INTERNATIONAL WOOL TEXTILE ORGANISATION

TECHNOLOGY & STANDARDS COMMITTEE **BIELLA MEETING**

Raw Wool Group **November 2005**

Chairman: A.C. BOTES (South Africa) **Report No: RWG 03**

Residues of Pesticides in Australian Wool 2004-2005: Results from AWI Survey

By

C.R.Nunn, I.M.Russell, S.Williams

CSIRO Textile and Fibre Technology	PO Box 21, Belmont, VIC, 3216 Australia
CSIRO Textile and Fibre Technology	PO Box 21, Belmont, VIC, 3216 Australia
Australian Wool Innovation Ltd	Level 6, 369 Royal Parade, Parkville, VIC,3052

SUMMARY

Australian Wool Innovation Ltd (AWI) sponsors an annual survey of pesticide residues in the Australian wool clip. Six hundred samples of fleece wool are selected at random by Australian Wool Testing Authority from sale lots submitted by growers for pre-sale testing. The sample numbers are divided in proportion to the number of samples from each state of Australia, on a monthly basis. Fifty gram sub-samples taken from the 'keeper' sample held by AWTA are transported to CSIRO TFT in Geelong, and analysed according to IWTO Draft Test Method 59.

This paper describes the results of the analysis of the 600 samples entering the AWTA system from the 2004-2005 wool clip. The paper also provides historical trends since 1992-1993. The average concentrations of organophosphates and synthetic pyrethroids have decreased progressively from 10 mg/kg and 6 mg/kg to around 1 mg/kg, however the hydrophobic insect growth regulators, diflubenzuron and triflumuron, have increased from when they were introduced in 1995, to 7 and 12 mg/kg respectively. These latter chemicals are found on just over 50% of the clip. Organochlorines have been eliminated from the clip.

A proportion of the clip (14%) has no reportable pesticides, and 42% of the clip meets the pesticide residue requirements for greasy wool in the EU eco-label criteria. Non-compliance with the EU eco-label in the clip is principally caused by use of diflubenzuron and triflumuron (52% of wool contains more than 2 mg/kg total), while 6% of the clip contains more than 2 mg/kg organophosphate and 6% contains more than 0.5 mg/kg of synthetic pyrethroid (some clips fail the EU eco-label criteria on more than one basis).

By analysis of the residue data and from knowledge of the rates of application and decay rates for different chemical treatments, some quite simple rules can be set up to assist wool producers to voluntarily identify that their wool will meet the EU eco-label requirements.

Roberts Ltd in Tasmania has used these rules to develop its current voluntary residue declaration scheme to identify wools that meet the EU eco-label criteria. While the scheme is based on farm records, it is backed by a chemical residue test taken at random on a single line of wool from each property. The residue test provides feedback to the producer to improve their knowledge of their farming practices, and it also provides assurance to buyers of the wool that the declaration is reliable. By extending this scheme to other producers on mainland Australia, and even without changing current treatment practices, it should be possible for Australia to be able to provide significant quantities of low residue wool meeting the EU eco-label criteria.

BACKGROUND

Since 1992, Australian Wool Innovation Ltd and its precursor organisations have sponsored an annual survey of pesticide residues in the Australian wool clip. Each year, six hundred samples of fleece wool are selected at random by Australian Wool Testing Authority from sale lots submitted by growers for pre-sale testing. The sample numbers are divided in proportion to the number of samples from each state of Australia, on a monthly basis. Fifty gram sub-samples taken from the 'keeper' sample held by AWTA are transported to CSIRO TFT in Geelong, and analysed according to IWTO Draft Test Method 59 and its precursor methods. This laboratory is accredited by NATA to ISO/IEC Standard 17025 for the analysis of greasy wool samples.

While the AWI contract for this testing program required only analysis and reporting of the common organophosphate (OP), synthetic pyrethroid (SP) and insect growth regulator (IGR) pesticides registered for use on sheep in Australia, the analytical method also detects all pesticides required by IWTO Draft Test Method 59, which also corresponds to those pesticides listed in the greasy wool criteria for the EU Eco-label for Textiles [1]. The analytical method used by CSIRO has a Limit of Reporting (LoR) for organophosphates and synthetic pyrethroids of 0.1 mg/kg, 0.05 mg/kg for most organochlorines, and 1 mg/kg for insect growth regulators (triflumuron, diflubenzuron, cyromazine and dicyclanil) and spinosad.

RESULTS FROM 2004-2005 SURVEY

This paper describes the results of the analysis of the 600 fleece wool samples selected randomly by AWTA from the 2004-2005 wool clip. The paper also provides the historical trends since 1992-1993.

