# CHANGES IN APPLICATION SYSTEM – INFLUENCE ON HERBICIDES RESIDUE IN SOIL AND SUGAR BEET ROOTS

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**Abstract:** The aim of performed investigation was to evaluate the influence of changes in herbicide application system on herbicide residues in soil and sugar beet roots. Chemical weed control in sugar beet was carried out by herbicides that included substances such as phenmedipham, desmedipham, ethofumesate, metamitron, triflusulfuron and surfactant adjuvant applied in three different systems: two times application at bare soil (preemergence) and postemergence application (weeds in phase of 2–4 leaves) – system "A", 3 times split, postemergence application (full dose of herbicide mixture) – system "B" and 4 times application at 7 to 10 day intervals starting at the beginning of weed emergence – system "C". Samples of soil and roots of sugar beet were taken at the day of lifting. Herbicide residues were analysed using HPLC with UV-detection. At lifting time, in soil samples, where herbicides were applied in system "A", the residues of metamitron amounted from 0.0097 to 0.0132 mg/kg. Sum of all detected residues of applied substances amounted 0.0341–0.0458 mg/kg. In sugar beet root samples, the residues amounted to respectively, 0.0049–0.0064 and 0.0136–0.0247 mg/kg. The application of herbicides in "B" and "C" systems caused a significant decrease of residues by about 50% (system "B") and 65% (system "C") on average, in comparison with results obtained for herbicide application in "A" system. Residues of active substances determined in roots of sugar beet did not exceed acceptable limits (MRLs).

**Key words**: adjuvant, application system, herbicide, micro-rates, residue, sugar beet

## INTRODUCTION

In soils, the biological activity of herbicides may be decreased by chemical or biological degradation of active ingredients. Adsorption by soil colloids, absorption by plants or leaching into lower layers of the soil profile influences also the biological activity of herbicides in the soil (Harris 1969). In plants, the biological activity of herbicides may by decreased by low retention and washing of herbicide from leaf surface by rain, dew and irrigation to the soil (Nalewaja *et al*. 1995).

In older systems used for weed control in sugar beets, herbicides were applied at a high, single dose. Usually, herbicides were applied two times – at bare soil (preemergence application) and postemergence application (weeds in phase of 2–4 leaves); (Dexter 1994; Woźnica *et al*. 2004). Herbicides are often applied at rates higher than required for weed control under ideal conditions. This is done primarily to compensate losses that occur at the target site in the plant (McMullan *et al*. 1998).

The newest system (micro-rates program) of herbicides used in sugar beets was developed and introduced to farmers by Dr Alan G. Dexter – professor from North Dakota State University, USA (Woźnica *et al.* 2004). This program is attractive from economical point of view and was accepted by most of sugar beets farmers in the USA. The micro-rate program uses low rates of herbicides in combination (phenmedipham + desmedipham + ethofumesate + triflusulfuron + clopyralid) applied 3 or more times at 5 to 7 day intervals starting at the beginning of weed emergence. Herbicides are used at rates reduced approximately by 2–3 times comparing to rates recommended in a conventional herbicide split application programs (Dexter *et al.* 1996; Dexter and Luecke 1998, 2001). Since 2003 the initial experiments with evaluation of micro-rates of herbicides in sugar beet crop were conducted in Poland (Woźnica *et al.* 2004; Domaradzki 2007).

The aim of the present investigation was to evaluate the influence of herbicide application system on herbicide residues in soil and sugar beet roots.

