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ASSESSMENT OF PESTICIDE

SPRAY DEPOSITS

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COMPARATIVE EVALUATION OF SOME TECHNIQUES
USED FOR AERIAL SPRAY DEPOSIT ASSESSMENT

bу

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ABSTRACT

Deposit assessment was made in a study with the ultra-low-volume (ULV) aerial application of two types of fenitrothion formulations. Deposits were collected on spruce foliage and on standard Kromekote® card glass plate collection units. Two different techniques were used to assess deposit on foliage: gas-liquid chromatography (GLC) for the active ingredient (AI) and spectrofluorometry for the fluorescence of the tracer dye, Rhodamine B; two additional techniques were used for the Kromekote® card glass plate units, viz. spot counting on Kromekote cards and use of the spread factor data, and spectrophotometry for the tracer dye on glass plates.

Spectrofluorometry proved unsuitable for assessing aerial spray deposits on foliage because of

its insensitivity to quantify the low concentrations of dye (ppb) encountered in ULV treatment. Of the four techniques used to quantify deposits on the ground sampling units, GLC, spectrofluorometry and colorimetry gave approximately the same values while the droplet stain counts provided slightly lower ones. Nevertheless, significant differences were not observed among values obtained from the four techniques.

An additional laboratory study was carried out to investigate the recovery efficiency of Rhodamine B and AI from spruce needles over different time intervals at 22°C using various concentrations of the dye and active ingredient. The minimum quantification limit was 10 ppm (at the recovery level of 78%) as determined by spectrofluorometry.

INTRODUCTION

Aerial application of pesticides onto forest trees does not usually result in a uniform distribution of spray droplets so that some trees or parts of trees are more heavily dosed than others (Hurtig et al. 1953; Sexsmith et al. 1957; Maksymiuk et al. 1975; Williams et al. 1978). Several meteorological and topographical factors contribute to the non-uniform spray deposition (Hurtig et al. 1953; Armstrong 1975, 1977; Armstrong and Yule 1978). However, successful insect control and foliage protection, requires fairly even coverage of the entire target area so that each target zone receives adequate dosage of pesticide. It is therefore essential to obtain accurate information on spray deposit concentrations on various target matrices of the forest environment.

Several investigators have explored the use of fluorescent tracers for studying spray droplet numbers and their sizes on foliage (Staniland 1959; Himel and Moore 1967; Barry et al. 1977; Barry and Ekblad 1978; Spillman and Joyce 1978). Sharp (1976) extracted and quantified fluorescent tracer dyes from foliar deposits of herbicide sprays in a small-scale study using individual bean plants. Maksymiuk and Orchard (1975), in a moderately large-scale aerial field trial, extracted and quantified the fluorescent dyes from oak leaves.

Armstrong and Yule (1978) used the chemical tracer Tris

(2-ethylhexyl) phosphate and measured foliar deposits by GLC. None of these studies, however, used more than one technique at the same time to compare and evaluate the efficiency of residue quantification.

The present paper describes the comparative efficiency of some analytical techniques currently in use for assessing aerial spray deposits of fenitrothion on crown foliage of spruce trees, and on sampling units placed at ground level. The objective is to compare accuracy, cost and speed of the operation in each case. Two types of studies were carried out; first, spiking experiments using individual spruce needles, and second, aerial spraying over a spruce plantation forest.

Important considerations included i) the time lapse between pesticide treatment on foliage and residue measurements at room temperature, and ii) the minimum concentration required on foliage for spray deposit assessment in a field study. Specifically, the study was designed to investigate the following:

- a) The efficiency of spectrofluorometry as compared to that of gas-liquid chromatography (GLC) for measuring foliar deposits.
- b) Comparative evaluation of the four techniques, spectrofluorometry, colorimetry, gas-liquid chromatography and droplet stain on Kromekote® cards, for measuring deposits on ground sampling units.

MATERIALS AND METHODS

1) Formulations

Two formulations of fenitrothion were applied: an emulsion formulation (F1) consisting of technical fenitrothion 10%, Atlox 3409 1% (Atlas Chemical Industries, Brantford, Ontario), Arotex 3470 1% (Texaco Canada Ltd.), water 87.4% and Rhodamine B 0.6% (a 20% solution in alcohol; Allied Chemical, Morristown, N.Y.), and the oil-based formulation (F2) contained 48.5% of technical fenitrothion, 1% of Automate B red (a 50% solution in xylene) (Morton Williams Ltd., Ajax, Ontario) and 50.5% of Arotex 3470 (all percentages expressed in v/v). Prior to application, samples of the formulations were analysed for insecticide and dye tracer by GLC and colorimetry, respectively.

