CLOPYRALID HERBICIDE RESIDUES IN STREAMWATER AFTER AERIAL SPRAYING OF A PINUS RADIATA PLANTATION

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ABSTRACT

Residues of clopyralid (also known as 3,6-dichloropicolinic acid or 3,6-DCPA) in streamwater were monitored after helicopter application of the herbicide LONTREL L at a rate of 2.5 kg a.i./ha to 56 ha of a **Pinus radiata** D. Don plantation at Archerton in north-eastern Victoria, Australia, to control the woody weed, silver wattle (**Acacia dealbata** Link). During and after the spraying, the streamwater was regularly sampled 0.5 km below the sprayed area and at a point 13 km downstream for a 19-day period, during which there were seven substantial rainfall events totalling 143 mm. The highest clopyralid concentration (0.017 mg/l) was detected just below the sprayed area soon after the start of the first rainfall event after spraying; this concentration is much lower than the maximum recommended level of 1 mg/l in potable water. At the downstream sampling point, the highest concentration detected was 0.001 mg/l. The results indicated that the main contamination was due to rainfall washing herbicide deposits from streamside vegetation that had intercepted minor amounts of spray drift.

The negligible concentrations of clopyralid found in streamwater during this study, despite substantial rainfall (72 mm) within 3 days of spraying, were attributed to several factors: (i) the small proportion of catchment (16%) that was sprayed, (ii) the presence of unsprayed streamside reserves, (iii) the use of techniques that ensured accurate spraying and minimised spray drift, and (iv) the pattern of rainfall after spraying that included low-intensity storms followed by high-intensity storms.

Keywords: herbicide; 3,6-dichloropicolinic acid; streamwater contamination; aerial spraying; **Acacia dealbata**.

INTRODUCTION

The relatively new herbicide clopyralid (3,6-dichloropicolinic acid, Haagsma 1975) has recently been identified as an effective chemical for the control of silver wattle which frequently competes strongly with *Pinus radiata* in plantations on former native forest sites in Victoria and south-eastern New South Wales (Flinn & Minko 1980; M. Hall, Forestry Commission of New South Wales, pers. comm.; Fagg & Flinn 1983). In *P. radiata* plantations in New Zealand, clopyralid has also been successful in controlling silver wattle, as well as other woody weeds such as gorse (*Ulex europaeus* L.) (Rutherford 1982).

This paper describes a study in which a 56-ha plantation of *P. radiata*, located within a domestic water supply catchment, was aerially sprayed with clopyralid, and streamwater was sampled to determine whether any stream contamination by clopyralid residues was within the allowable limit for potable water.

STUDY AREA

The sprayed plantation was at Archerton, in the headwaters of the catchment of Ryans Creek that supplies domestic water for Benalla (population 8500) in north-eastern Victoria (Fig. 1). The off-take for Benalla's water supply is at Loombah Weir

