

## Studies on potato sprout suppressants. 5. The effect of chlorpropham contamination on the performance of seed potatoes

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Accepted for publication: 20 November 1981

*Zusammenfassung, Résumé p. 55*

*Additional keywords:* isopropyl N-(3-chlorophenyl) carbamate, CIPC, isopropyl m-chlorocarbanilate

### Summary

Potatoes of cv. Désirée and Pentland Crown, treated in February at 0, 0.6, 1.2, 2.5, 5, 10, and 20 mg kg<sup>-1</sup> chlorpropham adsorbed on alumina, were sampled for analysis immediately before planting at the beginning of May. Chlorpropham was extracted using hexane as the solvent in the presence of anhydrous sodium sulphate. Emergence and yield data showed that low levels of chlorpropham (<0.3 mg kg<sup>-1</sup>) that had little effect on total emergence, increased the mean emergence time and reduced yield. High levels of chlorpropham drastically reduced total emergence and yield.

### Introduction

Chlorpropham (isopropyl N-(3-chlorophenyl) carbamate) is the most widely used potato sprout suppressant. Its high activity and volatility enables excellent sprout control to be maintained for several months by a single application as a thermal fog at a rate of 10–30 mg kg<sup>-1</sup>. It is never deliberately used on seed potatoes because it is a mitotic poison that can permanently impair subsequent field performance (Reeve et al., 1963; van Vliet & Sparenberg, 1970; Kim et al., 1972).

Accidental contamination of seed does occur, often resulting in crop failure but the origins of such contamination are not always evident (Anon., 1974, 1975). Because there is little detailed information about the effects of contamination on field performance, the current investigation was made to determine the levels of residues which significantly affect emergence, yield and size distribution of the subsequent crop.

This work demands an accurate, sensitive and highly specific method of determining chlorpropham residues. Methods with infrared spectroscopy (Nultsch, 1959; Ferguson et al., 1963) are too insensitive. None of the colorimetric methods (Bissinger & Fredenburg, 1951; Gard & Rudd, 1953; Merz & Kammerer, 1958; Gard, 1959; Gard et al., 1959; Kroeller, 1962; Gard & Ferguson, 1963; Gard & Ferguson, 1964; Koivistoinen & Karinpaa, 1965; Ferguson & Gard, 1969; Ercegovich & Witkonten, 1972) satisfy the

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above requirements. They involve hydrolysis followed by determination of the resultant 3-chloroaniline molecule and are liable to interference from other aniline-based herbicides often used on potatoes such as monolinuron. Of several gas chromatographic methods (Gutenmann & Lisk, 1964; Vogel & Deskusses, 1965; Romagnoli & Bailey, 1966; van Vliet & Hertog, 1966; Cerny & Blumenthal, 1972; Corsini et al., 1978), that of Cerny & Blumenthal was found to be the most satisfactory although modifications were needed to produce samples in which low levels of chlorpropham could be determined using non-specific gas chromatographic detectors.

## Materials and methods

### *Experimental treatments*

Certified seed was used for each of the two cultivars, Désirée and Pentland Crown. Batches, weighing 7.5 kg, were dusted with chlorpropham, adsorbed on alumina, to give treatment rates of 0 (control), 0.6, 1.2, 2.4, 5, 10, and 20 mg kg<sup>-1</sup>. There were duplicate batches for each rate and for each cultivar and the treated tubers were stored at 10 °C, in boxes with closely fitting lids, from the treatment date on 21 February 1977 until the controls had commenced sprouting. From this time (6 April 1977) they were stored under illuminated conditions to maintain sprouts at a manageable level until planting on 3 May 1977.

Immediately prior to planting, 1 kg samples were removed from each batch, washed and stored at -18 °C until required for analysis.

The site was planted in two independently randomised blocks. The plots were 3 drills wide (drill width 750 mm) by 6 m long, the tubers being planted at 330 mm spacing.

Emergence was scored at two-day intervals from 28 May until 27 July 1977. The crop was harvested on 5 October, graded and the yield of each drill within the plots recorded.

### *Extraction of chlorpropham from potatoes*

The washed 1 kg samples were removed from storage and, after thawing for 30 minutes, comminuted using a powerful mincer (Model AL 2-1, Bauknecht, W. Germany). The minced material was stirred vigorously to ensure its homogeneity and a 50 g sub-sample was extracted with anhydrous sodium sulphate - hexane (glass distilled - Rathburn Chem. Co., Scotland) (Cerny & Blumenthal, 1972). The extract was evaporated to ca. 1 cm<sup>3</sup> on a rotary evaporator keeping the temperature below 40 °C to prevent chlorpropham loss.

