

Risk Management Tools for the Recycled Organics Industry

Department of **Environment and Conservation** NSW



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Contents

Section 1 About this report.....	4
1.1 Objectives of the report	4
1.2 Who is the report for?	5
1.3 How to use the report	5
1.4 Terminology	6
1.5 Acknowledgement	6
Section 2 Introduction to persistent herbicides and the recycled organics industry	7
2.1 Background	7
2.2 Potentially persistent herbicides	7
Section 3 Raw materials	12
3.1 Introduction	12
3.2 Potentially contaminated raw materials	12
Section 4 Taking a Representative Sample for Testing	17
4.1 Introduction	17
4.2 Information Sheet 3-3: On-site Field Testing and Monitoring for Quality	17
Section 5 Sample Management for Consistent Analysis of Products and Raw Materials	24
5.1 Introduction	24
5.2 Information Sheet 3–11: Sample Management for Consistent Analysis of Products and Raw Materials	24
Section 6 Herbicide detection using plant bioassay	29
6.1 Introduction	29
6.2 Using this bioassay	31
6.3 Bioassay method for determination of herbicide toxicity to plants	31
Section 7 Commercial laboratories	40
7.1 Introduction	40
7.2 Virotec Global Solutions	40
7.3 State Chemistry Laboratory	41
Section 8 Acid herbicides analytical test method	43
8.1 Acid herbicides test	43
Section 9 Tolerance levels	45
9.1 Introduction	45
9.2 Sensitive crops and applications	45
9.3 Tolerant applications	48
9.4 Verification	50
Section 10 Recommendations	51
Section 11 References	52
Appendix 1 Glossary	55

Section 1 About this report

1.1 Objectives of the report

The *recycled organics industry* in New South Wales has received substantial State Government investment to support sustainable development objectives in the area of the diversion of *compostable organic materials* from landfill. The industry is reliant on the consistent production of quality *recycled organics* products, however, recent incidents overseas have indicated the potential for *herbicide* contamination of recycled organics products.

Whilst further research into this issue is conducted, it is important for processors and manufacturers of recycled organics to be aware of these potential threats. As a precautionary step, this report has been developed to inform the recycled organics industry of the potential threat from three specific herbicides to various crops and applications and to assist producers of recycled organics products to avoid the problems that have occurred overseas.

A risk assessment of potentially *persistent* herbicides in New South Wales was conducted in response to incidents of contamination reported internationally. This risk assessment identified that three potentially problematic herbicides, clopyralid, picloram and triclopyr, are registered for various applications in Australia (Recycled Organics Unit, 2002c). However this study also indicated that in New South Wales, there is currently:

1. No evidence of the use of these problematic herbicides in applications from which raw materials for commercial *compost* production are commonly sourced; and
2. No evidence of any herbicide contamination issues in commercial composts manufactured in New South Wales (or Australia).

However, as these *agricultural chemicals* are registered for use in Australia, it is prudent and responsible to address this potential risk in a pre-emptive manner to ensure that the problems experienced overseas are not repeated in Australia. Consequently, a risk management program was recommended to determine the threat to the recycled organics industry in Australia posed by these chemicals.

In the short term, the organics recycling and processing industry requires a range of resources and services to manage the potential risks posed by these potentially persistent chemicals. These resources and services will enable the organics recycling and processing industry to manage potential risk, whilst longer term and sustainable resolution of this issue can be addressed.

This report provides access to such resources and services as are required to manage these risks in the short term. The objectives of this report are:

- To identify and document raw materials (compostable organic materials) that are commonly processed into composts in New South Wales that could potentially contain specific herbicides known to be persistent beyond the commercial *composting* process;
- To document the tolerance levels at which different crops/applications are known to be susceptible to these specific herbicides that are known to be persistent beyond the commercial composting process;

- To ensure cost effective and appropriate commercial analytical testing services are available to the organics recycling and processing industry to enable the testing of both raw materials and composted products for presence of these specific herbicides known to be persistent beyond the commercial composting process;
- To clearly document a bioassay method for the testing of composted products for the presence of these specific herbicides known to be persistent beyond the commercial composting process. This bioassay shall be suitable for use by organics recycling and processing enterprises and/or commercial laboratories; and
- To define and document a sampling method for the representative sampling of composts from windrows for the purpose of testing composted products for the presence of herbicides known to be persistent beyond the commercial composting process.

1.2 Who is the report for?

This document has specifically been developed for commercial composting facilities and producers of recycled organics products. This report will be of direct interest to:

- Department of Environment and Conservation NSW
- Manufacturers of recycled organics products
- Recycled organics industry associations
- Marketers of recycled organics products
- Consumers of recycled organics products

1.3 How to use the report

The document is designed as an informative for the recycled organics industry to ensure raw materials and finished products for identified sensitive applications do not contain specific herbicides known to be persistent beyond the commercial composting process. The presence of these herbicides overseas in composts for agricultural/horticultural applications has threatened the viability of the recycled organics industry in parts of North America and New Zealand and resulted in significant losses to composting operations due to loss of market confidence.

This document contains a number of sections that detail the issue of persistent herbicide contamination of *feedstocks* and compost that has occurred internationally. A list of raw materials that have been found to contain these specific persistent herbicides overseas and/or have the potential to be contaminated are listed to allow composting facilities to be aware of such risks when considering the quality of new and/or existing sources of raw materials.

An information sheet detailing on-site methods for quality testing and sampling is included which provides guidance for producers of recycled organics products on how to representatively sample products for analysis. A second information sheet provides details on managing samples to support consistent analysis of quality of both raw materials and compost products.

A bioassay method is also included in the document that allows herbicide contamination of compost products to be detected via observing the germination and growth of seeds in a compost mix under a

controlled environment. Bioassay results that are cause for concern can be confirmed via laboratory analysis, and information on laboratories capable of testing for the presence of these specific persistent herbicides is included in the document with an outline of the method of analysis.

Finally, this document contains a table of tolerance levels for a variety of crops and applications to particular persistent herbicides. This will help in informing the possible uses of products that are contaminated with these specific persistent herbicides.

A glossary is contained at the end of the document detailing all terms used throughout the report.

This document should enable producers of recycled organics products to understand the potential risk to the recycled organics industry from these specific herbicides that have demonstrated persistence overseas, and allow measures to be taken to prevent such contamination risks and associated losses from occurring in Australia. Avoiding these risks will therefore assist in protecting the viability of the recycled organics industry in Australia.

1.4 Terminology

Terms used throughout this report have been officially adopted by the NSW Waste Boards in July 2000 in the form of the *RO Dictionary and Thesaurus: Standard terminology for the recycled organics industry* (Recycled Organics Unit, 2002b). This document can be freely downloaded from <http://www.rolibrary.com>.

1.5 Acknowledgement

The Recycled Organics Unit (ROU) would like to thank The Department of Environment and Conservation NSW for providing funding to make this research possible.

Dr William Brinton of Woods End Research Laboratory, United States, is thanked for his assistance in providing a draft bioassay method for acid herbicides and allowing this method to be adapted for the Recycled Organics Industry in New South Wales.

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Section 2 Introduction to persistent herbicides and the recycled organics industry

2.1 Background

Incidents in the United States and in New Zealand have revealed that there is a potential threat to the recycled organics industry in Australia from contamination of recycled organics products by a number of specific persistent herbicides. In response to these incidents, a risk assessment was conducted to identify chemicals available in New South Wales that may pose a threat to producers and consumers of recycled organics products in New South Wales (Recycled Organics Unit, 2002c).

This document provides information to producers of recycled organics products in regard to the susceptibility of crops and other applications to three specific persistent herbicides: clopyralid, picloram and triclopyr; and also the potential for a range of raw materials to be contaminated with these herbicides prior to processing. Although this document does not provide a solution to the problem of persistent herbicides, it provides risk management tools for processors to use to avoid potential contamination and associated economic losses whilst this issue is being addressed.

It should be noted that no contamination of recycled organics products by these specific persistent herbicides has been known to occur in Australia. However, the incidents reported internationally highlight the potential threat to the industry and the need for immediate action to avoid such risks to the recycled organics industry in Australia.

2.2 Potentially persistent herbicides

The recent risk assessment of potentially persistent garden maintenance chemicals identified three specific herbicides that could potentially cause a threat to the recycled organics industry in New South Wales. These herbicides are clopyralid, picloram and triclopyr (Recycled Organics Unit, 2002c). Clopyralid and picloram in particular, have been shown to persist beyond the commercial composting cycle in the United States and in New Zealand, and have resulted in significant economic loss to a number of composting enterprises in these countries. General descriptions of these herbicides including mode of action, use and commonly available commercial products are given below. Clopyralid, picloram and triclopyr are often referred to as *acid herbicides* or *auxin-like herbicides*.

2.2.1 Clopyralid

Clopyralid is a pyridine carboxylic acid herbicide used to control annual and perennial *broadleaf* weeds in turf, pastures and some agricultural crops such as wheat barley, oats sugar beets and mint. Clopyralid is a synthetic plant growth hormone that has some structural similarities to naturally occurring hormones called auxins. Clopyralid disrupts plant growth by binding to molecules that are normally used as receptors for the natural growth hormones and because clopyralid is more persistent in plant tissue than these auxins, the binding causes abnormal growth leading to plant death within a few days or weeks, depending on the species (Cox, 1998a).

Some general chemical properties of clopyralid are shown in Table 2.1. Plants subjected to clopyralid may appear almost normal but there is generally a loss of apical dominance that is likely to prevent fruit set. Some leaves that are supposed to be compound become single leaves and side shoots may develop where they should not (Figure 2.1). Cupping of the leaves is also a typical symptom for legumes (Bezdicsek *et al.*, 2001).

Table 2.1: General chemical properties of clopyralid.

Chemical name (IUPAC*)	Formula	Half-life (days)	Solubility	Volatility	K _{OC} **
3,6-dichloropyridine-2-carboxylic acid	C ₆ H ₃ Cl ₂ N ₂ O ₂	56-392 (Cox, 1998a)	1000 mg/L (very soluble) (Bezdicek <i>et al.</i> , 2001; Cox, 1998a)	Considered volatile by US EPA (Cox, 1998a)	6 (indicates mobility) (Vogue <i>et al.</i> , 1994; Fauci <i>et al.</i> , 2002)

* International Union of Pure and Applied Chemistry; **K_{OC} is the tendency of a pesticide to bind to soil particles.

Examples of products registered for use in Australia containing clopyralid are:

- Lontrel™ Herbicide (Dow AgroSciences) – Used to control broadleaf weeds in wheat, barley, oats, triticale, canola, pastures, fallow land, forests and industrial situations.
- Chipco Spearhead (Bayer CropScience) – Used to control broadleaf weeds in turf.

Figure 2.1. Typical clopyralid damage including cupping and slight twisting of leaves (Bezdicek *et al.*, 2000).



2.2.2 Picloram

Picloram is a pyridine herbicide that can be formulated as an acid (technical product), potassium or triisopropanolamine salt, or an isooctyl ester. Picloram is available as soluble concentrates, pellets or granular formulations (Extension Toxicology Network, 2002). Picloram is a *systemic* herbicide used for control of woody plants and a wide range of broadleaf weeds in pastures, rangeland, reforestation programs, uncultivated areas and along *rights-of-way* (Cox, 1998b; Bezdicek *et al.*, 2000).

Picloram kills plants by acting like plant growth hormones or auxins. Picloram is more persistent than auxins and so inhibits the enzymes that normally break down these auxins. This results in the disruption of normal plant growth and causes abnormal stimulation and maturation of tissues. Plant growth then stops and the roots of the plants deteriorate resulting in death (Cox, 1998b) as shown in Figure 2.2.

Figure 2.2. Typical picloram damage including leaf cupping and distortion of stems (Bezdicsek *et al.*, 2000).



Picloram is readily absorbed by plant roots, less so by the foliage, and is readily translocated throughout the plant. Picloram remains stable and intact within the plant (Extension Toxicology Network, 2002). Some general chemical properties of picloram are shown in Table 2.2.

Table 2.2: General chemical properties of picloram.

