# HERBICIDE RESIDUES – MEASURING HERBICIDE CARRYOVER

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#### TAKE HOME MESSAGES

- Crop toxicity thresholds for residues of clopyralid and imazapyr in soil have been developed.
- Clopyralid (Lontrel®) and imazapyr/imazamox (Intervix®) residues from 2019 application in barley did not affect the growth, yield or nodulation of following 2020 lentil crop at Curyo.
- There was significant variation in the breakdown of imazapyr/ imazamox applied in 2019 at four other sites but levels at sowing of 2020 crops in these paddocks would have unlikely caused damage to subsequent crops.

### **BACKGROUND**

Herbicides are a valuable tool for controlling weeds and reaching crop yield potential but herbicide residues in soils can limit crop performance if not managed correctly. The recently concluded Grains Research and Development Corporation (GRDC) project DAN00180 (Rose et al., 2019) found that between 5-15 % of surveyed paddocks (n=40) contained residues of sulfonylureas or trifluralin that could reduce seedling vigour of some crops. Damage was avoided in most cases by growing tolerant crops (e.g. cereals or tolerant legumes in paddocks with sulfonylurea/group B residues). Growers also identified imidazolinone (group B) and clopyralid (group I) residues as potentially damaging to crops or constraining rotation options. However, the exact loss of productivity due to herbicide residues as a soil constraint has not been accurately determined due to the lack of tools to measure herbicide residues and quantify herbicide damage. It is difficult for growers and advisors to know whether herbicide residues will cause issues beyond the 'label' plant-back period because the persistence and behaviour of these residues depends on numerous site-specific factors including soil chemistry, organic matter, microbial activity and climatic conditions.

There are currently very few tools to assist growers to determine the level of herbicide residues present and if they negatively affect soil and crop performance. This project will develop knowledge and tools to better understand the factors regulating herbicide persistence and bioavailability. This will give farmers an increased confidence in crop choice, timing of sowing and herbicide management to ensure soil and crop performance are not limited by herbicide residues.

This research was established in 2019 where a Spartacus barley crop was treated with herbicides containing imidazolinone and clopyralid active ingredients to look at the impact of their application on crop performance and breakdown patterns. This report summarised the first two years of the project.

In addition to the herbicide removal trial work, data is also presented on the monitoring conducted in commercial paddocks over 12 months to look at breakdown patterns of herbicides on different soils and in different environments. This is an extension of the work presented in the 2019 BCG Season Research Results (Rose et. al. 2019).

## **AIM**

To determine the persistence of imidazolinone and clopyralid herbicides over multiple seasons in different soil types and what levels incite crop damage functions.

## PADDOCK DETAILS

Location: Curyo

Crop year rainfall (Nov-Oct): 342mm

GSR (Apr-Oct): 205mm

Soil type: Sandy Loam

Paddock history: 2019 – Spartacus Barley (plots), 2018 – Scepter Wheat (farmer crop)

## TRIAL DETAILS

Crop type/s: Lentils, Jumbo 2 and Hallmark XT overlay on below

herbicide treatments

Treatments: Herbicide treatments included Nil control, Farmer practice all year

(imazamox + imazapyr), Farmer practice all year (clopyralid), no summer spray (imazamox + imazapyr), no summer spray (clopyralid).

Target plant density: 120 plants/m<sup>2</sup>

Seeding equipment: Knife points, press wheels, 30cm row spacing

Sowing date: 15 May 2020

Replicates: Four

Harvest date: 21 November 2020

Trial average yield: 2.2t/ha

# TRIAL INPUTS

Fertiliser: Granulock® Supreme Z + Impact @ 60kg/ha at sowing

Herbicide: See Table 1 for focus herbicides. Other products were used as per

standard farmer practice where applicable but are not listed here

as were not part of the primary trial outcomes.

Table 1. Treatment list and application dates for focus products.