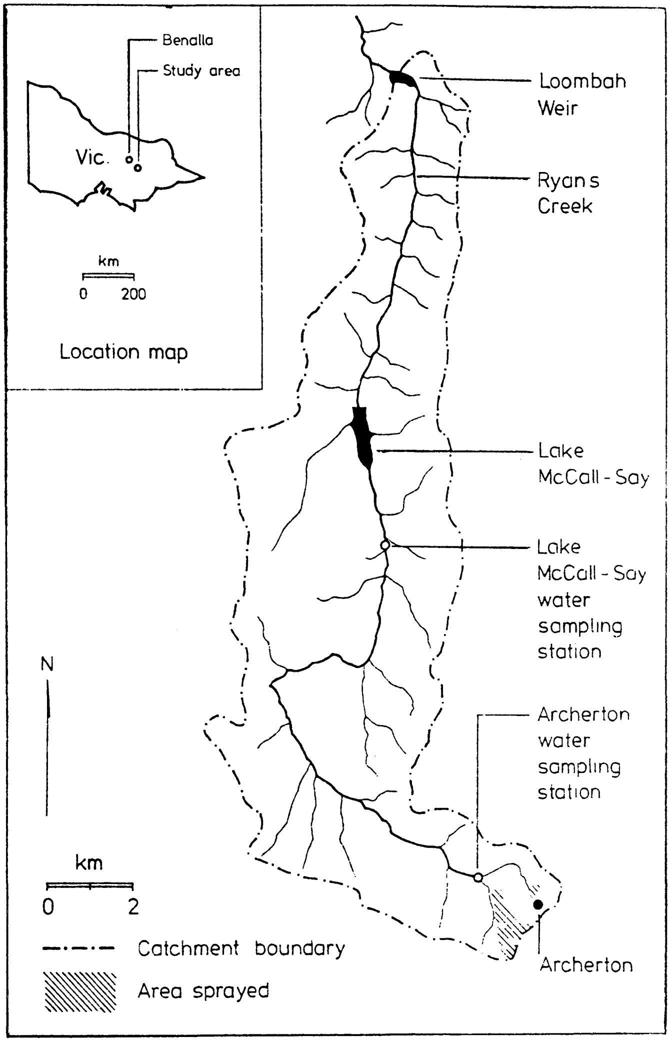

FIG. 1—The catchment of Ryans Creek above Loombah Weir, in north-eastern Victoria. Shown is the area sprayed near Archerton and the two water sampling stations used in the clopyralid monitoring study.

on Ryans Creek, 25 km downstream of the sprayed area. Lake McCall-Say is a man-made reservoir on Ryans Creek upstream of Loombah Weir.

Mean annual rainfall at Archerton is 1360 mm. Soils in the study area are gradational orange-red clay-loams of moderate erodibility, and elevations range from 890 m to 960 m with slopes up to 12° on westerly and north-westerly aspects. The *P. radiata* plantation was established between 1977 and 1980 on land previously used for grazing and potato cropping. A 20- to 90-m-wide reserve of either native vegetation or improved pasture has been maintained on both sides of the north and south branches of Ryans Creek, for stream protection and conservation of flora and fauna. Most of the vegetation of the Ryans Creek catchment above Loombah Weir is dry sclerophyll eucalypt forest.

At the time of spraying, there was a dense cover (average of 8000 stems/ha) over the majority of the treated area of silver wattle that had a predominant height of 3–4 m. Where the competition from the wattles was most intense, the *P. radiata* was stunted and spindly, and only 1–3 m tall.

Both the north and south primary tributaries of Ryans Creek are adjacent to the sprayed area, and were flowing at the time of spraying. The 56-ha sprayed area was 16.4% of the 342-ha catchment of the Archerton streamwater sampling station, and 1.4% of the 4050-ha catchment of the Lake McCall-Say streamwater sampling station (Fig. 1).

METHODS

Spraying

Clopyralid (as LONTREL L Selective Herbicide*, containing 300 g/*l* clopyralid present as the monoethanolamine salt) was applied by a Bell Jet Ranger II helicopter at a rate of 2.5 kg/ha in a spray volume of 60 *l*/ha that comprised 14% LONTREL L, 33% ULVAPRON* (an anti-evaporant paraffinic oil that enhances the efficacy of clopyralid on silver wattle, Fagg & Flinn 1983), and 53% water. The 10-m spray boom (outer nozzle to outer nozzle) was fitted with 28 nozzles (D7/56) spaced 30 cm apart and set in a nearly fully-trailing position. In addition, six similar nozzles (which pointed downwards in flight) were mounted on flexible feeder tubes beneath the 420-*l* spray tank.

The pilot was instructed to spray only the plantation area marked on a map given to him, which clearly showed the buffer strips and watercourses to be avoided, and a reconnaisance flight was made.

Spraying began at 1630 h and concluded at 1735 h on 21 April 1983. A light wind of 2–5 km/h blew from the north-east initially, though this ceased during the last 0.5 h of spraying. During the spraying operation, air temperature decreased from 12°C to 11°C, relative humidity increased from 58% to 60%, and cloud was high and light. Flight lines were not marked, though indistinct corners of the plantation were. In flight, the spray boom was generally 5–6 m above the tallest trees on the target area. It appeared from ground observation of the spraying operation that relatively

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uniform coverage and good cut-off of spray at boundaries were achieved although some spray mist, largely generated by the hanging nozzles, spread under the influence of the light wind in a westerly direction towards the creek.