Chemical name (IUPAC*)	Formula	Half-life (days)	Solubility	Volatility	K _{oc} **
4-amino-3,5,6-trichloropyridine-2-carboxylic acid	C ₆ H ₃ Cl ₂ NO ₂	20-300 (Extension Toxicology Network, 2002)	430 mg/L (highly soluble) (Bezdicsek <i>et al.</i> , 2001)	Practically nil (Extension Toxicology Network, 2002)	16 (indicates mobility) (Vogue <i>et al.</i> , 1994; Fauci <i>et al.</i> , 2002)

* International Union of Pure and Applied Chemistry; **K_{oc} is the tendency of a pesticide to bind to soil particles.

Examples of products registered for use in Australia containing picloram are:

- Tordon™ 75-D Herbicide (Dow AgroSciences) – Used to control a wide range of annual and perennial broadleaf weeds.
- Grazon™ DS Herbicide (Dow AgroSciences) – Used to control a wide range of environmental and noxious woody and herbaceous weeds.

2.2.3 Triclopyr

Triclopyr is a pyridine compound similar to clopyralid and picloram. Triclopyr is a *selective* systemic herbicide generally used for the control of woody and broadleaf plants typically along rights-of-way, in forests, industrial lands, grasslands and parks (Cox, 2000).

Triclopyr acts by imitating a plant hormone called indoleacetic acid, which is one of a number of plant hormones classified as auxins. Triclopyr causes the growing tips of a plant to elongate and become distorted resulting in withering of the plant and finally death. Triclopyr is selective, being mostly toxic to broadleaf plants, as grasses are quickly able to transform triclopyr into compounds that do not have hormonal activity (Cox, 2000). Some general chemical properties of triclopyr are shown in Table 2.3.

Table 2.3: General chemical properties of triclopyr.

Chemical name (IUPAC*)	Formula	Half-life (days)	Solubility	Volatility	K _{oc} **
3,5,6-trichloro-2-pyridyloxyacetic acid	C ₇ H ₄ Cl ₃ NO ₃	30-90 days (Extension Toxicology Network, 2002)	440 mg/L (soluble) (Extension Toxicology Network, 2002)	Very low (Information Ventures, 2002)	20 (indicates mobility) (Vogue <i>et al.</i> , 1994)

* International Union of Pure and Applied Chemistry; **K_{oc} is the tendency of a pesticide to bind to soil particles.

Examples of products registered for use in Australia containing triclopyr are:

- Hortico Blackberry and Tree Killer (Hortico Australia Pty Ltd) – Used to control blackberries and problem trees in the home garden.
- Chemspray Tree, Blackberry & Woody Weed Killer (Envirogreen Pty Ltd) – Used to control blackberry, eucalyptus, wattle and many other weeds.
- Garlon™ 600 (Dow AgroSciences) – Used to control a range of woody weeds and melons.

2.2.4 Other herbicides

There is no evidence of any herbicide contamination in commercially produced composts in Australia, and no evidence of any herbicides other than the three specified above being capable of surviving the composting process (Recycled Organics Unit, 2002c). Internationally, millions of tonnes of commercially produced compost have been sold over the past decade, yet there have been no problems reported arising from herbicide-contaminated composts prior to the advent of clopyralid, picloram and triclopyr.

Compost manufacturers may consider the risk (however remote) of general herbicide contamination via the *Toxicity Index* test specified in AS 4454 (Standards Australia, 1999). In the instance that compost products that meet maturity, pH and EC requirements fail this toxicity test, one reason for failure could be due to the presence of herbicides.

The NSW EPA *Utilisation of Treated Effluent by Irrigation* (1995) specifies a maximum permissible concentration of total “organochlorine pesticides” (including both insecticides and herbicides) in irrigation water of 0.001 ppm, and the ANZECC (1992) *Australian Water Quality Management Guidelines* specify a maximum permissible concentration of a range of commonly used herbicides of 0.1 ppm. As a result analytical laboratories offer standard general testing services for herbicides (e.g. glyphosate, paraquat, simazine, dicamba, atrazine etc.).

The difference between commonly used herbicides and the three specific herbicides that are shown to persist beyond the composting cycle is that clopyralid, picloram and triclopyr have the potential to cause economic damage to crops at such minute concentrations (1 ppb) that normal analytical tests are of no value.

As such, commonly available analytical tests for herbicides should be suitable for testing for potential herbicide concentration (in the unlikely event that this is required) for commonly used herbicides other than clopyralid, picloram and triclopyr.

Section 3 Raw materials

3.1 Introduction

This section provides a list of raw materials that have been found overseas to contain the specific persistent herbicides clopyralid, picloram and triclopyr and/or have the potential to be contaminated with these specific herbicides. This list allows composting facilities to be aware of such risks when considering the quality of new and/or existing sources of raw materials.

Processors of compostable organic materials accept raw materials from a range of sources in New South Wales. The extent of the threat from persistent herbicides in New South Wales is not clear at this stage. Consequently, it is important to be aware of the risks posed from these herbicides, to consider international experience, and the risk of such incidents occurring in Australia.

3.2 Potentially contaminated raw materials

A review of raw materials that have reportedly been contaminated with the herbicides clopyralid, picloram and triclopyr is given in Table 3.1. Also included in this table are raw materials that could potentially be contaminated with these herbicides. This list has been derived from a range of literature including published articles, herbicide *labels*, general herbicide characteristics and chemical manufacturer information and has been informed by the application for which these herbicides are registered in Australia. No confirmatory testing has been conducted in Australia, and comprehensive testing of raw materials has not been conducted overseas. This table is not a comprehensive review and should only be used as a guide.

Processors are advised to be diligent in investigating whether these persistent herbicides could be present in raw materials from various sources prior to accepting them for processing. Alternatively, laboratory analysis of raw material from new sources can be performed prior to accepting the material to confirm that the raw material does not pose a contamination threat to the facility. Details on sample management for raw materials, and for provision of analytical laboratory services are given in [Section 5](#) and [Section 7](#) respectively.

Table 3.1. Raw materials that risk potential contamination with the herbicides clopyralid, picloram and triclopyr. Note: Reported contamination by these chemicals is highlighted.

Raw material	CLOPYRALID		PICLORAM		TRICLOPYR	
		Details	Source		Details	Source
Barley	P	AUS: Application according to product label.	Dow AgroSciences (1998a)	P	AUS: Application according to product label.	Dow AgroSciences (1998b)
	✓	US: Contaminated barley used as a grain feed	Rynk (2002a)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)
Blackberry		--		P	AUS: Potential for contamination due to commercial products available.	e.g. Dow AgroSciences (2001)
Bracken and ferns		--		P	INTER: For example from grassland and non-cop areas	Tomlin (1997)
Canary grass		--		P	AUS: Potential contamination due to commercial products available.	Dow AgroSciences (1998b)
Canola	P	AUS: Application according to product label.	Dow AgroSciences (1998a)		--	--
Eucalyptus		--		P	AUS: Application according to product label.	Dow AgroSciences (1998b)
Garden organics (garden waste)	P	AUS: Potential for contamination due to commercial products available.	e.g. Bayer Cropscience Pty Ltd (2002)	P	AUS: Potential for contamination due to commercial products available.	e.g. Dow AgroSciences (2001)
	✓	US: Detected in raw feedstock material accepted at composting facility.	Rynk (2002a)			
	✓	NZ: Detected in raw feedstock material accepted at composting facility (predominantly from lawn clippings).	Fietje (2001)			
Lantana		--		P	AUS: Application according to product label.	Dow AgroSciences (1998b)
				P	AUS: For example from home gardens.	Hortico (Australia) Pty Ltd (no date)

Raw material	CLOPYRALID		PICLORAM		TRICLOPYR	
		Details	Source		Details	Source
Lawn clippings	P	AUS: Products used for the control of broadleaf weeds in turf/lawns.	Bayer Cropscience Pty Ltd (2002) plus others	P	AUS: Most grasses are resistant to picloram so it is used in range management programs.	(Extension Toxicology Network, 2002)
	✓	US: Clippings from lawns treated with clopyralid are accepted at commercial composting operations and have contaminated finished compost.	Washington State Department of Agriculture (2002)	P	INTER: Most grasses are resistant to picloram so it is used in range management programs.	(Extension Toxicology Network, 2002)
	✓	NZ: Clippings from lawns treated with clopyralid are accepted at commercial composting operations and have contaminated finished compost.	Fietje (2001)			
Leaves	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)		--	--
Lucerne		--		P	AUS: Potential contamination due to commercial products available.	e.g. Dow AgroSciences (2001)
Maize		--		P	AUS: Application according to product label.	Dow AgroSciences (1998b)
Manure (beef cattle)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)
Manure (chicken)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)		--	--
Manure (dairy cattle)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)
Manure (feedlot)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)		--	--
Manure (horse)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)	✓	US: Detected in raw material accepted at composting facility.	Rynk (2002a)
Mint sludge	P	US: Potentially contaminated.	Rynk (2000)		--	--

Raw material	CLOPYRALID		PICLORAM		TRICLOPYR	
	Details	Source	Details	Source	Details	Source
Non-woody plants	--		--		P INTER: For example, used to control annual and perennial herbaceous weeds in pastures, forestry, site preparation, conifer release, industrial sites and rangeland.	Tomlin (1997)
Oats	P AUS: Application according to product label.	Dow AgroSciences (1998a)	P AUS: Application according to product label.	Dow AgroSciences (1998b)	--	
Sorghum	--		P AUS: Application according to product label.	Dow AgroSciences (1998b)	--	
Spent mushroom media	P AUS: Potential contamination from contaminated straw.	AMGA (2002)	--		--	
	P US: Potentially contaminated	Rynk (2001)				
Straw	P AUS: Potential contamination due to commercial products available.	AMGA (2002)	--		--	
	✓ US: Detected in raw material accepted at composting facility.	Rynk (2002a)				
Straw (and manure)	✓ US: Sourced from fair, including manure.	Rynk (2002a)	--		--	
Sugar beet	P US: Potentially contaminated.	Rynk (2000)	--		--	
Sugar cane	--		P AUS: Application according to product label.	Dow AgroSciences (1998b)	--	
Tea tree	--		--		P AUS: For example from home gardens.	Hortico (Australia) Pty Ltd (no date)
Timothy hay	✓ US: Detected in raw material accepted at composting facility.	(Rynk, 2002a)	✓ US: Detected in raw material accepted at composting facility.	Rynk (2002a)	--	
Triticale	P AUS: Application according to product label.	Dow AgroSciences (1998a)	P AUS: Application according to product label.	Dow AgroSciences (1998b)	--	

Raw material	CLOPYRALID		PICLORAM		TRICLOPYR	
	Details	Source	Details	Source	Details	Source
Wattle	--		P AUS: Potential contamination due to commercial products available.	e.g. Dow AgroSciences (2001)	P AUS: For example from home gardens, parks, golf courses and factories.	Envirogreen Pty Ltd (2002)
Wheat	P AUS: Application according to product label.	Dow AgroSciences (1998a)	P AUS: Application according to product label.	Dow AgroSciences (1998b)	--	
Woody plants	--		P AUS: Potential contamination due to commercial products available.	e.g. Dow AgroSciences (2001)	P INTER: For example nettles, docks, brambles, gorse, broom from grassland, uncultivated land, industrial areas, coniferous forests, plantation crops and rice fields.	Tomlin (1997)
			✓ INTER: For example from grassland and non-crop areas	Tomlin (1997)		

KEY: P – potentially contaminated, ✓ – contamination has occurred and this contamination has been reported in the literature (shaded cells).
INTER – Potential/actual contamination has been reported in international literature; **AUS** – Potential/actual contamination has been reported in Australia, **US** – Potential/actual contamination has been reported in the United States; **NZ** – Potential/actual contamination has been reported in New Zealand.

NOTE: This table is not a complete list of all reports of contamination of these specific herbicides.
 This table provides a guide for the type of raw materials accepted at composting facilities that should be investigated for potential contamination by the herbicides clopyralid, picloram and triclopyr.
 Absence of data in this table does not indicate that risk of contamination from these specific herbicides does not exist, please note that no comprehensive studies of contamination of raw materials have been conducted anywhere in the world.