## MATERIALS AND METHODS

Field experiments were conducted during a three-year-period from 2006 until 2008 on arable fields localized in South-West Poland (brown soils, pH = 6.2–6.5, organic carbon content 2.14–2.30% and clay content 46–58%). The field trial was set up as a randomized complete block design with four replicates. Chemical weed control in sugar beet was carried out with herbicides containing substances such as phenmedipham, desmedipham, ethofumesate, metamitron, triflusulfuron and surfactant adjuvant (Table 1) applied in three different systems: two times application

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Table 1. Characteristics of preparations used in the experiments

| Common name of preparation | Active substance [a.s.] | IUPAC name | Content of [a.s.] |
|---|---|---|---|
| Betanal Progress 274 OF (Betanal Progress AM 180 EC) | phenmedipham | methyl 3-(3-methylcarbaniloyloxy) carbanilate | 91 g/l (60 g/l) |
| | desmedipham | 3-phenylcarbamoyloxyphenylcarbamate | 71 g/l (60 g/l) |
| | ethofumesate | (±)-2ethoxy-2,3-dihydro-3,3-dimethyl-benzofuran-5-yl methanesulfonate | 112 g/l (60 g/l) |
| Goltix 70 WP<br>Goltix 700 SC | metamitron | 4-amino-4,5-dihydro-3-methyl-6-phenyl-1,2,4-triazin-5-one | 70% (700 g/l) |
| Safari 50 WG | triflusulfuron | 2-[4-dimethylamino-6-(2,2,2-trifluoroethoxy)-1,3,5-triazin-2-ylcarbamoylsulfamoyl]-m-toluic acid | 50% |
| Break Thru S-240 | polymethylsiloxane copolymer | – | 240 g/l |

Table 2. Herbicide systems application

| Objects | Date of treatment | Herbicide dose per ha | Sum of a.s. [g/ha] | System |
|---|---|---|---|---|
| Goltix 70 WP<br>+ Betanal 180 EC | T-0<br>T-3 | 1 x 5.0 kg<br>1 x 5.0 l | 3500<br>900 | system "A" (preemergence application + postemergence application) |
| Betanal 274 OF<br>+ Goltix 700 SC<br>+ Safari 50 WG<br>+ Break Thru S-240 | T-1, 2, 3<br>T-1, 2, 3<br>T-1, 2, 3<br>T-1, 2, 3 | 3 x 1.0 l<br>3 x 1.0 l<br>3 x 30 g<br>3 x 0.25l | 822<br>2100<br>45<br>– | system "B" split postemergence application (3 times application) |
| Betanal 274 OF<br>+ Goltix 700 SC<br>+ Safari 50 WG<br>+ Break Thru S-240 | T-1, 2, 3, 4<br>T-1, 2, 3, 4<br>T-1, 2, 3, 4<br>T-1, 2, 3, 4 | 4 x 0.5 l<br>4 x 0.5 l<br>4 x 15 g<br>4 x 0.25 l | 548<br>1400<br>30<br>– | system "C" split postemergence application (reduced doses - 4 times application) |

T-0 preemergence application
T-1 weeds in cotyledonos phase
T-2 7–10 days after T-1 date
T-3 7–10 days after T-2 date (for ob. 1 – weeds in phase 2–4 of leaves)
T-4 7–10 days after T-3 date

Table 3. Recoveries and quantification limits of the analytical method

| Tested substance | Average recoveries [%] | | Limit of detection* [mg/kg] | |
|---|---|---|---|---|
| | soil | roots | soil | roots |
| Phenmedipham | 93 | 88 | 0.0001 | 0.0001 |
| Desmedipham | 92 | 85 | 0.0001 | 0.0001 |
| Ethofumesate | 86 | 78 | 0.0001 | 0.0001 |
| Metamitron | 83 | 85 | 0.0005 | 0.0005 |

* for 30 g of sample

– at bare soil (preemergence) and postemergence application (weeds in phase of 2–4 leaves) – system "A", 3 times split, postemergence application (full dose of herbicide mixture) – system "B" and 4 times application at 7 to 10 day intervals starting at the beginning of weed emergence – system "C" (Table 2).

Samples of soil and roots of sugar beet were taken at the day of lifting from the middle of each plot to avoid interference and side effects from the neighbouring plots. The soil samples were taken at a soil depth of 0–20 cm. Samples from each plot were well mixed and stored in polyethylene bags at minus 19°C until sample extraction. Soil moisture content was determined for each soil sample. The samples were dried at 105°C for 24 h.