The summarised results from the 2004-2005 survey are shown in Table 1. In the summarised tables of results, for the pesticides registered for use in Australia, the following data are reported:

mean residues on all wool tested in the survey;

mean residues when the specific pesticide is present above the Limit of Reporting (LoR);

median residue concentration,

highest concentration found,

percentage of lots with 'nil' residues, and

percentage of lots with residues above the LoR.

2004/2005 AWI Wool Residue Survey **% Compliant with EU eco-label for textiles** **42%** **Reason for non-compliance:**

OP	**6%**
SP	**6%**
DFB/TFM	**52%**

Number of Samples Analysed 600

Mean Pesticide Residues on Australian Fleece Wool 2004/2005 (mg/kg greasy wool)

	Organophosphates				Synthetic pyrethroids				IGRs			
	Total	**Diazinon**	**Propetamphos**	**Chlorfenvinphos**	**Total**	**Cypermethrin**	**Deltamethrin**	**Cyhalothrin**	**Cyromazine**	**Dicyclanil**	**Diflubenzuron**	**Triflumuron**
Mean Residue on all Wool (mg/kg)	**1.1**	1.1	0.0	0.0	**0.9**	0.9	0.0	0.0	9.2	3.1	7.2	12.3
% samples with residues>LoR	**36.3%**	34.7%	0.7%	1.8%	**9.2%**	9.0%	0.2%	0.0%	34.3%	13.5%	28.3%	34.5%
Mean residue when treated (mg/kg)		3.0	0.5	0.6		10.0	0.6	0.0	26.0	22.7	25.4	35.5
Median residue in survey (mg/kg)		0.5	0.5	0.2		1.5	0.6	0.0	12.0	9.5	20.0	30.0
Highest Residue in Survey (mg/kg)	70	70	1.0	4.6	100	100	0.6	0	260	200	140	260
Samples with 'nil residues'	83	14%										

Pesticide distribution on wool lots

	Organophosphates		Synthetic pyrethroid		Cyromazine		Dicyclanil		Diflubenzuron		Triflumuron	
Concentration range (mg/kg)	**% sales lots**	**% residue load**	**% sales lots**	**% residue load**	**% sales lots**	**% residue load**	**% sales lots**	**% residue load**	**% sales lots**	**% residue load**	**% sales lots**	**% residue load**
<1	86%	7%	94%	1%	65%	0%	86%	0%	72%	0%	65%	0%
1.0-9.9	12%	36%	4%	11%	16%	8%	7%	9%	9%	4%	7%	3%
10.0-24.9	1%	14%	1%	16%	8%	14%	3%	17%	8%	19%	8%	12%
25-49.5	1%	23%	1%	39%	6%	23%	2%	24%	7%	35%	11%	31%
>=50	0.3%	20%	0.3%	33%	6%	55%	2%	50%	4%	41%	9%	54%

Table 1. 2004/2005 AWI Wool Residue Survey Results

A proportion of the clip (14%) has no reportable pesticides, and 42% of the clip meets the pesticide residue requirements for greasy wool in the EU eco-label criteria. (The current EU eco-label requirements for pesticide concentration in greasy wool are total organochlorines 0.5 mg/kg, total synthetic pyrethroids 0.5 mg/kg, total organophosphates 2 mg/kg and 'diflubenzuron + triflumuron' 2 mg/kg. Dicyclanil, cyromazine and spinosad are exempt under the current criteria.)

Non-compliance with the EU eco-label in the clip is principally caused by use of diflubenzuron and triflumuron (52% of wool contains more than 2 mg/kg total), while 6% of the clip contains more than 2 mg/kg organophosphate and 6% contains more than 0.5 mg/kg of synthetic pyrethroid (some clips fail the EU eco-label criteria on more than one basis).

In Table 1, the percentages of wool with reported residues may represent an approximation of the market share held by a particular active agent, however it is possible that this figure underestimates the usage of pesticides that degrade reasonably rapidly. For instance, organophosphate and synthetic pyrethroid residues may have degraded to below the LoR on sheep treated very early in the growing season in hot sunny regions, or there may be wool lots from mixed flocks where only a proportion of sheep have been treated.

Results not shown in this paper confirm that organochlorines have been eliminated from the Australian wool clip.

Table 2 shows the historical trend results since 1992 – 1993. The average concentrations of organophosphates and synthetic pyrethroids have decreased progressively from 10 mg/kg and 6 mg/kg to around 1 mg/kg, however the hydrophobic insect growth regulators, diflubenzuron and triflumuron, have increased from when they were included in the survey (1996-1997), to 7 and 12 mg/kg respectively.