Section 4 Taking a Representative Sample for Testing

4.1 Introduction

Compost or raw material samples can be tested to identify potential contamination by herbicides or other substances. However, a representative sample must be taken for the analysis of the material to be valid. The Recycled Organics Unit has developed an information package to assist producers of recycled organics products. This information package includes guidance in taking representative samples for laboratory analysis and is titled *Producing Quality Compost* (Recycled Organics Unit, 2002a).

The *Producing Quality Compost* package contains a number of information sheets that inform the consistent production of quality compost and other recycled organics products. Information Sheet No. 3-3 *On-Site Field Testing and Monitoring for Quality* in this package includes a method for taking a representative sample of compost.

4.2 Information Sheet 3-3: On-site Field Testing and Monitoring for Quality

The relevant sections of this information sheet are reproduced here to provide a method for taking a representative sample, which is the first step required for on-site and/or laboratory analysis of a compost sample. The subsequent section, [Section 5](#), details associated methods and protocols for sample management.

The entire package of the *Producing Quality Compost* information sheets can be freely downloaded from <http://www.recycledorganics.com>

Information Sheet No. 3–3

On-site Field Testing and Monitoring for Quality

Information Sheet No. 3–3

December 2002

Inside This Sheet

1 Why is on-site field testing and monitoring important?

Easy-to-do field tests

2 Definitions

3 Field testing safety tips

Sampling

5 Notes

Important references and acknowledgement

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Why is on-site field testing and monitoring important?

On-site field testing procedures are employed at a composting facility to maintain *process control* and to generate products of consistent quality.

Testing and monitoring is a core component of a *Quality Management System (QMS)*, required to ensure that the systems in an organisation are performing effectively.

Process control is defined as the stringent and documented monitoring of all critical control points in a composting operation so as to minimise defects and make products which can be guaranteed to customers (Recycled Organics Unit, 2002).

Products that vary in quality over time may result in customer dissatisfaction and loss of business. This can occur when manufacturing systems are not regularly checked and when products are not tested against pre-determined quality criteria.

To avoid releasing non-conforming products containing recycled organics into the market place — that is, product which does not conform to specification — testing and monitoring procedures need to be employed at all stages of production.

There are two categories of testing that can be performed on-site.

Rapid, on-site testing can be done in the field (e.g. at the composting pile) with lightweight and portable scientific equipment.

Field tests can be performed quickly, allowing the immediate identification of problems. This allows problems to be quickly rectified, thus minimising impacts on final product quality.

The second type of on-site testing possible at a composting facility is laboratory testing. This involves the analysis of samples in a dedicated room, usually with non-portable and more accurate equipment than that employed for field testing.

On-site laboratory tests that can be used to maintain process control and product quality at a composting facility are discussed in Information Sheet No. 3-4.

This Information Sheet reviews easy, inexpensive field tests that can be used to form the basis of a testing program at a composting facility.

Easy-to-do field tests

Field testing should be performed at all stages of the production cycle, though the stages will vary depending on types of *feedstocks* processed, how they are managed on-site, and what type of products are manufactured.

Please note that tests described in this Information Sheet are targeted towards composting operations.

Tests performed in the field can also play an important role in environmental management. For example, monitoring of oxygen content within a *turned pile* can indicate when odour formation is

likely to occur. Interventions, such as aeration or turning may be needed so that anaerobic or low oxygen conditions do not form, thereby minimising the potential for odour production and impacts on air quality.

Tests used do not necessarily have to conform to those outlined in an Australian Standard or other relevant standard.

What is important is that tests are employed regularly and consistently to maintain product quality, most effectively achieved through the application of a well documented QMS.

It is important to note that not all tests can be done in the field or in an on-site laboratory. When a new type of feedstock is accepted at a composting facility, for example, nutrient content may have to be analysed by an off-site laboratory to allow a compost recipe to be formulated.

Field tests described here are quick and easy to perform. Tests are described for:

- temperature;
- moisture content;
- pH;
- maturity; and

between 1.5 and 3 m in height. Turning assists in: aeration and oxygen re-supply; eliminating odours; reducing consolidation, and moisture and nutrient re-distribution.

Pasteurisation

The process whereby organic materials are treated to kill plant and animal pathogens and weed propagules.

In-vessel

System of composting involving the use of an enclosed chamber or vessel in which (in most cases) the composting process is controlled by regulating the rate of mechanical aeration. Aeration assists in heat removal, temperature control and oxygenation of the mass. Aeration is provided to the chamber by a blower fan which can work in a positive (blowing) and/or negative (sucking) mode. Rate of aeration can be controlled with temperature, oxygen or carbon dioxide feedback signals.

- oxygen status.

Suppliers and approximate prices for scientific equipment recommended for each of these field tests is shown in Figure 1.

Definitions

Process Control

Stringent and documented monitoring of all critical control points in a composting operation so as to minimise defects and make products which can be guaranteed to customers.

Quality Management System (QMS)

Is a set of procedures an organisation establishes to guarantee its products will satisfy consumers.

Feedstock

Organic materials used for composting or related biological treatment systems. Different feedstocks have different nutrient concentrations, moisture, structure and contamination levels (physical, chemical and biological).

Turned Pile

System of composting involving the periodic turning of piles of organic matter with mechanical equipment (e.g. front-end loaders or specialised windrow turners)

Figure 1. Equipment suppliers and approximate prices

Temperature meters

- Bimetal windrow thermometer: analogue type, stem length 91 to 183 cm, stainless steel probe, crystal face, hermetically sealed. REOTEMP Instrument Corporation, USA. Cost \$223 to \$554.
- Digital hand held composting thermometer: water resistant digital meter, LCD display, 91 to 183 cm stainless steel compost probe. REOTEMP Instrument Corporation HI9063, USA. Cost from \$944, depending on probe length (see Plate 4).

pH kits

- pH test kit: pocket sized with enough tablets to perform 50 tests, and calibration chart, pH range 4-8. Palintest SL150, available from Crown Scientific, NSW. Cost \$115 (see Plate 11).

Maturity kits

- Compost maturity test kit: measures carbon dioxide and ammonia concentrations based on gel colorimetric analysis. Kit for 6 analyses, manufactured by Solvita, available through Biotec Pacific, Victoria. Cost \$198 (see Plate 15).

Oxygen meters

- Combined oxygen and temperature meter: portable, stainless steel probe, 9V battery, galvanic cell type oxygen detector, LCD display, resolution 1%, accuracy ± 1%. Manufactured by Demista, USA and available through Enviromulch Pty Ltd, VIC. Cost about \$2376 (see Plate 20).

Field testing safety tips

The field tests discussed here are not hazardous, but a few safety precautions need to be observed.

- **Gloves:** These should be worn at all times to prevent injury from sharp objects (e.g. glass and metal in contaminated feedstocks).
- **Safety Glasses:** Safety glasses or goggles need to be worn during all testing procedures.
- **Ventilation:** All testing areas, particularly enclosed rooms, need to be well ventilated.
- **Equipment Precautions:** Observe all safety precautions associated with equipment used for sampling and testing.
- **Hygiene:** If any materials are handled including raw feedstocks and compost during field testing procedures, hands should always be washed with soap and hot water afterwards.

Sampling

The first step in testing is to obtain a representative sample, or to sample from representative locations.

The sample (or sampling points) should reflect the overall characteristics of the material being tested.

Testing performed on a sample that is not representative of the bulk material will produce unreliable results.

Collect a number of samples from different, representative locations in a pile and/or from several piles to ensure that a representative sample is obtained. Mix these samples together well and then draw sub-samples to be tested from the mixture.

Avoid taking a disproportionate number of samples from the centre, edges, and outer surface, which are likely to have different qualities from the bulk of the material in the pile.

A recommended sampling procedure for testing has been reported in AS 4419 (2002) for Soils for Landscaping and Garden Use. This method was developed for sampling a batch of product ready for sale, though it can be easily adapted for use as in-process sampling method.

The method shown below is appropriate for field-testing procedures that require a sample of material for analysis.

1. From each batch of material (e.g. a windrow, pile or from a vessel) to be tested, collect 20 random samples at various depths, each having a volume of not less than 1 L (Plate 1).
2. Blend these samples to prepare a composite sample of not less than 20 L (Plate 2).

Plate 1. Random sampling of finished compost from 20 different points in the pile.



Plate 2. Blending of random samples to form a 20 L sample of homogenous, finished compost representative of the bulk material.



3. A sub-sample should be taken which is convenient to work with and suited to the testing equipment and containers. Sub-sampling can be done by the 'coning and quartering method'. Form the combined sample into a conical shape on a clean surface;
4. Quarter and mix the sample in successive steps until the sample volume is reduced to a suitable size (Plate 3).

Usually 6-10 L of sample is required for a full range of tests.

In the time that elapses between collecting and testing, it is possible for samples to lose moisture and undergo other changes. Therefore, samples should be collected shortly before testing.

If samples must be collected some time in advance, they should be refrigerated ($\sim 2^{\circ}\text{C}$) in a sealed plastic container or bag (NRAES, 1992).

If samples are to be analysed by an off-site laboratory, seal the sample in a plastic bag and dispatch the same day of sampling.

Note that if the sample is to be analysed for organic contaminants, a 1 L sample needs to be placed into a glass jar and sent to the laboratory as well. This is because the organic contaminants can react with plastic polymers in the bag.

A convenient way of posting the packaged samples is in a polystyrene box.

Plate 3. Sub-sampling of the combined sample by coning and quartering. The combined sample is formed into a cone, and divided into quarters.



Section 5 Sample Management for Consistent Analysis of Products and Raw Materials

5.1 Introduction

Laboratory analysis of compost or raw material samples can be used by facilities to indicate contamination by herbicides or other substances. However, organic materials are biologically active and many compounds degrade under unfavourable environmental conditions. Therefore, samples must be handled correctly to ensure the sample reaches the laboratory in a suitable condition and that the material/product that is analysed is representative of the original material/product sampled. The Recycled Organics Unit has developed an information package titled *Producing Quality Compost* (Recycled Organics Unit, 2002a) designed to assist producers in preparing a sample for laboratory analysis.

The *Producing Quality Compost* package contains a number of information sheets that inform the consistent production of quality compost and other recycled organics products. Information Sheet 3-11 in this package titled *Sample Management for Consistent Analysis of Quality* includes a method of storing, packaging and dispatching a sample for laboratory analysis.

5.2 Information Sheet 3–11: Sample Management for Consistent Analysis of Products and Raw Materials

The relevant sections of this information sheet are reproduced here to provide a method for preparing a sample for laboratory analysis. This sheet is directly related to the information sheet discussed in the previous section, [Section 4](#), detailing the method for taking a representative sample.

The entire package of the *Producing Quality Compost* information sheets can be freely downloaded from <http://www.recycledorganics.com>

Inside This Sheet

- 1** Need for standard sample management procedures
- 2** Taking a representative sample of “compost” product
 - Packaging and dispatch of the ‘compost’ product samples
 - Definitions
- 3** Receipt and processing of “compost” product samples at the laboratory
 - “Compost” maturity testing
- 4** Packaging and sample management for raw materials
 - Important references
 - Acknowledgement

Information Sheet No. 3–11

Sample Management for Consistent Analysis of Products and Raw Materials

Need for standard sample management procedures

Laboratory testing of recycled organics products is required to provide assurance that products meet minimum quality requirements, are fit for purpose, and/or are consistent with customer specifications.

The purpose of this information sheet is to define standard sample management procedures from the point of dispatch at the manufacturing facility right through to the point of testing at a laboratory.

Assessing compliance to an Australian or industry standard via off-site (independent) laboratory testing provides independent verification of the quality of the product(s) supplied to customers.

Independent quality testing of products also provides an important management tool for organics processing facilities by providing a check on the reliability and performance of the process control system for product manufacture.

However, non-representative sampling of product from windrows (for example), or poor sample handling and management by either the organics processing facility or the laboratory may produce unreliable testing results that do not accurately reflect the overall characteristics of the product available for sale.

It is important to note that recycled organics products (eg. composts) are ‘biologically active’. The chemical, physical and biological properties of the product can change after sampling, if samples are not managed correctly.