2) Laboratory experiments using individual spruce needles treated with emulsion formulation (F1)

i) Spectrofluorometry

The extraction efficiency of the fluorescent dye Rhodamine B was investigated at i) various time intervals and ii) various concentrations applied to spruce needles. Triplicate samples of 100 needles each were spiked with 11 or 22 μ l of the emulsion formulation to give a foliar concentration of 13 or 26 μ g of dye per 100 needles (20 or 40 μ g per g of needles). After each of 0.5, 1.0, 2.0, 3.0, 5.0 and 6.0 hr periods (samples were maintained at

room temperature of 22°C during this interval), the dye was extracted by shaking the needles gently for 1 min. with 2 x 5 ml of water containing 0.01% Atlox 3409 emulsifier. The extracts were decanted and their fluorescence was measured in a Turner 111 fluorimeter using the primary and secondary filters 7-60 and 22, respectively. The instrument had previously been calibrated using the emulsion formulation containing 0.12% (w/v) of Rhodamine B as the standard. Aqueous extracts (with 0.01% Atlox) of unsprayed spruce needles were used to determine the blank fluorescence, which was subtracted from the readings of the treated samples. Results are recorded in Tables 1 and 2. The use of 1:1 (v/v) ethanol-water mixture did not improve the recovery of the dye due to the interference of the coextracted fluorescent pigments from the foliage. The recovery levels were not improved significantly by prolonged extraction times. The use of the Atlox wetting agent at 0.01% improved the recovery, but at higher concentrations, foliar pigments were also extracted contributing to high blank fluorescence and nullifying the effect of the wetting agent.

ii) Gas-liquid chromatography

As in the previous test, 100-needle samples were spiked with 11 or 22 μ 1 of formulation F1 and the dye at each of 0.5, 1.0, 2.0, 3.0, 5.0 and 6.0 hr after treatment was extracted while samples were maintained at 22°C. To

measure the AI, the 100-needle samples (three replicates) were washed with 4 x 5 ml of ethyl acetate in a stoppered measuring jar. It was known that this procedure would only extract the fenitrothion residues on the foliar surface (surface residues) and not the tissue residues which penetrated the leaf cuticle (Sundaram and Sundaram 1982). Nevertheless, this procedure was employed to study the fraction of the spiked formulation that penetrated the leaf cuticle and/or metabolized within the 6 hr period when samples were kept at 22°C (in the study quoted above, foliar samples frozen within 1 hr after treatment contained less than 1.8% of the total residues as tissue residues). The recovery of fenitrothion in the 100-needle samples were quantitated by GLC after appropriate dilution following the procedure described by Sundaram (1974). Results are presented in Table 3.

3) Aerial spray application over a plantation forest

i) Experimental plots

The experimental spray plots P1 and P2, each covered about 16 ha and contained 80% spruce (Picea spp.), 15% pine (Pinus spp.) and 5% miscellaneous species. The plots were situated in a plantation forest of area of about a thousand ha and the distance between them was sufficient to preclude contamination by drift. The conifer component of the forest averaged 8 to 12 m in height and up to 23 cm diameter at breast height. Maple,

birch and poplar provided a scattered overstory. A light to moderate understory of willow, alder and spruce regeneration was found throughout the area. Grasses and scattered patches of mosses covered the forest floor.

ii) Experimental design

Dominant spruce trees (12 in plot P1 and 15 in plot P2), measuring about 10 m in height with a 23 cm diameter at breast height, were randomly selected in each plot. The area surrounding each sample tree was cleared for a radius of about 5 m. In each opening, four Kromekote® card glass plate units were placed at the ground level, one at each quadrant of the clearing.

iii) Spray application

The aircraft used was a Pawnee 235 fitted with four "Micronair" AU3000 spray units. A sensor attached to each "Micronair" mounting indicated the speed of rotation of the cage. The spray was applied at an approximate height of 15 m above the canopy. Plot Pl was treated with the emulsion formulation at the rate of 0.21 kg AI per ha in 1.46 L, and Plot P2 received the oil-based formulation at the rate of 0.28 kg AI per ha in 0.44 L.