Mean pesticide residues in Australian fleece wool (mg / kg greasy wool)

Year	OPs	SPs	Cyromazine	Dicyclanil	Diflubenzuron	Triflumuron
1992/93	10.2	5.8	-	-	-	-
1993/94	9.0	6.6	-	-	-	-
1994/95	4.3	5.7	5.2	-	-	-
1995/96	4.4	5.5	6.3	-	-	-
1996/97	4.5	3.8	8.7	-	1.2	3.5
1997/98	5.8	3.3	5.8	-	3.6	6.1
1998/99	2.4	1.6	7.4	-	3.5	7.6
1999/00	2.2	2.0	5.1	0.1	2.9	9.0
2000/01	1.6	1.3	10.2	0.4	5.5	8.8
2001/02	1.4	0.8	8.6	1.8	4.3	11.5
2002/03	1.4	0.7	6.0	2.1	7.2	11.1
2003/04	1.1	1.1	7.5	1.5	5.9	9.7
2004/05	1.1	0.9	9.2	3.1	7.2	12.3

Table 2. Historical Data: 1992-93 to 2004-05 AWI Wool Residue Survey

Synthetic pyrethroids

Apparent usage of synthetic pyrethroids on Australian wool continued to decline. Residues were reportable on 9% of the samples tested, with an average concentration of 0.9 mg/kg. Cypermethrin was the major synthetic pyrethroid used. The highest synthetic pyrethroid residue found was 100 mg/kg of cypermethrin. Where high concentrations of cypermethrin were found, they were predominantly as alpha-cypermethrin, and this is consistent with the formulations registered for late-season use. As with the organophosphates, these few high residue lots remain a problem. Just over 2% of fleece wools contained residues above 10 mg/kg, and these lots held 90% of the residue load.

Cyromazine and dicyclanil

The average concentration for cyromazine was 9.2 mg/kg, and for dicyclanil was 3.1 mg/kg. The highest residue found for dicyclanil was 200 mg/kg, with the highest cyromazine concentration being 260 mg/kg. Use of these treatments is dependent on climatic conditions during the year. Cyromazine was found on 34% and dicyclanil found on 14% of the sales lots. However these fly-specific chemicals are not included in the EU eco-label criteria.

Spinosad

Spinosad was not detected on any samples. This may be due to its low market share, or, more probably, to its rapid degradation kinetics.

Organophosphates

Organophosphate residues above the LoR were reported on 36% of the clip, however because much of the diazinon use is early in the growing season, and since diazinon degrades reasonably rapidly on sheep under Australian wool-growing conditions, this probably underestimates the frequency of its use. Diazinon is the predominant organophosphate treatment used. The average concentration of organophosphates on the wool clip was just over 1 mg/kg, and because they are reported on around 1/3rd of Australian wool, the average concentration when they are reported was 3 mg/kg.

A detailed analysis of the diazinon residues in the survey indicates that its occurrence is highly skewed. It was found, at or above its Limit of Reporting (0.1 mg/kg), in 214 of the 600 samples tested (Figure 1). Only around 50 of the 600 samples exceed the EU eco-label target of 2 mg/kg. Most usage of diazinon (producing residues both above and below the LoR) is within the EU eco-label requirement.

The highest diazinon residue found was 70 mg/kg. It is likely that this, and the few other high residue results, arose from a treatments applied late in the wool growing season. These few high residue lots remain a problem and just over 2% of the samples tested contained nearly 60% of the residue load.

