Ingo Meiners

Management of black-grass (*Alopecurus myosuroides* Huds.) in winter wheat and taking into account the soil activity of post-emergence herbicides

Inaugural Dissertation
submitted in fulfillment of the partial requirements for
a doctoral degree in agriculture (Dr. agr.)
to the Faculty of Agricultural and Nutritional Sciences,
and Environmental Management of the
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Management of black-grass (*Alopecurus myosuroides* Huds.*)* in winter wheat and taking into account the soil activity of post-emergence herbicides

Management von Ackerfuchsschwanz (*Alopecurus myosuroides* Huds.*)* in Winterweizen unter besonderer Berücksichtigung der Bodenwirkung von Nachauflauf-Herbiziden

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by

Ingo Meiners

aus Bad Soden am Taunus

Gießen 2014

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List of abbreviations

ACCase	Acetyl coenzyme A carboxylase,
a.i. /a.s.	Active ingredient/active substance
ALS	Acetolactate synthase
BBCH	Code scheme for plant growth stages (JKI, 2001)
clodinafop	clodinafop-propagyl
Conc	Concentration
C-org	Organic material (carbon)
CS	Capsule suspension
DAA	Days after application
D/d	Days
df	Degree of freedom
DT	Degradation time
dt/ha	Decitons per hectare (metric)
dw	Dry weight
EC	Emulsifiable Concentrate
EMR	Enhance metabolic resistance
EPSP	5-enolpyruvylshikimate-3-phosphate synthase
ES	Early sowing of winter wheat
EWRS	European Weed Research Society
EXP	Experiment
F	Full recommended field dosage
fenoxaprop-P	fenoxaprop-P-ethyl
FHS	Formulierhilfsstoff = Biopower
fig	Figure
flupyrsulfuron	flupyrsulfuron-methyl
Form	Formulation
FW	Fresh weight
g/L	Grams per litre
GH	Greenhouse (glass-house)
GI	Gießen
GB	Great Britain
GS	Growth stage
GST	Glutathione-S-transferase
GY	Grain yield
HE	Herbicidal efficacy
ha	Metric hectar
HRAC	Herbicide Resistance Action Committee
iodosulfuron	iodosulfuron-methyl-sodium
Irr	Irrigation
JKI	Julius-Kühn-Institut
Kd	Soil-water partition coefficient
Koc	Soil organic carbon-water partition coefficient
L/kg/ha	Litre/kilograms per hectare
LS	Late sowing time of winter wheat
LSD	Least significance difference
logKoW	Octanol/water partition coefficient
mesosulfuron	mesosulfuron-methyl
min	Minute

MINTIL	Minimum tillage using a chisel plough
Mio	Million
MOA	Mode of action
n	Number of results
N	Nitrogen
n.a.	Not available/assessed
OD	Dispersible oil
PBDB	Pesticide Properties Database
pl	Plants
PLOUGH	Mouldboard ploughing
ppm	Parts per million
propoxycarbazone	propoxycarbazone-sodium
PSII	Photosystem II
RH	Rauischholzhausen
red	Reduction
rpm	Revolutions per minute
s	Safener
S	Sulfur
SC	Soluble Concentrate
SG	Water soluble granules
SL	Soluble concentrate
t	Metric tons
tab	Table
TGW	Thousand grain weight (mass)
TM	Trade name
TR	Trebur
TSR	Target-site resistance
UTC	Untreated check/control
UPLC	Ultra Performance Liquid Chromatography
WG	Water-Dispersible Granule
WH	Heads of wheat
WI	Wicker
WI-STR	Wicker-Staßenmühle
WI-Ken	Wicker-Kennel
WI-JOH	Wicker-Johannespfad
WP	Wettable Powder

1 Introduction

Alopecurus myosuroides Huds. (black-grass) is one of the main graminaceous weeds in Germany and other Western European countries such as the UK and France. *A. myosuroides* is a predominant annual grass weed which is well adapted to current agricultural production systems, such as early sowing dates, non-ploughing cultivations, high nitrogen levels and crop rotations dominated by winter cereals (Knab and Hurle 1988, Amann 1991, Hurle 1993). Due to it`s well adapted growth behavior to winter cereals, *A. myosuroides* can be very competitive and consequently needs to be controlled regularly.

Due to economic and practical factors, farmers in Europe are still relying almost exclusively on herbicides for weed management in many cropping systems, such as winter cereal monocultures. However, the indiscriminate and repetitive use of similar active ingredients over the last 20 years has led to the appearance and spread of herbicide resistant biotypes in intensive production systems (Moss 1987, Gressel and Segel 1990). Today, black-grass populations which are resistant to acetyl Co-A carboxylase (ACCase) inhibitors and to acetolactate-syntase (ALS) inhibitors are increasing in middle European countries (Deyle et al. 2007, Moss and Hull 2009, Krato and Petersen 2010, Gehring et al. 2012). Herbicide resistances often result in declining profits for the farmer and higher costs for weed control measures. In addition, there is political pressure in European countries to ban and restrict the usage of certain herbicides, and also a lack of new innovative active ingredients from the industry. Because of the rapid decline in the number of effective herbicides for the control of *A. myosuroides,* it is necessary to develop alternative strategies for managing weeds by using all currently available tools.

The most promising approach is integrated weed management; a strategy which includes a combination of various indirect and direct control measures to keep the weed populations low. As a part of integrated weed management, and in addition to crop rotation, primary tillage is known to have a drastic effect on annual grass weeds (Hurle 1993, Grundy and Froud-Williams 1993). The reductive effect of ploughing cultivation compared to tine and direct drilled cultivations on infestations with black-grass has been reported in several studies (Moss 1980, Pollard et al. 1982, Chauvel et al. 2001). Also, delaying the sowing date of winter cereals can have some positive effects on reducing grass weeds. Studies with *A. myosuroides* have shown that later sowing dates can reduce seedling emergence, yield losses caused by black-grass competition, and *A. myosuroides* seed production compared to 3-4 weeks earlier sowing (Moss 1985a, Amann et al. 1992, Melander 1995). Additionally, there are many new models available which simulate the effects of non-chemical weeding strategies and farming practices, such as the cropping system, on black-grass infestations. However, these models are usually theoretical and based on estimations or random studies. Overall, there is a lack of information on the effect of integrated weed control measures, such as the sowing date, cultivation system and herbicide applications on heavy infestations with both resistant and sensitive black-grass populations.

Currently *A. myosuroides* can be controlled by either pre- or post-emergence herbicide applications. However, the most efficient application timing has not yet been defined due to varying biological, agronomic and climatic factors. In many cases, the efficacy of post-emergence herbicides is not maintained long enough to provide sufficient control. Late flushes of *A. myosuroides* occur especially in years with a high level of seed dormancy. *A. myosuroides* dormancy varies every year and is highly influenced by the weather conditions during maturation (Fenner 1991, Moss et al. 2006). It has been reported that black-grass is capable of emerging slowly over a longer period of time and is dependent on the cropping practice (Menck 1968, Kampe 1975). Further, it is reported that *A. myosuroides* is able to emerge at very low temperature regimes such as 2°C (Menck 1968) or 3°C (Colbach et al. 2002a). Colbach et al. (2002a) even estimates a minimum temperature requirement for the germination of *A. myosuroides* at 0°C. The importance of herbicides with residual activity to cover the prolonged period of weed emergence becomes obvious in years with mild winters and a high level of seed dormancy. Currently, there is a lack of information on the residual activity of widely used post-emergence herbicides to control black-grass.

