of each of these samples was sent to the FDA/FCC for testing and the remainder of the sample was retained and analyzed in our laboratory. All samples in this report were collected without prior knowledge of pesticide application or potential microbial contamination. A total of twenty-one fresh and twenty-four dried samples were collected for this work.

B) Pesticide Methods – Connecticut Agricultural Experiment Station (CAES)

i. Sample Homogenization

Fresh herb samples were homogenized prior to extraction using a 3 quart robot coupe® food processor. A portion of each sample was retained in a frozen state in plastic Whirl-Pak® bags until analysis and reporting of the results were completed. Dried herb samples were mixed in their original container with a spatula, and weighed directly into Falcon Tubes for extraction. Excess sample was retained in the original container at room temperature until analysis and reporting of the results were completed. These samples were then sent to the FDA/FCC for analysis.

ii. Sample Extraction

The Quick, Easy, Cheap, Effective, Rugged, Safe (QuEChERS; pronounced "catchers") multi-residue methodology described by Anastassiades et al. (Anastassiades, 2003; AOAC, 2007) was modified for this work. For dried herbs and spices, three grams of material was weighed into a 50 mL Falcon Tube. Distilled-Deionized (DI/DI) water was added to give a final weight of 15 g. For fresh herbs and spices, five grams of material was weighed into a 50 mL Falcon Tube, and DI/DI water added to give a final weight of 15 g. [U-ring]-$^{13}C_6$-Alachlor Internal Standard (IS) (60 µL of 10 part per million (ppm) solution in toluene; i.e. 600 ng/15g), prepared from material purchased

from Cambridge Isotope Laboratories was spiked onto the samples prior to the addition of water. Anhydrous magnesium sulfate (6 g), anhydrous sodium acetate (1.5 g) and acetonitrile (15 mL) all available from Mallinckrodt Baker, Inc., were added. The mixture was shaken on a Burrell Model 75 Wrist Action Shaker (ca 1h). The mixture was centrifuged using a Thermo IEC Centra GP6 Centrifuge at 3000 rpm for 10 min to separate the acetonitrile from the aqueous phase and solids. Acetonitrile (10 mL) was decanted into a 15 mL polypropylene Falcon® centrifuge tube containing magnesium sulfate (1.5 g), together with Primary and Secondary Amine (PSA) bonded silica (0.5 g) and toluene (2.0 mL). The mixture was shaken by hand (ca 5 min) and centrifuged at 3000 rpm for 10 min. Exactly 6.0 mL of the extract was added to a concentrator tube and blown down to just under 1 mL (but not to dryness) under a stream of nitrogen at 50 °C. The concentrated solvent was reconstituted to a final volume of 1.0 mL with toluene. It should be noted that this extraction method results in a five-fold concentration of the original sample.

iii. Instrumental Analysis

Samples extracted by the QuEChERS method were concomitantly analyzed by Gas Chromatography (GC) and Liquid Chromatography (LC). For the GC analysis, an Agilent 6890 plus GC equipped with: dual 7683 series injectors and a 7683 auto sampler (collectively known as an Automatic Liquid Sampler (ALS)); Agilent model number G2397A micro Electron Capture Detector (µECD) and a 5973 Mass Spectral (MS) Detector; a Programmable Temperature Vaporization (PTV) port on the front inlet leading to the MS, and a Merlin MicroSeal® system on the rear inlet leading to the µECD; and dual J&W Scientific DB-5MS+DG (30 m x 250 µm x 0.25 µm) columns. Two microliter injections were made simultaneously onto both columns, and all data were collected and analyzed using Enhanced MSD Chemstation Software version E.02.00.493. Deconvolution and identification of pesticides in the mass spectra of samples were aided by the use of the Automated Mass spectral Deconvolution and Identification System (AMDIS) with a user constructed library.

The LC analyses were made using an Agilent 1200 High Pressure Liquid Chromatograph (HPLC) equipped with a Zorbax® SB-C18 Rapid Resolution (2.1 mm x 50 mm, 1.8µ) column using a 3µL injection volume. The mobile phase conditions were as follows: avflow rate 0.45 mL/min; gradient flow 95% A (H_2O/0.1N HCOOH) to B (100% MeOH/0.1N HCOOH) over 15 min in several steps; hold 100% B for 7 min. The column eluent was interfaced to a Thermo-Electron LTQ ion trap mass spectrometer. The mass spectrometer was operated in the positive ion electrospray mode with most pesticides being determined using MS/MS selective reaction monitoring. Data were collected and analyzed using Xcalibur® software version 2.0. Alternatively and usually concurrently, LC analyses were made employing a Thermo Scientific Exactive Orbitrap MS run by Thermo Xcalibur® version 2.1.0.1140 with ToxID® version 2.1.2. The software controlled the MS and the Agilent 1200 Series HPLC used for the chromatographic resolution. The HPLC was equipped with a Thermo Hypersil gold aQ column (2.1 mm x 100 mm x 2.1 µ); 2µL injection volume; flow rate 0.25 mL/min; column temperature 40 °C; gradient flow: initial 99% A (water with 0.1% formic acid), 1% B (acetonitrile with 1% formic acid), hold 1 min, 1-10 min 99% A to 5% A, hold 5 min, 15.1 – 21.5 min 99% A. The column eluent was interfaced to the MS. The mass resolution was set to 100,000

with balanced settings and an injection time of 20 milliseconds (ms). The mass range of 75 – 1500 atomic mass units (amu) was monitored.

iv. Detection Limit of Pesticide Residues

All pesticide residue levels are reported in parts per million (ppm) based upon the fact that the EPA tolerance levels are established using this convention. The CAES reports all pesticide residues which are confirmed by MS to an arbitrarily set limit of 0.001 ppm (one part per billion (ppb)). There are many pesticide residues seen below this level, especially using LC/MS, which are not included in this work. As part of ongoing laboratory accreditation work, limits of detection (LOD) and limits of quantitation (LOQ) for individual pesticide Active Ingredients (AI's) will be determined.

v. Reproducibility of Results

All samples examined in this work were individually homogenized, extracted and analyzed by GC and LC once. Statistical analysis obtained through inter and intra-laboratory studies over a wide range of pesticides, pesticide concentrations, and matrices have demonstrated that this is sufficient to obtain accurate quantitation of pesticide residue concentrations from the extract of a single sample (AOAC, 2007). Further proof of this was obtained in unpublished work conducted in our laboratories on violative samples. All violative samples were re-extracted, analyzed, and quantitated in duplicate using portions of the original sample retained from homogenization step. One of the duplicate samples was spiked with the pesticide(s) in question at a concentration slightly above the originally determined value. Quantitative values of these extracts were compared to the concentration found in the original analysis. High resolution (four decimal) exact mass spectra are obtained, employing the Exactive MS, as confirmation of all violative residues.

