

Australian national residue survey – closing the loop on pesticide residue risk management for Australian grain

Reichstein, I.*^{#1}, Healy, K.¹, James, A.¹, Murray, B.²

¹ National Residue Survey, Australian Government Department of Agriculture, Fisheries and Forestry, PO Box 858, Canberra, ACT, 2601, Australia. Email: ian.reichstein@daff.gov.au

² WJ Murray Consulting Services, 22 Thornley Close, Ferntree Gully, Victoria, 3156, Australia

* Corresponding author

Presenting author

DOI: 10.5073/jka.2010.425.122

Abstract

Australia exports a major proportion of its agricultural production and is highly dependent on maintaining and developing access to, and competitiveness in, export markets. To preserve Australia's status as a provider of high quality grain, the majority of Australian primary producers rely on pesticides to protect their crops from pests and diseases, particularly in post-harvest situations. The Australian Pesticides and Veterinary Medicines Authority (APVMA) supports Australian agriculture by registering and allowing the supply of safe and effective animal health and crop protection products.

A residue risk management continuum is established when the effectiveness of chemical registration and control of chemical use regulations is assessed through residue monitoring programs. Programs assess good agricultural practice and provide traceback capacity to investigate areas of concern. Risk communication provides opportunities for continuous improvement. In the early 1960s, the Australian Government established a non-regulatory body, the national residue survey (NRS). In 2008-2009, random monitoring programs were conducted for over 50 commodities (21 grains, five horticultural commodities, 11 fish species, 12 animal species, honey and egg with over 20,000 samples collected for analytical testing. The NRS grain residue monitoring program is presented as a case study of the residue risk management continuum demonstrating to overseas markets the high level of residue integrity of Australian grain. Over 4,000 grain samples are collected and analysed per annum. Most of the samples are collected in the bulk export program where samples are collected from every hatch of every ship loaded at the seventeen Australian grain export terminals. The chemical screens have expanded beyond the multi-residue screen (MRS) insecticides, fungicides and herbicides, to include phosphine, additional herbicides (not included in the MRS), heavy metals and mycotoxins. In its current form, the NRS grains program provides 15 years of residue testing data which demonstrates a very high degree of conformance with Australian MRLs and the import tolerances of overseas trading partners. In addition, trends in residue testing data demonstrate a decline in the frequency of residue detections and the levels of residue detected. To be confident that residue testing results meet the requisite standards, the reliability of the Australian analyses must be assured. The NRS laboratory performance evaluation system has been developed to provide that assurance, using a range of proficiency tests and other techniques in the selection of laboratories for NRS work. Residue testing results are reported against both Australian MRLs and the international MRLs which apply in the relevant export market. NRS maintains databases of overseas MRLs and compares its residue testing results of exported commodities against those standards. Grain marketers receive certificates of analysis, in the form of NRS residue testing results, for each shipment prior to arrival at the overseas market.

Keywords: Grain, Pesticide, residue, monitoring program, residue risk management

1. Introduction

Pesticides (insecticides, fungicides, herbicides and fumigants) are widely used in Australia as an essential tool in broadacre farming. Pesticides are used at all stages of crop production from seeding through to post-harvest protection from stored grain pests, to maximise production and improve productivity from weed control and control of micro- and macro-organism pests. Pesticides are registered for use by the Australian Pesticides and Veterinary Medicines Authority (APVMA) which takes into account the potential risks from the incorrect use of pesticides. The APVMA has responsibility to ensure that pesticide products registered for use in Australia are suitably formulated and properly labelled and, when used according to instructions, are safe to the plant host, the user, consumers and the environment;

efficacious (that is, the product does the job it claims it shall do); and not unduly prejudicial to trade. Registration helps to ensure that unacceptable residues from the chemicals used in agriculture do not appear in food for human or animal consumption in Australia or in Australia's export markets. As part of the registration process, APVMA recommends maximum residue limits (MRLs). MRLs reflect the maximum residues of pesticides which may occur in foods when registered chemicals are used in accordance with Good Agricultural Practice (GAP). Good Agricultural Practice is defined as the nationally recommended, authorised or registered use-pattern of chemicals, that is necessary for effective and reliable pest control under actual conditions at any stage of the production, storage, transport, distribution and processing of food commodities and animal feed. MRLs are not health standards *per se*, however they are assessed against health standards, to ensure that foods containing residues at the MRL are fit for human consumption. These considerations apply to the current methodology used to set Australian MRLs and the MRLs of other countries, as well as MRLs set by the Codex Alimentarius Commission.

Residue monitoring programs are an essential component of the Australian national residue risk management framework. The closed loop approach ensures that pesticide registration and farm chemical use is audited through residue monitoring programs which in turn provide feedback with information supporting the original registration decision. Traceback investigations on non-compliant residues provides additional information which allows regulators to address problems and minimise reoccurrences.