Thesis objectives

The overall purpose of this research is to get a better understanding of the biology of *A. myosuroides* and to improve the management strategies (including both non-chemical and chemical measures) for the control of black-grass in the field. Lab and agricultural field experiments were conducted in order to identify and characterize the soil activity of post-emergence herbicides against black-grass. This thesis has two major topics with herein objectives:

A: Soil activity of post-emergence herbicides

- Characterization of the natural emergence of *A. myosuroides* in the field.
- Clarify the possible soil activity of post-emergence herbicides (ACCase- and ALS-inhibitors) against *A. myosuroides.*
- Evaluate the soil activity of post-emergence herbicides under different conditions and assess their ability to provide residual activity.

B: Management of black-grass

- Evaluate the efficacy of different herbicide strategies for black-grass control.
- Review the success of integrated weed management strategies (both chemical and non-chemical practices), including the influence of sowing time and cultivation systems on the infestation and development of *A. myosuroides* populations (incl. herbicide resistant) in winter wheat.
- Identify options to manage herbicide resistant populations.

2 Literature overview

2.1 Black-grass (*Alopecurus myosuroides*) as a weed

2.1.1 Biology and Importance of black-grass (*A. myosuroides*)

Since 1960, black-grass has been a common annual grass weed in autumn sown crop rotations in Atlantic European regions such as the UK, Belgium, the Netherlands, France and Germany (Kemmer and Koch 1980). In Europe, the distribution and severity of black-grass infestations are mainly determined by soil type and cropping system rather than by climate, although it prefers cool and humid conditions (Balgheim 2006, Moss 2013).

Black-grass (*Alopecurus myosuroides* Huds.), also known as slender foxtail, germinates mainly in autumn and ranks with loose silky bent grass (*Apera-spica venti*) among the most important grass weeds in winter cereal production in Germany (Hurle 1993). *A. myosuroides* is well adapted to current cultural practices such as short rotations with frequent winter cereals, early sowing dates, and reduced tillage which have contributed to the spread of black-grass (Knab and Hurle 1988, Amann 1991, Amann et al. 1992, Hurle 1993).

Biology

Black-grass generally favors water retentive soils, which is why it is mainly a problem in heavier clay and silt soils with moderate to high humus and nitrogen contents (Thursten 1972, Moss 2013). Mature plants are 20-80 cm tall with erect hairless leaves. Leaves are up to 15 cm long and leaf sheathes are smooth green, sometimes purplish around the base. Having no ears, the blunt, finely toothed, and the membranous ligule is about 2-5 mm long (Kemmer and Koch 1980, Partzsch et al. 2006). Black-grass usually flowers from May to August and the panicles (florescences, seed heads) are spike-like, from 2-12 cm long and 3-6 mm wide, tapering towards the top. The seed heads (infl are sometimes pale green, but often purplish in colour when mature (Partzsch et al. 2006). Black-grass is an annual grass-weed propagated solely by seeds. The number of tillers (seed heads) per plant varies considerably, depending mainly on crop competition and the germination date, i.e. late spring emerging plants have few or no tillers. Typical numbers in competitive winter cereal crops are 2-20 heads per plant. Black-grass has the ability to produce large numbers of seeds (ca. 40-400 seeds/plant) (JKI 2008). Each head contains 80-150 seeds, and with populations of 500 heads/m^2 'seed return can easily exceed 50,000/m^2. Black-grass is a cross-pollinating species and the viability of seed is typically 40-60% (Moss 2013).

Most black-grass seeds are shed between June and August, with the majority shed prior to the harvest of winter cereal crops (Moss 1983). Seeds have a relatively short period of primary dormancy (several weeks) which prevents them from germinating immediately (Moss 1980a, Moss 1980b, Colbach et al. 2002a), although this is reduced when seeds mature under hot/dry conditions (Moss et al. 2006).

The germination of black-grass seeds is stimulated by light, and along with moisture and warmth, the emergence of seedlings is optimized at around 10-15°C (Menck 1968, Froud-Williams et al. 1984, Moss 1985c).

Around 80% of seedling potential will emerge in the autumn (Thurston 1972, Moss 2013), from September to November, and consequently black-grass is mainly associated with autumn sown crops, especially cereals (Figure 1). Whilst this pattern is dependent on both soil and climatic conditions, in the majority of instances black-grass emerges along with or before the crop. Consequently, a delayed sowing time and spring sown crops tend to be less vulnerable to black-grass, as the majority of weed plants will have emerged before sowing and can be destroyed by seedbed cultivations or use of a total herbicide, such as glyphosate.

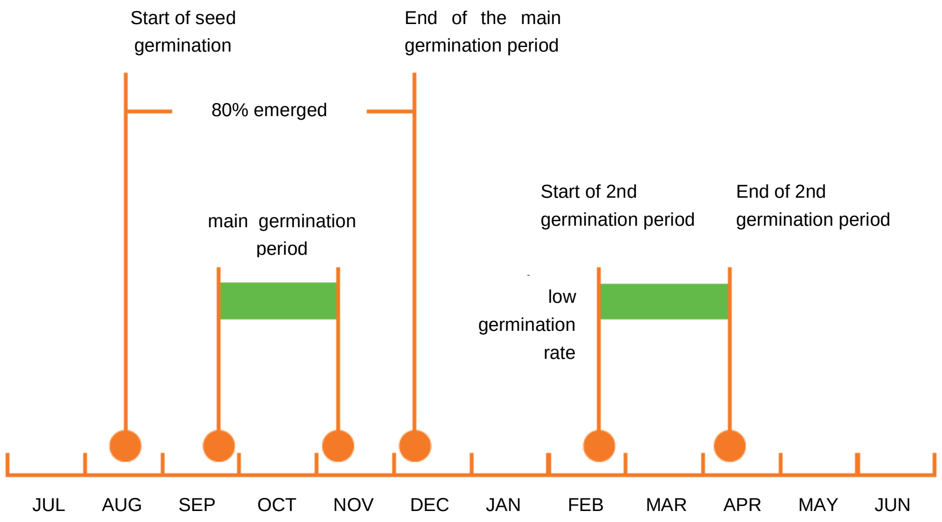

Figure 1: Emergence pattern of black-grass in Germany (Bayer Cropscience 2004)

Generally black-grass plants can only emerge successfully from seeds retained within the top 5 cm of soil, and that is the reason why minimum tillage encourages black-grass growth. Ploughing however, buries the fresh seeds deeper and prevents them from germinating during the next season, although some viable seeds may be returned to the soil surface if the land is ploughed in subsequent years (Cussans and Moss 1990).

Generally *A. myosuroides* has a low seed persistence in the soil (Jensen 2009). Survival of buried seeds in the soil is about 20-30% per year, so after 3 to 4 years burial, only about 1%-3% of seeds will still be viable (Moss 2010, Moss 2013), –

although this may still represent a considerable number of seeds. Buried seeds have the ability to remain dormant and viable in the soil, in some cases for up to 9-11 years (Menck 1968, Thursten 1972), although further cultivations may break this dormancy, resulting in the emergence of black-grass. If uncontrolled, populations can build up very rapidly given favourable conditions, by over 30 fold per annum (Moss 2013).