C) Microbiological Methods – Department of Public Health (CT DPH)

All samples were processed using the FDA's Bacteriological Analytical Manual (BAM, 8th Edition, Revision A, 1998). The twenty-six produce samples included in this report, and the fourteen herb samples reported elsewhere (Krol *et al.*, 2013) collected by DCP were delivered to the DPH laboratory and were processed with an amended FDA procedure. Briefly, the samples were weighed out in 1:10 aliquots and soaked in a selective pre-enrichment media. Following this pre-enrichment incubation, a Polymerase Chain Reaction (PCR) screening method, using a DuPont Qualicon BAX® detection system, was performed targeting the presence of *Salmonella* spp., *E. coli* 0157:H7 (STEC), and *Listeria monocytogenes* Deoxyribonucleic Acid (DNA). Simultaneously, conventional microbiology was performed on the enriched samples, which involved culture plating onto selective agars, Enzyme Linked Immunoassays (ELISA), biochemical and confirmation testing. All samples were streaked onto 1) Xylose lysine Deoxycholate (XLD) agar for isolation of *Salmonella* spp. colonies 2) MacConkey with Cefixime and Tellurite agar and MacConkey Sorbitol Agar for *E. coli* 0157:H7 and STEC colonies and 3) modified Oxford agar for suspect *Listeria* colonies. Any suspect colonies were further characterized using biochemical and confirmation testing. ELISA was performed for the confirmation of *Salmonella* spp. and STEC. Following identification and confirmation; all isolates were sent for Pulsed-Field Gel

Electrophoresis (PFGE) for DNA fingerprinting using the Centers for Disease Control (CDC) PulseNet protocol. The PFGE laboratory results were compared to the DNA fingerprints in both the local and national databases which contain images obtained from clinical, environmental and food isolates.

D) Dart Protocol – Food and Drug Administration / Forensic Chemistry Center (FDA/FCC)

i. Sample Preparation

A solvent mixture composed of equal parts methanol, isopropanol, ethyl acetate, hexanes, and acetone was lightly spritzed onto a fresh oregano sample, such that the surface of the sample was wetted but not dripping. A piece of polyester fabric (Polywip-C Heatseal, Contec, Spartanburg, SC) was held with tweezers and wiped over the surface of the oregano. This was repeated with ten pieces of material until the entire sample was thoroughly swabbed. Each piece of polyester was placed vertically on a DART module that was designed to orient the material such that the He beam passes directly through the center of the swab. The module was then placed on the DART rail for introduction into the MS.

ii. Mass Spectrometer Parameters

These analyses were performed using a Thermo Scientific Exactive Orbitrap mass spectrometer (Thermo Scientific, Waltham, MA, USA) in positive ion mode. Data acquisition was accomplished with Thermo Scientific Xcalibur software and data processing was undertaken using Thermo Scientific ToxID software (Version 2.1.1.56), which screens data against a user created library of compounds to determine which analytes are present based on the accurate mass of the [M+H]+ pesticide ions, as well as the [M+H+1] ions (replacing a C with a 13C) and the [M+H+2] ions (either replacing another carbon or else chlorine (37Cl), bromine (81Br), or sulfur (34S). The instrument parameters used during data acquisition were as follows: polarity, positive; scan range, m/z 120-1000; resolution, 100,000; automatic gain control (AGC) target, 1,000,000; maximum inject time, 1000 ms; acquisition time, 7.1 minutes; capillary temperature, 150 °C; capillary voltage, 44 V; tube lens voltage, 120 V; skimmer voltage, 26 V.

iii. Direct Analysis in Real Time (DART) Ionization Source

The DART source was obtained from IonSense Inc., model number SVP100. The position of the DART was set at 0 cm vertically and 6 - 8 mm horizontally. The original inlet ceramic tube of the DART was exchanged for a stainless steel tube (O.D.: 6.35 mm, I.D.: 4.60 mm, L: 42.5 mm). A linear rail ran between the DART source and the stainless steel tube. A module capable of holding ten round foam pieces was attached to the rail.

A temperature gradient method (100 – 350 °C over 7.1 minutes), was used to increase the temperature across the ten pieces of polyester. The grid voltage was set to 300 V.

Results and Discussion

In the past, extraction of herb samples followed by GC/MS analysis was problematic and extremely unproductive. Herbs, by their nature, contain high numbers of

volatile components. These volatile components are typically present in far greater amounts (by orders of magnitude) than any pesticide residue which may be present. These volatiles typically mask the presence of pesticide residues when performing an analysis using GC/MS. As described in the accompanying report (Krol *et al.*, 2013) our pesticide program has undergone numerous changes since 2006, most notably to include the use of the QuEChERS extraction protocol and the use of LC/MS to complement our GC/MS analysis of pesticide residues

The current work underscores the unproductive nature of testing herbs employing GC/MS and the impressive power of the LC/MS for the determination of residues. In this study, 2011 there were a total of 210 individual pesticide residues found on 40 of the 45 (88.9%) of the samples tested. Of these 210 residues, there were nine found by GC/MS and 208 found by LC/MS. There were only two (0.952%) residues found by GC/MS alone; permethrin (0.039 ppm) on a sample of parsley and cypermethrin (1.5 ppm) found on a sample of tarragon.

Results from DPH – CAES Samples

The current study was initiated as part of our ongoing program with the CT DPH. Our inspector from DCP initially collected two herb samples, cilantro and parsley, which were spilt between DPH and CAES. Nine pesticide residues were found on these samples. The cilantro sample was found to contain three violative residues; the parsley sample was found to contain six allowable residues.