The key objective of Australia's national grain residue testing program is to audit the effectiveness of regulatory controls that are in place to ensure that pesticides applied in broadacre farming are used according to GAP. This provides assurances to the grain industry that farming practices are appropriate and that pesticide residues levels in grain are compliant with relevant MRLs, do not pose a threat to human health and have no adverse impact on trade.

The specific pesticides registered for use in each country and GAP for the use of those chemicals varies from country to country on the basis of differing agricultural pest pressures and diseases and their significance in food production. GAP is also affected by the nature of agricultural business, geography and environmental considerations. Therefore, variances in MRL values for particular chemical-commodity combinations from country to country can be expected and commodity exporters need to be aware of those differences.

The grain industry is becoming increasingly aware of overseas MRLs and varying overseas marketing requirements when considering pesticide use in crops and in post-harvest situations. Given the high importance placed on food safety and market access, the National Residue Survey (NRS) in cooperation with the grain industry have established a comprehensive national grain residue monitoring program to verify the pesticide residue integrity of Australian grain from farm receipt to point of export. The NRS Export Grains Residue Testing Program involves grain sampling at container and bulk out-turn as a final check of residue integrity prior to export and is considered the primary residue testing program for Australian grain. The NRS is a government agency, located in Canberra within the Australian Government Department of Agriculture, Fisheries and Forestry. Since 1993, the NRS has operated on the basis of full cost recovery.

In 1993, the Grains Council of Australia, in full consultation with the grain industry, elected to establish a 0.015% ad valorem grain grower levy to fund the operation of the NRS Grains Residue Testing Program. The grain industry requested that the program be operated primarily as a market access project focussed on collecting grain samples at the point of export and at the point of domestic receipt for processing.

2. Materials and methods

2.1. Pesticide residue management in Australia grain

The responsibility, in regard to pesticide residue matters in grain, is shared between the Commonwealth and State governments and the grain industry. The Commonwealth government's responsibilities cover registration of pesticides, establishment of MRLs and the inspection and phytosanitary certification requirements for export. State government agencies are responsible for the control-of-use of pesticides and the inspection and certification requirements for the domestic market. Grain marketers and bulk

handling companies (BHCs) within Australia conduct rigorous testing from the first point of farm receipt through to grain aggregation into bulk storage facilities. BHCs monitor all grain quality parameters including pesticide residues.

2.2. *The grains pesticide residue testing program*

The NRS Grains Program is designed to provide an unbiased estimate of the frequency of residues in Australian grain as a whole. The program requires randomised sampling of grain from throughout Australia from as many grain streams as is possible. The NRS Grains Program is structured to give due consideration to the number of different grain commodities and the many export and domestic grain streams. The NRS Grains Program currently consists of the following series of sub-programs:

- Bulk Export - All 17 grain exports terminals with a representative grain sample collected from every hatch loaded on every ship leaving Australia
- Export Container - Representative grain samples collected whilst grain is loaded into bags and shipping containers destined for export
- Domestic (Milled Products) - Samples of wheat, maize, soybean, triticale and corresponding milled derivatives are collected.
- Domestic (Maltsters) - Samples at grain receipt to malting barley plants.
- Domestic (Oat Processors) - Samples collected at grain receipt to rolled oat processing plants
- Domestic (Oilseed Crushers) - Samples collected at grain receipt to crushing plants.
- Domestic (Feed mills) - Samples collected at grain receipt to stockfeed-mill site.
- Domestic (Feedlots) - Samples collected at grain receipt to cattle feedlots

All tradable grains are included in the NRS Grains Program. These are wheat (including durum), barley, oat, sorghum, maize, triticale, canola, soybean, sunflower, safflower, linseed, chickpea, field pea, cow pea, lentil, lupin, mung bean, faba bean, navy bean, vetch and pigeon pea. This paper focuses on the NRS Export Grain Program covering bulk shipments.

2.3. *Sampling methodology – bulk export program*

Approximately 3,000 samples of export grain are collected annually on the basis of a sample from every hatch from every bulk shipments of grain loaded at the 17 export terminals around Australia. The sample numbers are therefore determined by shipping throughput. Grain is collected at outturn at a rate of 2.25 L per 33 t using automated sampling equipment and a representative composite sample is collected from each hatch. Sample collectors are provided with a sample collection and despatch manual, sample forms, plastic sample bags, security satchel, freight satchels addressed to NRS contract laboratory and reply paid envelopes for return of the 'original' form.