Importance of Black-grass

A. myosuroides can seriously reduce crop yields through competition for nutrients, especially nitrogen. The tillering capacity, and thus competitive ability of black-grass, depends strongly on the vigour of the crop. Consequently, yield losses from *A. myosuroides* in winter cereals are highly variable within years and sites (Blair et al. 1999, Kötter 1991) and are very dependent on factors like crop, crop density, sowing time and nitrogen level (Hurle 1993). Even at low densities black-grass can cause significant yield losses. Populations as low as 12 plants/m^2 have been shown to reduce winter wheat yields in England by between <5 and 15% on average, and yield losses of 4 – 8 dt wheat/ha can be expected at black-grass populations of 12 – 25 plants/m^2, but much higher losses of over 20 dt wheat/ha at densities of over 100 plants/m^2 (Moss 2013). In German field trials conducted by Kötter (1991), the yield of winter wheat can be reduced by 4-5 dt/ha per 100 heads of *A. myosuroides,* while Machefer et al. (1998) found an average reduction by 1.3 dt/ha per 100 black-grass heads.

For black-grass in winter wheat in England, a weed density of 12 plants/m^2 which causes on average 5% yield loss is often used as a threshold value at which herbicidal control is justified to prevent unacceptable yield loss. Depending on the economical value of wheat in Germany, this threshold is considered to be 5-30 plants/m^2 (Obst and Gehring 2002). Nevertheless, according to Moss (2013) control of black-grass densities of less than 1 plant/m^2 may be justified in high risk situations (minimally cultivated, early autumn sown crops on heavy soils).

2.1.2 Chemical control of black-grass

For a long period of time herbicides with only two different modes of actions were the most effective and preferred chemical solution to control *A. myosuroides*: Photosystem II- inhibitors (PSII inhibitors) such as isoproturon or chlortoluron and inhibitors of Acetyl-CoA Carboxylase (ACCase) such as fenoxaprop and clodinafop were the prioritized choice of farmers. Black-grass resistances to those herbicides have been widely confirmed first in 1982 in the UK (Moss and Cussans 1985) and shortly after that in intensive cereal grown regions in the North of Germany (HRAC 2013).

The introduction of ALS-inhibitor herbicides (inhibitors of the Acetolactate Synthase) like flupyrsulfuron in the mid 1990s (Troltzsch 1998, Drobny et al. 2012) and mesosulfuron in 2003 (Brink et al. 2002, Hacker et al. 2002), offered new chemical solutions with a different mode of action class that were highly effective against black-grass and economically attractive.

The rapid adoption of this chemistry in farming practice led to the detriment of compounds with alternative mode of actions such as prosulfocarb or pendimethalin. As a consequence, the first cases of resistance to ALS-inhibiting herbicides in Germany were reported in 2001 (Niemann et al. 2002).

The introduction of flufenacet in the 1990s (Bayer 2000, PPDB 2012) has added a new tool with an alternative mode of action which is nowadays a fixed key active ingredient in programs for the control of heavy infestations of grass weed population in autumn. Pre-emergence herbicides, such as flufenacet, pendimethalin, prosulfocarb, can give a good level of black-grass control in cereals under favoarable conditions. Their efficacy is usually not or only partially reduced by resistance (Moss and Hull 2009).

In conclusion, the use of intensive chemical treatments further led to the expansion of herbicide-resistant populations (Moss 1987, Gressel and Segel 1990), which at present can be controlled only by adapting cultural practices, together with appropriate herbicide treatments (Clarke and Moss 1991, Chauvel et al. 2001, Balgheim 2006, Wolber 2009, Wolber 2011, Gehring et al. 2012).

2.1.3 Herbicide Resistance

Due to economic and practical factors, farmers in Europe are still relying almost exclusively on herbicides for weed management in many cropping systems such as winter cereal monocultures. However, the indiscriminate and repetitive use of similar active ingredients over the last 20 years has led to the appearance and spread of herbicide resistant biotypes in intensive production systems (Moss 1987, Gressel and Segel 1990, Menne et al. 2008). Resistant populations of black-grass have been confirmed in many countries. Resistance is widespread in England and increasing in France and Germany. In a survey conducted by the European Weed Research Society`s Herbicide- Resistance Working group, black-grass was rated as the most important herbicide-resistant weed in Europe (Tatnell et al. 2007). *A. myosuroides* was considered a problem mainly in central and northern European countries, and resistant biotypes have been reported in 10 countries: Belgium, Bulgaria, Denmark, France, Germany, Netherlands, Spain, Sweden, Switzerland and England.

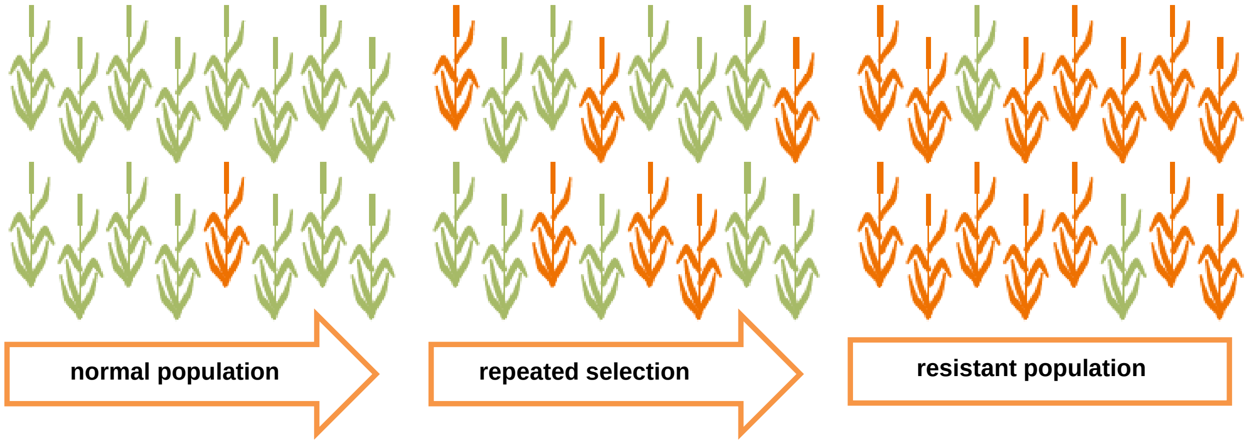

Figure 2: Evolution of Herbicide Resistance (mod. from Bayer Cropscience 2004)

A certain proportion of resistant biotypes are naturally present within a black-grass population in agricultural fields (see Fig. 2). Herbicide resistance is inherited and occurs through selection of plants that survive herbicide treatment. With repeated selection with the same herbicide and/or same herbicide mode of action, resistant plants multiply until they dominate the population (JKI 2008, Moss 2013). The two main mechanisms of resistance are:

Enhanced metabolism resistance (EMR)

EMR causes more rapid herbicide detoxification within resistant plants and is the most common resistance mechanism in black -grass. The herbicide is effectively de-activated by the excess enzyme before getting to the target site. The involvement of cytochrome P450 mono-oxygenases (P450s) and glutathione-S-transferase (GST) enzymes has been implicated in enhanced metabolism of herbicides in several resistant weed species (Moss 1987, Hall et al. 1997,Cobb et al. 2001, Moss 2002, Powles and Yu 2010). With enhanced metabolism resistance, the herbicides usually work to some extent and only in very severe cases does it result in complete loss of control. There is, however, less flexibility relating to growth stage and other conditions that would normally be associated with acceptable levels of control. EMR may affect partial resistance in a wide range of herbicides with different modes of action (Cross-resistance). For example, resistant plants may be able to detoxify both ALS- and ACCase-inhibitors as well other herbicide groups, including substituted ureas (Moss 2002) or pre-emergence herbicides (flufenacet, prosulfocarb, and others) (Petersen et al. 2012). Generally, this form of resistance tends to develop quite slowly over a period of years. The proportion of target-site resistance is lower than of non-target-site mechanisms, especially in Germany (Drobny et al. 2006).