As a follow up, our inspector collected and split an additional five herb samples between DPH and CAES. This included one sample labeled as organic. There were a total of thirty-eight residues discovered on these five samples. Of these, twenty-eight (73.7%) individual residues were found to be violative. All the samples tested were found to contain at least one violative residue. There were a total of ten residues on the organic sample, seven of them were violative.

A final sampling of seven additional samples split between DPH and CAES in 2011. A total of twenty-six different residues were found on six of the seven samples. One of the samples contained two non-violative residues, and the remaining five samples contained a total of twenty-four residues of which sixteen were found to be violative.

Of the fourteen samples of herbs split between the DPH and the CAES in 2011, eleven (78.5%) contained at least one violative residue (See Table 1). This was an extraordinarily high rate. Between 1990 and 2011, the average violation rate among all samples tested at the CAES (n=6048) was 2.95 percent. This anomaly led us to study the twenty-one fresh and twenty-four dried samples in this report.

In the current work the DPH tested for *E. coli* O157:H7, *Salmonella* spp., *Listeria monocytogenes* and Shiga-toxin producing *E. coli* (STEC). A single sample of fresh chives from Chile was found to contain *L. monocytogenes* in addition to seven pesticide

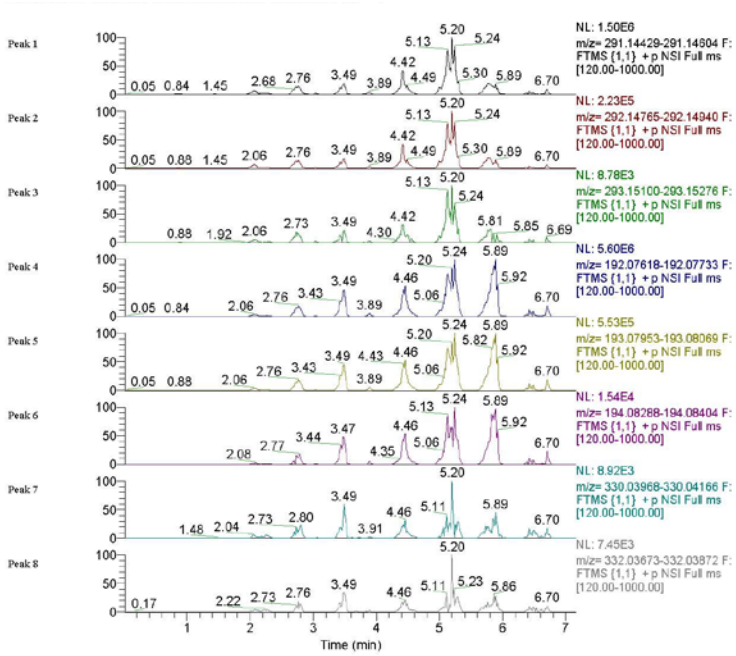
residues of which five were violative. The finding of bacterial contamination led the FDA to recall the chives.

Results from FDA-FCC – CAES Samples

We have worked with the FDA-FCC in our capacity as a Food Emergency Response Network (FERN) laboratory since 2006. In 2011, the FDA-FCC made contact with us in search of foods containing large numbers of field incurred pesticide residues. Specifically, The FDA-FCC was interested in comparing findings they obtained using a direct analysis in real time (DART) mass spectrometer (MS) with results that were obtained by a conventional extraction technique followed by LC/MS and GC/MS analysis. Owing to the exceedingly high number of residues found in herbs during the first part of this 2011 study, these commodities seemed ideal for the purpose. To this end, an additional 32 samples comprised of seven fresh and twenty-four dried herb samples were collected by our DCP inspector and divided at the CAES for analysis by the two agencies.

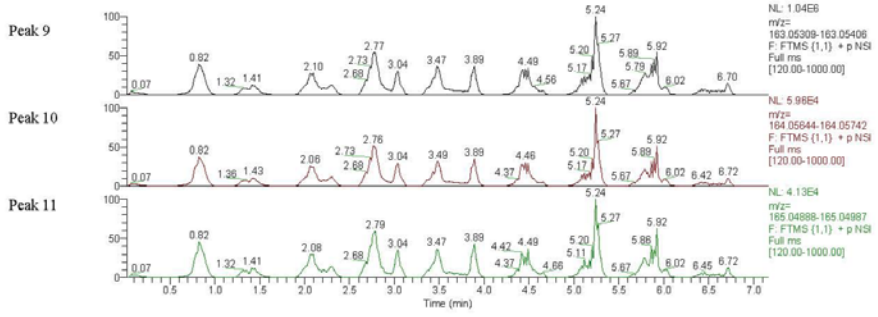
The CAES findings for these 32 samples are detailed in Tables 1 & 2. The FDA-FCC was able to confirm residues of the pesticides benomyl, carbendazim, iprodione and methomyl employing their DART instrument. Their results are included in the tables 1A and 1B below, and were presented at the American Society of Mass Spectrometry (ASMS) meeting held in Vancouver, Canada in May of 2012 (ASMS, 2012).

TABLE 1AB. Extracted Ion chromatograms of the pesticides detected employing the DART instrument.



#	Comp. Index	Compound Name	Formula	Detected m/z	Delta (ppm)	Expected RT	Actual RT	Intensity	Adducts			Fragments		
									H+	NH4+	Na+	1	2	3
1	1	Benomyl	C14H18N4O3	291.14523	0.2	0.00	5.20	1500633	Y*	-	-	-	-	-
2	2	Benomyl (M+1)	C13[13]C1H18N	292.14853	0.0	0.00	5.20	222864	Y*	-	-	-	-	-
3	3	Benomyl (M+2)	C12[13]C2H18N	293.15186	-0.1	0.00	5.20	8731	Y*	-	-	-	-	-
4	4	Carbendazim	C9H9N3O2	192.07672	-0.2	0.00	5.89	5600747	Y*	-	-	-	-	-
5	5	Carbendazim (M+1)	C8[13]C1H9N3	193.07997	-0.7	0.00	5.24	552502	Y*	-	-	-	-	-
6	6	Carbendazim (M+2)	C7[13]C2H9N3	194.08304	-2.2	0.00	5.24	15190	Y*	-	-	-	-	-
7	7	Iprodione	C13H13Cl2N3O3	330.04034	-1.0	0.00	5.20	8880	Y*	-	-	-	-	-
8	9	Iprodione (M+2)	C13H13Cl[37]C	332.03720	-1.6	0.00	5.20	7446	Y*	-	-	-	-	-

TABLE 1B.