In order to detect any spray drift into the stream, the streamside reserve, or adjacent private property at Archerton, a total of 43 spray-sensitive KROMEKOTE cards were set out along five transects on the morning of the spraying, and were retrieved within 1.5 h of spraying being completed. The transects ran approximately at right angles from inside the plantation, across the reserve to the stream.

Streamwater Sampling

Samples of streamwater were collected from Ryans Creek at two stations: Archerton Station was immediately below the confluence of the upper branches, about 0.5 km from the closest area sprayed, and Lake McCall-Say Station was at the most accessible point above Loombah Weir and approximately 13 km downstream of the sprayed area (Fig. 1). Sampling began 4.5 h before spraying on 21 April 1983, and concluded on 9 May 1983, when it was considered that enough rain (143 mm) had fallen to flush any mobile herbicide into the stream.

An ISCO 1680 automatic water sampler, containing glass bottles to receive the water samples, was installed at Archerton Station and continuous sampling was undertaken at intervals ranging from 0.25 h to 4 h. The most intensive sampling was during aerial spraying and for 6 h afterwards, and during the four storms that occurred within a few days of spraying. The intake hose of the automatic sampler was positioned so that thoroughly mixed samples of streamwater were obtained. Streamwater at Lake McCall-Say Station was manually sampled using a USDA DH-48 depth-integrating sampler; generally, sampling was 2-hourly during daylight, giving four samples per day, though this was extended on wet days to provide six samples per day.

The samples were transferred to and subsequently stored in 500-ml, brown, soda-glass bottles with aluminium foil under the plastic caps. To avoid contamination of samples, the funnel was rinsed with deionised water after each transfer, and the collection bottles were similarly rinsed and shaken "dry" before being returned to the automatic or manual samplers. Samples were placed in cool, dark storage at 4°C within 4 days of collection.

Rainfall and Streamflow

A Commonwealth Bureau of Meteorology rain station is located at Archerton (Fig. 1) and rainfall was measured daily at 0900 h. To supplement this record, a MORT pluviometer was installed in a clearing adjacent to the sprayed area, 2 km west of the Archerton rain station.

A 90° V-notch weir, a staff, and a peak-stage indicator were installed at Archerton Station so that streamflow at the time of servicing and peak flow between visits could be estimated from a theoretical stage-streamflow rating curve (U.S. Geological Survey 1977). Flow depth at Lake McCall-Say Station was recorded at each visit to collect water samples, and stream cross-sections and velocities were measured at a range of flows to provide estimates of streamflow.

Analysis of Water Samples

Selection of samples for analysis

A total of 416 individual samples were collected from the two stations on Ryans Creek over the 19-day monitoring period. Both individual and composite samples were chosen for analysis in such a way that all rainfall events were represented (Table 1). A larger proportion of samples was selected for analysis from the events that were most likely to result in contamination of streamwater, viz, the period during and immediately after spraying, and the storms (defined as discrete rainfall events of >5 mm) that occurred within 4 days of spraying. On days with less than 5 mm rainfall, the samples taken nearest to 0900 h were selected for individual analysis. Aliquots of the majority of the remaining samples, covering both storm and non-storm periods, were combined to form composite samples for analysis.

As budget constraints meant that only a certain number of samples could be analysed, samples were selected for analysis using a two-stage process. Initially 32 individual samples from Archerton Station were selected to cover the main rainfall events and analysed for the presence of clopyralid. Using the trends found in these results as a basis for selection, a further 65 samples (44 individual, 21 composite) from Archerton Station, and 30 samples (27 individual, 3 composite) from Lake McCall-Say Station were selected and analysed (Table 1).

Chemical analysis

Analyses for clopyralid were carried out in June and July 1983, resulting in storage of samples for 8 to 12 weeks.

The analytical method, adapted from procedures supplied by Dow Chemical (Australia) Limited, was as follows: a 10-ml aliquot of each sample was acidified with 4 ml of 2M sulphuric acid, saturated with sodium chloride (4.5 g), and extracted with 10 ml and 5 ml, respectively, of diethyl ether. The ether extracts were dried by passing through anhydrous sodium sulphate, combined, and evaporated to 1 ml with a stream of dry air. Methyl derivatives were formed by addition of 1 ml of diazomethane reagent in hexane, mixing, and standing the sample for 1 h at room temperature.