Phenmedipham, desmedipham, ethofumesate and metamitron residues were analysed using high performance liquid chromatography (SHIMADZU HPLC measuring set: pump LC-10AT, degasser DGU-4A) with UV-detection (SPD-10A). Because of low dose use and lack of analytical method the triflusulfuron residues were not determined. The recoveries of the active substances were determined by fortification of soil and root samples at concentrations of 0.0005, 0.001, 0.01 and 0.1 mg/kg in three replicates. The average recoveries and quantification limits of the methods for all concentrations are given in table 3. Analytical procedures were performed at the Institute in Laboratory of Residue Research (Kucharski and Sadowski 2001; Kucharski 2007). All experimental data were calculated using the statistical program Statgraphics Centurion, version XV.

## RESULTS

In soil samples, where herbicides in old system „A" were applied, residues of metamitron at the lifting time amounted from 0.0097 to 0.0132 mg/kg. Sum of all detected residues of applied substances (phenmedipham, desmedipham, ethofumesate and metamitron) amounted 0.0341–0.0458 mg/kg. The application of herbicide in system "B" caused the decrease of residues about 55% for metamitron and about 35% for sum of all substances. The decrease of residues level was statistically significant. Residues detected in samples of system "C" (micro-rates program) were on average 60% lower in comparison with results for samples of old system "A". Residues of metamitron amounted from 0.0034 to 0.0043 mg/kg. Sum of all detected residues of applied substances amounted 0.0158–0.0221 mg/kg.

In sugar beet roots samples, residues of active substances were lower than in soil. For samples where herbicides in "A" system were applied, residues of metamitron amounted from 0.0049 to 0.0064 mg/kg. Sum of all detected residues of applied substances amounted 0.0136–0.0247 mg/kg. The application of herbicide in system "B" caused the decrease of residues about 50% for metamitron and for sum of all substances. The decrease of residues level was statistically significant. Residues detected in samples of system "C" (micro-rates program) were on average 70% lower in comparison with results for samples of old system "A". Residues of metamitron amounted from 0.0008 to 0.0010 mg/kg. Sum of all detected residues of applied substances amounted 0.0035–0.0095 mg/kg.

Results obtained from all experiments are shown in table 4 and 5.

Table 4. Residues of active substances in soil

| System | Residues* [mg/kg] | | |
|---|---|---|---|
| | metamitron | phenmedipham + desmedipham + ethofumesate | Sum of residues |
| 2006 | | | |
| "A" | 0.0132 | 0.0326 | 0.0458 |
| "B" | 0.0058 | 0.0244 | 0.0302 |
| "C" | 0.0043 | 0.0178 | 0.0221 |
| LSD (0.05) | 0.00106 | 0.00637 | 0.01246 |
| 2007 | | | |
| "A" | 0.0118 | 0.0278 | 0.0396 |
| "B" | 0.0054 | 0.0207 | 0.0261 |
| "C" | 0.0038 | 0.0145 | 0.0183 |
| LSD (0.05) | 0.00114 | 0.00522 | 0.00682 |
| 2008 | | | |
| "A" | 0.0097 | 0.0244 | 0.0341 |
| "B" | 0.0049 | 0.0195 | 0.0244 |
| "C" | 0.0032 | 0.0126 | 0.0158 |
| LSD (0.05) | 0.00148 | 0.00617 | 0.00884 |

* average residues for 4 replications
A, B, C – herbicide systems of application (see Table 2)

Table 5. Residues of active substances in sugar beet roots

| System | Residues* [mg/kg] | | |
|---|---|---|---|
| | metamitron | phenmedipham + desmedipham + ethofumesate | sum of residues |
| 2006 | | | |
| "A" | 0.0064 | 0.0183 | 0.0247 |
| "B" | 0.0032 | 0.0109 | 0.0125 |
| "C" | 0.0010 | 0.0085 | 0.0095 |
| LSD (0.05) | 0.00146 | 0.00487 | 0.00937 |
| 2007 | | | |
| "A" | 0.0056 | 0.0097 | 0.0153 |
| "B" | 0.0022 | 0.0056 | 0.0068 |
| "C" | 0.0008 | 0.0039 | 0.0047 |
| LSD (0.05) | 0.00093 | 0.00294 | 0.00574 |
| 2008 | | | |
| "A" | 0.0049 | 0.0086 | 0.0136 |
| "B" | 0.0020 | 0.0049 | 0.0060 |
| "C" | 0.0009 | 0.0028 | 0.0035 |
| LSD (0.05) | 0.00088 | 0.00248 | 0.00581 |