Treatment ID	Treatment	Focus Chemical and rate	Date of Application
А	Nil herbicide	-	-
В	Clopyralid in crop – no summer spray	Lontrel® Advanced @ 30ml/ha	27 June 2019
С	Imazamox/imazapyr in crop – no summer spray	Intervix® @ 750ml/ha	27 June 2019
D	Clopyralid in crop + summer farmer practice	Lontrel® Advanced @ 30ml/ha	27 June 2019
Е	Imazamox/imazapyr in crop + summer farmer practice	Intervix® @ 750ml/ha	27 June 2019

#### **METHOD**

This trial was established in 2019 and sown to Spartacus barley. Herbicide treatments were applied in season (Table 1.) according to farmer practice with a focus herbicide which in this case was identified to be either Imazamox/imazapyr or clopyralid. Soil sampling was undertaken quarterly at two depths (0-10cm and 10-30cm) to determine rates of breakdown over time using gas/liquid chromatographymass spectrometry methods. Crop performance was also measured. In 2020, plots were split and sown to either Hallmark XT or Jumbo2 lentils. Assessments were carried out on crop biomass at flowering, nodulation, N-fixation and yield.

In conjunction with the field trials, dose-response bioassays were conducted for clopyralid and imazapyr in soil taken from the trial site. Large volume topsoil samples taken in 2019 prior to herbicide application were transported to NSW DPI Wollongbar, air dried, homogenised and sieved to <2mm. Subsamples (20 kg) were then spiked with increasing concentrations of either herbicide, with six levels ranging from 0 – 100 ng/g (for clopyralid) or 0 – 30 ng/g for imazapyr. Residue levels were confirmed by liquid chromatography/mass spectrometry. Wheat (cv. Scepter) or lentils (cv PBA Bolt) were sown into pots (dimensions 65 mm by 65mm and 160mm depth, filled with 140mm soil kept moist to 80% field capacity) and harvested after 21 days after sowing. Shoots were cut at the soil surface, weighed, dried at 60°C for 2 days and then re-weighed to determine dry weight. Dose-response thresholds were determined by fitting shoot dry weight data to soil clopyralid concentrations using 4 parameter log-logistic curves using the package 'drc' (Ritz et al., 2015) in the R statistical software environment (R Core Team, 2019). The 'effective dose' for 20% shoot biomass reduction (ED<sub>20</sub>) was calculated.

The persistence of imazapyr was also measured at four field sites during the 2019 growing season through until mid-2020. Four soil samples comprising of homogenised sub-samples each were taken within a 100 m by 100 m georeferenced grid at participating farmer paddocks prior to sowing the 2019 winter crop (Mar-April 2019), at two depths: 0-10 cm and 10-30 cm. Repeated soil sampling occurred throughout the year following application of the imazapyr according to the following schedule: 1, 7, 21, 42, 84, 168, 364 days after herbicide application. Soil samples were refrigerated and transported to NSW DPI, where they were dried at 40°C and then stored frozen until analysis for herbicide residues. Herbicides were extracted from soils and analysed via LC-MS/MS, with spike-recoveries for each soil type to ensure satisfactory sensitivity, accuracy and precision.

#### RESULTS AND INTERPRETATION

#### Dose-response bioassays for soilborne clopyralid and imazapyr

Lentils were not sensitive to imazapyr ( $ED_{20} > 30$  ng/g), but were sensitive to clopyralid in the sandy loam, with an  $ED_{20}$  of 2.5 ng/g at which seedling shoot biomass was estimated to be 20% lower than in control soil with no clopyralid (Table 2).

In contrast, wheat was sensitive to imazapyr, with an  $ED_{20}$  of 4.2 ng/g in the Curyo sandy loam. As expected, wheat was tolerant to all residue levels of clopyralid (up to 100 ng/g tested).

Table 2. Toxicity thresholds for 20% shoot dry weight reductions ( $ED_{20}$ ).