Target-site resistance (TSR)

Target-site resistance is the result of a modification of the herbicide-binding site after point mutation, which precludes a herbicide from effectively binding or acting on its target enzyme in order to kill the plant. This usually results in complete resistance to herbicides acting on that specific site, but not to herbicides acting on different targets (Hall et al. 1997, Powles and Yu 2010). Resistant populations can also increase rapidly.

Target-site resistances have been identified in Germany for various herbicides, for example ALS-inhibitors e.g. sulfonylurea herbicides, and ACCase-inhibitors e.g. fop/dim/den herbicides (Gehring et al. 2012, HRAC 2013). Examples of these herbicides are shown in Table 1.

Current situation in Germany

A. myosuroides is the most problematic weed linked with herbicide resistance in Germany. Nevertheless, reports about ALS-inhibitor resistance in *A. spica-venti* occur as well (Niemann and Zwerger 2006). In different monitoring programs conducted by both the industry and governmental institutes in all regions of Germany, about 75% of all collected seed samples of black-grass and about 30% of *Apera-spica-venti* samples were resistant to herbicides (Gehring et al. 2012). Black-grass biotypes resistant to ACCase-inhibitors occurred in 50% of the samples, whereas about 35% were resistant to ALS-inhibitors, which shows an increase over the last years. A further 15% of the samples were resistant to PSII-inhibitors.

Cross- and multiple-resistances have been observed in black-grass populations due to enhanced metabolism (EMR) or changes in the target protein by genetic mutations (TSR), or both (Hall et al. 1997, Krato and Petersen 2010, Menne and Hogrefe 2012). In Germany the most herbicide resistance cases which occur for black-grass are due to enhanced metabolism (Balgheim 2006, Menne et al. 2008, JKI 2008, Petersen et al. 2012, Gehring et al. 2012).

Conservative estimations assume a resistance infestation level of 5 to 10% on the German arable land with naturally occurring *A. myosuroides* populations (Petersen and Wagner 2009). The distribution of resistant black-grass is concentrated mainly in Northern marsh landscapes along the coast and in the intensive grown cereal regions in Baden-Würtemberg, Bayern, Niedersachsen, Nordrhein-Westfahlen and Schleswig-Holstein (see. Fig.3). In Germany, an area of about 2,500.000 ha is infested with *A. myosuroides* (Petersen 2014). Black-grass resistance is estimated to be a problem on about 875.000 ha of arable land in Germany (35%), of which about 80-90% is affected by ACCase-inhibitor resistance and about 20-30% is affected by resistance to ALS-inhibitors (Raffel 2013, Petersen 2014). According to Gehring et al. (2012) an area of about 200-250.000 ha is affeced by black-grass resistance in Germany.

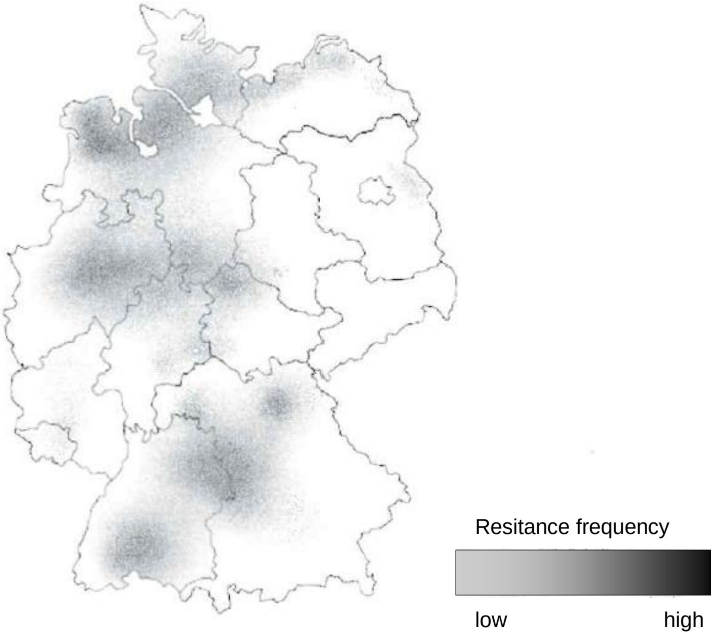

Figure 3: Distribution of ACCase-inhibitor resistant black-grass in Germany (Gehring et al. 2012)

As a result of herbicide resistance, additional costs for more intensive herbicide management for weed control (herbicide, higher rates, frequency, change in crop rotation etc.) is estimated to exceed 15-20 Mio. €. In addition to the economic problem, herbicide resistance may lead to an ecological problem. Higher herbicide intensity may result in higher herbicide entry into the environment and also increase the risk of soil erosion by intensified soil tillage.

The loss of the herbicide activity due to resistances is expected to decrease the yearly cereal production in Germany by 200-600 t. Overall, it is estimated that the resistance costs about 20-100 Mio. € a year (Gehring et al. 2012).

Herbicides are often considered the primary method of weed control, but the loss of existing herbicides through regulatory action, lack of new modes of action/new herbicides and increasing resistance means that non-chemical methods will become more important in the future.

Therefore, industrial and official advisors are trying to implement anti-resistant strategies based on appropriate cropping strategies combined with suitable and specific herbicide management strategies for the avoidance of resistance (Cavan et al. 2000, Balgheim 2006, Menne et al. 2008, JKI 2008, Gehring et al. 2012).

2.1.4 Management of herbicide- resistant black-grass (chemical and non-chemical)

Herbicide management

Application technique can significantly affect herbicide performance, especially for small weeds early in the season. In order to maximize herbicide performance in the field the following factors must be considered (JKI 2008, Gehring et al. 2012):

- Optimized product use (e.g. no reduction in rate)
- Timing of the application (weed size, weather conditions etc.)
- Adapted application technology for high levels of deposits of active ingredient (e.g. right droplet size, controlling spray drift, water volume)

In order to prevent or delay the build up of resistant weeds, herbicides within the same mode of action group e.g. A, B, C1, C2 etc. (compare Tab. 1) should not be applied as the solo treatment year after year on the same target in the same crop (Balgheim 2006, JKI 2008, Wolber 2009). Moreover, a systematic alteration of mode of actions within a crop rotation should be established in order to reduce the selection pressure on a certain weed population. Use of mixes (products or tank mixes) or programs (e.g. sequences) of herbicides based on different modes of action will help to improve the overall control and slow down resistance developing (Moss 2013). A typical sequence starts with pre-emergence herbicides (e.g. flufenacet, pendimethalin, prosulfocarb) followed by post-emergence herbicides ACCase (fops, dins, dens) and/or ALS-inhibitors (e.g. sulfonylureas) and is often promoted as a way of reducing the risk of herbicide resistance (Gressel and Segel 1990, Tatnell et al. 2007).

Residual herbicides such as flufenacet or pendimethalin can provide good control of black-grass emerging after application and as such are a valuable component of an autumn control program when applied either pre-emergence or early post-emergence. For any soil acting herbicide, seedbed preparation is particularly important to get the optimal level of control. Accumulations of straw, crop residue or ash should be buried, or spread as thinly as possible, following cultivation. Ideally soil clods should be small and the seedbed consolidated. For pre-emergence applications a moist soil surface with some rainfall soon after application will also help maximize the level of control provided by residual herbicides (Wolber 2009, Gehring et al. 2012). Heavy populations or re-emerged black-grass may then be controlled in the spring with ACCase- and ALS-inhibitors.