This means that standard procedures are required to ensure correct and consistent sampling and sample management. If these procedures are not followed consistently, laboratory test results will be unreliable.

For example, if a 20 litre sample of *composted soil conditioner* is sampled from a compost windrow, and is left to stand for 2 weeks in a sealed plastic bag in a site office or laboratory, the properties of the product are likely to change.

Plate 1. Finished batch of composted soil conditioner ready for off-site laboratory testing according to Australian Standard AS 4454 (2002).



Microorganisms in the biologically active compost product will continue to decompose some of the organic matter, resulting in the consumption of oxygen in the air contained in the bag. Eventually all of the oxygen in the sample bag will be consumed, and bacteria present in the sample that favour low oxygen (*anaerobic*) conditions will start to thrive.

Anaerobic microorganisms will continue to break down the organic product, and will tend to produce compounds such as volatile fatty acids (which can smell if high enough in concentration, particularly for relatively immature *pasteurised* products).

These compounds can lower the pH of the sample, resulting in a pH test result that is not representative of the pH of the compost in the windrow.

Clearly, if manufacturers of recycled organics products are to derive consistent results from off-site laboratory testing, samples sent for analysis need to be managed in a standard manner to minimise variation in the product caused by transport, storage and handling.

Testing laboratories also need to recognise that proper sample management and prompt testing of samples following receipt is required to provide consistent and relevant results.

By following these methods, changes in the properties of samples can be minimised, thus improving the consistency and reliability of results generated from laboratory testing.

It is recommended that compost manufacturers demand assurance from their commercial laboratory that they follow the procedures recommended here, or such other procedures as are relevant to the needs of the specific manufacturer's context, product or material.

Taking a representative sample of 'compost' product

The sample (or sampling points) should reflect the overall characteristics of the "compost" product being tested.

Laboratory analysis of a sample that is not representative of the bulk product will produce unreliable, non-representative results.

A method of taking a representative sample from a large batch of product for testing is provided in Information Sheet No. 3-3 in this series.

By following this sampling procedure, a sample which reflects the overall characteristics of the bulk product can be obtained.

Packaging and dispatch of the 'compost' product samples

To minimise changes that can occur between collecting the "compost" sample and laboratory analysis of samples, samples should be taken, packaged and sent to the laboratory on the same day by overnight courier.

A number of courier services can provide overnight delivery, and these can be found in the Yellow Pages™.

Depending on the range of tests to be performed, and the relevant Standard to be tested against, usually between 6 and 10 litres of representatively sampled product is required.

A procedure for packaging the sample is as follows:

1. Place the 6 – 10 litre sample of product into a plastic bag. Loosely tie the end of the bag.
2. Prick approximately 20 small holes in the bag with a small sharp point (eg. the point of a cheap school compass) to allow the contents of the bag to breathe during transport.

Definitions

Composted Soil Conditioner

Any composted product, including vermicast, manure and mushroom substrate, that is suitable for adding to soils. This term also includes 'soil amendment', 'soil additive', 'soil improver' and similar terms, but excludes polymers which do not biodegrade, such as plastics, rubber and coatings. Soil conditioner has not more than 20% by mass of particles with a maximum size above 16 mm.

Anaerobic

In the absence of oxygen, or not requiring oxygen. Composting systems subject to anaerobic conditions often produce odorous compounds and other metabolites that are partly responsible for the temporary phytotoxic properties of compost. Anaerobic conditions are important for anaerobic digestion systems.

Pasteurised

An organic product that has undergone controlled aerobic and thermophilic biological transformation to achieve pasteurisation, but is relatively immature and lacking in stability compared to compost.

3. If the sample is to be analysed for organic contaminants, a representative 1 litre sample needs to be placed into a sterile glass jar and sent to the laboratory as well. This is because the organic contaminants can react with plastic polymers in the bag.
4. Place sample/s into an appropriately sized eski or polystyrene box (e.g. similar to ice-packed broccoli box).
5. Insert relevant documentation into the polystyrene box and **seal** the lid to the box and any air holes with masking tape.
6. Place a label on the top of the box and address it to the relevant laboratory marked '**Urgent Sample for Analysis**'.
7. Arrange for the collection of the parcel by a courier for overnight delivery to the laboratory.

8. Store the sample box in a cool location out of direct sunlight whilst awaiting collection.

Polystyrene is a good insulator, and is recommended for transport as sealed insulated boxes can be effective in reducing temperature extremes during transport.

The temperature in the cargo bays of aircraft can often fall below – 20°C, which can have an impact on the biological properties of products containing compost.

Transport of samples in sealed polystyrene boxes can therefore assist in reducing the impact of air transport on samples.

A test that may be impacted by this form of transport is the self heating test (AS 4454, 2002).

This test is based on the principle that actively decomposing or immature products liberate heat, due to high levels of microbial activity.

Microbial activity responsible for generating this heat may be compromised for a period of time following such transport.

However, packaging of the sample into polystyrene can minimise such extreme temperatures, producing more reliable results.

Samples should be protected from light, as some compounds that may be present in the sample can be degraded by light.

If the sample cannot be sent the day of collection, it is recommended that the bagged sample be stored under refrigerated conditions (2-3°C) for no more than 3 days prior to packaging (as specified above) for dispatch.

For extended storage of product, product should be frozen (~0°C) to halt all microbiological activity.

Storage under these conditions will affect the microbial population in the product, and may affect the reliability of stability tests (eg. self heating test).

Receipt and processing of 'compost' product samples at the laboratory

Laboratories should minimise delays in processing of samples, in particular, for those tests that are likely to be affected by inconsistent and/or excessive storage time.

AS 4454 (2002) requires that *products shall be tested as soon as possible after receipt at a laboratory, and certainly within four days of receipt.*

It is recommended here that tests which can be influenced by continued biological activity in the sample should be performed within a two days of receipt at a laboratory.

Tests that should be performed within this two day period of receipt of the sample, based on (normative) tests specified in the Australian Standard AS 4454 (2002) include:

1. Appendix H: moisture content and level of physical contamination.
2. Appendix A: pH, electrical conductivity, ammonium, nitrate and soluble phosphorus content.
3. Appendix E: toxicity to plants.
4. Appendix K: self heating test.
5. Appendix M: Presence of plant propagules in pasteurised products.

If the tests specified above will not be performed within this two day period, it is recommended that the insulated container be opened, and that the bagged sample should be stored under refrigerated conditions (2-3°C) whilst awaiting analysis.

Samples should be protected from light, as some compounds that may be present in the sample can be degraded by light.

Other tests defined in the Australian Standard AS 4454 (2002) do not need to be undertaken as promptly, as the results are unlikely to be as affected by storage for durations exceeding two days. For these other tests, it is appropriate for the sample to be stored in the sample bag under conditions consistent with those specified in the relevant Australian Standard (AS 4454, 2002).

AS 4454 (2002) requires that *if [samples] must be stored after receipt, a sub-sample is to be taken for as-received moisture determination. If the sample is drier than a moisture content of 40-50%, water is to be added to bring it into this range and the sample stored in a large container that is loosely sealed to minimise water loss but to allow oxygen entry, [samples should be stored in cool conditions] at 18-25°C.*

These other tests should be performed within a two-week period.

Perforations inserted into the side of the bag should be effective in maintaining relatively aerobic conditions in the bag, whilst reducing moisture loss. If perforations have not been inserted into the bag, the laboratory technician should do so as defined in the previous section.

'Compost' maturity testing

There are two tests specified in the relevant Australian Standard (AS 4454, 2002) that relate directly to product stability and maturity. These tests are:

- Appendix E (normative): toxicity to plants.
- Appendix K (normative): self heating test.

For reliable results from the self heating test, the test should be commenced within one week of sampling. The sample should not be refrigerated, and should be stored, as specified above, in a plastic bag, in cool conditions below 25°C (Crouchley, 2001).

The bag allows dissipation of heat and retention of moisture, and should have air holes as previously specified to allow circulation of air to support oxygen entry and maintenance of aerobic conditions.

For reliable results from the toxicity test, where sample product has been refrigerated, the sample should be left to stand at room temperature until product reaches ambient temperature before initiating this test. This is because rate of seed germination can be influenced by the temperature of the growing media (Handreck and Black, 1994).

Packaging and sample management for raw materials

The sampling and analysis of compostable organic materials (raw materials) is often necessary when establishing the suitability of a raw material for processing, and for establishing a suitable compost recipe that incorporates the specific raw material.

The types of analysis that may be conducted relate to moisture content, carbon to nitrogen ratio, and potentially to the screening for contaminants such as heavy metals, organochlorine compounds and pesticide/herbicide residues.

The goal of testing of raw materials, particularly unstable materials, should be to analyse the material in the state in which it is likely to arrive at the processing facility.

It is recommended that for unstable raw materials such as lawn clippings and food waste, samples should be managed in the following manner:

1. Obtain a representative sample of sufficient size for the tests being conducted (ask laboratory for guidance on this).
2. Place the sample of raw material into a plastic bag, remove excess air and tie the end of the bag closed. Alternatively, "ziplock" resealable bags can be used.
3. Refrigerate the sample without freezing (2-3 °C) as soon as possible after sampling.
4. If the sample is to be analysed for organic contaminants, a representative 1 litre sample needs to be placed into a sterile glass jar and sent to the laboratory as well. This is because the organic contaminants can react with plastic polymers in the bag. This sample should also be refrigerated prior to shipping.
5. Arrange for the collection of the parcel by a courier for overnight delivery to the laboratory.

6. Place the cool sample/s into an appropriately sized eski or polystyrene box.
7. Place an ice-pack into the eski to maintain cool conditions during transport. This is particularly important where overnight/same day delivery is not possible.
8. Insert relevant documentation into the polystyrene box and **seal** the lid to the box and any air holes with masking tape.
9. Place a label on the top of the box and address it to the relevant laboratory marked '**Urgent: Sample for Analysis**'.
10. Store the sample box in a cool location out of direct sunlight whilst awaiting collection.
11. Upon arrival at the laboratory, the laboratory should remove the sample from the eski and keep the sample refrigerated and protected from direct light until ready to analyse.
12. Products shall be tested as soon as possible after receipt at the laboratory and certainly within four days of receipt.
13. Tests that can be influenced by continued biological activity in the sample should be performed within two days or receipt at the laboratory.

Important references

- Crouchley, T. (2001). Evaluation of Dewar Flask Self Heating Test. Submission to Australian Standards committee CS-037-01 Composts and Mulches from Richgro Garden Products, Western Australia.
- Handreck, K. and Black, N. (1994). Growing Media for Ornamental Plants and Turf. University of New South Wales Press, Sydney, Australia
- Standards Australia (2002). AS 4454—Composts, soil conditioners and mulches. Standards Australia, Homebush, NSW.

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Section 6 Herbicide detection using plant bioassay

6.1 Introduction

A bioassay involves growing sensitive plants in a sample of growing media, for example a compost mixture, in a controlled environment and observing the growth of the plant for indications of the presence or absence of stress. The time required for a bioassay generally depends on the type of plant used. Indicator plants are species sensitive to the compound that is being tested for, for example clover, tomatoes, beans and peas have been used in bioassays for clopyralid and picloram.

A number of bioassay techniques have been recently developed in response to the clopyralid contamination issues that have occurred in the United States and in New Zealand. The Washington State University composting facility uses a bioassay test to identify contaminated soil (Bezdicek *et al.*, 2001). A new bioassay is currently being developed in New Zealand to reduce the time taken to establish compost contamination by clopyralid. Tomato plants have been used traditionally for bioassay indications, however, these plants can take six to eight weeks before symptoms may appear, far too long for commercial composting facilities (Fietje, Personal Communication, 2002). Fauci *et al.* (2002) have also developed a plant bioassay to detect clopyralid and picloram herbicide contamination of compost. This bioassay for finished compost involves a bean or tomato grown in 25% test sample with 75% potting mix (v/v). Plant bioassays can be cost effective, however, they require several weeks to gauge plant response and if not conducted carefully, can produce false results.