iv) Meteorological conditions

Optimal weather conditions existed throughout the spray operation and the measured parameters are given below:

Plot	Wind	Relative	Temp. °C		
No.	speed (m/sec)	humidity	6 m	27 m	
P1	1.37	~ 91%	21.3	21.0	
P 2	1.5	~ 93%	13.6	14.2	

v) Deposit collection

One hour after spraying, foliage samples were clipped by pole-pruner from each quadrant of the tree (a 25 cm tip of a branch) about 1.5 m below the apex. All four samples from each tree were pooled, placed in plastic bags and stored immediately in styrofoam coolers containing dry ice. These were transported to the laboratory on the same day and stored in a freezer until analysis.

The ground sampling units were harvested 1 hr after spray application. Spray deposits were removed from the glass plates by washing with 3 x 2 ml of solvent. Methanol was used for the aqueous formulation containing the fluorescent tracer dye Rhodamine B, and toluene for the oil-based formulation containing the non-fluorescent dye Automate B red. The eluants of all four plates from each sample tree were pooled and stored in a freezer to await analysis.

vi) Deposit assessment

a) Foliar deposits

Foliar deposits were quantitated by two techniques, GLC for the active ingredient and spectrofluorometry for the fluorescent tracer dye Rhodamine B. Both techniques were used for the emulsion formulation F1, but GLC alone could be used for the oil-based formulation because Automate B red dye is non-fluorescent.

ai) Spectrofluorometry

For measuring the fluorescent dye residue on foliage, 10 g aliquots of spruce needles (about 1500 needles) were washed with 2 x 10 ml of water containing 0.01% Atlox emulsifier (after shaking for 1 min.). The extracts were read in the Turner 111 fluorimeter following the procedure described earlier. Since the readings were not significantly higher than the blank fluorescence readings obtained from untreated spruce needles, the aqueous extracts were concentrated to half of their original volumes (50%) and were read again. This failed to improve the efficiency, indicating the insensitivity and unsuitability of spectrofluorometry for field experiments involving ultra-low-volume (ULV) spray application where foliar concentrations of dye are likely to be very low. Results are given in Table 6.

aii) GLC measurements

For measuring foliar deposits by GLC, aliquots of foliage weight ca. 10 g were washed with 4 x 30 ml ethyl acetate in a Sorvall-Omni-Mixer (10 min., speed setting 7), to extract the total residues of fenitrothion. The extracts were quantitated by GLC following the procedure described by Sundaram (1974). Results are presented in Tables 6 and 7.

ii) Deposits on glass plates

The eluants from the glass plates were measured

by GLC, spectrofluorometry and spectrophotometry. For formulation F1, all three techniques were used but, for F2, only GLC and spectrophotometry could be used because of the non-fluorescent dye added to the spray mix. The GLC and spectrofluorometry measurements were carried out in the same manner as described under "foliar deposits". The spectrophotometric measurements were carried out as described by Armstrong and Randall (1969). From the measured concentration, the volume of spray that deposited on the glass plates was calculated in units of g AI/ha (deposit density). Results are given in Tables 6 and 7.

111) Deposits on Kromekote Cards

Droplet density and the size spectrum of sprays reaching the forest floor were evaluated by counting the droplet stains on Kromekote® cards and measuring their diameters under magnification. Spread factor data for the formulation were obtained in the laboratory, using the rotary device described by Rayner and Haliburton (1955) for the stain size range obtained on Kromekote® cards. These data were used to assess the aerodynamic diameters of droplets after correcting the stain sizes for the evaporation rates of droplets (see Tables 4 and 5 for the limiting values) and using the spread values obtained in the laboratory for each formulation. From the mean values of droplet density of four cards collected from each sample tree the volume of spray reaching the card

was calculated in units of g AI/ha (deposit density). The data are presented in Tables 4 and 5.

RESULTS AND DISCUSSION

A. Laboratory experiments using individual spruce needles treated with formulation F1

Ai) Spectrofluorometry

Table 1 presents recovery efficiencies of the spiked Rhodamine B at various foliar concentrations. It is evident that the recovery decreased gradually with the concentration and, below 20 ppm, the standard deviation of the mean value of recovery is appreciable, indicating the low precision of the technique. The minimum quantification limit of the method is 10 ppm at 78% recovery level.