Non-chemical methods

Very high levels of control (97%) are needed to prevent *A. myosuroides* from increasing in winter wheat grown in non-inversion tillage systems (Moss et al. 2013). Nowadays, achieving such high levels of control by using solely herbicides is a challenge in many areas. Today, herbicide resistance is already widespread and increasing, leading to unreliable control by most herbicides, especially from post-emergence herbicides such as ACCase- and ALS-inhibitors (Gehring et al. 2012). But also pre-emergence herbicides (flufenacet, prosulfocarb, and others) can be affected due to strong metabolic resistances (Petersen et al. 2012) and therefore only a few herbicides are left which are still effective on *A. myosuroides.* Further, no new innovative herbicides or modes of action are likely to become available in the near future, and some existing herbicides may be withdrawn or restricted in European countries for regulatory reasons. Also, the EU-Sustainable use of Pesticides Directive (2009/128) requires farmers to give priority to non-chemical methods of plant protection. As a result of the drastically reduced number of effective herbicides for the control of *A. myosuroides,* in particular herbicide resistances*,* it becomes necessary to develop sustainable strategies for managing grass weeds by using also non-chemical weed control methods. The most promising approach is integrated weed management, a strategy which includes a combination of various indirect and direct control measures to keep the weed populations low (Clarke and Moss 1991, Balgheim 2006, Chauvel et al. 2009, Gehring et al. 2012, Moss et al. 2013).

Crop rotation

The prevalence of autumn sown crops is the main reason why black-grass is an increasing problem and widespread in intensive cereal grown areas (Balgheim 2006, Gehring et al. 2012). The inclusion of spring sown crops is likely to help control grass-weeds such as *A. myosuroides* (Hurle 1993). Growing non-cereal crops in rotation enable both different cultural methods and herbicides to be used, thus forming the basis of a strategy to reduce or delay the development of resistance black-grass populations.

Soil cultivation

Primary tillage has a very drastic effect on annual grass weeds (Hurle 1993). With ploughing, the fresh seeds from one season are buried and small seeds such as *A. myosuroides* have practically no chance of germinating (> 5 cm). Black-grass seeds are relatively non-persistent in the seed bank (70-80% decline per year), so usually fewer, old, buried seeds are brought back up to the surface, especially if ploughing is conducted on a rotational basis, once every 3-6 years (Moss et al. 2013). The reductive effect of ploughing cultivation compared to tine and direct drilled cultivations on infestations with black-grass has been reported in different studies (Moss 1980, Amann 1991, Amann et al. 1992, Pollard et al. 1982, Chauvel et al. 2001). On the contrary, with reduced tillage seeds remain in the upper layer and contribute to the infestation immediately (Amann 1991).

After the harvest a first shallow stubble cultivation can be useful to trigger and promote the emergence of fresh black-grass seeds prior to sowing, which can then be controlled by a non-selective herbicide (JKI 2008, Landschreiber 2014).

Delaying the sowing

Delaying the sowing time allows more black-grass seedlings to emerge and be controlled by seedbed preparation or non-selective herbicide application before sowing (Hurle 1993, Landschreiber 2014). Studies with *A. myosuroides* have shown that later sowing dates can reduce seedling emergence, yield losses caused by black-grass competition and *A. myosuroides* seed production compared with 3-4 weeks earlier sowing (Moss 1985a, Amann et al. 1992, Melander 1995).

Further, later emergence of new seedlings is likely to be reduced, although this is dependent on the dormancy level of black-grass (Fenner 1991, Moss et al. 2006). Moreover, residual pre-emergence herbicides can be more effective when applied in later sown crops, because soil conditions are usually more favorable for good activity. Delaying the drilling, however, carries obvious risks, such as soil cultivation being no longer possible due to changing soil conditions. Because about 80% of black-grass emergence occurs in autumn (Moss 2013), spring sown crops tend to be much less affected and have often shown a good reduction in black-grass infestations in field trials (Chauvel et al. 2009, Moss et al. 2013). However, establishing spring crops can be difficult, in particular on heavy soils, and the herbicide choice is more limited.

Competitive Crops

Competitive crops can help greatly in suppressing black-grass (JKI 2008, Cussans 2009, Gehring et al 2012). Methods such as higher seed rates of winter cereals, more competitive crops or varieties, narrow row spacing, improved drainage and good seedbeds can improve the competiveness of crops that are better able to suppress weeds (Moss 1985d, Grundy and Froud-Williams 1993, Hurle 1993, Seavers and Wright 1999, Moss 2013, Moss et al. 2013).

Higher winter wheat populations (e.g. > 300 plants/m^2) are much more competitive than low populations (e.g. 100 plants/m^2), but excessively high seed rates increase the risk of lodging (Moss 2013).

2.2 HRAC Classification System of Herbicides

The Herbicide Resistance Action Committee (HRAC) is an international body founded by the agrochemical industry as part of the Global Crop Protection Federation (GCPF) organization. The aims of the HRAC are to support cooperative approaches to the management of herbicide resistance by fostering understanding, cooperation, and communication between industry, government, and farmers (HRAC 2014). The global HRAC group proposed a classification system for herbicides according to their target sites, to support the use of herbicides suitable for resistance management (Table 1). The HRAC classification should help farmers, advisors and researchers to know which herbicides are best suited to combat specific resistant weeds in crops. In order to prevent or delay the buildup of resistant weeds, herbicides within the same mode of action group e.g. A, B, C etc., should not be applied more than once to control the same target weeds in the same crop (Balgheim 2006, JKI 2008).

Because this research is focused mainly on post-emergence herbicides, the ACCase-inhibitors as well as the ALS-inhibitors are described in more detail below.

Table 1: Key grass weed herbicide groups by mode of action and active ingredient (HRAC 2014)

HRAC-group	Site of Action	Chemical Family	Active Ingredient
A	ACCase inhibitors (inhibitors of Acetyl-CoA Carboxylase)	Aryloxyphenoxy-propionate ‘FOPs’	clodinafop-propargyl fenoxaprop-P-ethyl
		Cyclohexanedione ‘DIMs’	clethodim* cycloxydim*
		Phenylpyrazoline ‘DEN’	pinoxaden
B	ALS inhibitors (inhibitors of Acetolactate Synthase)	Sulfonylurea	flupyrsulfuron-methyl- iodosulfuron-methyl-Na mesosulfuron-methyl sulfosulfuron
		Triazolopyrimidine	florasulam pyroxsulam
		Sulfonylamino-carbonyltriazolinone	propoxycarbazone-Na
C1	Photosystem II-inhibitors	Triazinones	metribuzin*
C2	Photosystem II-inhibitors	Urea	isoproturon chlorotoluron
F1	Inhibition of Pigment Synthesis (Inhibition of PDS)- Bleaching Herbicides	Pyridinecarboxamide	diflufenican picolinafen
G	Inhibition of EPSP synthase	Glycine	glyphosate*
K1	Microtubule assembly inhibition	Dinitroaniline benzamide	pendimethalin propyzamid*
K3	Inhibition of VLCFAs	Oxyacetamide	flufenacet
N	Inhibition of lipid synthesis	Thiocarbamate	prosulfocarb

* = Not for use in cereals; PDS = phytoene desaturase, EPSP = 5-enolpyruvylshikimate-3-phosphate

2.2.1 Inhibitors of the Acetyl-CoA Carboxylase (ACCase)

Fatty acid biosynthesis is important in providing phospholipids used in building new membranes required for plant growth and development, and also for replacing lipids damaged by active oxygen species (AOS) such as superoxide, peroxide anions and hydrogen peroxide. The first step in fatty acid synthesis is carried out by the enzyme acetyl-CoA carboxylase (ACCase). The acetyl-CoA carboxylase catalyzes the carboxylation of acetyl-CoA, which results in the formation of malonyl-CoA (Harwood 1999, Reade and Cobb 2002). In plastids, this reaction is the initial step of de novo fatty acid biosynthesis and is, therefore, of high importance in plant metabolism.