Clopyralid and similar herbicides affect varying plant species to different degrees. For example, plants in the grass family are not affected by moderate to low levels of clopyralid compared to broadleaf plants such as beans, peas, potatoes and tomatoes, which tend to be severely affected (Rynk, 2002a). Clopyralid and picloram are reportedly active at low concentrations of less than 1 ppb and 0.3 ppb, respectively (Rynk, 2000; Bezdicek *et al.*, 2001; Bezdicek *et al.*, 2002) (see [Section 9](#)).

Laboratory analysis for clopyralid has required testing to be performed at the part per billion (ppb) level, as opposed to the more common part per million (ppm) range (Rynk, 2002a). Concentration levels for detection of clopyralid can seemingly vary depending on the extraction method. For this reason, bioassays can more accurately reflect the real situation. Bioassays are a less expensive method to determine clopyralid damage, however, damage may be a result of other substances, such as high soluble salts (Rynk, 2002a). Fauci *et al.* (2002) report that a pea bioassay can be more sensitive than an analytical test, detecting picloram or clopyralid to less than 1 ppb. An example of the damage to peas observed during a bioassay is shown in Figure 6.1.

Figure 6.1. Example of a pea bioassay showing response to symptoms of clopyralid damage of varying concentration. R=0 is an uncontaminated control and R=5 shows severe damage (Washington State University, 2003). Observable physiological effects as a result of herbicide damage to the plants range from slight cupping and twisting of leaves, curled cotyledons and first leaves, crooked or bent stems, reduced development of leaves and loss of apical dominance, to extreme cases of poor germination, no leaf development and cessation of plant growth.



The ROU has directly consulted the developers and practitioners of various bioassays for these persistent herbicides and has chosen to adapt and document a bioassay that:

- Assesses the growth of clover which is highly sensitive to these herbicides (observable effect at 1 to 2 ppb);
- Provides observable results within 14 days (shortest time frame);
- Provides readily observable plant response;

- Is relevant for all three persistent herbicides (clopyralid, picloram and triclopyr); and
- Is cheap to conduct.

It should be noted that although the observable plant physiological effects for the clover-based bioassay are well specified in the following bioassay method, there are no photos available for clover. However, observable effects from these herbicides are similar for most plants and the response of peas, shown in Figure 6.1, indicates typical herbicide damage caused by these specific herbicides.

6.2 Using this bioassay

This bioassay can be used to screen finished compost prior to selling. In the instance that herbicides are found/suspected to be present, the compost should:

- Be sampled for confirmatory laboratory analysis (refer [Section 7](#) and [Section 8](#));
- The results of this analysis be cross referenced against the “tolerance levels” in [Section 9](#) to identify potential applications for the contaminated batch that are less sensitive to the specific herbicides;
- This may require blending of the compost product to dilute contamination levels. Note that it is strongly recommended that the bioassay be conducted again on any product that results from blending for dilution of herbicide concentration.

6.3 Bioassay method for determination of herbicide toxicity to plants

6.3.1 Acknowledgement

This method has been adapted from a Standard Operating Procedure developed by Woods End Research Laboratory (2002) in the United States. The ROU would like to thank Dr William Brinton of Woods End Research Laboratory for providing this information and allowing this method to be adapted for use by the recycled organics industry in Australia.

6.3.2 Scope

This bioassay method aims to determine whether a recycled organic product contains auxin-like herbicides (specifically clopyralid and picloram), to a level that would be damaging if applied to sensitive plant species. These specific herbicides have been found to persist beyond the commercial composting cycle, and the application of contaminated composts has resulted in commercial damage to a range of sensitive plants/crops overseas. This method allows for the identification of the presence of these specific herbicides in composts, and allows a distinction to be made between herbicidal damage, on the one hand, versus other abnormalities that may be caused by product immaturity and/or high salt content.

This bioassay method is similar to, and can be performed with some of the apparatus required, for the *Method for Determination of Toxicity to Plants* (Appendix E) from Australian Standard AS 4454 (Standards Australia, 1999).

This test is only relevant for mature composted products according to Australian Standard AS 4454 (Standards Australia, 1999). Pasteurised products require analytical laboratory analysis for the

determination of any herbicide contamination (see [Section 7](#) in *Risk Management Tools for the Recycled Organics Industry*, Recycled Organics Unit, 2003).

6.3.3 Principle

A specific variety of seed is germinated under controlled conditions over a 14 day period and early growth (meristematic tissue) is assessed for typical and visible auxin-like herbicide damage. The seeds are germinated in two different growth mediums, one a peat based seed raising mix that has no potential for herbicide contamination (control), the other a compost based mix made from the recycled organic product (compost) being tested for potential herbicide contamination.

6.3.4 Definitions

Compost test product. Sample of recycled organics (e.g. compost) to be tested for herbicide contamination. Any herbicide concentration within this test product is referred to as $[H]_C$.

Compost growth media: Blend of test product and peat used to grow the seeds for determination of herbicide damage. Any herbicide contamination within this media referred to as $[H]_D$

Control growth media: Commercially available peat based seed raising mix.

6.3.5 Apparatus and materials

A list of suppliers and equipment options is provided at the end of this method in [Section 6.3.7](#). The following apparatus and materials are required:

- (a) A reference sample of the compost product that is to be tested. This material is called the *compost test product*. See Information Sheet 3-3 from the *Producing Quality Compost* (Recycled Organics Unit, 2002a) series of information sheets available from www.recycledorganics.com. A copy of this information sheet is also included in [Section 4](#) of *Risk Management Tools for the Recycled Organics Industry* (Recycled Organics Unit, 2003) for more details on how to take a representative sample.
- (b) Means of determining *electrical conductivity* (EC) (see relevant method in the Australian Standard AS 4454 for composts, soil conditioners and mulches, Standards Australia, 1999).
- (c) Sieve with apertures of 6.70 mm.
- (d) Peat (sphagnum moss) with pH of between 5.5 and 6.5.
- (e) 1 L calibrated beaker.
- (f) Peat based seed-starting medium (*control growth media*).
- (g) Two growing containers with individual cell diameter of approximately 50 mm. Six cells of one growing container will be used for the *compost test product* and six cells of the second growing container will be used for the *control growth media*. Additional products can be tested at the same time with only one control required.

- (h) Red clover seeds (*Trifolium pratense*, variety Astred). 84 seeds are required for testing one *compost test product* (42 seeds for control, 42 seeds for product). Testing for additional *compost test products* will require 42 seeds per product. Advice from overseas laboratories is that any red/white clover with rapid germination rate and that is more tolerant to salt levels should be reasonable suitable for this bioassay. We are currently awaiting NSW Agriculture advice.
- (i) Balanced nutrient solution.
- (j) Seed germinating cabinet or an enclosure capable of maintaining consistent air temperature of 25 ± 2 °C and providing a 16h day/8h night cycle. If an enclosure is used, a fluorescent light with timer for illuminating the enclosure from a height of about 25 cm shall be used to provide this day/night cycle.
- (k) Deionised or distilled water.

6.3.6 Procedure

The procedure shall be as follows:

Compost growth media preparation

- (a) Determine that the *compost test product* is sufficiently mature by testing in accordance with requirements of the Australian Standard AS 4454 (Standards Australia, 1999). Proceed only if the *compost test product* is sufficiently mature.
- (b) Determine the electrical conductivity (EC) of the *compost test product* according to Australian Standard AS 4454 (Standards Australia, 1999). Compost with high salt content may have a detrimental effect on the germination of the seeds. It is therefore necessary to ensure that poor germination rate has not simply resulted from high EC. Refer to Table 6.1 to assess the suitability of EC levels in the *compost test product* for this bioassay method and to determine whether the test product requires dilution to achieve a suitable salt content.
- (c) Clean growing containers, glassware and sieve. To clean, wash with water and brush all equipment to remove particles, then sterilise with disinfectant (e.g. methylated spirits) and triple rinse with deionised or distilled water. These auxin-type herbicides are water-soluble and this method ensures there is no potential for contamination or presence of plant pathogens from equipment.
- (d) Screen a 1 L sample of the *compost test product* to a particle size of less than 6.70 mm using sieve.
- (e) Screen approximately 500 mL of peat to a particle size of less than 6.70 mm using sieve.
- (f) Prepare 1 L of the *compost growth media* by blending the screened *compost test product* and peat according to the proportion P_M in Table 6.1 to create a growth media with an EC of no more than 2 dS/m. Place the measured volumes of screened *compost test product* and screened peat into a calibrated beaker and tap the base sharply three times to settle material. Mix thoroughly.

Table 6.1. Dilution procedure to standardise *compost test product* for suitable electrical conductivity prior to performing bioassay (from Woods End Research Laboratory, 2002).

Test sample EC (dS/m)	P_M Proportion of test product in growth media (%)	Volume of components	
		Test product (mL)	Peat (mL)
≤ 2.0	75	675	225
3.0	67	600	300
4.0	50	450	450
5.0	40	360	540
6.0	33	300	600
7.0	29	257	643
8.0	25	225	675
10.0	20	180	720
15.0	13	120	780

Establishment of bioassay cultivars

- (g) Fill six cells of a clean and dry growing container with the prepared *compost growth media* (*compost test sample* mixed with peat). Fill the containers evenly by tapping gently three times to settle the material. Label these with appropriate sample/batch details and with the bioassay start date.
- (h) Screen approximately 1 L of *control growth media* (peat based seed raising mix) to a particle size of less than 6.70 mm using sieve.
- (i) Fill six cells of a growing container with the screened *control growth media* (peat based seed raising mix) and tap gently three times to settle the material. Label these as the control and with the bioassay start date.
- (j) Plant each of the cells with seven seeds of red clover (*Trifolium pratense*, variety Astred). Arrange seeds in a circular pattern near the perimeter of each cell. Plant seeds by pushing 5 mm below the surface of the growth media.
- (k) For initial watering only, water each cell to field capacity with a balanced nutrient solution.
- (l) Maintain environmental conditions at 25 ± 2 °C and illuminate on a 16h day/8h night cycle with appropriate fluorescent lighting for plant growth.
- (m) Water the seeds and growth media daily to field capacity with deionised or distilled water to maintain in a moist but not saturated condition.

NOTE: Do not over water as leaching/drainage from base of cells is undesirable due to the high water solubility of these herbicides. This bioassay aims to identify the presence/absence of herbicide in the compost and it is self-defeating therefore to risk leaching of herbicides from the compost test mix.

Data collection

- (n) After 14 days, count and record the number of fully germinated plants in each cell.
- (o) Evaluate the leaf-shape noting any curling or puckering of the leaf margins for the germinated plants grown in the *compost test product*. Note any differences between the plants grown in the *control growth media* versus those grown in the *compost test product*.
- (p) Indicate the possible presence of auxin-like herbicide by comparing observations of the plants grown in the *compost test product* with the descriptions in Table 6.2. Determine the mean range of the estimated auxin-like herbicide levels in the six cells of the growing container $[H]_M$.

Table 6.2. Observed plant effects due to presence of auxin-type herbicide contamination of growth media (adapted from Woods End Research Laboratory, 2002).

Observed effect on red clover	Estimated concentration of auxin-like herbicide present in growth media (ppb)
None – normal plant development, cotyledons and first leaf flat, stems erect.	< 2
Slight – slightly down-curved cotyledons and first leaf, stems erect, continued growth.	2 – 10
Medium – most cotyledons curled down in half circle, most first leaflets not opening, stems erect, many or most stems crooked or bent, growth ceases.	10 – 25
Severe – cotyledons curled into half or full circle, few leaves, stunted growth ceases.	25 – 100
Extreme – little stem elongation or cotyledon enlargement, no leaves, few stems, growth ceases.	100 – 500
Extreme – poor germination, little elongation or cotyledon enlargement, no leaves, few stems, growth ceases.	> 500

Calculation

- (q) Determine the approximate auxin-like herbicide level in the original (undiluted) *compost test product* $[H]_C$, by dividing the estimated concentration $[H]_M$ by the percentage of compost in the *compost growth media* as determined from Table 6.1, according to Equation 6.1:

$$[H]_C = \frac{[H]_M}{P_M}$$

Equation 6.1

Where:

$[H]_C$ = auxin-like herbicide concentration in original *compost test product*

$[H]_M$ = mean of auxin-like herbicide concentration in diluted *compost growth media*

P_M = proportion of *compost test product* in *compost growth media* (%) from Table 6.1.