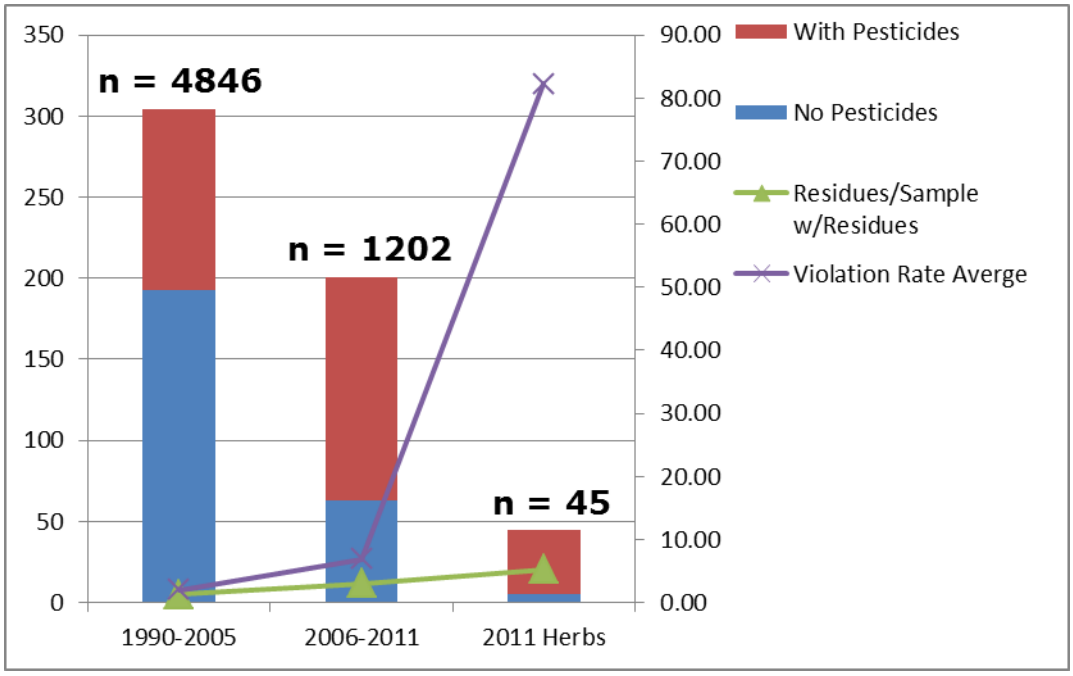


#	Comp. Index	Compound Name	Formula	Detected m/z	Delta (ppm)	Expected RT	Actual RT	Intensity	Adducts			Fragments		
									H+	NH4+	Na+	1	2	3
9	10	Methomyl	C5H10N2O2S	163.05357	-0.0	0.00	5.24	1037487	Y*	-	-	-	-	-
10	11	Methomyl (M+1)	C4[13]C1H10N2	164.05685	-0.5	0.00	5.24	59455	Y*	-	-	-	-	-
11	12	Methomyl (M+2)	C5H10N2O2[34]	165.04935	-0.1	0.00	5.24	41200	Y*	-	-	-	-	-

Combined CAES – DPH and FDA-FCC Results

Of the forty-five samples tested, forty (88.9%) contained at least one pesticide residue. This is markedly higher than the number of samples found to contain residues in any other year of our survey (average 2006 – 2010 = 67.37%). The primary factor for isolating this herb data from the 2011 market basket survey was the fact that the pesticide residue violation rate (82.2%) was dramatically different than the typical 6.86% rate encountered between 2006 and 2010 (Table 2) and also because of the targeted nature of the study with regard to commodity.

TABLE 2 Comparison of market basket results to herb results



The number of residues on the 45 herb samples tested in 2011 (5.25 residues / sample) was higher than that found in the 212 samples tested as part of our market basket survey in 2011 (3.77 residues / sample). It was also higher than the 2006-2010 'QuEChERS' average (2.8 residues / sample). The average residue level found in the herb samples was 1.05 ppm for the combined LC and GC analysis, 0.911 ppm by LC alone, and 4.24 ppm by GC alone. Between 2006 – 2011, the average residue level across all samples tested was 0.10 ppm for the combined LC and GC analysis, 0.08 ppm by LC alone, and 0.24 ppm by GC alone. There were a total of 60 different AI's found on the forty five samples of herbs tested. In 2011, there were a total of 65 different AI's found on 212 samples across all 43 different commodities. The number of different AI's found in the current report, 65, is the most ever found per annum in our survey. Furthermore as of this writing, only 48 different pesticide AI's are registered for use on herbs (Pest Chem. News Guide, 2013).

In 2009 the United States Department of Agriculture (USDA) pesticide data program (PDP) tested 184 samples of cilantro for pesticide residues. The study found at least one pesticide residue in 174 (94.6%) of the samples tested. Of these 174 samples, 125 (71.8%) samples contained at least 1 violative residue (PDP, 2011). Of the eight samples of cilantro tested in the current work, seven (87.5%) were found to contain pesticide residues. Of these seven samples, six (85.7%) samples were found to contain violative residues.

Conclusions:

Based upon our findings it is clear that the majority of the fresh and dried (domestic, imported?) herbs sold in the Connecticut marketplace contain pesticides, and the vast majority of those contain violative pesticide residues (see Tables 1 & 2). The PDP targeted study has also confirmed these findings on cilantro (PDP, 2011). There are very few pesticide AI's registered for use on herbs, likely owing to the fact of the high and likely prohibitive cost involved to register an AI on a small commodity crop such as herbs. Growers therefore may be using pesticides not registered for use in order to harvest a profitable crop.

In the past, analysis of herb samples for pesticide residues has been hampered by methodology used in the analysis. The introduction of LC/MS/MS and High Resolution LC/MS in our laboratory in recent years. In addition, the recent acquisition of a new GC Triple Quad in 2013 should continue to improve program performance.