Crop	Herbicide	ED <sub>20</sub> (ng/g)
Lentil	Clopyralid	2.5
	lmazapyr	>30
Wheat	Clopyralid	>100
	lmazapyr	4.2

#### Herbicide Removal Trial 2019-20

Behaviour of herbicide in the soil

In January 2020, approximately six months after the application of herbicides, residues of imazapyr/imazamox and clopyralid were still detectable in plots receiving each of the herbicides. However, by April 2020 clopyralid was no longer detectable (<1 ng/g in all but one plot) and imazapyr/imazamox in the top 10 cm of soil had declined to approximately 4 ng/g. By the time of sowing, (15<sup>th</sup> May 2020) residues of all focus herbicides had declined to levels below the estimated ED $_{20}$  for lentils.

Notably, there was no significant difference in herbicide dissipation between plots receiving summer fallow sprays vs plots that only received in-crop sprays. This suggests that soil microbial populations that help to breakdown residual herbicides were not significantly impacted by summer fallow sprays.

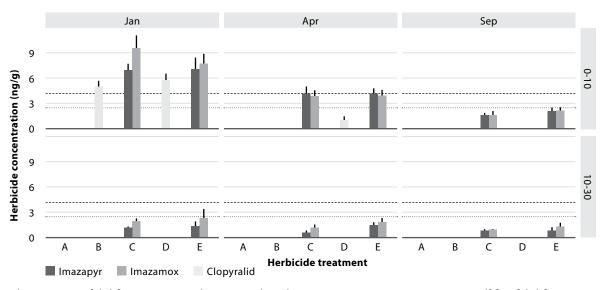


Figure 1. Herbicide concentration over time in 2020. Treatments were: A = Nil herbicide; B = Clopyralid in crop – no summer spray; C = Imazamox/imazapyr in crop – no summer spray; D = Clopyralid in crop + summer farmer practice; E = Imazamox/imazapyr in crop + summer farmer practice.

Influence of herbicide on crop biomass and yield

From results collected in 2019, there was no influence on crop performance of Spartacus CL barley from herbicide use (data not shown). This is as would be expected as the crop type used has recommended use patterns to clopyralid and is tolerant of the use of imazamox and imazapyr (Intervix®) as part of a herbicide program. Demonstrating the reason it is commonplace in the program in this area, as it gives flexibility to manage weeds with unconventional chemistries due to breeding for crop tolerance.

In 2020, when the trial was sown to a more susceptible crop (lentils) there was no difference in performance between varieties and herbicide treatments that was statistically significant (Table 3), although there was a trend that not using herbicides and allowing weeds to impact crop performance as happened in the trial, is detrimental to yield as would be expected and why herbicides are an important part of farming systems. There was no significant interaction noted so only main effects are presented. Although it could have been expected that Lontrel® or Intervix® would have influenced one or both varieties in terms of performance, the seasonal conditions that were presented over the period of the trial were conducive to sufficient breakdown of the herbicides, limiting the effect on crop performance as discussed above.

Table 3. Crop biomass and yield results from variety and herbicide treatments in 2020.

	Crop biomass (t/ha)	Grain yield (t/ha)
Variety		
Hallmark XT	1.2	2.21
Jumbo 2	1.15	2.18
Herbicide Treatment		
Nil Herbicide	1.19	1.91
Clopyralid in crop – no summer spray	1.17	2.36
lmazamox/imazapyr in crop – no summer spray	1.16	2.27
Clopyralid in crop + summer farmer practice	1.11	2.12
Imazamox/imazapyr in crop + summer farmer practice	1.23	2.32
Sig. diff.	NS	NS
LSD (P=0.05)	NS	NS

*Influence of herbicide on nodulation and N fixation* 

Nodulation and N fixation effects of herbicide treatment was also measured and showed no difference as a result of herbicide treatments (data not shown). This is not surprising given the favorable conditions that likely allowed enough breakdown and lack of effect on biomass and crop yield. However herbicidal effects that influence pulse crops ability to nodulate should remain a consideration, as it may have flow on effects to subsequent crops due to increased nitrogen requirements to meet the needs of the crop. Further work in this area is required to understand the unmeasured effects of herbicide use that may have longer term impacts on the system.