Concentrations of methyl 3,6-dichloropicolinate in hexane were determined by the injection of 1–4 μl of this solution into a Varian Research Model 2740 gas chromatograph fitted with a 1.5 m × 3-mm i.d. glass column, silanised and packed with 10% silicone OV 225 on Chromosorb W HP 100/120 mesh and a ^{63}Ni electron capture detector. High-purity nitrogen (44 ml/min) was the carrier gas recommended by Varian, requiring no quench gas. The temperatures were column 200°C, injector 250°C, and detector 300°C. The instrument was calibrated using standards of methyl 3,6-dichloropicolinate in hexane, equivalent to clopyralid concentrations of 0.020 and 0.100 mg/*l*. Recoveries based on eight solutions of water and clopyralid, 0.002–0.050 *mg/l,* were 90% ± 10%. The minimum detectable concentration of methyl 3,6-dichloropicolinate for the above method was taken as 0.001 mg/*l,* based on a peak height of 1 cm for injection of 4 μl of derivatised extract from a sample containing 0.001 mg/*l* clopyralid.

TABLE 1—Distribution of streamwater samples selected for analysis of clopyralid residues from periods before, during, and after aerial spraying at Archerton, north-eastern Victoria, on 21 April 1983

Event	Automatic sampling 0.5 km below sprayed area (Archerton Station)				Manual sampling 13 km below sprayed area (Lake McCall-Say Station)	
	Duration of sampling (h)	No. of samples analysed		Interval between individual samples selected for analysis (h)	No. of samples analysed	
		Individual	Composite		Individual	Composite
Before spraying	4.5	1	1	—	1	0
During spraying and for 6 h after	7.0	10	1	0.25–0.9	3	1
Storms* 1 and 2	12.5†	16	2	0.25–1	2	0
Storms 3 and 4	17.5†	19	2	0.5–1, except for one interval of 3 h	7	1
Storm 5	12.0	4	1	4	1	0
Storm 6	22.5	10	1	1–2, except for first 7 h of light rainfall	6	0
Storm 7	12.0	4	1	4	0	1
Days with <5 mm rainfall	12 days	12	12	24	7	0
Total No. of samples		76	21		27	3

* Discrete rainfall events of >5 mm.

† Sampling continued for 6 h after cessation of rainfall, to ensure that peak flows were sampled.

To determine any loss in concentration of clopyralid during storage, solutions of LONTREL L at concentrations of 0.100, 0.010, and 0.001 mg clopyralid/*l* in uncontaminated streamwater were analysed after 5 weeks' storage. In addition, a solution of LONTREL L (1.00 mg clopyralid/*l* in distilled water) was analysed after 12 weeks' storage.

RESULTS

Spray Drift

The KROMEKOTE cards indicated that, along three of the five transects, there was little spray drift into the adjacent vegetation. Herbicide was detected immediately adjacent to running water on only one of four streamside cards.

Rainfall and Streamflow

A total of 133.8 mm of rainfall was recorded at Archerton rain station, with the rain falling on 12 of the 19 days of the monitoring period. For the same period, 142.7 mm was recorded by the pluviometer located adjacent to the sprayed area. The seven distinct storm events in this short period had a wide range of rainfall totals, durations, and intensities, as measured by the pluviometer (Table 2). Of special note was Storm 4 (34.6 mm) because, not only was it particularly intense, but it closely followed Storm 3 (21.4 mm) and therefore overland flow within the catchment would have been maximised. This intense rain caused serious erosion of ploughed paddocks at Archerton, resulting in large amounts of topsoil being deposited along the main road, and the water in Ryans Creek became extremely turbid.

Streamflow at Archerton Station varied from 2 *l*/s at the time of spraying to 210 *l*/s soon after Storm 4; corresponding flows at Lake McCall-Say Station were 120 *l*/s and 3700 *l*/s. Peak flows at Archerton for each storm are presented in Table 2.