The work presented herein is intended to inform the consumer, not to frighten or scare them. Although most of the herbs tested contain violative pesticide residues, the consumer should rest assured that herbs themselves are only a very small portion of one's diet, and play only a small part in the overall risk cup of pesticide exposure. (See note) Herbs are typically highly diluted prior to consumption, as are any pesticide residues they might contain.

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Table 1: Summary of Pesticides Found in Fresh Herbs Sold in Connecticut in 2011.

Commodity / Origin / Pesticide	Samples with Residues (Total)	Found by LC, GC or Both	Number of Times Detected	Residue Range (ppm)	Average Residue (ppm)	EPA Tolerance (ppm)
Basil (4 Samples; 2 Foreign; 2 Unknown; **3 Violations**; 4 Bacterial Analyses)						
Foreign						
Columbia	2 (2)					
Atrazine		**LC**	**2**	**0.001-0.006**	**No Tolerance**	**0**
Carbendazim (Metabolite)		**LC**	**2**	**0.054-2.200**	**No Tolerance**	**0**
Chlorothalonil		**LC**	**1**	**0.082**	**No Tolerance**	**0**
Cyprodinil		Both	1	1.134		15
Fludioxonil		Both	1	0.670		10
Imidacloprid		LC	1	0.001		8
Metalaxyl		**Both**	**1**	**0.675**	**No Tolerance**	**0**
Methomyl		**LC**	**1**	**1.157**	**No Tolerance**	**0**
Spinetoram		LC	1	0.557		3
Unknown (US)	1 (2)					
Carbendazim		**LC**	**1**	**2.100**	**No Tolerance**	**0**
Metalaxyl		**LC**	**1**	**0.002**	**No Tolerance**	**0**
Methomyl		**LC**	**1**	**0.018**	**No Tolerance**	**0**
Chives (2 Samples; 2 Foreign; **2 Violations**; 1 Bacterial Analysis)						
Foreign						
Columbia	1 (1)	L. *monocytogenes*				
Acephate		**LC**	**1**	**0.015**	**No Tolerance**	**0**
Azoxystrobin		Both	1	0.007		50
Carbendazim (Metabolite)		**LC**	**1**	**0.675**	**No Tolerance**	**0**
Difenoconazole		**LC**	**1**	**0.003**	**No Tolerance**	**0**
Fipronil		**LC**	**1**	**0.170**	**No Tolerance**	**0**
Imidacloprid		LC	1	0.003		8
Pyraclostrobin		**LC**	**1**	**0.002**	**No Tolerance**	**0**
Israel	1 (1)					
Azoxystrobin		LC	1	0.002		50
Carbendazim (Metabolite)		**LC**	**1**	**0.002**	**No Tolerance**	**0**
Thiamethoxam		**LC**	**1**	**0.018**	**No Tolerance**	**0**
Coriander / Cilantro (3 Samples; 2 Unknown; **2 Violations**; 3 Bacterial Analyses)						
Florida	1 (1)					
Acetamiprid		**LC**	**1**	**0.016**	**No Tolerance**	**0**
Atrazine		**LC**	**1**	**0.022**	**No Tolerance**	**0**
Pendimethalin		**LC**	**1**	**0.002**	**No Tolerance**	**0**
Unknown (US)	2 (2)					
Azoxystrobin		LC	1	0.001		30
Boscalid		**LC**	**1**	**0.615**	**No Tolerance**	**0**
Carbendazim (Metabolite)		**LC**	**1**	**0.008**	**No Tolerance**	**0**
Imidacloprid		LC	1	0.003		8
Prometryn		LC	1	0.004		3.5

Mint (1 Sample; 1 Unknown; **1 Violation**; 1 Bacterial Analysis)

Unknown (US) 1 (1)					
Azoxystrobin	LC	1	0.002		30
Imidacloprid	**LC**	**1**	**0.007**	**No Tolerance**	**0**
Thiamethoxam	LC	1	0.033		1.5

Marjoram / Oregano (2 Samples; 1 Foreign; **2 Violations**)

Foreign (Peru) 1 (1)					
Atrazine	**LC**	**1**	**0.001**	**No Tolerance**	**0**
Azoxystrobin	LC	1	0.003		50
Buprofezin	**LC**	**1**	**0.003**	**No Tolerance**	**0**
Carbaryl	**LC**	**1**	**0.006**	**No Tolerance**	**0**
Carbendazim (Metabolite)	**LC**	**1**	**12.00**	**No Tolerance**	**0**
Cyprodinil	LC	1	0.014		3
Difenoconazole	**LC**	**1**	**0.005**	**No Tolerance**	**0**
Emanectin Bromide	**LC**	**1**	**0.014**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.277		8
Indoxacarb	**LC**	**1**	**0.006**	**No Tolerance**	**0**
Iprodione	**LC**	**1**	**40.00**	**No Tolerance**	**0**
Metalaxyl	**LC**	**1**	**0.004**	**No Tolerance**	**0**
Methamidophos	**LC**	**1**	**0.004**	**No Tolerance**	**0**
Methomyl	**LC**	**1**	**20.00**	**No Tolerance**	**0**
Methoxyfenozide	LC	1	0.004		10
Hawaii 1 (1)					
Azoxystrobin	LC	1	0.292		50
Carbaryl	**LC**	**1**	**1.100**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.571		8
Piperonyl Butoxide	**LC**	**1**	**0.035**	**No Tolerance**	**0**
Spinetoram	LC	1	0.065		3

Parsley (1 Sample; 1 Bacterial Analysis)

Florida 1 (1)					
Azoxystrobin	LC	1	0.076		30
Flonicamid	LC	1	0.012		4
Linuron	LC	1	0.007		0.25
Malathion	LC	1	0.005		8
Permethrin	LC	1	0.039		20
Pyraclostrobin	LC	1	0.084		29

Rosemary (2 Sample; 2 Foreign; **2 Violations**)

Foreign (Mexico) 1 (1)					
Azoxystrobin	LC	1	0.002		50
Chlorpyrifos	**LC**	**1**	**0.036**	**No Tolerance**	**0**
Dimethoate	**LC**	**1**	**0.002**	**No Tolerance**	**0**
Foreign (Peru) 1 (1)					
Carbendazim (Metabolite)	**LC**	**1**	**0.046**	**No Tolerance**	**0**