# **HERBICIDE PERSISTENCE 2019-20**

There was significant variation in the persistence of imazapyr at the four different sites where imazapyr was applied and measured. Dissipation of imazapyr at Horsham and Kinnabulla was relatively rapid (Table 3, Figure 2), where >150 mm rain was recorded in the six months following application.

In contrast, the two sites where imazapyr persistence was >100 days (Jil Jil and Brim) had <100 mm rain for the 6-month period following application. Both these sites also have a higher clay content, which may increase sorption and therefore decrease the herbicide bioavailability for microbial breakdown. The lower amount of rainfall also restricts microbial activity and can therefore lead to longer herbicide persistence in soil. The pesticide properties database (PPDB, 2015) lists the field half-life of imazapyr as 6-142, supporting our results that there is large variation across different sites.

It is likely that the Horsham, Kinnabulla and Jil Jil sites would not have experienced any plant-back toxicity in the 2020 season, as levels of imazapyr (and imazamox where it was co-applied) were <1ng/g by sowing in 2020. Although imazapyr residue levels were between 3-4 ng/g at sowing in 2020 at the Brim site, it is likely that phytotoxicity thresholds would be higher than shown in Table 2 due to the higher soil clay content and predicted lower bioavailability. Research into the effects of sorption on herbicide bioavailability in different soil types will attempt to confirm or reject this prediction.

Table 3. Soil properties, imazapyr application date and imazapyr dissipation at four monitoring sites.

Location	Soil Clay (%)	Soil pH	Soil OC (%)	Date of application	Precipitation 0-180 d post-spray (mm)	Estimated 1st-order half-life (d)
Horsham	9	7.6	1	6 June 2019	230	11
Kinnabulla	14	7.4	1.1	1 June 2019	150	13
Jil Jil	29	8	1	22 July 2019	94	118
Brim	19	7.5	1	2 August 2019	99	320

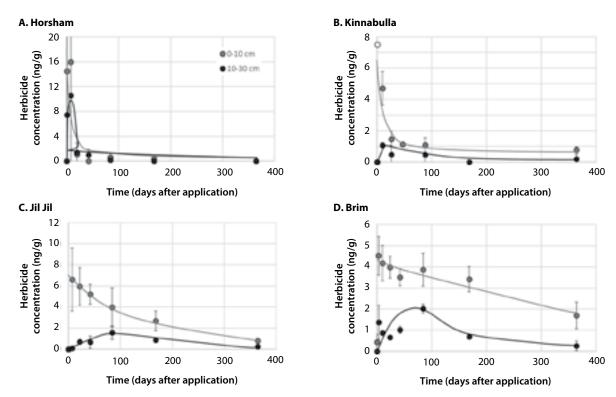


Figure 2. Imazapyr residue concentrations in 0-10 cm layer (grey points) and 10-30 cm layer (black points) at four BCG monitoring sites. Points represent average residue levels of three field replicates. Smoothed splines have been overlaid as a visual guide and are not statistical model fits.

## COMMERCIAL PRACTICE AND ON-FARM PROFITABILITY

Herbicide residues continue to be a large consideration when making crop rotation selections in regions where rainfall can be unpredictable and sporadic. As expected under good seasonal conditions, breakdown of otherwise persistent herbicides can occur quite quickly but these conditions or more the exception than the norm.

Tools to assess the risk posed by residual herbicide have not been available and this project aims to offer opportunities to make more accurate decisions around the risks presented from application of herbicides known to be persistent in certain environments and/or soil types. It is hoped that through a better understanding of factors that contribute to persistence combined with modelling, the concern around how long is 'long enough' before putting a susceptible crop into the rotation will be less about gut feel and more about educated decisions.

#### REFERENCES

Rose M., Van Zwieten L., Angel K., 2019, 2019 BCG Season Research Results, 'Herbicide Residues – Measuring Herbicide Carryover' pp 99-103.

PPDB (2015) Pesticides Properties Database, available online at <a href="http://sitem.herts.ac.uk/aeru/ppdb/">http://sitem.herts.ac.uk/aeru/ppdb/</a>>.

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