2.4. *Analytical methodology*

A 20-g portion of each grain sample is subjected to a 30 minute sonication extraction in acetone with 12 h soaking. All extracted samples are applied to the contract laboratory's LCMSMS technique. The analytical parameters are as follows: Flow rate: 0.22 mL min⁻¹, Oven temp: 40°C, Column: Luna 3 μ PFP (150 x 3.00 mm), Mobile phase A: 0.1% FA in water, Mobile phase B: 0.1% FA in MeOH: ACN (2:3), LOQ: 0.01 mg kg⁻¹. If required, the laboratory utilises its confirmatory LCMSMS technique for purposes. The analytical parameters are as follows: Flow rate: 0.40 mL min⁻¹, Oven temp: 40°C, Column: Luna 3 μ PFP (150 x 3.00 mm), Mobile phase A: 0.2% FA in 10 mM NH₄OAc, Mobile phase B: 0.1% FA in MeOH: CAN (2:3), LOQ: 0.01 mg kg⁻¹.

2.5. *Analytical screen*

All grain samples are subjected to a multi-residue screen which is based on a risk profile that considers the following criteria:

- all pesticides registered for use in Australia on grain;
- known use patterns including timing of application in the growing season and repeat applications
- potential for residues in grain commodities;
- availability of suitable analytical methods, testing capacity and laboratory testing proficiency arrangements; and
- perceived risks to international trade and overseas market concerns.

- The analytical screen is as follows:
- Post-harvest grain protectants: chlorpyrifos-methyl, fenitrothion, pirimiphos-methyl, dichlorvos, methoprene, deltamethrin, spinosad
- Organophosphate insecticides: azamethiphos, chlorfenvinphos, chlorpyrifos, diazinon, dimethoate, ethopros, malathion, methacrifos, omethoate, phosmet, profenofos, terbufos, trichlorfon
- Synthetic pyrethroids insecticides: bifenthrin, bioresmethrin, cyfluthrin, cyhalothrin, cypermethrin, fenvalerate, permethrin, phenothrin, piperonyl butoxide
- Other insecticides: acetamiprid, amitraz, carbaryl, diflubenzuron, endosulfan, fipronil, imidacloprid, indoxacarb, methomyl, thiodicarb, pirimicarb, pyriproxyfen, triflumuron.
- Fungicides: azoxystrobin, captafol, carbendazim, cyproconazole, difenoconazole, epoxiconazole, etridiazole, fluquinconazole, flutriafol, hexaconazole, iprodione, penconazole, propiconazole, prothioconazole, tebuconazole, thiabendazole, triadimefon, triadimenol, triticonazole
- Herbicides: 2,4-D, atrazine, bromoxynil, carfentrazone-ethyl, chlorsulfuron, clethodim, clodinafop-propargyl, clopyralid, dicamba, diflufenican, diuron, iodosulfuron-methyl, MCPA, metolachlor, metosulam, metsulfuron-methyl, pendimethalin, picloram, propyzamide, simazine, tralkoxydim, triasulfuron, triclopyr, trifluralin
- Environmental contaminants: aldrin, chlordane, DDT, dieldrin, endrin, HCB, HCH, heptachlor, lindane, methoxychlor, mirex, oxychlordane
- Randomly selected grain samples are also subjected to additional analytical screens which include analytes not able to be included in the MRS. These analytical screens are as follows:
- Fumigants: phosphine
- Additional herbicides: amitrole, diclofop-methyl, diquat, fenoxaprop-P-ethyl, flamprop-M-methyl, fluazifop-P-butyl, glufosinate, glyphosate, haloxyfop, paraquat
- Heavy metals: cadmium, lead, mercury

2.6. Analytical laboratory proficiency

NRS awards analytical laboratories contracts on the basis of: performance in a pre-requisite NRS proficiency testing round; assessment of laboratory management; National Association of Testing Authority accreditation; accreditation for the relevant analytical test; quality assurance and control systems; previous performance; and value for money. The NRS laboratory performance evaluation activities are designed to maintain an up-to-date and continuing assessment of the proficiency of laboratories analysing samples for NRS. In simple terms, proficiency testing involves sending verified residue-free grain samples, which have been spiked with known concentrations of pesticides, to analytical laboratories which have indicated interest in tendering for the NRS Grains Program laboratory contract. Analytical laboratories must demonstrate the capacity to identify all spiked pesticides and quantify to 85 to 115 percent of the spike level. Ongoing proficiency of contract laboratories is closely monitored by NRS through a structured quality assurance program that includes biannual proficiency tests, bimonthly check samples, random audits and the use of 'blind' samples. Blind samples contain incurred residues and are indistinguishable from normal grain samples. These blind samples are sent to contract laboratories through the routine courier system in place for normal grain samples. Failure to meet the required standard during ongoing proficiency testing can result in the termination of a laboratory contract.