TABLE 2—Storm event parameters at Archerton and corresponding peak flows in Ryans Creek during the 19 days in which streamwater samples were collected

Storm number	Start time & date	Duration of rainfall (h)	Rainfall (mm)	Max. rainfall intensity for 30 min (mm/h)	Peak flow: Archerton Station (l/s)	Peak flow: Lake McCall Say Station (l/s)
1	0040 h 23.4.83	1.7	8.9	6.6	7	330
2	0520 h 23.4.83	3.25	6.9	4.0	9	330
3	0030 h 24.4.83	3.0	21.4	18.2	140	3700
4	0820 h 24.4.83	3.0	34.6	35.6	210	3700
5	1320 h 1.5.83	8.5	7.6	3.0	20 (est.)	600 (est.)
6	0930 h 2.5.83	16.5	35.8	14.8	80	2300
7	2300 h 3.5.83	21.0	12.2	2.6	60 (est.)	1700

Herbicide Concentrations

Streamflow, hourly rainfall, and concentrations of herbicide residues in streamwater at Archerton Station from the start of spraying to the end of the main storm events are shown in Fig. 2. As expected, the samples collected at both stations before spraying were uncontaminated by herbicide. In the period during spraying and for 6 h afterwards at Archerton Station, the last two of the 10 individual samples analysed were slightly contaminated (containing 0.002 mg/*l* clopyralid*), though no herbicide was detected in the corresponding composite sample. In the equivalent period at Lake McCall-Say Station (it is estimated that this streamwater took 24 h to flow from the sprayed area to Lake McCall-Say Station), one of the three individual samples and the one composite sample likewise had low levels of contamination (0.001 mg/*l*).

During Storms 1 and 2, the concentrations of herbicide in the streamwater increased rapidly at Archerton Station (Fig. 2), reaching a peak of 0.017 mg/*l* 3.5 h after Storm 1 began and 35 h after spraying had finished. This maximum level was well below the maximum allowable residue limit of 1.0 mg clopyralid/*l* in potable water, as recommended by the National Health and Medical Research Council (1985), though herbicide concentrations may have been higher adjacent to the sprayed areas because only 16% of the catchment area draining to the sampling point was sprayed (Fig. 1). Even if it was assumed that the maximum detected concentration was proportional to the area sprayed, the theoretical maximum concentration adjacent to the sprayed areas would still be far below the maximum residue limit. Residue levels gradually declined over the next 12 h to around 0.003 mg/*l*.

During the very high flows associated with Storms 3 and 4, herbicide residue levels at Archerton Station peaked at 0.004 and 0.002 mg clopyralid/*l*, respectively (Fig. 2), but were below the limit of detection during the other three storms of the monitoring period.

At Lake McCall-Say Station, clopyralid was detected in only one of the 19 streamwater samples (individual and composite) representing all storm events, and this was only 0.001 mg/*l*, associated with Storm 3.

Of the 24 streamwater samples analysed from Archerton Station for days with less than 5 mm rainfall, only four contained herbicide (all 0.001 mg/*l*), whilst only one of the seven Lake McCall-Say Station samples from the same periods contained herbicide (0.001 mg/*l*). Movement of herbicide in non-storm periods was therefore negligible.

The solution of LONTREL L (1.00 mg clopyralid/*l* distilled water) that was analysed after 12 weeks' storage contained 0.90 mg clopyralid/*l*. Other solutions of LONTREL L, at concentrations of 0.100, 0.010, and 0.001 mg clopyralid/*l* uncontaminated streamwater, that were analysed after storage for 5 weeks, contained respectively 0.095, 0.009, and 0.001 mg clopyralid/*l*. As the analytical recovery rate was 90% ± 10%, it is clear that the concentrations of clopyralid in the 127 samples that were analysed would have decreased negligibly during the 8–12 weeks of storage.

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* Analytical values corrected (increased by 11%) on the basis of the 90% mean recovery rate, although this makes no difference to values $<$0.005 mg/l.