For example:

If the estimated concentration of persistent herbicide present in the *compost growth media* $[H]_M$, according to Table 6.2, is in the range 2–10 ppb, and the compost was diluted to 25% (1:3 v/v) determined from Table 6.1, then using Equation 6.1:

$$[H]_{Lower} = \frac{[H]_M}{P_M} = \frac{2}{0.25} = 8 \qquad [H]_{Upper} = \frac{[H]_M}{P_M} = \frac{10}{0.25} = 40$$

Therefore, the herbicide concentration of the original product $[H]_C$ is in the range of 8–40 ppb.

Confirmatory laboratory analysis as required

- (r) Determine the requirement for confirmatory laboratory analysis by evaluating the potential auxin-like herbicide concentration of original (undiluted) test product $[H]_C$ with the recommended requirement for laboratory analysis in Table 6.3.
- (s) If the bioassay indicates the possible presence of auxin-like herbicides and confirmatory laboratory analysis is required, refer to [Section 7](#) for laboratory contact details and [Section 8](#) for details on the laboratory analysis required in the document *Risk Management Tools for the Recycled Organics Industry* (Recycled Organics Unit, 2003).
- (t) Use analytical results from the laboratory to determine which markets are safe for the sale of the contaminated compost by reference to [Section 9](#) of *Risk Management Tools for the Recycled Organics Industry* (Recycled Organics Unit, 2003).

Table 6.3. Estimated concentration of auxin-like herbicide in original (undiluted) test product $[H]_C$ and requirements for confirmatory laboratory analysis.

Estimated concentration of auxin-like herbicide present in original (undiluted) test product $[H]_C$ (ppb)	Confirmatory laboratory analysis required
< 2	No Unless reason to suspect contamination due to source of raw materials, see Section 3 .
2 – 10	Yes
10 – 25	Yes
25 – 100	Yes
100 – 500	Yes
> 500	Yes

6.3.7 Equipment suppliers

A list of equipment suppliers is given in Table 6.4 to assist producers of recycled organics in purchasing apparatus and materials required for successful investigation of potential auxin-like herbicide contamination of recycled organics products using a bioassay. The equipment required and listed below can be acquired from a number of sources and is not limited to those listed. These suppliers are suggested only and this information should only be used as a guide. Refer your chosen supplier to the specifications in [Section 6.3.5 Apparatus and Materials](#) above.

Table 6.4: Supplier information for equipment required for bioassay. This information is a guide only.

Item	Description	Price	Suggested supplier contact details
De-ionised or distilled water	Available from most hardware stores (4 L)	\$5.90	Hardware stores e.g. Bunnings Warehouse Web: www.bunnings.com
Enclosed seed raising environment	Controlled greenhouse environment for seed raising with fluorescent lights to maintain set day/night period. Example product: Clone Dome	Clone Dome: \$175 or	Hydroponic suppliers e.g. Ezy-Grow Hydroponics Shop 3/340 Windsor St, Richmond NSW Phone: (02) 4588 5826 17 Mount Druitt Road, Mt Druitt NSW Phone: (02) 9832 1610
	Simple mini greenhouse for seed raising (requires heat mat and light)	Mini greenhouse: \$25	
	Heat-mat to maintain a constant temperature within a seed raising environment such as the Clone Dome.	Heat mat: \$75	
	Light with 2-foot twin fluorescent bulbs for illuminating seed raising container. Requires timer (some containers have in built lights so may not be required).	Light: \$75	
Growth cells	Tray of 40 individual 50 mm wide cells to fit into enclosed seed-raising environment.	\$3.50	Hydroponic suppliers e.g. Ezy-Grow Hydroponics Shop 3/340 Windsor St, Richmond NSW Phone: (02) 4588 5826 17 Mount Druitt Road, Mt Druitt NSW Phone: (02) 9832 1610
Peat	Bagged peat (5 L)	\$5.90	Landscaping suppliers e.g. St George Landscape Gardening Supplies Ph: (02) 9529 6512 Email: sales@stgeorgelandscape.com.au Web: www.stgeorgelandscape.com.au
Peat-based seed raising mix	Bagged seed raising mix (5 L)	\$5.90	Landscaping suppliers e.g. St George Landscape Gardening Supplies Ph: (02) 9529 6512 Email: sales@stgeorgelandscape.com.au Web: www.stgeorgelandscape.com.au
Red clover seeds (<i>Trifolium pratense</i> , variety Astred)	84 seeds required per compost test product. If Astred variety unavailable, Colenso would also be suitable.	Variable	Seed suppliers e.g. Auswest Seeds Ph: (02) 6852 1500

Continued over page

Item	Description	Price	Suggested supplier contact details
Self-contained propagation system	Single tray heated propagation tray with thermostat. Clear dome enclosure. Light kit. Product codes for suggested items: TPS030 (single propagation tray) TPSTOP (dome enclosure) TPSKIT (light kit)	Tray: \$140.25 Enclosure: \$16.50 Light kit: \$41.25	Hydroponic suppliers e.g. Thermofilm Australia Pty Ltd. 27 Rosalie St, Springvale VIC 3171 Phone: (03) 9562 3455 Fax: (03) 9548 3979 Web: www.thermofilm.com.au
Sieve 6.70 mm	Used to sieve products to ensure particle size of maximum size. Product codes for suggested items: 200 SBW 6.70 (stainless steel, 200 mm diameter) 200 BBW 6.70 (brass, 200 mm diameter)	Stainless steel: \$268.00 Brass: \$229.90	Scientific suppliers e.g. Crown Scientific Phone: 1300 727 696 Email: crownsci@crownsoci.com.au Web: www.crownscientific.com.au
Thermometer	Basic thermometer to measure temperature within the seed-raising container to ensure constant conditions. Temperature range of – 10 to 50 °C. Products containing low toxicity components are recommended. Product code for suggested item: 44/800/8	\$10.00	Scientific suppliers e.g. Crown Scientific Phone: 1300 727 696 Email: crownsci@crownsoci.com.au Web: www.crownscientific.com.au

Note: Product codes only apply to suggested supplier.

Section 7 Commercial laboratories

7.1 Introduction

The issue of persistent herbicide contamination of compost has not yet occurred in Australia. As a consequence, commercial laboratories have not been required to perform testing to the tolerance limits of as low as 1 to 2 ppb as required for sensitive crops such as tomatoes (see [Section 9](#)). To provide relevant analytical services, laboratories must be firstly equipped to perform these analyses and to perform them to these low detection limits.

Commercial laboratories were approached as to whether clopyralid, picloram and triclopyr could be detected in composted materials to the required detection limits. These herbicides can be detected in an acid herbicides test, however, the low detection limits required further development of the method and therefore limited the number of laboratories that are potentially capable of performing the analysis.

The following laboratories responded positively to the requirements for testing and details of these laboratories are given below.

7.2 Virotec Global Solutions

7.2.1 General information

Virotec Global Solutions specialise in commercialising new environmental technologies that tackle the problems of environmental degradation and the accumulation of human and industrial wastes. Virotec Global Solutions provide products and services that are practical solutions to touch environmental problems.

Virotec Global Solutions provide new technologies for:

- Industrial wastewaters
- Mining
- Sewage treatment
- Soils

Virotec Global Solutions provide advanced laboratory analysis of organic contaminants. Australian Government Analytical Laboratories (AGAL) subcontract analyses requiring high performance liquid chromatography (HP/LC) and liquid chromatography mass spectrometry (LC/MS) to Virotec Global Solutions.

7.2.2 Accreditation

The National Association of Testing Authorities (NATA) is Australia's Government-endorsed provider of accreditation for laboratories and similar testing facilities. By obtaining NATA accreditation, a laboratory can obtain an internationally recognised means of evaluating the competence of the organisation to perform specific tests, calibrations, measurements and inspections.

Virotec Global Solutions is a NATA accredited laboratory but does not hold accreditation for the acid herbicides analysis. This is due to this issue and test being new to Australia, and due to the limited number of these analyses performed. Virotec Global Solutions have a strict quality control and quality assurance program followed for every analysis. A minimum of three to four standards is run on each sample as well as a quality control standard (Marie Hendriks, Personal Communication, 2002).

7.2.3 Persistent herbicides testing information

Virotec Global Solutions can confidently perform an acid herbicides test for clopyralid, picloram and triclopyr to a reliable detection limit of 5 ppb. This method is currently being refined with a goal of achieving detection limits as low as 1 to 2 ppb, however at time of writing this cannot be guaranteed. The ROU is currently working with Virotec as the laboratory service provider for a subsequent composting trial, and the samples tested for this trial will enable Virotec to refine their method to achieve the lowest possible detection limit. Turn-around time is generally around three to four weeks depending on the batch size.

7.2.4 Contact details

Virotec Global Solutions
34 Norfolk Court
Coburg, Victoria, 3058
Australia

Telephone: (03) 9350 4800

Facsimile: (03) 9350 4871

Email: lab@virotec.com

Website: www.virotec.com

7.3 State Chemistry Laboratory

7.3.1 General information

The State Chemistry Laboratory (SCL) supports the scientific activities of Agriculture Victoria, Government Departments, industry and the public through the provision of specialist analytical, research, investigation and consultancy services in chemistry and related services.

SCL offer the following services:

- Soil, plant and environmental chemistry
- Pesticide and organic residue analysis
- Food and public health services
- Human and animal nutrition
- OH&S audits and consultancies

- Land use assessment
- Waste utilisation
- Export certification
- Certification of composition
- Soil physics

SCL has state of the art equipment for a wide range of analyses including organic residues, melts, soil and plant nutrients, and toxins. Major institute programs employed by SCL include general analytical chemistry services; soil acidity and acidification; assessment of herbicide persistence in soils; and value adding to industrial, domestic and agricultural wastes.

Clients of SCL include Agriculture Victoria, Environment Protection Authority, Department of Conservation and Natural Resources, Australia Pesticides and Veterinary Medicines Authority, Universities, Hospitals, local government authorities, major industrial and commercial firms, farming community and the general public.

7.3.2 Accreditation

SCL is a NATA accredited laboratory for testing herbicides in biota, which includes composts, however, the acid herbicide analysis is a new method and therefore does not hold NATA accreditation.

7.3.3 Persistent herbicides testing information

SCL can perform an acid herbicides test on composted material that will detect clopyralid, picloram and triclopyr as well as 2,4-D, MCPA and dicamba in a single screen. This is a standard test with a detection limit of down to 10 ppb using a gas chromatography (GC), mass spectrometry (MS). Upon consultation with SCL, they anticipate that this detection limit can be lowered to 5 ppb by performing GC/MS/MS (Colin Cook, Personal Communication, 2002).

7.3.4 Contact details

State Chemistry Laboratory
Corner Sneydes and South Road
Werribee, Victoria, 3030
Australia

Telephone: (03) 9742 8755

Facsimile: (03) 9742 8700

Email: scl.enquiries@nre.vic.gov.au

Section 8 Acid herbicides analytical test method

8.1 Acid herbicides test

The ROU has contacted a number of laboratories in Australia to instigate the development of methods for analysis of compost and similar organic materials for acid herbicides that may be persistent and potentially problematic if contained in recycled organics products. The laboratories identified as being capable of conducting this analysis are detailed in [Section 7](#).

These laboratories can perform an *acid herbicides test* on organic materials to analyse for the presence of the herbicides clopyralid, picloram and triclopyr (and others). This analysis is relatively new and the test itself is not NATA certified as yet. However, as detailed in [Section 7](#), the laboratories themselves hold NATA certification and follow strict quality assurance procedures to ensure sample quality and results are not compromised.

The specific acid herbicide analytical test method is the intellectual property of the laboratory due to method development required to reach the low tolerance levels for these persistent herbicides. Virotec Laboratory has provided an overview of the method they have developed in consultation with the ROU for this acid herbicide test (Dr Marie Hendriks, Personal Communication, 2003).