Table 2 presents recovery efficiencies for dye concentrations of 20 and 40 ppm at various time intervals after treatment, when samples were maintained at 22°C. It is evident that no loss of dye was noted for up to 6 hr after treatment, either by cuticle penetration or by metabolic processes.

The present results indicate that the use of a dilute solution (0.01%) of Atlox® 3409 provides satisfactory recoveries of the dye from foliar surface at levels of 20 and 40 ppm up to 6 hr after treatment. Lower concentrations (e.g., 10 ppm) were not tried because of the poor recoveries noted in Table 1.

Aii) Gas-liquid chromatography

Table 3 presents recovery efficiencies of the fenitrothion active ingredient (AI) at the 2000 and 4000 ppm levels, at various time intervals after treatment when samples were maintained at 22°C. A significant and gradual loss of the insecticide is noticeable over the 6 hr period indicating either cuticular absorption or metabolic breakdown when samples were stored at 22°C. The degree of loss was ca. 14%, indicating a rapid interaction between spruce foliage and fenitrothion in presence of the additives in formulation F1.

The present study indicates that the components of spruce foliage rapidly interacts (either physically or chemically) with fenitrothion but not as rapidly, with the tracer dye Rhodamine B.

B. Aerial spray application over a plantation forest

B1) Foliar deposit assessment: Comparative efficiencies of spectrofluorometry and GLC

Table 6 presents foliar concentrations of dye (ppm) extracted with 0.01% of aqueous Atlox solution from sampling trees in plot Pl. It is clear that the method is highly insensitive and unsuitable for samples from aerial spray trials, as evident from the large deviations in dye concentrations from the theoretical values listed in parenthesis in column B. The fluorometric readings were entirely due to sporadic variations in background fluores-

cence of control foliar samples, and bore no relationship to the AI values determined by GLC. It is evident that, at the observed AI levels the foliar dye concentrations are 100 times lower, ranging from ~ 0.05 to 0.1 ppm. These values are far below the minimum quantification limit (10 ppm) of the method as evident from the spiking studies.

B2) Deposits on ground sampling units: Comparative evaluation of GLC, spectrofluorometry, colorimetry and droplet stain counting techniques

Results of this aspect of the study are presented in Tables 6 and 7 in columns C, D, E and F. Table 6, column D expresses ground deposits in g AI/ha as measured by spectrofluorometry of the eluants of glass plates.

Data in Tables 6 and 7 were subjected to analysis of variance. No significant difference was observed among values obtained from different techniques, although spectrofluorometry in Table 6 yielded values slightly lower than those of GLC or colorimetry. The droplet stain counting technique yielded the lowest values in both tables. This can be attributed to errors in the experimental techniques (in droplet sizing and spread factor measurements), and to errors arising from categorization of droplets by sizerange and to using the mean of the range for spray volume and mass deposit calculations.

Correlation coefficients were obtained between GLC, the most accurate method, spectrofluorometry (r = 0.957 in

Table 6 for C vs. D), and colorimetry (C vs. E = 0.982 in Table 6 and 0.869 in Table 7). Correlation was found to be quite good, considering the variations in sampling and analytical procedures. This illustrates the suitability of both spectrofluorometry and colorimetry for quantifying deposits on ground sampling units. In addition, both of these methods were cheaper and less time-consuming than GLC.

Correlation coefficients between droplet stain counting (the least accurate technique) and other techniques indicate a good correlation, but however, due to the consistently lower mean values in both tables, this technique was considered unsuitable for deposit assessment procedures in spray operations. Instead, Kromekote® cards should be used only for assessing droplet number per unit area and droplet size spectrum.

cent pigments or by spectrofluorometry of extracts, were reported in literature. Staniland (1959) traced fluorescent dyes on foliage following high volume applications (100 gal./acre). Joyce and Beaumont (1978) sprayed pine trees with a fenitrothion formulation containing 2.5% (w/v) of a fluorescent pigment Rocket Red 20 under the conditions of ULV application, and failed to detect fluorescent markings on pine needles; the authors assumed that the pigment particles were absorbed into the leaf tissue. Maksymiuk and Orchard (1975) recovered and quantified Brilliant Sulpho-

flavine and Rhodamine B Extra S from oak leaf deposits following a low-volume application, at the rate of 2 gal/acre and at the foliar recovery level of 10% of the applied amount (or ~ 2 µg of dye per 100 cm² area of oak leaf). However these levels are higher than the calculated values of dye concentrations (Table 6) deposited on spruce foliage in the present study. Additional factors might also have played a role contributing to the lack of detection of fluorescent dye on foliage. Due to the large time interval between foliar sampling and fluorescence analysis (this is inevitable in a large scale field study), foliar absorption and penetration might have occurred contributing to low % recovery. Metabolic and degradative changes might also have occurred contributing to low foliar concentration of the intact dye at the time of measurement.