There are three different chemical groups of ACCase-inhibitors, the aryloxyphenoxypropionates (FOPs) and the cyclohexanediones (DIMs), and phenylpyrazoline (DENs), which have developed in the past 15 to 30 years into a very important herbicide family with selective action on a broad spectrum of grass weed species (Zwerger and Ammon 2002). Examples of these ACCase-inhibitors are given in Table 1. These herbicides are collectively known as graminicides, or sometimes "grass killers".

Broadleaf species are naturally resistant to ACCase-inhibitor herbicides because of an insensitive ACCase enzyme. The ACCase- inhibiting herbicides inhibit only the plastidic homomeric ACCase in grasses (Poaceae), but not the plastidic heteromeric form of other mono- and dicotyledonous species nor the homomeric ACCase in the cytosol. Therefore, these herbicides selectively have a lethal effect only on grass species, while they are tolerated by other monocotyledonous and by dicotyledonous species (Burton 1997, Zwerger and Ammon 2002).

Fops were first developed in Japan and later in Germany, and diclofop-methyl was the first one commercially marketed in 1979. Since most ACCase-inhibitors have very low water solubility, they are formulated and sold as esters which also supports the penetration via the foliage. Once absorbed into plant tissues, the esters are rapidly cleaved to produce the free acids, which are the herbicidal form of the molecules (Krähmer et al. 2003).

Most of the herbicides that inhibit this site of action have little to no soil residual activity, so the majority of the activity comes from foliar applications. ACCase-inhibitor herbicides produce an increase in cell permeability and, later, a breakdown of membrane structure (Crowley and Pendeville 1979). Such actions lead to the obvious signs of graminicide damage in sensitive plants within the first week of application, as exemplified by first chlorosis followed by necrosis and death of the rapidly growing meristematic tissues. Complete control of susceptible species may require two to three weeks following applications (Hofer et al. 2006). Crop tolerance of ACCase-inhibitors within monocotyledonous species is based on different metabolic kinetics (Burton 1997). Tolerance in cereal crops, however, is typically insufficient to provide an agronomically adequate margin of crop safety. Co-application of the safener cloquintocet-mexyl or mefenpyr induces metabolic enzymes specifically in the crop species resulting in degradation of the herbicide to non-phytotoxic compounds before damage can occur to the crop. The safener does not affect metabolism in grass weeds generally. Herbicides based on ACCase-inhibitors such as Axial, Topik or Ralon Super are therefore always co-formulated with the safener (Hoechst 1988, Krähmer et al. 2003, Hofer et al. 2006, Fortmeier et al. 2006).

2.2.2 Inhibitors of the Acetolactate synthase (ALS)

The acetolactate synthase (ALS) is a key enzyme in the biosynthesis of the branched-chain amino acids isoleucine, leucine and valine. The ALS is located in the chloroplast, although the gene encoding it is nuclear. In the pathway leading to valine and leucine, ALS catalyzes the formation of 2-acetolactate from two pyruvate molecules, and in the pathway to isoleucine the formation of 2-acetohydroxybutyrate from 2-ketobutyrate and pyruvate (Brown 1990, Shaner and Singh 1997, Reade and Cobb 2002). Due to this double function, the enzyme is also referred to (with a more general term) as acetohydroxyacid synthase (AHAS). Inhibition of this enzyme disrupts the ability of the plants to produce proteins, thus inhibiting their growth. The visible symptoms of herbicidal action are arrested growth within the first days after application, accompanied by chlorosis and sometimes anthocyanin discolouration of the foliage and the appearance of chlorotic patches, followed by slow necrosis leading finally to plant death. Competition with crops by weeds for nutrients and water ceases usually within a few hours of application. Susceptible plants stop growing almost immediately after post-emergence application, allowing crops to grow further without weed competition. Depending on the environmental conditions weeds will be completely killed 4 to 6 weeks after application (Brown 1990, Reade and Cobb 2002, Krähmer et al. 2003).

ALS is inhibited by a number of structurally diverse groups of herbicides, mainly the sulfonylureas, imidazolinones, triazolopyrimidines, pyrimidinylthiobenzoates, and sulfonylamino-carbonyltriazolinones. Examples of these herbicides are given in Table 1. The herbicidal activity of certain sulfonylurea compounds, as the first ALS-inhibiting herbicides, were discovered by George Levitt (first patent awarded by DuPont in 1978) and after subsequent development metsulfuron-Methyl (Gropper) became the first registered product in Germany in 1985 (Drobny et al. 2012). The drastically reduced use rates compared with standard products of the time (below 10 g/ha), the entirely new mode of action (inhibition of the ALS), flexible application (pre- or post-emergent), and the outstanding safety to the applicator and the environment set a new standard for herbicides. A total of over 30 molecules have been commercialized from this herbicide class worldwide, with uses in practically all major agricultural crops.

Residual activity, crop selectivity and the spectrum of weeds controlled can vary greatly depending on the herbicide selected. ALS-compounds, such as mesosulfuron, act on the target weeds both via the foliage and the soil, usually with predominantly foliar action (Köcher 2005). iodosulfuron and flupyrsulfuron also act via foliar uptake but also offers a significant residual activity component via soil activity (Teaney et al. 1995, Trabold et al. 2000). An example of a sulfonylurea which is taken up mainly by the roots is imazosulfuron (Drobny et al. 2012), whereas thiameturon-methyl can be regarded as a mainly foliar-active herbicide because of its rapid degradation (Kudsk et al. 1989). The corn herbicide foramsulfuron provides weed control primarily through foliar activity after penetration into the leaves of grasses and broad-leaved weeds (Collins et al. 2001, Collins et al. 2003). Thiencarbazone-methyl and metosulam achieve equivalent levels of weed control after uptake from the soil after pre-emergence or after foliar uptake from post-emergence spraying (Kinzel 1994, Santel 2012.).

In general ALS-inhibiting herbicides are weak acids with intermediate water solubility. Making these herbicide's salts or formulating at neutral or high pH increases their water solubility (Strek and Green 2001). ALS-inhibiting herbicides enter plants through foliage and through the root and are translocated in both the symplast (phloem) and in the apoplast (xylem). Sulfonylureas degrade in soils mainly through microbial action or through chemical hydrolysis; degradation rates and pathways are specific to the molecule, soil and climate (Drobny et al. 2012).

Selectivity is achieved through the rapid metabolism of the active substance to non-toxic metabolites within the crop before it can act (Reade and Cobb 2002). Nevertheless, Co-application of safeners like cloquintocet-mexyl or mefenpyr increase the crop selectivity through both reduction of herbicide uptake and translocation within the crop, and enhancement of herbicide degradation in the crop. Thus, herbicides based on ALS-inhibitors such as Atlantis or Broadway are therefore co-formulated with the safener. Some herbicides are formulated with activator adjuvants to enhance foliar activity while others must be tank-mixed with external adjuvants to ensure activity.