The method is as follows:

- Laboratory receives representative sample/s of compost and/or raw materials. Section 4 of this report reproduces ROU Information Sheet No. 3-3 *On-site Field Testing and Monitoring for Quality* which documents the procedure for taking a representative sample from a compost windrow. Section 5 of this report reproduces ROU Information Sheet No. 3-11 *Sample Management for Consistent Analysis of Products and Raw Materials*, which includes packaging and shipping instructions for compost facilities.
- The laboratory manages the sample/s in accordance with the procedures documented in Section 5 of this report: ROU Information Sheet No. 3-11 *Sample Management for Consistent Analysis of Products and Raw Materials*.
- A minimum sample size of 100g is required for compost products, and a minimum sample size of 50g is required for other raw materials such as plant tissues and lawn clippings.
- The sample is blended (size reduced) and mixed to form a homogeneous material to ensure results are representative of the sample provided.
- A random sub-sample of approximately 10 g is taken from the homogenised sample for analysis.
- Herbicides are extracted from the sub-sample using an appropriate solvent.
- The extract is passed through a solid phase extraction cartridge to concentrate and “clean up” the extract. This process removes contaminants such as amino acids that are commonly found in plant materials that can interfere with accurate detection of the relevant herbicide compounds.
- The extract is injected into a LC/MS (liquid chromatography/mass spectrometry) instrument for analysis.

- Standard herbicide solutions of known concentrations are run through the instrument that correspond to instrument reading thereby allowing the unknown concentration of herbicide in the sample to be determined.
- Any indication of the general range of herbicide contamination, as determined by performing a bioassay (see [Section 6](#)), should be provided to the laboratory to assist them in selecting the appropriate sample preparation method and thereby allowing a more accurate determination of herbicide concentration.
- Currently, Virotec Laboratory can analyse samples reliably and confidently to report results to a minimum detection level of 5 ppb¹.

¹ Virotec Laboratory aim to achieve the 1 to 2 ppb threshold level of detection claimed by a commercial laboratory in New Zealand (Recycled Organics Unit, 2002c). However a greater number of samples of lower concentrations than those initially provided for method development are required to refine the method in order to achieve a lower threshold level of detection. Virotec Laboratory will be providing the analytical testing services for the applied *Assessment of Herbicide Breakdown During Composting* trial, and will continue method development during this period until they reach the lowest achievable threshold level of detection that can provide reliable results.

Section 9 Tolerance levels

9.1 Introduction

The susceptibility of plants to specific herbicides varies due to the type of plant and type of herbicide. Herbicides can be selective, meaning that they are designed to target specific types of plants, for example broadleaf plants; or herbicides can be non-selective, meaning they kill any plant on contact. The concentration of the herbicide can also influence the toxicity of the substance on a plant with some herbicides requiring only very low concentrations to result in plant damage or death.

9.2 Sensitive crops and applications

The herbicides clopyralid, picloram and triclopyr are non-selective herbicides and are therefore toxic to a wide range of plants. For example, plant families sensitive to clopyralid (Dow AgroSciences, no date-b) include:

- Legumes: peas, beans, lentils, clover
- Solanaceous: potatoes, tomatoes
- Asteraceae: sunflower, thistle, dandelion

A review of the tolerance levels of sensitive crops and applications to these herbicides is given in Table 9.1. This data has been collected from a number of sources including the US Environmental Protection Agency (US EPA, 2002), herbicide information sheets, chemical manufacturers and literature reviews. The table is not a comprehensive summary of all plants sensitive to these chemicals and should be used as a guide only.

If herbicide contamination has been detected in a recycled organic product through bioassay and/or laboratory analysis, this information can be used to determine which crops are sensitive to particular concentrations of these specific herbicides, and which are therefore to be avoided.

Table 9.1: Tolerance levels for crops/applications known to be susceptible to the potentially problematic chemicals clopyralid, picloram and triclopyr.

Crop/Application	Clopyralid		Picloram		Triclopyr	
	Tolerance	Source	Tolerance	Source	Tolerance	Source
Asters	Sensitive	Dow AgroSciences (no date-b)	--	--	--	--
Asparagus	1.0 ppm	US EPA (2002)	--	--	--	--
Barley	1.0 – 9.0 ppm	Bezdicsek (2002); US EPA (2002)	0.5 – 3.0 ppm	US EPA (2002)	--	--
Bean	<1 ppb	Bezdicsek (2002)	<1 ppb	Rynk (2000)	--	--
Bean, Faba	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Bean, Navy	--	--	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Bean, Pinto	1 ppb	Bezdicsek (2002)	--	--	--	--
Beetroot	4.0 ppm	US EPA (2002)	--	--	--	--
Brassica	2.0 ppm	US EPA (2002)	--	--	--	--
Canola	1.0 – 6.0 ppm	Bezdicsek (2002); US EPA (2002)	--	--	--	--
Carrot	Sensitive	Dow AgroSciences (no date-b)	--	--	--	--
Chickpea	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Clover	1 ppb	Bezdicsek (2002)	Sensitive	(Woods End Research Laboratory, 2002)	Sensitive	Tomlin (1997)
Common buckwheat	Sensitive	Dow AgroSciences (no date-b)	--	--	--	--
Corn	1.0 – 10.0 ppm	US EPA (2002)	--	--	0.01 – 0.3 ppm	US EPA (1998)
Cotton	10 ppb	Bezdicsek (2002)	0.05 ppm	Cox (1998b)	Sensitive	Cox (2000)
Cranberry	2.0 – 4.0 ppm	US EPA (2002)	--	--	--	--
Cucumber	Sensitive	(Woods End Research Laboratory, 2002)	Sensitive	(Woods End Research Laboratory, 2002)	Sensitive	(Woods End Research Laboratory, 2002)
Flax	0.5 – 6.0 ppm	US EPA (2002)	--	--	--	--
Fruit tree	Sensitive	(Dow AgroSciences, 1998a)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Grass, forage	500 ppm	US EPA (2002)	80 ppm	US EPA (2002)	500 ppm	US EPA (2002)
Hops	--	--	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Legumes	--	--	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)

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Crop/Application	Clopyralid		Picloram		Triclopyr	
	Tolerance	Source	Tolerance	Source	Tolerance	Source
Lentil	<1 ppb	Bezdicek (2002)	--	--	--	--
Lettuce	<1 ppb	Bezdicek (2002)	Sensitive	(Woods End Research Laboratory, 2002)	Sensitive	(Woods End Research Laboratory, 2002)
Linseed	0.1 ppm	Bezdicek (2002)	--	--	--	--
Lucerne	Sensitive	(Dow AgroSciences, 1998a)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Lupins	Sensitive	Dow AgroSciences (no date-b)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Medics	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Mint, hay	3.0 ppm	US EPA (2002)	--	--	--	--
Mustard	3.0 – 5.0 ppm	US EPA (2002)	--	--	--	--
Mycorrhizal fungi	--	--	--	--	0.1 ppm	Cox (2000)
Oats	3.0 – 12.0 ppm	US EPA (2002)	0.5 – 3.0 ppm	US EPA (2002)	--	--
Ornamentals	Sensitive	(Dow AgroSciences, 1998a)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	Tomlin (1997)
Pea	<1 ppb	Bezdicek (2002)	<1 ppb	Rynk (2000)	Sensitive	(Dow AgroSciences, 1999)
Pea, Field	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Capsicum (bell peppers)	<10 ppb	(Rynk, 2002b)	--	--	--	--
Potatoes	10 ppb	Bezdicek (2002)	Sensitive	Cox (1998b)	Sensitive	(Dow AgroSciences, 1999)
Rapeseed	3.0 ppm	US EPA (2002)	--	--	--	--
Rice	--	--	--	--	0.3 ppm	US EPA (2002)
Rice, straw	--	--	--	--	10 ppm	US EPA (2002)
Safflower	Sensitive	Dow AgroSciences (no date-b)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Sorghum	--	--	0.2 – 0.5 ppm	US EPA (2002)	--	--
Soybeans	--	--	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Spinach	5.0 ppm	US EPA (2002)	--	--	--	--
Stone fruit	0.5 ppm	US EPA (2002)	--	--	--	--
Strawberry	1.0 ppm	US EPA (2002)	--	--	--	--
Sub-clover	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Sugar beet	10 ppb	Bezdicek (2002); US EPA (2002)	Sensitive	(Dow AgroSciences, 1997)	0.2 ppb (germination)	US EPA (1998)
Sunflower	<1 ppb	Bezdicek (2002)	Sensitive	(Dow AgroSciences, 1997)	4 ppb	US EPA (1998)

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Crop/Application	Clopyralid		Picloram		Triclopyr	
	Tolerance	Source	Tolerance	Source	Tolerance	Source
Tobacco	10 ppb	Bezdicek (2002)	<1 ppb	Cox (1998b)	Sensitive	(Dow AgroSciences, 1999)
Tomato	1 ppb	Bezdicek (2002)	<1 ppb	Rynk (2000)	Sensitive	(Dow AgroSciences, 1999)
Turnip	1.0 – 4.0 ppm	US EPA (2002)	--	--	--	--
Vegetables	Sensitive	(Dow AgroSciences, 1998a)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Vines	Sensitive	(Dow AgroSciences, 1998a)	Sensitive	(Dow AgroSciences, 1997)	Sensitive	(Dow AgroSciences, 1999)
Wattle	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--
Wheat	1.0 – 12.0 ppm	Bezdicek (2002); US EPA (2002)	1.0 – 3.0 ppm	US EPA (2002)	--	--
White clover	Sensitive	(Dow AgroSciences, 1998a)	--	--	--	--

NOTE: This table is not a complete list of all crops/applications that are sensitive to clopyralid, picloram and triclopyr. This list should not be used as a replacement for product labels. Tolerance levels down to ppb are highlighted. *Sensitive* indicates that some sensitivity/phytotoxicity has been reported but no actual tolerance limits published. Crops not listed but in the same family are likely to have similar responses.

9.3 Tolerant applications

Dow AgroSciences reports the “plants are most tolerant to herbicide applications, including clopyralid, when they are established and have formed some woody tissue around the stem/trunk” (Dow AgroSciences, no date-a). There are also a variety of applications for which these herbicides are designed, and therefore, there may be a range of applications that are suitable for use of products contaminated with these specific persistent herbicides. These applications are likely to include forestry, orchard, established perennial landscapes, and pasture/lawn top-dressing, however, actual levels of tolerance for such applications are not accurately known.

Use of clopyralid-contaminated compost on crops and other applications is dependent on the application rate and concentration of the clopyralid present in the compost. Consequently, results can vary for similar crops as the type of product, concentration of herbicide and application rate vary. The safest use of clopyralid-contaminated compost is lawn and turf as this application is the major application internationally (although not in Australia). However, if lawn clippings are removed from areas of herbicide application and added to the garden organics stream for processing into compost, this application of contaminated compost merely perpetuates the original contamination problem. Consequently, use on lawns and turf would only be advised if clippings are not removed from the application area or are only used on-site.

Rynk (2002c) states that compost contaminated with clopyralid can be incorporated into soil or blended with clean compost to lower the clopyralid concentrations. However, compost with 50 ppb or more of clopyralid would have to be spread evenly in a thin layer (<20 mm) assuming the compost

was mixed with the soil to a depth of 150 mm. These low application rates are easier and more typical for farms than gardens.

Research into the clopyralid issue internationally has resulted in limited information on the use of the contaminated compost product. Industry professionals and researchers have been contacted in regard to this issue to identify suitable uses and application rates of this contaminated material. Table 9.2 summarises this information primarily derived from personal communication with industry and researchers.

Table 9.2. Suggested applications for compost contaminated with the herbicide clopyralid. Note: whilst these potential applications and tolerances represent the best information currently available, it is sourced from literature and direct consultation with relevant researchers and effected organics processing facilities. The ROU has not confirmed any of these potential applications and tolerance levels, and recommends that compost producers assess the suggestions documented here via small-scale applications in the first instance.