CONCLUSIONS

In conclusion, the present study indicates the following:

- i) GLC is the only accurate and sensitive technique available to date for measuring aerially-sprayed ULV pesticide deposits on conifer foliage.
- ii) Spectrofluorometry is relatively insensitive and is therefore unsuitable for quantifying fluorescent tracer dyes from foliar deposits in ULV aerial spray operation, where the foliar concentrations of deposits are usually low.

- iii) Both spectrofluorometry and spectrophotometry were sufficiently accurate for purposes of deposit assessment on ground sampling units used in the study.
 - iv) Droplet stain counting appears to be the least sensitive and least accurate method of deposit assessment, although significant difference was not observed between it and the other techniques studied.

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Table 1. Recovery of Rhodamine B from 100 spruce needles spiked at various levels

Amount spiked (µ1)	Dye concn. (ppm)	% recovery (mean* ± SD)		
1	1.8	55.5 ± 6.35		
5.5	10	77.9 ± 1.30		
11	20	88.2 ± 0.35		
22	40	91.8 ± 0.50		

^{*} Mean of three replicates

Table 2. Recovery of Rhodamine B from 100 spruce needles following spiking with 11 or 22 µl of formulation F1 at various intervals

Time (hr)	% recovery (mean* ± SD) (11 μ1 or 20 ppm)	% recovery (mean* ± SD) (22 µl or 40 ppm)
0.5	103 ± 2.0	110 ± 2.00
1	97 ± 5.0	107 ± 2.50
2	104 ± 6.0	103 ± 0.60
3	102 ± 2.5	104 ± 6.10
5	99 ± 1.4	106 ± 0.50
6	98 ± 2.8	99 ± 0.75

^{*} Mean of three replicates; values were corrected for % recovery using values from Table 1

Table 3. Recovery of Fenitrothion at various intervals from needle surface (100 needles) treated with 11 or 22 $\mu \dot{\textbf{l}}$ of formulation F1

Time (hr)	% recovery (mean* ± SD) (11 μ1 or 1700 ppm)	% recovery (mean* ± SD) (22 μ1 or 3400 ppm)
0.5	98 ± 0.8	97 ± 1.6
1	94 ± 0.5	95 ± 0.8
2	93 ± 1.0	93 + 1.0
3 .	90 ± 1.5	91 ± 1.2
5	88 ± 0.5	88 ± 1.3
6	87 ± 1.0	86 ± 1.0

^{*} Mean of three replicates

Table 4. Droplet density a and size spectrum of sprays collected on Kromekote cards at ground level (emulsion formulation F1)

Stain size ^d range (µm)	Spread factor	Drop size range (µm)	Mean drop diameter (μm)	Frequency percent ^b	Volume percent
1 - 75	1.62	1 - 50	36	27.4	1.4
76 - 150	1.65	51 - 100	75	33.2	14.8
151 - 250	1.68	101 - 150	125	31.0	63.2
251 - 350	1.72	151 - 200	175	8.4	20.7
351 - 450	1.76	201 - 250	0	0	0

a Droplet density = 16 droplets/cm²

The error in these data is up to 15%

 $b_{\text{NMD}} = 60 \, \mu \text{m}$

 $c_{\text{VMD}} = 105 \, \mu \text{m}$

Stain sizes are values corrected for in-flight droplet evaporation (a limiting value of $\sim 15\%$ of the original droplet volume)

1

Table 5. Droplet density a and size spectrum of sprays collected on Kromekote cards at ground level (oil-based formulation F2)

Stain size ^d range (µm)	Spread factor	Drop size range (μm)	Mean drop diameter (μm)	Frequency percent	Volume percent
100 - 175	3.63	1 - 50	35	38.5	· 2
176 - 250	3.70	51 - 75	60	38.5	10
251 - 350	3.81	76 - 100	85	19.0	28
351 - 450	3.90	101 - 125	110	4.0	60
451 - 550	4.03	126 - 150	130	0.0	0