2.3 Soil activity of herbicides

2.3.1 Factors affecting the soil activity of herbicides

In general, the soil activity of herbicides are only able to exist when the active ingredient is capable of being taken up by either the roots or the hypocotyls of the weed species. Further, the herbicide must be available for transport in the water phase and be present in the root zone of the weed in order to be absorbed. Several factors are affecting the availability and persistence of a herbicide in the weed/soil environment, thus determining the activity of soil applied herbicides. On the one hand this is highly dependent on characteristics of the chemical itself such as the mobility, soil adsorption, water solubility and the degradation stability (DT50). On the other hand a lot of environmental factors such as the soil properties (organic matter, biological activity, temperature, clay content, structure), precipitation and plant growth conditions have a big impact (Devine et al. 1993, Aldrich and Kremer 1997). In the following section the main factors which determine soil activity are described in more detail.

2.3.2 Chemical properties of herbicides which determine soil activity

The chemical properties of the herbicide itself determine its persistence and mobility in the soil. Properties of particular importance are the vapour pressure, water solubility, soil sorption coefficient (Kd or Koc) and the molecules' susceptibility to chemical or microbial degradation (half-life: DT50) in soil.

The **sorption coefficient (Koc or Kd)** describes the tendency of a pesticide to bind to soil particles (e.g. inorganic clay minerals, humic matter C_{org}). Sorption properties are usually measured in a laboratory by shaking a solution of the herbicide with air-dried, sieved soil. Equilibrium sorption is usually achieved and measured within 24 hours. Those herbicides with higher sorption values are more strongly sorbed to soil and are less likely to be available in the soil solution for transport/movement, and may also increase persistence because the pesticide is protected from degradation. Many soil and herbicide factors may influence the actual sorption of a pesticide to soil, which varies depending on the soil type (Strek 1998a, Strek 1998b, Haider and Schäffer 2000, Gaibhiye and Gupta 2002).

The degradation pathways and rate of a herbicide is dependent on its chemical properties and is influenced by environmental factors such as soil moisture, biological activity and temperature in soils. **The half-life (DT50)** can be measured in the laboratory, usually at a standard temperature (20 °C) and soil moisture content (40-

50%), and using sieved soil. The time it takes for half of the original concentration to undergo degradation is measured. Field dissipation studies may also be made and represent more realistic conditions (Yutai et al. 1999, Carter 2000, Haider and Schäffer 2000).

Water solubility describes the amount of herbicide that will dissolve in a known amount of water. Most of the values reported were determined at room temperature (20°C or 25°C). The higher the solubility value, the more soluble the herbicide. Highly soluble herbicides are more likely to be removed from the soil by runoff or by moving below the root zone with excess water (leaching) (Fryer and Makepeace 1977).

The **vapor pressure** of a herbicide determines its volatility. Volatilization is the process by which chemicals go from a solid or liquid state into a gaseous state and are released into the atmosphere. Volatile herbicides (those with higher vapor pressures) generally dissipate more rapidly than herbicides with lower vapor pressures. Volatilization increases with temperature and moisture. Most herbicides, however, are relatively nonvolatile under normal field-use conditions. 2,4-D and dicamba are commonly used chemicals that are known to be subject to volatilization (Aldrich and Kremer 1997).

2.3.3 Environmental factors affecting the soil activity

Several factors are affecting the availability of the herbicide to plants and thus influence the level and duration of weed control achieved by soil-applied herbicides. The major factors which are presented in more detail below are: **Adsorption, Climatic factors, leaching and degradation**.

Soil composition is a physical factor determined by the relative amounts of sand, silt, and clay in the soil texture, as well as by the organic matter content. Soil composition affects herbicidal activity and persistence through soil-herbicide adsorption, leaching, and vapor loss. **Herbicide adsorption (binding)** is generally greater in fine-textured soils high in organic matter than in coarse-textured soils low in organic matter (Carter 2000). Because of its much greater surface area and chemically higher activity, the clay fraction of the inorganic material is much more important than other inorganic fractions in adsorption in soils. Organic matter at advanced stages of decomposition contains particles colloidal in size. The colloidal fraction of organic matter which is most responsible for adsorption of herbicides is the humic matter (C_{org}) derived from organic materials most resistant to decomposition in soil. This material also has charged external and internal surfaces capable of adsorbing herbicides to the same extent or more as expanding clays (e.g. Montmorillonite). Inorganic clays and organic matter together provide soil colloids that are most important in adsorption of herbicides (Schmidt 1972, Fryer and Makepeace 1977, Aldrich and Kremer 1997, Haider and Schäffer 2000, Gaibhiye and Gupta 2001).

Early in the modern herbicide era, it was shown that a lower proportion of herbicide is available for plant uptake in soil with a high organic matter and clay content, thus a higher herbicide application rate was required to provide the same level of weed control as in coarse-textured and high organic soils (Düfer and Heitefuss 1992, Schmidt 1972). At the same time, the chance of crop injury is greater on coarse-textured soils low in organic matter because a higher proportion of the applied herbicide is available for plant uptake (Hurle 1973).

A study implied that, because of its moderate to high adsorption, flufenacet is likely to persist in soils for some time (Gaibhiye and Gupta 2001). Besides the texture and organic matter, other factors also affecting degree of adsorption include soil pH, soil drying-rewetting cycles, presence of crop residues, temperature, herbicide solubility, and degree of ionization (Aldrich and Kremer 1997). Walker et al. (1989) and Walker and Welch (1989) found a highly significant negative correlation between chlorsulfuron and metsulfuron adsorption and soil pH, and consequently that phytotoxicity was greater at high soil pH. Sorption of sulfonylurea to soil is stronger at low pH. Moreover, soil moisture is important because it influences herbicide adsorption to soils and thus the availability of herbicide molecules for plant uptake. Water molecules compete with herbicide molecules for adsorption sites on soil particles and organic matter. Therefore, herbicide adsorption is highest under dry soil conditions, and lowest in moist soils. Consequently, weed control is generally best with moist soil conditions because more herbicide is in the soil solution or gaseous phase and available for plant uptake (Aldrich and Kremer 1997).

Climatic factors involved in herbicide availability and persistence in soils are moisture, temperature, and sunlight. Climatic conditions around the time of application are generally considered one of the key parameters affecting herbicide activity and the cause of much variation in herbicide performance (Lundkvist 1997, Kudsk 2002, Collings et al. 2003). Climatic conditions affect the growth and physiological status of the weed and crop, the herbicide and thus the plant-herbicide interactions.

Temperature affects the activity of soil-applied herbicides primarily because of its influence on the rate of seed germination, emergence, and growth. Seedling plants tend to be more susceptible to soil-applied herbicides under cool conditions than under warm temperatures because plant emergence is delayed and metabolism is slowed. Also, with warm conditions seedling roots can quickly grow away from the soil zone, where the highest concentration of herbicide is present. Temperature has a major influence on plant development and can influence herbicide performance.

Higher transpiration rates of plants at adequate soil moisture levels generally increase adsorption and translocation throughout the plant (Fryer and Makepeace 1977). Herbicide degradation rates generally increase as temperature and soil moisture increase, because both chemical and microbial decomposition rates increase with higher temperatures and moisture levels. Cool, dry conditions slow down herbicide degradation (Walker et al. 1989, Strek and Green 2001). Generally, soil-applied herbicides require adequate soil moisture to activate and must be in soil solution for the roots to absorb them. In the presence of adequate soil moisture, less herbicide is adsorbed onto the soil and more is available in the soil solution for plant uptake, thus weed control. Under dry conditions, herbicides are tightly adsorbed by soil colloids, and insufficient amounts may be available to provide acceptable weed control.