Application	Comments	Source
Large-scale agriculture	Applications rates up to 20 t/ha with clopyralid concentration of less than 70 ppb.	George Fietje, Technical Manager of Living Earth Ltd New Zealand, Pers. Comm., 2003.
Lawns and turf	The safest use of clopyralid-contaminated compost is lawns and turf if the clippings are not returned to commercial facilities for composting. Lawn and turf species tolerant to clopyralid-contaminated compost include, but not limited to: bentgrass, buffalograss, fescues, Kentucky bluegrass, ryegrass.	George Fietje, Technical Manager of Living Earth Ltd New Zealand, Pers. Comm. (2003); Robert Rynk, BioCycle, Pers. Comm. (2003); Gill Pontin, Living Early Ltd. New Zealand, Pers. Comm. (2003); Dow AgroSciences (no date-a).
Ornamentals	Including, but not limited to, species of: azalea, apple, cranberry, fir, juniper, maple, oak, pine, rhododendron, rose, spruce, walnut.	Dow AgroSciences (no date-a); Jeff Gage, Compost Design Services, US, Pers. Comm. (2003).
Other applications	Fallow cropland, forestry, industrial and storage sites, non-cropland, permanent pastures, rangeland, rights-of-way.	Dow AgroSciences (no date-a).
Other plants	Including, but not limited to: asparagus, barley, Christmas trees, field corn, eucalyptus trees, grass grown for seed or sod, mint, oats, oilseed rape, poplars, sugar beets, wheat.	Dow AgroSciences (no date-a).
Sensitive vegetable and ornamentals (e.g. lettuce, zinnia and marigold)	Lettuce, zinnia and marigold plant growth and vigour improved with increasing rate of compost due to the nutrients supplied. Compost with 25 ppb of clopyralid was applied in proportions of 10, 20, 30 and 50% compost.	Bary (2002).
Sensitive vegetables (e.g. tomato, bean and pea)	12 ppb clopyralid in compost applied at 25 mm produced tomato and pea plants that were equal or superior to unamended controls. 25 ppb clopyralid in compost applied at 0, 14, 25, 50 mm to tomato, bean and pea with no symptoms or plant damage.	Cogger (2002). Mary Fauci, Washington State University, US, Pers. Comm. (2003).
Woody landscape plants	Woody landscape plants seem to be tolerant due to maturity.	Robert Rynk, BioCycle, US, Pers. Comm., 2003.

A study by Brinton and Evans (2002) of Woods End Research Laboratory in the US, examined the dilution effects of compost contaminated with clopyralid after application at varying depths. The results of this study are shown in Table 9.3. These results indicate potential application rates of clopyralid-contaminated compost that can be safely applied. Whilst only preliminary results, this data could be useful in advising application rates of clopyralid-contaminated compost to ensure detrimental effects to crops and applications do not occur.

Table 9.3. Dilution effects of application of compost contaminated with clopyralid (from Brinton and Evans, 2002)

Compost application rate		Clopyralid in compost (ppb)		
t / ha	depth (mm)	10	50	200
		Resulting concentration in soil (ppb)		
475	80	1	5	20
120	2	0.25	1.25	5
24	shallow	0.05	0.25	1

9.4 Verification

Note that Section 9 of this report has been submitted to Dow AgroSciences, and is currently being reviewed. The Recycled Organics Unit hopes to receive a response from Dow AgroSciences by the end of April 2003.

Section 10 Recommendations

An herbicide bioassay has been developed as a risk management tool for the organics processing industry in NSW in order to avoid the problems that have arisen overseas in relation to three specific herbicides known to be persistent beyond the commercial composting cycle. The herbicide bioassay has been adapted from direct consultation with international sources to provide a bioassay for the recycled organics industry that:

- Assesses the growth of clover, which is highly sensitive to the three specific herbicides of known persistence (clopyralid, picloram and triclopyr);
- Provides observable results within 14 days (shortest period of time compared to other species);
- Provides readily observable plant response at lowest levels of contamination (compared to other species);
- Is relevant for all three persistent herbicides (clopyralid, picloram and triclopyr); and
- Is cheap to conduct.

However, the clover variety upon which the specified bioassay is based is not available in Australia. The bioassay therefore requires validation to ensure that the documented method using a different clover variety is effective, and that the observable effects in terms of plant response at various concentrations of contamination accurately correspond to the specified descriptions. In addition, validation of the bioassay method is required to provide photos of plant response to varying concentrations of herbicide, as none are currently available (for clover). Such pictures would provide a resource for industry (and laboratories) to accurately identify herbicide damage using the specified bioassay.

The ROU has evaluated the photos available for other herbicide-affected plants, including tomatoes, peas and beans. The available pictures are neither large enough to clearly identify the physiological impacts, nor well labelled, and are therefore not considered suitable as pictorial guides for supporting application of a bioassay. It is also recommended that internet pictures, such as those currently available, are of inadequate resolution for use, and that internet is not the appropriate medium for laboratory/field use.

It is recommended therefore that the documented bioassay be validated for observable plant physiological response at different levels of concentration of these specific herbicides, and that these effects be documented in a clearly labelled pictorial poster for industry use in identification of plant physiological effects. This poster should be made available to the commercial composting industry and to commercial laboratories for use with the documented bioassay method.

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Appendix 1 Glossary

All terms defined in this glossary are given in the Recycled Organics Industry Dictionary and Thesaurus, 2nd Edition (Recycled Organics Unit, 2002b) unless otherwise noted.

Term	Definition
Acid herbicides	Grouping of herbicides including clopyralid, picloram and triclopyr used to define a laboratory analytical method: acid herbicides analysis (Marie Hendriks, Personal Communication, 2003).
Agricultural chemical product	A substance or mixture of substances that is represented, imported, manufactured, supplied or used as a means of directly or indirectly: destroying, stupefying, repelling, inhibiting the feeding of, or preventing infestation by or attacks of, any pest in relation to a plant, a place or a thing; or destroying a plant; or modifying the physiology of a plant or pest so as to alter its natural development, productivity, quality or reproductive capacity; or modifying an effect of another agricultural chemical product; or attracting a pest for the purpose of destroying it (National Registration Authority, 2001).
Agricultural organics	Any residual organic materials produced as by-products of agricultural and forestry operations, including: weeds (woody and non-woody); animals (processing residuals, stock mortalities, pests), and crop residuals (woody and non-woody), and manures.
Auxin-like herbicides	Herbicide grouping used to define herbicides with a mode of action similar to auxins (from Woods End Research Laboratory, 2002).
Broadleaf plants	Dicotyledonous plants, including both herbaceous and woody species, which have wide, rounded or flattened leaves and netted veins, as distinct from grasses and grass-like plants (National Registration Authority, 2001).
Chemical persistence	The time a chemical remains essentially unaltered in the environment.
Compost	An organic product that has undergone controlled aerobic and thermophilic biological transformation to achieve pasteurisation and a specified level of maturity. Compost is suitable for the use as soil conditioner or mulch and can improve soil structure, water retention, aeration, erosion control, and other soil properties.
Compostable organics	Compostable organics is a generic term for all organic materials that are appropriate for collection and use as feedstocks for composting or in related biological treatment systems (e.g. anaerobic digestion). Compostable organics is defined by its material components: residual food organics; garden organics; wood and timber; biosolids, and agricultural organics.
Composting	The process whereby organic materials are pasteurised and microbially transformed under aerobic and thermophilic conditions for a period not less than 6 weeks. By definition, it is a process that must be carried out under controlled conditions yielding mature products that do not contain any weed seeds or pathogens.
Cotyledon	First seedling leaves that serve as food-storing organs or may develop the ability to photosynthesis as the seed germinates (NSW Agriculture, 1999).
Electrical conductivity (EC)	A measure of the ability of a solution to carry an electrical current; varies both with the number and type of ions contained in the solution. Usually measured in deci-Siemens per metre (dS m ⁻¹).
Feedstock	Organic materials used for composting or related biological treatment systems. Different feedstocks have different nutrient concentrations, moisture, structure and contamination levels (physical, chemical and biological).

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Term	Definition
Forage crop	A crop grown specifically for the purpose of being grazed by, or fed to, livestock, but excluding pasture. The term excludes crops such as cereals, oil sees, vegetables and cole crops, which may be grazed as opportunity crops. If any of these other crops are to be grown for forage they should be specifically referred to as, for instance 'cereals for forage' (National Registration Authority, 2001).
Garden organics	The garden organics material definition is defined by its component materials including: Putrescible garden organics (grass clippings); non-woody garden organics; woody garden organics; trees and limbs; stumps and rootballs. Such materials may be derived from domestic, commercial and industrial and commercial and demolition sources. Garden organics is one of the primary components of the compostable organics stream. Garden organics is the standard material description from the Australian Waste Database.
Herbicides	A material that will kill plants. Herbicide may kill virtually all plants (non-selective) or by quite <i>selective</i> in the way they work. They may be knockdown (short-lived) or residual in the soil.
Label	The written, printed and related graphic matter on, or attached to, the container in which a product is directly packed and the outside container or wrapper of the retail package, if there be any. A label includes tag, leaflet, brand, stamp, mark, stencil or written statement (National Registration Authority, 2001).
Manure	Refers to all faecal and urinary excretion of livestock and poultry that are appropriate for collection and use as feedstock materials for composting or in related biological treatment systems. This material may also contain bedding, spilled feed, water or soil. See also agricultural organics. Such material may be derived from agricultural sources. These materials form one of the material description subcategories within the <i>Agricultural Organics</i> material description.
Non-crop areas	Areas of land not being used or not intended to be used for cropping. These areas include industrial sites, timber yards, areas around farm buildings, along fences and roadsides, rights-of-way, storage areas, wastelands, vacant lots, cemeteries etc. (National Registration Authority, 2001).
Non-woody garden organics	Refers to leafy and/or succulent compostable organic plant materials that generally have a diameter of less than 5 mm that are appropriate for collection and use as feedstock materials for composting without necessarily requiring size reduction. Some materials including vines and tussocky grasses may be relatively unchanged through size reduction processes, and loads can often contain inorganic soil, rubble and physical contaminants. Such material may be derived from domestic, agricultural, forestry, C&D or C&I sources. Non-woody garden organics form one of the material description subcategories within the <i>Garden Organics</i> material description.
Pastures	Herbage grown specifically for the purpose of being grazed by, or fed to, livestock. Pastures include lucerne, medics, clovers and grasses, whether for grazing or seed crops. The word 'herbage' excludes crops such as cereals, oilseeds, vegetables and cole crops (National Registration Authority, 2001).
ppb	Parts per billion
ppm	Parts per million

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Term	Definition
Recycled organics	The term Recycled Organics has been adopted by NSW Waste Boards (now Resource NSW) and EcoRecycle Victoria as a generic term for a range of products manufactured from compostable organic materials (garden organics, food organics, residual wood and timber, biosolids and agricultural organics). Specific recycled organic (RO) products are defined in the following Australian Standards and NSW EPA guidelines: AS 4454 (2001-draft) Composts, mulches and soil conditioners; AS 3743 (1996) Potting mixes; AS 4419 (1998) Soils for landscaping and garden use; AS/NZS 4422 (1998) Playground surfacing: specifications, requirements and test methods; NSW EPA (1997) Environmental guidelines: use and disposal of biosolids products. Whilst quality standards exist, there are also many raw RO products that are not defined in any standard and are completely unregulated, certain risks are associated with their use.
Recycled organics industry	A range of related business enterprises involved in the processing of compostable organics into a range of recycled organics products, and the development, assessment, marketing, promotion, distribution and application of those products.
Rights-of-way	Roads, stock routes, pathways, railways, power lines, telephone lines, fuel and water pipelines (National Registration Authority, 2001).
Selective herbicide	An herbicide that kills only certain groups of plants (NSW Agriculture, 1999).
Systemic	Entering the system of a plant and freely transported within its tissues (Oxford University Press, 2002).
Volatilisation	The conversion of a chemical from liquid or solid to a gas or vapour (NSW Agriculture, 1999).
Woody garden organics	Refers to all compostable organic plant materials that have a diameter of between 5 and 150 mm that are appropriate for collection and use as feedstock materials for composting or in related biological treatment systems. Such material may be derived from domestic, agricultural, forestry, construction and demolition or commercial and industrial sources. These materials contain a significant wood or cellulose component, requiring different size reduction technology from non-woody garden organics. Examples include: branches; twigs and bark. Woody Garden Organics forms one of the material description subcategories within the <i>Garden Organics</i> material description.