2.7. Reports

The NRS is bound by Australian privacy legislation which stipulates that detailed specific residue testing data must only be disseminated to the grain marketer and the grain handler. By agreement with the grain industry, NRS is required to provide a certificate of analysis within 14 d of an export sample being collected. This timeframe takes into account sample courier from grain establishment to the contract laboratory and a laboratory turn-around time of three working days. In addition, NRS publishes a Export Grains Program results reports each financial year. Each year, the NRS published its Annual Report which covers all residues testing program managed by NRS. These Annual Reports are available on the NRS website (www.daff.gov.au/nrs).

3. Results

As shown in Table 1, 3012 grain samples were collected for analyses during the 2008-2009 financial year from throughout Australia as part of bulk and container export residue testing programs. In 2008-2009, all bulk export grain samples were 100% compliant with relevant Australian Standards. Overall, the export container grain samples were 98.2% compliant with the relevant Australian standards. A Phosphine Residue Testing Program has been conducted since 2002 with no residues detected in export grain samples above the Australian MRL. There has been a progressive decline in the frequency of violative residues detected in Australian grain exported in bulk shipments as shown in Table 2. The table illustrates the very high compliance rates against Australian standards. These figures provide sound evidence that pesticide use in-crop and post-harvest applications are in accordance with good agricultural practice as specified on the pesticide chemical product label and instructions for use. Tighter marketing controls and improved grain storage facilities have contributed to this nation-wide trend towards residue-free exported grain. The introduction of smaller production volume grains into the export container program in 2006 has had a minor impact on the compliance rate trend shown in Table 3. The grain industry and NRS expect a return to 100 percent compliance when traceback investigations, education programs and quality assurance feedback mechanisms with grain growers take effect.

Table 1 NRS Export Grains Program for 2008-2009.

Commodity	Bulk export samples	Container export samples
wheat	1684	258
barley	505	44
sorghum	246	1
Other cereals	15	16
oilseeds	134	31
pulses	37	41
TOTAL	2621	391

Table 2 Residue violations in bulk export grain from 1996-2009.

Year	Samples	>MRL	Compliance %
1996-97	5746	22	99.6
1997-98	4420	20	99.7
1998-99	4972	6	99.9
1999-00	4758	13	99.8
2000-01	4559	2	99.9
2001-02	4436	0	100.0
2002-03	3233	0	100.0
2003-04	3822	0	100.0
2004-05	3659	2	99.9
2005-06	2953	0	100.0
2006-07	2085	0	100.0
2007-08	2055	0	100.0
2008-09	2621	0	100.0

Table 3 Residue violations in container export grain from 2004-2009.

Year	Samples	Compliance %
2004-05	77	100.0
2005-06	89	100.0
2006-07	168	100.0
2007-08	565	99.6
2008-09	391	98.2

When a pesticide residue is detected in a grain sample that is above the relevant Australian MRL, the contract laboratory immediately notifies NRS. Under NRS-laboratory contract arrangements, the contract laboratory is required to retest the grain sample to confirm the first result. If confirmation is received, the relevant Australian state or territory government and the grain owner/handler and marketer are notified of the residue violation. Should the need arise, the NRS Export Grains Program includes a traceback investigation function which allows State/Territory government officers, in cooperation with the particular grain company, to conduct an examination of any residue detection in grain over the MRL. The investigators trace the grain sample back through transporter to the property of origin to determine the cause of the residue violation. Subsequent actions depend on both the chemical detected, the levels detected and the commodity in which it is found, and are specified by the relevant state and territory government authority legislation. Action can vary from simple advice to the grain grower in the case of a minor problem to legal action where the residue violation has resulted from gross misuse of an agricultural chemical. NRS is provided with report of the traceback investigation and general details are provided in annual reports to industry. NRS forwards residue monitoring results along with traceback investigation report to the APVMA to assist existing chemical reviews processes and close the risk management framework loop.

4. Conclusions

The approach taken by the Australian grain industry to manage pesticide residues has evolved over the past 17 years. The consultative and cooperative framework established between the APVMA, NRS, state government departments of primary industries, Grains Council of Australia and grain marketing organisations has facilitated enhancements to the comprehensiveness of the NRS Grains Program. The expansion of the NRS Grains Program to include all tradable grains and to cover all trading streams has helped raise the program's profile both within Australia and with trading partners. Moreover, the move to assessing and reporting residue testing results against relevant international MRLs, in addition to the standard checks against Australian standards, has provided Australian grain marketers with a complete set of information with which to make appropriate grain marketing decisions. The grain industry has progressively become more involved in managing pesticide residue issues and residue testing data provided by the NRS Grains Program. This has assisted the development of on-farm quality assurance programs and enabled industry to demonstrate long term improvements in pesticide residue management. The residue testing results derived from the NRS Grains Program over the past 12 years and the integration of this information into market access and quality assurance initiatives gives strong guarantees that Australia's grain contains very low and ever declining levels of pesticide residues.