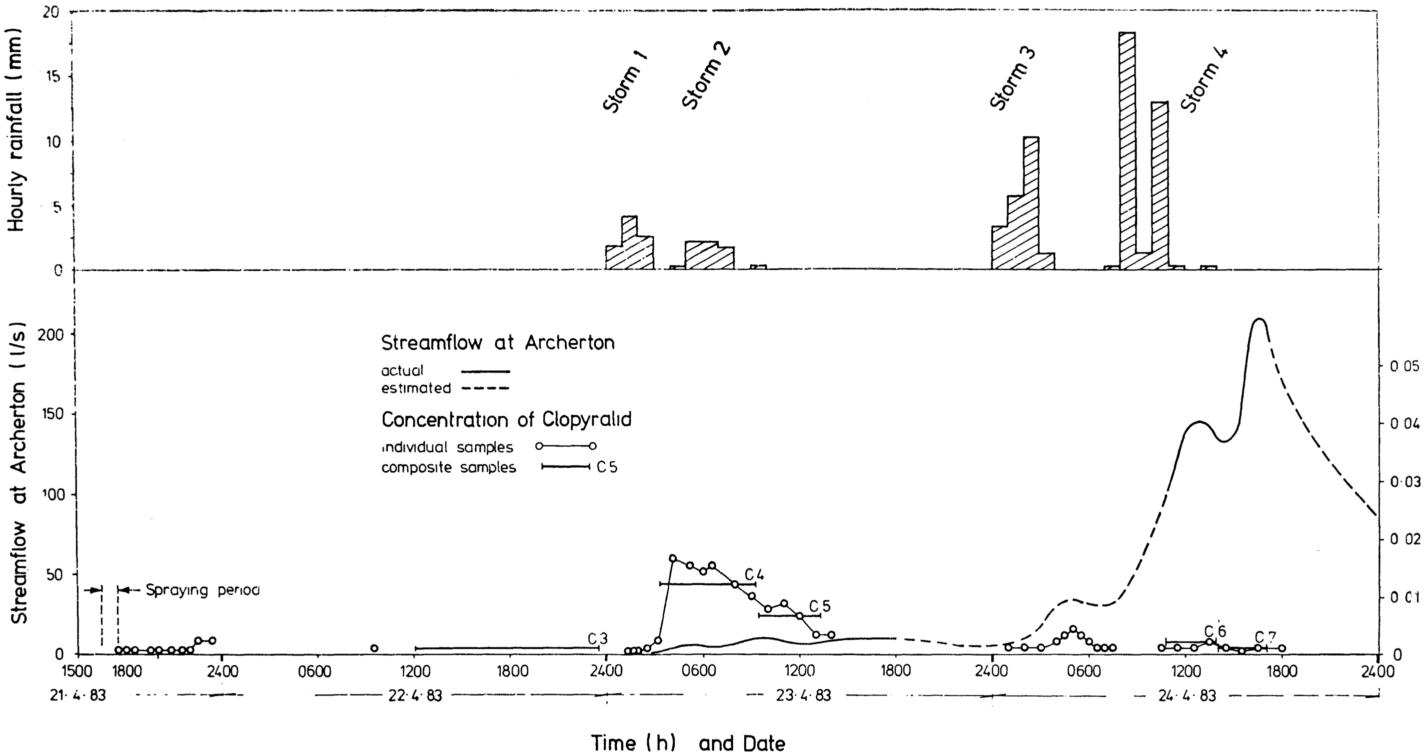

FIG. 2—Hourly rainfall, streamflow, and concentrations of clopyralid in water samples collected from Ryans Creek at Archerton Station during the 72 h after the spraying of LONTREL L.

DISCUSSION

The slight contamination of streamwater that occurred within 6 h of spraying (prior to any rainfall) was probably due to spray drift falling directly into the stream along the sections where there was little overstorey (as detected on one KROMEKOTE card). It was observed during spraying, and when viewing an 8-mm film of part of the operation, that the light north-easterly wind caused noticeable movement of fine droplets towards the southern branch of the creek. The quantity of fine (driftable) droplets was larger than planned, due to the nozzles which pointed downwards in flight causing their spray streams to suffer strong shearing forces. Ideally, these nozzles should have been fully trailing, but unlike the boom nozzles, they could not be adjusted to this position.

The first rainfall occurred 31 h after the spraying operation. As it was of low intensity, it did not cause a significant rise in stream levels (Fig. 2). Therefore, the stream contamination (up to 0.017 mg clopyralid/l) during this period (Storms 1 and 2) was more likely due to spray drift deposits being flushed from streamside vegetation, rather than herbicide being transported to the stream in overland flow.