^a Droplet density = 18 droplets/cm^2

The error in these data is up to 15%

 $b_{\text{NMD}} = 45 \, \mu \text{m}$

 $^{^{}c}$ VMD = 90 μ m

d Stain sizes are values corrected for in-flight droplet evaporation (a limiting value of $\sim 52\%$ of the original droplet volume)

Table 6. Field Study. Plot P1: Spray deposit on crown foliage and at ground level in adjacent openings after an aerial application of an emulsion formulation of fenitrothion at 0.21 g AI in 1.46 L/ha

Sampling tree No.	Fo11	Foliage concn (ppm)		ound deposit in	Correlation between		
	GLC ^a	Spectro- b fluorometry	GLC [©]	Spectro-d fluorometry (D)	Colorimetry ^e (E)	Spot counting f	deposits in columns C to F (r = corr. coef.
P1-1	4.20	0.80 (0.04) ^h	291	220	313	207	
P1-2	4.74	0.70 (0.05)	212	180	217	159	C vs D: 0.957
P1-3	5.90	ND ^g (0.06)	72.1	101	77.0	103	C vs E: 0.982
P1-4	7.08	0.15 (0.07)	62.3	45.7	51.1	35.3	C vs F: 0.897
P1-5	5.63	0.20 (0.06)	199	200	174	109	D vs E: 0.929
P1-6	4.84	ND (0.05)	169	126	153	101	D vs F: 0.893
P1-7	8.98	1.20 (0.09)	182	145	204	124	E vs F: 0.944
P1-8	3.64	0.65 (0.04)	120	107	154	141	
P1-9	8.78	ND (0.09)	335	229	358	201	
P1-10	3.80	0.66 (0.04)	69.3	74.8	65.4	66.5	
P1-11	3.04	0.32 (0.03)	24.4	31.2	38.5	41.3	
P1-12	5.39	ND (0.05)	162	122	140	95	
Mean	5.50	0.39 (0.04)	158	132	162	115	
± sp	± 1.92	± 0.40 (± 0.04)	± 94.4	± 65.3	± 100	± 55.2	

a Data from "active ingredient" on foliage

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b Data from "spectrofluorometry" for Rhodamine B on foliage

^c Data from "active ingredient" on glass plates

d Data from "spectrofluorometry" for Rhodamine B on glass plates

Bata from "spectro photometry" for Rhodamine B on glass plates

f ,Data from "droplets/cm2" on Kromekote cards after correcting for droplet evaporation

g ND--None detected

h Values in parenthesis represent theoretical values of dye concentration calculated using the GLC values

Table 7. Field study. Plot P2. Spray deposit on crown foliage and at ground level in adjacent openings after an aerial application of an oil-based formulation of fenitrothion at 0.28 kg AI in 0.44 L/ha

Sampling tree No.	Foliage concn (ppm)	Ground	d deposit in ad (g AI/ha)	Correlation between	
	GLC	GLC ^a (C)	Colorimetry ^b (E)	Spot counting (F)	deposits in columns C to F (r = corr. coef.)
P2-1	4.32	65.1	46.2	43.3	C vs E: 0.869
P 2 – 2	2.49	146	155	144	C vs F: 0.869
P2-3	7.08	40.6	32.3	31.2	E vs F: 0.996
P2-4	2.87	244	254	240	
P 2 – 5	4.58	75.6	141	126	
P2-6	2.29	230	188	187	
P2-7	3.76	221	155	145	
P2-8	4.08	258	169	148	
P2-9	8.52	60.9	48.3	27.3	
P2-10	2.55	38.5	27.3	25.4	
P2-11	8.03	99.4	41.3	32.9	
P2-12	9.82	240	281	267	
P2-13	4.02	83.3	32.2	31.9	
P2-14	7.79	134	160	153	
P2-15	3.09	202	238	210	
Mean	5.02	143	131	121	
± sp	± 2.52	± 82.1	± 87.9	± 83.8	

a Data from "active ingredient" on glass plates

b Data from "Automate B red" dye on glass plates

^c Data from "droplets/cm²" on Kromekote cards after correcting for droplet evaporation