Explanation as for Table 4

## DISCUSSION

To evaluate efficacy of reduced herbicide rates (micro-rates) for weed control in sugar beet field experiments were conducted in Poland since 2003. Micro-rates system gives good results when all farming activities are carried out in accordance with conventional agricultural practice and in line with recommendations for split-reduced rates application (Woźnica *et al.* 2004, 2007; Domaradzki 2007).

Prevention of nozzle plugging from herbicide precipitation in a tank by application of specific, based on fatty acids methylated esters adjuvant, plant oil derivatives and surfactants plays an important role for low herbicide rates weed control increase (Warner and Dexter 1995; Dexter and Zollinger 2001; Wilson *et al.* 2005). Properties of adjuvant increase herbicide activity through mechanisms such as droplet adhesion, retention, spreading, deposit formation, uptake and translocation (Bruce and Carey 1996; Sharma *et al.* 1996). Moreover some research indicates that adjuvants can reduce leaching of herbicide through the soil profile (Reddy 1993).

In our experiment residues of active substances determined in roots of sugar beet did not exceed maximum residue limits (Rozporządzenie 2004, 2007). The changes in herbicide application system, especially the micro-rates program used allowed to reduce a herbicide dose with no weed control efficacy loss. Thus the agricultural environment contamination risk was limited.

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Rozporządzenie Ministra Zdrowia z dnia 16 kwietnia 2004r. w sprawie najwyższych dopuszczalnych poziomów pozostałości chemicznych środków ochrony roślin, które mogą znajdować się w środkach spożywczych lub na ich powierzchni (Dz. U. Nr 85, poz. 801, z późn. zm.).

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## POLISH SUMMARY

### ZMIANY W SYSTEMIE APLIKACJI – WPŁYW NA POZOSTAŁOŚCI HERBICYDÓW W GLEBIE I KORZENIACH BURAKA CUKROWEGO

Celem badań była ocena wpływu sposobu stosowania herbicydów na ich pozostałości w glebie i korzeniach buraka cukrowego. Zabiegi chwastobójcze wykonywano herbicydami zawierającymi: fenmedifam, desmedifam, etofumesat, metamitron i triflusulfuron. Herbicydy aplikowano 3 różnymi systemami: aplikacja dwukrotna – przedwschodowo i powschodowo (chwast w fazie 2–4 liści) – system „A", zabieg dzielony, powschodowy 3-krotny (mieszanina herbicydów) – system B i aplikacja 4-krotna w odstępach 7–10 dniowych począwszy od fazy liścieni chwastów (mikrodawki herbicydów) – system C. Próby gleby i korzeni buraka pobierano w czasie zbioru rośliny uprawnej. Pozostałości herbicydów analizowano techniką HPLC z detekcją UV. W czasie zbioru rośliny uprawnej, pozostałości metamitronu w glebie dla systemu A wynosiły 0,0097–0,0132 mg/kg. Suma pozostałości wszystkich badanych substancji wynosiła 0,0341–0,0458 mg/kg. W próbach korzeni buraka pozostałości wynosiły odpowiednio 0,0049–0,0064 i 0,0136–0,0247 mg/kg. Stosowanie herbicydów w systemach „B" i „C" znacząco wpłynęło na obniżenie pozostałości, średnio o 50% (system „B") i 65% (system „C"), w porównaniu z wynikami uzyskanymi dla systemu „A". Wykrywane pozostałości herbicydów w korzeniach buraka cukrowego nie przekraczały wartości dopuszczalnych (NDP).