The first heavy rainfall occurred during the third day after spraying, causing widespread overland flow and minor flooding of stream channels. A total of 56 mm of rain was received in 11 h, with a peak intensity in Storm 4 of 36 mm/h for 30 min (Table 2). During the height of this storm, a large proportion of any herbicide that had not been absorbed by foliage or adsorbed by soil would probably have been flushed into the stream system. However, only low concentrations of residues were detected in streamwater during and after this storm, and it is estimated that only 12 g clopyralid left the study area in streamflow during the high flows on 23–24 April; this represents less than 0.01% of the 140 kg clopyralid applied to the 56 ha of plantation a few days earlier. Such a low loss contrasts with high losses (>2%) often recorded where applications of other herbicides have been followed by severe storm events (Wauchope 1978). Further experimentation would be required to determine the possible reason(s) for this low result.

Residue levels in the stream were insignificant throughout the remainder of the 19-day study period, during which time there were three further storms (Table 2), so it is apparent that the clopyralid became immobilised in vegetation and/or soil a short time after spraying.

In the soil, clopyralid may have undergone some degradation over the 19 days, as one study (Anon. 1981) indicated that its median half-life was only 26 days, although its lasting hormonal effect on silver wattle foliage was obvious for up to 12 months after spraying in the present study.

The principle of dilution of streamwater contaminants with distance down a catchment was demonstrated by the decrease in residue levels between Archerton and Lake McCall-Say Stations, which were approximately 13 km apart. As the off-take for Benalla's water supply is 12 km downstream from Lake McCall-Say Station, it is likely that the contamination of Benalla's domestic water would have been less than 0.001 mg clopyralid/l (the detectable limit) at all times throughout the study period.

The effect of the maximum recorded levels of clopyralid in this study (0.017 mg/l) on stream fauna is likely to have been negligible, because the fish species, trout and bluegill, have 96-h LC_{50} concentrations of 104 and 125 mg clopyralid/*l* respectively, and the aquatic invertebrate species, daphnids, have an LC_{50} value of 230 mg/*l* for 48 h exposure (T. L. Batchelder, Dow Chemical, pers. comm.).

The very low or non-detectable concentrations of clopyralid in the streamwater samples relative to the recommended maximum limit for potable water are attributed to four main factors. Firstly, the sprayed area comprised less than 17% of the 342-ha catchment from which streamwater was monitored. Secondly, 20- to 90-m-wide vegetation reserves adjacent to the perennial streams separated the sprayed area from the watercourses, and to some extent protected them by intercepting spray drift, as also found by Leitch & Flinn (1983). Thirdly, accurate spraying was achieved by a skilful pilot, using a helicopter (instead of a fixed-wing aircraft), and spray drift was minimised (though not eliminated) by using procedures recommended by Newton & Knight (1981) that included spraying under almost calm conditions, using a system that emitted relatively large droplets, and using an anti-evaporant petroleum oil in the spray mix. Fourthly, although the initial low-intensity rainfall was sufficient to flush some herbicide from vegetation, it did not cause overland flow.

The results showed a similar trend to that reported by McKimm & Hopmans (1978) who found low 2,4,5-T residues in streamwater after rain only on the seventh and eighth days after spraying a young *P. radiata* plantation. Leitch & Flinn (1983) similarly found insignificant residues of hexazinone in streamwater after aerial spraying. Both these studies employed 30- to 40-m wide forested buffer strips to separate the sprayed area from the stream.

CONCLUSION

Despite intense rainfall occurring within only 3 days of spraying, there was no significant movement of clopyralid into Ryans Creek. The maximum concentration of clopyralid found in the samples, 0.017 mg/*l* streamwater, is only one fifty-ninth of the maximum acceptable residue limit (1 mg/*l*) recommended by the Australian National Health and Medical Research Council for potable water. Furthermore, this concentration was detected close to the sprayed areas, and subsequent dilution would have ensured that herbicide residue levels at the domestic water off-take point were insignificant.

The results strongly indicate that the main source of the minor contamination of the streamwater by herbicide was streamside vegetation that had intercepted spray drift. Thus it is clear that minimisation of spray drift towards streams is important if streamwater contamination is to be kept to low levels at all times during and after an aerial spraying operation.

It is concluded that aerial spraying of clopyralid at rates up to 2.5 kg/ha and according to the methods described, over proportionally small areas of *P. radiata* plantations in water supply catchments, poses no discernible hazard to people or wildlife using the water.

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