Sage (1 Sample; 1 Foreign; **1 Violation**)

Foreign (Mexico) 1 (1)					
Carbendazim (Metabolite)	**LC**	**1**	**0.460**	**No Tolerance**	**0**
Fluopicolide	**LC**	**1**	**0.372**	**No Tolerance**	**0**

Forchlorfenuron	**LC**	**1**	**0.017**	**No Tolerance**	**0**
Metalaxyl	**LC**	**1**	**0.008**	**No Tolerance**	**0**
Methomyl	**LC**	**1**	**1.900**	**No Tolerance**	**0**
Oxamyl	**LC**	**1**	**0.121**	**No Tolerance**	**0**
Prometryn	**LC**	**1**	**0.086**	**No Tolerance**	**0**
Propamocarb	**LC**	**1**	**1.400**	**No Tolerance**	**0**

Tarragon (2 Samples; 1 Foreign; 1 *Organic*; **2 Violations; 1 National Organic Program {NOP} Violation**; 2 Bacterial Analyses)

California 1 (1)					
Azoxystrobin	LC	1	0.002		50
Difenoconazole	**LC**	**1**	**0.005**		**0**
Diuron	**LC**	**1**	**0.010**		**0**
Foreign, *Organic* 1 (1)	**NOP Violation; NOP Violative Residues Denoted by ***				
Columbia	**Indicates Residue Present at Greater than 5% of Tolerance**				
Azoxystrobin*	Both	1	12.00		50
Carbendazim (Metabolite)*	**LC**	**1**	**0.003**	**No Tolerance**	**0**
Carbofuran*	**LC**	**1**	**0.050**	**No Tolerance**	**0**
Chlorpyrifos*	**LC**	**1**	**0.072**	**No Tolerance**	**0**
Cypermethrin*	**GC**	**1**	**1.500**	**No Tolerance**	**0**
Difenoconazole*	**Both**	**1**	**8.000**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.014		8
Methomyl*	**LC**	**1**	**2.000**	**No Tolerance**	**0**
Propiconazole*	**Both**	**1**	**10.00**	**No Tolerance**	**0**
Spinetoram	LC	1	0.092		3

Thyme (3 Samples; 2 Foreign; 1 Unknown; **3 Violations**; 2 Bacterial Analysis)

Foreign (Mexico) 1 (1)					
Methomyl	**LC**	**1**	**0.593**	**No Tolerance**	**0**
Foreign (Columbia) 1 (1)					
Acephate	**LC**	**1**	**0.029**	**No Tolerance**	**0**
Carbendazim (Metabolite)	**LC**	**1**	**16.00**	**No Tolerance**	**0**
Difenoconazole	**LC**	**1**	**0.007**	**No Tolerance**	**0**
Fipronil	**LC**	**1**	**0.078**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.868		8
Phosmet	**LC**	**1**	**0.212**	**No Tolerance**	**0**
Unknown (US) 1 (1)					
2,6 Dichlorobenzamide (met)	**LC**	**1**	**0.203**	**No Tolerance**	**0**
Azoxystrobin	LC	1	0.010		50
Boscalid	**LC**	**1**	**0.010**	**No Tolerance**	**0**
Carbendazim (Metabolite)	**LC**	**1**	**0.100**	**No Tolerance**	**0**
Cyprodinil	LC	1	0.342		15
Cyromazine	**LC**	**1**	**0.295**	**No Tolerance**	**0**
Dinotefuran	**LC**	**1**	**0.104**	**No Tolerance**	**0**
Flonicamid	**LC**	**1**	**0.275**	**No Tolerance**	**0**
Fludioxonil	LC	1	0.025		10
Flutriafol	**LC**	**1**	**0.219**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.868		8
Oxamyl	**LC**	**1**	**0.812**	**No Tolerance**	**0**
Profenofos	**LC**	**1**	**0.182**	**No Tolerance**	**0**

Pronamide	**LC 0**	**1**	**0.049**	**No Tolerance**
Pyraclostrobin	**LC 0**	**1**	**0.305**	**No Tolerance**
Thiamethoxam	**LC 0**	**1**	**0.089**	**No Tolerance**

FRESH TOTALS:	**SAMPLES**	**21**	
	WITH RESIDUES	**20**	**(95.2%)**
	VIOLATIVE SAMPLES	**18**	**(85.7%)**
	ORGANIC SAMPLES	**1**	
	ORGANIC VIOLATIVE	**1**	**(100%)**
	BACTERIAL ANALYSES	14	
TOTAL DIFFERENT ACTIVE INGREDIENTS FOUND:		**48**	
TOTAL NUMBER OF RESIDUES FOUND:		**109**	
TOTAL NUMBER OF VIOLATIVE RESIDUES FOUND:		**74**	**(67.9%) Of residues found**

ADD DISCUSSION
CLOTHIANADIN .586 = THIAMETHOXAM .565 METABOLITE
CARBENDAZIM = BENOMYL .294; THIOPHANATE METHYL .371 METABOLITE
2,6 DICHLOROBENZAMIDE = DICHLOBENIL .231; FLUOPICOLIDE .627 METABOLITE
CONSIDER USING STAR * TO DENOTE METABOLITES
none* -- There is no US tolerance for carbendazim. Carbendazim has been used as a standalone pesticide in the past; however it is also a metabolite of the insecticides Thiophanate methyl and benomyl both of which undergo rapid degradation in the field to carbendazim. When 'none' is used, it indicates that the commodity has a tolerance for either/both benomyl and/or Thiophanate methyl. Provided the level of carbendazim is below the tolerance level of these pesticides on the specific commodity of interest, it is not considered a violation. When '0' is used it indicates that the metabolite carbendazim is not allowed because there is no tolerance for benomyl or Thiophanate methyl on these commodities. For a more comprehensive discussion on this subject the reader is referred to Krol *et al*, 2007.

Table 2: Summary of Pesticides Found in Dried Herbs Sold in Connecticut in 2011.