### *Clean up*

A slurry of alumina (neutral - activity grade V, Woelm) in hexane was poured into a column, 300 × 9 mm, to a depth of 150 mm. A 10 mm plug of anhydrous sodium sulphate was added to the top of the column to maintain anhydrous conditions and the column equilibrated by running 20 cm<sup>3</sup> of hexane through it at a rate of 0.75 cm<sup>3</sup> min<sup>-1</sup>. The concentrated extract and washings were applied and eluted through the column with hexane at a maximum flow rate of 1.0 cm<sup>3</sup> min<sup>-1</sup>. The first 80 cm<sup>3</sup> of eluate were discarded, the next 120 cm<sup>3</sup> collected, concentrated to a small volume and made up to 2 cm<sup>3</sup>.

#### *Chlorpropham determination*

Samples of 0.5–5  $\mu\text{l}$  ( $\text{mm}^3$ ) of the purified extract were injected into a Pye Series 104 gas chromatograph equipped with flame ionisation detectors (FID). 2 m  $\times$  4 mm i.d. columns of 5% OV 17 on 100/120 mesh Gas Chrom Q, held at a temperature of 195 °C, were used. The injection ports were maintained at 220 °C and the detector temperature was 275 °C. The nitrogen flow rate was 40  $\text{cm}^3 \text{min}^{-1}$ . The concentration of chlorpropham was determined by comparison with standard solutions. Triangulation was used to measure peak areas.

#### *Recovery*

1 mg amounts of chlorpropham were applied to 5 separate batches of diced potatoes prior to blending and, by the procedure described above, recovery was found to be 91.2% s.d.  $\pm$  4.9. All results were corrected for this factor.

### **Results and discussion**

#### *Analytical method*

Prior to the use of the dry hexane extraction technique, other solvents were considered including ethyl acetate, dichloromethane and chloroform, but due to the formation of stable emulsions during blending they were abandoned. Van Vliet & Hertog (1966) encountered a similar problem when using dichloromethane. Extraction with methanol or ethanol followed by partitioning into hexane was also examined but the distribution of chlorpropham between the aqueous and organic phases resulted in poor recovery rates. The dry extraction method of Cerny & Blumenthal (1972) overcomes these problems and has the further advantage of using hexane as the solvent resulting in cleaner extracts and allowing the use of an electron capture detector fitted to a gas chromatograph.

However, the column clean-up described by Cerny & Blumenthal (1972), although suitable for a nitrogen specific detector, did not provide clean chromatograms with FID. Other types and grades of alumina were examined and it was found that decreasing the activity of the alumina rather than increasing the polarity of the eluent lead to cleaner chlorpropham extracts. The use of neutral Woelm alumina of activity grade V resulted in clean chromatograms, allowing the determination of low levels of chlorpropham by flame ionisation.

#### *Chlorpropham residues*

The experimentally determined chlorpropham residue levels associated with the various chlorpropham treatments are shown in Table 1, together with emergence and yield data of the resultant crop.

Analysis of variance of the residue values revealed significant ( $P < 0.01$ ) cultivar, treatment and interactive terms. Generally, the cv. Désirée had higher chlorpropham residues than Pentland Crown. Similar cultivar differences have been reported by Dalziel et al. (1980) with the sprout suppressant tecnazene.

As expected, treatment with chlorpropham produced a highly significant ( $P < 0.01$ ) effect on mean emergence time and total emergence. The effects of chlorpropham on the cv. Pentland Crown were more severe despite it having lower residue levels. In similar

Table 1. The effect of chlorpropham contamination on the performance of seed potatoes.

