

Oxytetracycline Residue Study

David Granatstein

Tree Fruit Research and Extension Center, Washington State University

Wenatchee, WA 98801 USA

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Oxytetracycline is a commonly used pesticide in Washington State and other regions to control the disease fire blight (*Erwinia amylovora*) on commercial apples and pears. It has been allowed for use in organic orchards as well since the establishment of the National Organic Program in 2002, and prior to that by a number of certifiers in the US. The National Organic Standards Board (NOSB) voted for an expiration of the use of oxytetracycline (OTC) in organic orchards starting in October 2014. A petition was submitted to the NOSB by the tree fruit industry in 2012 to extend that expiration date for 2 or 3 more years to allow research underway on non-antibiotic control measures to be completed and for the results to be made available to growers.

One of the issues raised by the NOSB was the presence of residues of OTC on fruit at harvest and the potential to expose organic consumers to these residues. The only published residue data that could be found were from studies arranged by the US Environmental Protection Agency (USEPA) as part of the regulatory process to extend the label use of OTC to apples. Field trials were conducted by the registrant in apple orchards in various regions of the country. Oxytetracycline (as the formulated material Mycoshield) was applied six times; the first five sprays were applied at 3 to 6 day intervals and the sixth spray was applied 49 to 50 days before harvest (the legal pre-harvest interval is 60 days). Among 15 trials, the total amount of Mycoshield applied within a season varied from 0.765 to 7.65 pounds a.i./acre, or 0.5 to 11 times the proposed seasonal rate of 1.53 lb a.i./acre. Apples were harvested, frozen, and residues from samples were extracted with water. The limit of quantification of oxytetracycline was 0.013 ppm. Most samples were at or below the limit of detection, while the highest residue level detected was 0.25 ppm in two fruits of the 128 fruits tested. None of the samples had residues at the permitted rate of 0.35 ppm. Several dose rates were tested in the field trials and no correlation between amount of Mycoshield applied and detectable residues was observed. No data were reported for trees treated only once or twice during bloom, which is the most common use pattern in the western U.S. (Stockwell and Duffy 2012).

In order to generate additional data reflective of actual orchard use patterns, a residue sampling study was conducted in fall 2012. An agreement with Pacific Agricultural Laboratory (PAL, Portland, OR) was reached in mid-October for them to do the study. Fruit from seven different orchards in south-central Washington were picked from the orchard on October 24-26, (10-15 fruit per orchard from different trees), placed in plastic bags, kept cool, and delivered to Cascade Analytical Inc. in Wenatchee, WA on October 26 for sample preparation. Samples were prepared, homogenized, blended, and frozen per instructions from PAL, and received at PAL on Nov. 13, 2012, where they were kept frozen at -15C prior to analysis.

PAL used the Quick Method for the Analysis of Residues of Highly Polar Pesticides in Foods of Plant Origin Involving Simultaneous Extraction with Methanol and LC-MS/MS Determination (QuPPE-Method), Version 6, August 2011 (M. Anastassiadou et al., 2011). PAL used a Waters MS/MS system equipped with a Xevo triple quadrupole mass spectrometer, Acquity UPLC and computerized Lynx data acquisition software. An initial demonstration of capability (IDOC) was

conducted prior to analysis of samples. The IDOC included the acquisition of an analytical reference standard material, validation of initial instrument conditions, a method detection limit (MDL) study, and an initial performance and recovery (IPR) study. Satisfactory completion of the IDOC occurred prior to the study samples being analyzed and reported. An MDL of 0.00090 mg/kg (ppm) was established for Oxytetracycline, and a limit of quantitation (LOQ) was established at 0.010 mg/kg (ppm). PAL does not generally report data below a LOQ, and estimated results are not generated between the MDL and the LOQ.

Upon successful completion of the IDOC, sample analysis was conducted. Extraction was performed on 2/13/13 and analysis performed on 2/14/13. Results were reported on 2/25/13, PAL project P121557. No Oxytetracycline residues were detected in any of the seven samples submitted for analysis down to an LOQ of 0.010 mg/kg (ppm). The LOQ of 0.010 mg/kg is sufficiently low to meet all default MRL tolerance criteria both foreign and domestic.

A table of results is presented below. This exploratory study suggests that with the typical OTC use pattern in orchards (primarily treatments during the bloom period at 150+ days before harvest, with no fruit yet formed), residues of OTC would not be found on or in the fruit at harvest.

Table 1. Details of apple samples used for oxytetracycline residue testing, Washington State.

Orchard	Location	Cultivar	Material	Rate	Dates	Residue
1	Zillah	Pink Lady	5 applications Mycoshield™	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-mid May	n.d.
2	Zillah	Pink Lady	3 applications Mycoshield™	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-late April	n.d.
3	Desert Aire	Pink Lady	4 applications Mycoshield™ ; + 2 applications every other row	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-mid May	n.d.
4	Mattawa	Pink Lady	3 applications Mycoshield™	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-late April	n.d.
5	Mattawa	Pink Lady	4 applications Mycoshield™	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-mid May	n.d.
6	Pasco	Fuji	2 applications Mycoshield™	1 lb a.i./100 gal water; tree row volume , 58-65 gal mixture/acre	Early April-mid April	n.d.
7	Royal City	Pink Lady	5 applications Mycoshield™	1 lb a.i./100 gal per acre	4/20, 4/24, 4/28, 5/2, mid-July*	n.d.
<p>The Mycoshield label calls for a maximum of 5 applications at not more than 1.0 lb of product per application, with a 200 ppm solution (1 lb in 100 gal. water).</p> <p>*this orchard experienced hail damage in mid-July which was followed immediately by an OTC treatment.</p>						

Citations

- Anastassiades, M., Kolberg, D.I., Mack, D. Sigalova, I., Roux, D., Fügél, D. 2011. Quick Method for the Analysis of Residues of Highly Polar Pesticides in Foods of Plant Origin Involving Simultaneous Extraction with Methanol and LC-MS/MS Determination (QuPPE-Method), Version 6, August 2011. EU Reference Laboratories for Residues of Pesticides. 37 pp.
<http://www.eurl-pesticides.eu/docs/public/home.asp?LabID=100&Lang=EN>
- Stockwell, V.O. and Duffy, B. 2012. Use of antibiotics in plant agriculture. *Rev. Sci. Tech. Off. Int. Epiz.* 31(1):199-210. <http://www.oie.int/doc/ged/D11800.PDF>
- USEPA. 2005. Oxytetracycline. Section 3 Use on Apples. Summary of Analytical Chemistry and Residue Data. HED Records Center Series 361 Science Reviews, File R104981. Environmental Protection Agency, Washington, DC.
http://www.epa.gov/pesticides/chem_search/cleared_reviews/csr_PC-006304_3-Jan-05_a.pdf