Arizona Iceberg Lettuce Research Council

Research Report

Pesticide Diagnostics Laboratory

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Purpose

Hundreds of soil and tissue samples are sent every year from the low deserts in Arizona to out-of-state laboratories for pesticide analysis. There are three primary needs for this information:

1. Vegetable crops in Arizona are intensively grown and of high value. Producing these crops commonly involves the use of herbicides, insecticides and fungicides to manage pests. There is a low tolerance for crop injury from pesticide misuse, soil carryover or off-target drift. Diagnosing these problems requires accurate and precise detection methods.
2. Advances in analytical chemistry have produced cost-effective techniques for the detection of pesticides in crops, soil and water. As these technologies have been developed the demand for information by government regulators, the public and the agricultural industry has also grown.
3. University extension and research programs can no longer rely on imprecise and subjective field evaluations. The agriculture industry has become increasingly sophisticated and more precise and accurate diagnostic techniques are required.

Cost effective analytical techniques are now available that allow us to detect minute amounts of pesticides in plant tissue, soil and water. These techniques generally involve solvent extraction of the pesticide followed by the use of specialized equipment to detect very small amounts. The most common techniques currently used are gas and liquid chromatography. The standard unit of measure using this equipment is parts per million (equivalent to one drop in 50 L) although parts per billion (equivalent to one drop and 50,000 L) is becoming more frequent.

Hundreds of samples are sent from the low desert vegetable growing regions of Arizona to out of state laboratories for pesticide analysis.

Almost every state has University related or private laboratories offering plant disease and soil testing. Arizona does not have a University or public Laboratory offering pesticide analysis to the public. Most laboratories in Arizona send samples to out of state laboratories. The cost of this service ranges from $100-$300 per sample.

The time it takes to receive results varies from 1 to 3 weeks or longer. In most cases more timely results are needed to take remedial action or make management plans. Timing is critical in the management of short season high-value crops. Some of these crops are harvested within 30 days of planting.

The purpose of this project was to establish a pesticide analytical Laboratory at the University of Arizona Yuma Agricultural Center that could process samples for the Arizona lettuce industry in a timely and cost-effective manner.

Procedure

This project was initiated in the summer of 2012 with the support of the Arizona Iceberg Lettuce Research Council. Both gas and liquid chromatograph machines were available for use in the food safety laboratory at the Yuma Agriculture Center. Two part-time students were hired to work with us to develop techniques to analyze plant tissue and soil for the three primary herbicides used in the production of lettuce; Pronamide, Benefin, and Bensulide. The next nine months were spent testing various procedures for detecting these three herbicides. Many people and organizations assisted in this effort. These included Frank Jaime (Gowan CO.), Sean Kurokawa (Primus Labs), Larry Evanicky (Shimadzu Corporation), Paul Martin (USDA Desert Research Center), Danielle Martin (Gowan CO.). Jaime Archuleta (Shimadzu Corporation), Steve Castle (USDA Desert Research Center) and Kurt Nolte (U of A Cooperative Extension).

Four field trials were established at the Yuma Agriculture Center, three trials in the greenhouse and three in the laboratory to produce plants and soil containing variable levels of these herbicides for analysis. Hundreds of samples were processed to evaluate various extraction and detection methods.

There were two components to this project:

1. Methods to extract three herbicides; Pronamide, Benefin and Bensulide from plant tissue and soil.
2. Methods to detect them in the extraction.

What is detected depends on the sampling and the extraction procedures that have been used. Several techniques were tested for extracting Pronamide, Benefin and Bensulide from soil and plant tissue. We found that so analysis is far more accurate and consistent than is tissue analysis for these three herbicides. All three are applied to soil where day are picked up by the weeds and the crop. Only Pronamide moves much in the plant. Benefin and Bensulide move very little.

What we detected in the plant tissue had been filtered through the soil and plant and was between 0.1 ppm and 3.0 ppm.

Seedling plants are hard to sample and the amount we found in them was extremely variable. In contrast, we found between three and 150 ppm of these three herbicides in the top inch of the soil and it was more consistent than what we found in the plant.

The soil is easier to sample and more consistent. The sampling guidelines that we currently give to people wanting us to analyze lettuce fields for Pronamide, Benefin or Bensulide is to collect a composite sample of 200 g taken from the top inch of soil. We ask also that they collect separate samples from affected and unaffected areas of the field if possible, for comparison. We have determined that a modified QUECHERS solid phase extraction procedure works well for these three herbicides.

The detection process was more straightforward although we had good assistance from both the hardware and software people at Shimadzu Corporation the manufacturers of the liquid and gas chromatographs machines and from the chemists at Gowan Company. After much experimentation and repair, we decided that the high-pressure/UV liquid chromatograph worked well for the three lettuce herbicides. Samples were spiked by the chemists at Gowan Company and provided to us as unknowns to verify our results. Our detections were within accepted Industry standards and we know have good confidence that our extraction and detection procedures are accurate.