| Cultivar <sup>1</sup> | Chlorpropham applied <sup>2</sup><br>(mg kg <sup>-1</sup> ) | Residue at planting <sup>3</sup><br>(mg kg <sup>-1</sup> ) | Mean emergence time (days) <sup>4</sup> | Total emergence <sup>5</sup><br>(%) | Yield per drill            |                             |               |
|-----------------------|---|--|---|-------------------------------------|----------------------------|-----------------------------|---------------|
|                       |   |  |   |                                     | large <sup>b</sup><br>(kg) | medium <sup>c</sup><br>(kg) | total<br>(kg) |
| Désirée               | 0.0   | 0.07   | 29.7                                    | 100.0                               | 16.8                       | 3.2                         | 20.1          |
|                       | 0.6   | 0.06   | 30.1                                    | 100.0                               | 18.1                       | 2.7                         | 20.8          |
|                       | 1.2   | 0.22   | 36.4                                    | 100.0                               | 15.2                       | 2.2                         | 17.5          |
|                       | 2.5   | 0.46   | 44.2                                    | 92.6                                | 12.5                       | 2.1                         | 14.6          |
|                       | 5.0   | 0.84   | 54.6                                    | 83.3                                | 9.2                        | 1.7                         | 10.9          |
|                       | 10.0  | 1.55   | 61.4                                    | 60.2                                | 4.8                        | 1.1                         | 5.9           |
|                       | 20.0  | 2.60   | 64.9                                    | 41.7                                | 3.9                        | 1.3                         | 5.2           |
| Pentland Crown        | 0.0   | 0.10   | 30.6                                    | 100.0                               | 21.5                       | 1.6                         | 23.1          |
|                       | 0.6   | 0.13   | 31.5                                    | 99.1                                | 21.2                       | 1.9                         | 23.2          |
|                       | 1.2   | 0.21   | 39.1                                    | 94.4                                | 18.2                       | 1.4                         | 19.7          |
|                       | 2.5   | 0.28   | 43.6                                    | 95.4                                | 17.3                       | 1.5                         | 18.8          |
|                       | 5.0   | 0.52   | 51.1                                    | 73.2                                | 13.1                       | 2.2                         | 15.3          |
|                       | 10.0  | 1.15   | 62.7                                    | 51.9                                | 7.3                        | 1.5                         | 8.8           |
|                       | 20.0  | 1.77   | 68.2                                    | 24.1                                | 2.1                        | 0.6                         | 2.7           |
| LSD <sup>d</sup>      | 0.05  | 0.36   | 4.8                                     | 12.0                                | 3.5                        | 0.7                         | 3.3           |

<sup>a</sup> Total emergence: % of plants emerged by the final emergence count 75 days after planting – *Gesamtauflauf: Pflanzen in %, die bei der endgültigen Zählung des Auflaufs 75 Tage nach dem Pflanzen aufgelaufen waren* – *Levée totale: % de plantes levées à la levée finale prise 75 jours après la plantation.*

<sup>b</sup> >50 mm.

<sup>c</sup> 32–50 mm.

<sup>d</sup> LSD: Tukey multiple comparison procedure which allows simultaneous comparison between all pairs of treatments – *Grenzdifferenz: Methode des multiplen Vergleichs nach Tukey, die den gleichzeitigen Vergleich zwischen allen Behandlungen erlaubt* – *LSD: méthode de comparaison multiple de Tukey qui permet la comparaison simultanée de traitements à répétitions.*

<sup>1</sup> *Sorte – Variété*; <sup>2</sup> *Aufwandmenge von Chlorpropham – Dose appliquée de chlorprophame*; <sup>3</sup> *Rückstände bei der Pflanzung – Résidus à la plantation*; <sup>4</sup> *Mittlere Auflaufzeit (Tage) – Durée de levée moyenne (jours)*; <sup>5</sup> *Gesamtauflauf – Levée totale*; <sup>6</sup> *Ertrag pro Reihe – Rendement par calibre*; <sup>7</sup> *Gross-Gros*; <sup>8</sup> *Mittel – Moyen*; <sup>9</sup> *Gesamt – Total*; <sup>10</sup> *Grenzdifferenz – pps*

Tabelle 1. Einfluss einer Kontamination mit Chlorpropham auf die Leistung von Pflanzkartoffeln.  
Tableau 1. Effet de la contamination de chlorprophame sur la potentialité des pomme de terre de semence.

experiments Dalziel et al. (1980) has shown that, in comparison with the emergence of other cultivars, Pentland Crown is affected to a greater extent by residues of tecnazene.

Analysis of variance also revealed significant treatment, cultivar and interactive effects on the yields of large and of medium tubers and total yield. At higher chlorpropham levels these effects were particularly severe.

#### *Possible sources of contamination*

Agricultural Development and Advisory Service (ADAS) reports and unpublished work from our laboratory have shown that chlorpropham can be found encrusted on the blades of ventilation fans installed in, and in dust, wood, wood shavings and paint

scrapings etc. taken from buildings where chlorpropham has been used or where crops treated with chlorpropham have been stored, all obvious sources of low level contamination. Chlorpropham vapour may also drift from one section of a store used to keep ware potatoes to another section where seed is kept. The chlorpropham levels in seed resulting from such exposures should be low although its effects could be noticeable in the field.

High levels of contamination could be produced by granular chlorpropham formulations being mistakenly applied to seed potatoes, resulting in residues of ca. 3 mg kg<sup>-1</sup> in the whole tuber, sufficient to drastically reduce total emergence and yield.

### Acknowledgements

This work was supported by a grant from United Biscuits (Agriculture) Ltd.