Commodity / Origin / Pesticide	Samples with Residues (Total)	Found by LC, GC or Both	Number of Times Detected	Residue Range (ppm)	Average Residue (ppm)	EPA Tolerance (ppm)
Basil (4 Samples; 1 Foreign; 3 Unknown; **3 Violations**)						
Foreign	1 (1)					
Mediterranean						
Carbendazim (Metabolite)		**LC**	**1**	**0.013**	**No Tolerance**	**0**
Myclobutanil		**LC**	**1**	**0.007**	**No Tolerance**	**0**
Unknown (US)	1 (1)					
Carbendazim (Metabolite)		**LC**	**1**	**0.001**	**No Tolerance**	**0**
Chlorpyrifos		**LC**	**1**	**0.009**	**No Tolerance**	**0**
Metalaxyl		**LC**	**1**	**0.005**	**No Tolerance**	**0**
Pendimethalin		**LC**	**1**	**0.007**	**No Tolerance**	**0**
Unknown	1 (2)					
Chlorpyrifos		**LC**	**1**	**0.028**	**No Tolerance**	**0**
Metalaxyl		**LC**	**1**	**0.014**	**No Tolerance**	**0**
Chives (3 Samples; 3 Unknown; **3 Violations**)						
Unknown (US)	1 (1)					
Acephate		**LC**	**1**	**0.123**	**No Tolerance**	**0**
Acetamiprid		**LC**	**1**	**0.248**	**No Tolerance**	**0**
Carbofuran		**LC**	**1**	**0.052**	**No Tolerance**	**0**
Chlorpyrifos		**LC**	**1**	**0.027**	**No Tolerance**	**0**
Difenoconazole		**LC**	**1**	**0.045**	**No Tolerance**	**0**
Dimethomorph		**LC**	**1**	**0.012**	**No Tolerance**	**0**
Imidacloprid		LC	1	0.203		48
Iprodione		**LC**	**1**	**0.396**	**No Tolerance**	**0**
Metalaxyl		**LC**	**1**	**0.023**	**No Tolerance**	**0**
Oxadixyl		**LC**	**1**	**0.014**	**Revoked 9/03**	**0**
Pyrimethanil		**LC**	**1**	**0.228**	**No Tolerance**	**0**
Thiophenate Methyl		**LC**	**1**	**3.250**	**Not Tolerance**	**0**
Unknown	2 (2)					
Acephate		**LC**	**2**	**0.088-0.144**	**No Tolerance**	**0**
Acetamiprid		**LC**	**2**	**0.182-0.221**	**No Tolerance**	**0**
Carbofuran		**LC**	**2**	**0.053-0.057**	**No Tolerance**	**0**
Chlorpyrifos		**LC**	**1**	**0.063**	**No Tolerance**	**0**
Difenoconazole		**LC**	**2**	**0.078-0.121**	**No Tolerance**	**0**
Dimethomorph		**LC**	**2**	**0.295-0.421**	**No Tolerance**	**0**
Imidacloprid		LC	2	0.208-0.241		48
Iprodione		**LC**	**2**	**0.237-0.296**	**No Tolerance**	**0**
Metalaxyl		**LC**	**2**	**0.023-0.027**	**No Tolerance**	**0**
Methomyl		**LC**	**2**	**0.017-0.028**	**No Tolerance**	**0**
Oxadixyl		**LC**	**2**	**0.028-0.031**	**Revoked 9/03**	**0**
Propiconazole		**LC**	**2**	**0.011-0.028**	**No Tolerance**	**0**

Pyrimethanil	**LC**	**2**	**0.519-0.574**	**No Tolerance**	**0**
Thiophenate Methyl	**LC**	**2**	**2.600-2.700**	**No Tolerance**	**0**

Coriander / Cilantro (5 Samples; 5 Unknown; **4 Violations**)

Unknown 4 (5)

Azoxystrobin	LC	1	0.030		260
Carbendazim (Metabolite)	**LC**	**1**	**0.040**	**No Tolerance**	**0**
Difenoconazole	**LC**	**1**	**0.154**	**No Tolerance**	**0**
Dimethomorph	**LC**	**1**	**0.063**	**No Tolerance**	**0**
Linuron	**LC**	**1**	**0.133**	**No Tolerance**	**0**
Metalaxyl	**LC**	**1**	**0.034**	**No Tolerance**	**0**
Pendimethalin	**LC**	**3**	**0.024-0.034**	**No Tolerance**	**0**
Trifloxystrobin	**LC**	**1**	**0.008**	**No Tolerance**	**0**

Marjoram / Oregano (1 Sample; 1 Foreign; **1 Violation**)

Foreign (Turkey) 1 (1)

Methidathion	**LC**	**1**	**0.033**	**No Tolerance**	**0**

Parsley (4 Samples; 2 Foreign; 1 *Organic*; 2 Unknown; **4 Violations; 1 National Organic Program {NOP} Violation**)

Foreign (Europe) 1 (1)

Azoxystrobin	LC	1	0.752		260
Bromacil	**LC**	**1**	**0.027**	**No Tolerance**	**0**
Carbendazim (Metabolite)	**LC**	**1**	**0.040**	**No Tolerance**	**0**
Chlorpyrifos	**LC**	**1**	**0.070**	**No Tolerance**	**0**
Dimethomorph	**LC**	**1**	**0.906**	**No Tolerance**	**0**
Linuron	**LC**	**1**	**0.005**	**No Tolerance**	**0**
Malathion	**LC**	**1**	**0.124**	**No Tolerance**	**0**
Metalaxyl	**LC**	**1**	**0.004**	**No Tolerance**	**0**
Methomyl	**LC**	**1**	**0.007**	**No Tolerance**	**0**
Pendimethalin	**LC**	**1**	**0.054**	**No Tolerance**	**0**
Pirimicarb	**LC**	**1**	**0.005**	**No Tolerance**	**0**
Triadimenol	**LC**	**1**	**0.264**	**No Tolerance**	**0**

Foreign, *Organic*

Europe 1 (1) **NOP Violation; NOP Violative Residues Denoted by * Indicates Residue Present at Greater than 5% of Tolerance**

Bromacil*	**LC**	**1**	**0.041**	**No Tolerance**	**0**
Carbendazim (Metabolite)*	**LC**	**1**	**0.041**	**No Tolerance**	**0**
Chlorpyrifos*	**LC**	**1**	**0.011**	**No Tolerance**	**0**
Malathion*	**LC**	**1**	**0.110**	**No Tolerance**	**0**
Pendimethalin*	**LC**	**1**	**0.006**	**No Tolerance**	**0**