We felt confident enough in our ability to process samples to begin informing the industry that could process samples in September 2013. We have informed people through the vegetable IPM advisories and presentations at some vegetable production meetings. We hope to build a database of results and gain experience with our procedures this season, and we have decided to process samples free of charge this year. A summary of our activity follows. Our turnaround time in processing samples has been less than one day and a sample of one of our reports is included with this report. We expect the demand for this service will increase if we can continue to provide timely and accurate results.

Number of samples processed	Compounds analyzed for	Location of samples collected	Number of growers assisted	Number of pest control advisors assisted
112	Pronamide Glyphosate Bensulide DCPA EPTC Oxyfluorfen Benefin Carfentrazone Triflulfuron	Yuma Valley 49 Gila Valley 19 Wellton Mohawk 13 Yuma Mesa 3 Yuma Ag. Center 18	19	14

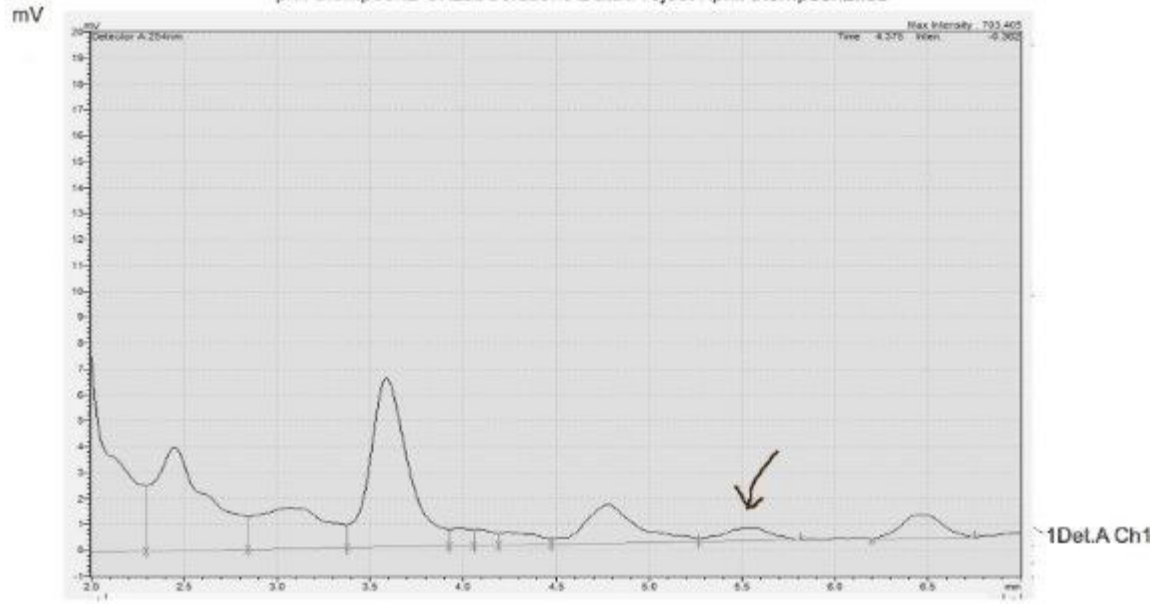
==== UA Yuma Agricultural Center Analysis Report ====

Sample Information

Sample Name	: phil thompson2
Sample ID	: phil thompson2
Vail#	: 1
Injection Volume	: 1 uL
Data Filename	: phil thompson2.lcd
Method Filename	: Isocratic 65-35 acn manual 7min.lcm
Batch Filename	:
Report Filename	: Default.lcr
Date Acquired	: 10/28/2013 2:42:37 PM

Chromatogram

phil thompson2 C:\LabSolutions\Data\Project1\phil thompson2.lcd



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Area %
1	0.542	5132904	61.442
2	0.690	1385735	16.588
3	0.860	500139	5.987
4	0.992	266368	3.189
5	1.096	461367	5.523
6	1.650	138628	1.659
7	1.831	56233	0.673
8	1.952	128019	1.532
9	2.443	79418	0.951
10	3.059	41705	0.499
11	3.584	93584	1.120
12	3.974	5196	0.062
13	4.092	4412	0.053
14	4.238	6729	0.081
15	4.775	30035	0.360
16	5.563	9295	0.111
17	6.456	14240	0.170

PPM:

We are finding a peak at 5.563 minutes which turned out to be to small to see because of the relative size of the image.Regardless we can ,with confidence, say that there a trace amounts of Oxyfluroefen being deteced through HPLC/UV analysis.