### Zusammenfassung

*Untersuchungen über Keimhemmungsmittel. 5. Der Einfluss einer Kontamination mit Chlorpropham auf Leistung von Pflanzkartoffeln*

Chlorpropham (Isopropyl-N-(3-chlorophenyl) carbamat) ist ein weitverbreitetes Keimhemmungsmittel, das niemals bei Pflanzkartoffeln angewendet wird, da es ein die Zellteilung hemmendes Gift ist. Zufällige Kontamination kann jedoch erfolgen und es war daher der Sinn dieses Versuches, die Mengen an Chlorpropham zu bestimmen, die nachweislich den Auflauf, den Ertrag und die Grössenverteilung der nachfolgenden Ernte beeinflussen.

Zwei Proben von je 7,5 kg der Sorten Désirée und Pentland Crown wurden mit Chlorpropham in den Aufwandmengen von 0, 0,6, 1,2, 2,5, 5, 10 und 20 mg/kg bestäubt. Nach der Lagerung bei 10 °C in fest verschliessbaren Behältern wurden sie zwei Wochen vor dem Pflanzdatum vorgekeimt und 1 kg für die Rückstandsanalysen abgenommen. Der Rest wurde gepflanzt und Auflauf und Ertrag bestimmt.

Die Extraktion von Chlorpropham erfolgte

nach der Methode von Cerny & Blumenthal (1972) und es wurde eine neue Reinigungsmethode entwickelt, die den Einsatz eines Gaschromatographen mit einem Flammenionisationsdetektor erlaubt. Tabelle 1 zeigt die Rückstandswerte und die entsprechenden Angaben für den Feldbestand.

Der Einfluss von Chlorpropham war bei der Sorte Pentland Crown deutlicher, aber allgemein verlängerten sehr kleine Rückstandswerte (<0,3 mg/kg) bereits die durchschnittliche Auflaufzeit und verringerten leicht den Ertrag, obwohl die gesamte Auflaufzeit nahe bei 100% blieb. Höhere Werte (0,3–1 mg/kg) verzögerten den Auflauf und reduzierten den Ertrag. Rückstände über 1 mg/kg führten zu geringem Auflauf und einer drastischen Ertragseinbusse.

Lagerhäuser in denen Chlorpropham angewendet wird oder in denen behandelte Knollen gelagert werden sind offensichtlich Quellen der Kontamination mit geringen Mengen.

### Résumé

*Etude d'inhibiteurs de germination de la pomme de terre. 5. L'effet d'une contamination de chlorprophame sur la potentialité de pomme de terre de semence*

Le chlorprophame (isopropyl-N-(3 chlorophenyl) carbamate) est un inhibiteur de germination très employé mais jamais sur pomme de terre de semence parce que c'est un poison mi-

totique. Une contamination accidentelle peut avoir lieu et l'objet de cette expérimentation était de déterminer les taux de chlorprophame qui affectent de façon significative la germina-

tion, le rendement et la distribution par calibre de la culture suivante.

Une expérimentation à 2 répétitions a été faite avec du chlorprophame par poudrage à différentes concentrations: 0, 0.6, 1.2, 2.5, 5, 10, et 20 mg/kg sur des échantillons de 7.5 kg pour les variétés Désirée et Pentland Crown. Ces échantillons ont été conservés à 10 °C dans des boîtes fermées hermétiquement et prégermés pendant 2 semaines avant la date de plantation tandis qu'un échantillon de 1 kg a été prélevé pour une analyse de résidus. Les tubercules restants ont été plantés et les notations de levée et de rendement ont été effectuées.

La méthode de Cerny & Blumenthal (1972) a été employée pour extraire le chlorprophame et une nouvelle technique de lavage a été imaginé afin d'obtenir des échantillons adaptés à la chromatographie en phase gazeuse utilisant la

détection thermoionique. Les teneurs en résidus et les notations au champ correspondantes sont indiquées dans le tableau I.

L'effet du chlorprophame était plus accentué pour les variétés Pentland Crown mais, en général, de faibles taux en résidus (<0.3 mg/kg) retardaient la durée de levée moyenne et réduisaient légèrement les rendements quoique la levée totale approchait 100 %. Les taux de résidus plus élevés (0.3 – 1.0 mg/kg) retardaient également la levée et diminuaient les rendements. Les résidus supérieurs à 1.0 mg/kg provoquaient une mauvaise levée et des réductions sérieuses de rendement.

Les locaux utilisés pour l'application du chlorprophame ou pour la conservation de tubercules traités sont indiscutablement des sources faibles de contamination.

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