Unknown (US) 1 (1)

Azoxystrobin	LC	1	2.700		260
Chlorpyrifos	**LC**	**1**	**0.074**	**No Tolerance**	**0**
Linuron	**LC**	**1**	**0.489**	**No Tolerance**	**0**
Malathion	**LC**	**1**	**0.027**	**No Tolerance**	**0**
Pendimethalin	**LC**	**1**	**0.044**	**No Tolerance**	**0**

Unknown 1 (1)

Azoxystrobin	LC	1	0.010		260
Boscalid	**LC**	**1**	**0.014**	**No Tolerance**	**0**
Chlorpyrifos	**LC**	**1**	**0.010**	**No Tolerance**	**0**

Diazinon	**LC**	**1**	**0.108**	**No Tolerance**	**0**
Imidacloprid	LC	1	0.049		48
Malathion	**LC**	**1**	**0.023**	**No Tolerance**	**0**
Methoxyfenozide	**LC**	**1**	**11.60**	**Over Tolerance**	**10**
Pyraclostrobin	**LC**	**1**	**0.642**	**No Tolerance**	**0**

Rosemary (1 Sample)

US 0 (1)

Tarragon (4 Samples; 4 Unknown; **3 Violations**)

Unknown 4 (4)

Amitraz	**LC**	**1**	**0.470**	**No Tolerance**	**0**
Azoxystrobin	LC	2	0.013-0.024	0.019	260
Carbendazim (Metabolite)	**LC**	**2**	**0.008-0.023**	**No Tolerance**	**0**
Diazinon	**LC**	**2**	**0.046-0.388**	**No Tolerance**	**0**
Linuron	**LC**	**1**	**0.236**	**No Tolerance**	**0**
Metolachlor	**LC**	**1**	**0.023**	**No Tolerance**	**0**
Propiconazole	**LC**	**2**	**2.000-10.00**	**No Tolerance**	**0**
Pyraclostrobin	**LC**	**1**	**0.007**	**No Tolerance**	**0**

Thyme (2 Samples; 2 Unknown; 1 *Organic*; **1 Violation; 1 National Organic Program {NOP} Violation**)

Unknown (US) 0 (1)

Unknown, *Organic* 1 (1) **NOP Violation; NOP Violative Residue Denoted by ***

Indicates Residue Present at Greater than 5% of Tolerance

Carbendazim (Metabolite)*	**LC**	**1**	**0.009**	**No Tolerance**	**0**

DRIED TOTALS:	**SAMPLES**	**24**	
	WITH RESIDUES	**20**	**(83.3%)**
	VIOLATIVE SAMPLES	**19**	**(79.2%)**
	ORGANIC SAMPLES	**2**	
	ORGANIC VIOLATIVE	**2**	**(100%)**
	BACTERIAL ANALYSES	0	
TOTAL DIFFERENT ACTIVE INGREDIENTS FOUND:		**30**	
TOTAL NUMBER OF RESIDUES FOUND:		**101**	
TOTAL NUMBER OF VIOLATIVE RESIDUES FOUND:		**91**	**(90.1%) Of residues found**

ADD DISCUSSION
CLOTHIANADIN .586 = THIAMETHOXAM .565 METABOLITE
CARBENDAZIM = BENOMYL .294; THIOPHANATE METHYL .371 METABOLITE
2,6 DICHLOROBENZAMIDE = DICHLOBENIL .231; FLUOPICOLIDE .627 METABOLITE
CONSIDER USING STAR * TO DENOTE METABOLITES

none* -- There is no US tolerance for carbendazim. Carbendazim has been used as a standalone pesticide in the past; however it is also a metabolite of the insecticides Thiophanate methyl and benomyl both of which undergo rapid degradation in the field to carbendazim. When 'none' is used, it indicates that the commodity has a tolerance for either/both benomyl and/or Thiophanate methyl. Provided the level of carbendazim is below the tolerance level of these pesticides on the specific commodity of interest, it is not considered a violation. When '0' is used it indicates that the metabolite carbendazim is not allowed because there is no tolerance for benomyl or Thiophanate methyl on these commodities. For a more comprehensive discussion on this subject the reader is referred to Krol *et al*, 2007.

FRESH TOTALS:	**SAMPLES**		**21**	
	WITH RESIDUES	**20**	**(95.2%)**	
	VIOLATIVE SAMPLES		**18**	**(85.7%)**
	ORGANIC SAMPLES		**1**	
	ORGANIC VIOLATIVE		**1**	**(100%)**
	BACTERIAL ANALYSES		14	
TOTAL DIFFERENT ACTIVE INGREDIENTS FOUND:			**48**	
TOTAL NUMBER OF RESIDUES FOUND:			**109**	
TOTAL NUMBER OF VIOLATIVE RESIDUES FOUND:			**74**	**(67.9%) Of residues found**

DRIED TOTALS:	**SAMPLES**		**24**	
	WITH RESIDUES	**20**	**(83.3%)**	
	VIOLATIVE SAMPLES		**19**	**(79.2%)**
	ORGANIC SAMPLES		**2**	
	ORGANIC VIOLATIVE		**2**	**(100%)**
	BACTERIAL ANALYSES		0	
TOTAL DIFFERENT ACTIVE INGREDIENTS FOUND:			**30**	
TOTAL NUMBER OF RESIDUES FOUND:			**101**	
TOTAL NUMBER OF VIOLATIVE RESIDUES FOUND:			**91**	**(90.1%) Of residues found**

FRESH AND DRIED HERBS

SUM TOTALS:	**SAMPLES**		**45**	
	WITH RESIDUES	**40**	**(88.9%)**	
	VIOLATIVE SAMPLES		**37**	**(82.2%)**
	ORGANIC SAMPLES		**3**	
	ORGANIC VIOLATIVE		**3**	**(100%)**
	BACTERIAL ANALYSES		14	
TOTAL DIFFERENT ACTIVE INGREDIENTS FOUND:			**60**	
TOTAL NUMBER OF RESIDUES FOUND:			**210**	
TOTAL NUMBER OF VIOLATIVE RESIDUES FOUND:			**165**	**(78.6%) Of residues found**

NOTES