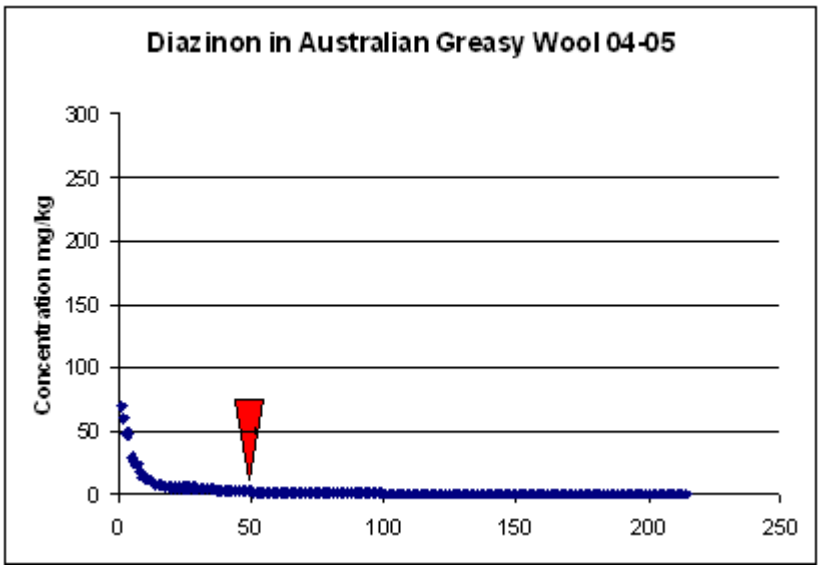

Figure 1: Diazinon reported in 600 Australian Fleece Wools 2004-2005 (the abscissa has been truncated). Marker shows samples above and below EU eco-label criteria (2 mg/kg).

Diflubenzuron and triflumuron

Triflumuron maintains a larger market share than diflubenzuron (35% compared with 28%). The highest individual residues were 140 mg/kg for diflubenzuron and 260 mg/kg for triflumuron, and again, given the known degradation rates for these compounds, these residues are likely to be from late season treatments.

The majority of treatments with diflubenzuron and triflumuron are usually applied off-shears or very early in the growing season and with fixed volume applicators. The average concentrations on the clip where these treatments were detected were 25 and 36 mg/kg respectively (Table 1). A detailed analysis of the residue patterns shows that almost all usage of either of these materials exceeds the EU eco-label targets (Figure 2). These materials are more persistent on sheep that the organophosphates and synthetic pyrethroids.

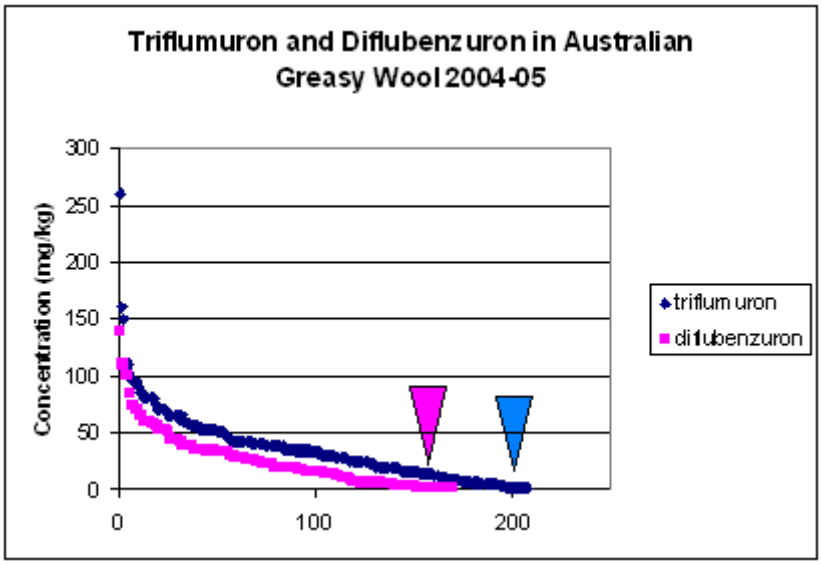

Figure 2: Diflubenzuron and triflumuron reported in 600 Australian Fleece Wools 2004-2005 (the abscissa has been truncated). Marker shows samples above and below EU eco-label criteria (2 mg/kg).

CONCLUSION

Australia consistently produces the cleanest wool in the world, and significant quantities of low residue wools are being produced. The data for 2004/2005 shows that 42% of the fleece lines tested have residue levels less than the pesticide criteria for the EU Eco-label for Textiles. As environmental pressure in Europe grows with the implementation of the Integrated Pollution Prevention and Control (IPPC) legislation by 2007 in all EU member states, wool processors will need to source low residue wools (greasy as well as scoured) to meet their reporting needs under this legislation.

However, the organophosphates are the only group of pesticides where the mean residue concentration in 2004/2005 is less than the EU eco-label criteria. For the synthetic pyrethroids, and the hydrophobic IGRs diflubenzuron and triflumuron, the average concentrations in the overall survey are significantly higher than the eco-label criteria. This means that an EU eco-label compliant processing lot cannot be assembled by random selection of clips, as the inclusion of one farm lot containing high residues would invalidate the total wool lot. Australia is working to develop mechanisms for pre-sale identification of the low residue wool lots in the market place.

REFERENCES

1. http://europa.eu.int/comm/environment/ecolabel/product/pg_clothing_textiles_en.htm