Soil-applied herbicides, particularly the ones absorbed mainly via the roots (e.g. the ureas and triazines), may fail totally if the soil is dry around the time of application. This is because the herbicide does not move into the upper centimeters of the soil where most weed seeds germinate and because the roots of weed plants do not explore dry soil. Herbicides absorbed primarily by the shoots (e.g. trifluralin, pendimethalin and propachlor) tend to be less affected by soil moisture deficit, although high soil moisture will also promote the performance of this group of herbicides (Kudsk 2002).

Adequate soil moisture ensures ALS-inhibiting herbicide translocation throughout the weed and subsequent control (Olson et al. 1999), metabolic inactivation and crop tolerance (Olson et al. 2000). Soil moisture has previously been demonstrated to be important for isoproturon activity for *A. myosuroides* control in pot experiments in a greenhouse (Blair 1985). In previous research, adequate soil moisture has been shown to improve herbicide efficacy of foliar applied herbicides such as fenoxaprop, fluazifop-P, haloxyfop imazethapyr, sethoxydim (Boydston 1990), imazethapyr (Nalewaja et al. 1990), glyphosate (McWhorter and Azlin 1978), dichlorprop-P/MCPA and tribenuron-methyl (Lundkvist 1997) and chlorsulfuron (Nalewaja and Woznica 1985).

Ideally, a herbicide applied to the soil to prevent weed emergence would be placed in the soil zone from which the weeds originate and remain there until all of the non-dormant seed had germinated. Soil-applied herbicides usually require a certain amount of precipitation to move them from the soil surface into the zone where weed seeds germinate and emerge. Herbicides need to be distributed via water movement through 5 cm or more of the surface layer of soils from where the weeds are emerging to be most effective (Aldrich and Kremer 1997). Adequate rainfall and the maintenance of a high soil moisture content in the surface layers has been shown to maximize the activity of soil-applied herbicides by dissolving and moving the herbicide into the top few centimeters of soil (Blair 1985, Malfyt and Quakenbush 1991, Lundkvist 1997, Kudsk 2002).

On the other hand, free movement with percolating rain water can transport the herbicide beyond its required soil zone and thereby cause the herbicide to be ineffective. **Leaching** is the term applied to the movement for herbicides with water in soil, and is dependent on the solubility of a herbicide in water, herbicide-soil binding properties, soil physical characteristics, and rainfall frequency and intensity (Oppong and Sagar 1992, Carter 2000). In general, herbicides that are highly soluble in water and weakly absorbed to soil particles are more likely to leach, particularly in wet years. Overabundant soil moisture reduces activity by promoting leaching out of the zone of greatest effectiveness near the surface or enhancing degradation (Strek and Green 2001). Nevertheless, poor weed control because of inadequate herbicide movement is a much more common reason for failure than movement of the herbicide beyond the effective root zone (Aldrich and Kremer 1997).

Degradation, the conversion or breakdown of the herbicide compound by simpler and more frequent nontoxic compounds, can occur via several pathways including: microbial degradation, chemical degradation and photo degradation. The kinetics of herbicide degradation in the soil are very complex. Depending on the properties of the chemical itself and the environmental conditions such as light, temperature, soil moisture and pH, different pathways may be dominant and affect the degradation rate (Mueller et al. 1992, Dinelli et al. 1998, Yutai et al. 2000, Strek 2005).

Microbial degradation occurs when soil microorganisms use the herbicides as a food source and is the most important pathway responsible for the breakdown of many herbicides. The types of microorganisms (fungi, bacteria, protozoans etc.) and their relative numbers determine how quickly decomposition occurs. Microorganisms require certain environmental conditions for optimal growth and utilization of any herbicide. Factors that affect population levels and activity of these microorganisms are food supply, soil moisture, temperature, pH, oxygen, and organic matter content. Usually, a warm, well-aerated and fertile soil with a near-neutral pH is most favourable for microbial growth and for herbicide breakdown (Fryer and Makepeace 1977, Aldrich and Kremer 1997, Dinelli et al. 1998, Gaibhiye and Gupta 2002).

Chemical degradation occurs when a non-biological chemical reaction dissipates the herbicide active ingredient into non-active secondary molecules. The most common form of chemical degradation is hydrolysis, a process in which the herbicide molecules react with water. In general, chemical bonds in the herbicide molecule are broken and one or more atoms or groups of atoms in the herbicide molecule are replaced by hydroxyl ions (OH-) from water. This change in molecular structure can inactivate the herbicide. Even very dry field soil has enough moisture for some hydrolysis to occur (Strek 2005). Hydrolysis together with microbial degradation are the major methods by which sulfonylurea herbicides such as chlorsulfuron are degraded in soils (Beyer et al. 1988, Brown 1990, Strek 1998a, Strek 2005). The major hydrolytic pathway in soil for the sulfonylurea herbicides is the cleavage of the sulfonylurea bridge, resulting in sulfonamide and heterocyclic amine molecules. Cleavage by hydrolysis is pH sensitive. Soil pH influences the availability and persistence of herbicides such as atrazine or sulfonylureas in the soil. Soil pH can alter the ionic nature of the herbicide molecule, which influences adsorption, solubility and rate of herbicide breakdown through aqueous hydrolysis. As the soil pH increases, the rate of chemical hydrolysis in the soil usually decreases. Therefore, sulfonylurea herbicides degrade more rapidly at a lower soil pH than at a higher soil pH. Thus, in alkaline soils where chemical hydrolysis is minimized, herbicides such as chlorsulfuron, triasulfuron and metsulfuron-mehtyl can persist long enough to injure certain rotational crops (carry over) (Walker and Welch 1989, Walker et al. 1989, Kotoula-Syka et al.1993, Strek 1998a, Strek 1998b, Sarmah et al. 2000). In addition, chemical hydrolysis of sulfonylurea herbicides is faster during the summer when soil temperatures are warm than in autumn and winter when soils are cooler. Low sorption properties of sulfonylureas at higher pH values (Walker et al. 1989) is likely to cause greater leaching after heavy rainfall (Nicholls and Evens 1985). At high pH levels in soils those herbicides as anions are very mobile. This could also lead to increased persistence of residues over a long period of time as the microbial activity would be expected to be much lower with increasing depth. Degradation rates of chlorsulfuron in soil have been shown to be negatively correlated with soil pH and positively correlated with temperature, soil moisture content, organic matter content and microbial biomass in soil (Walker et al. 1989, Dinelli et al. 1998, Strek 1998a).

Photo degradation can have distinct effects on the success of many soil-applied herbicides. Photochemical degradation occurs when ultraviolet (UV) light breaks chemical bonds of the herbicide’s active ingredient. Secondary molecules resulting from the cleavage of the parent molecule are usually less effective in providing weed control. Herbicides found to be sensitive to photo degradation are for example trifluralin, the s-triazines, paraquat and diquat (Aldrich and Kremer 1997).

3 Material and methods

3.1 Description of experimental locations

Field experiments were conducted on agricultural fields at different locations within Hessen. The soil properties and cultivation methods for each experimental site are listed in Tab. 2. The field trial locations in Wicker (WI), Gießen (GI) and Rauischholzhausen (RH) are described in more detail below.

Table 2: Soil properties and cultivation methods at experimental locations

Location	Sand (%)	Silt (%)	Clay (%)	C-org (%)	pH	Cultivation
RH	7	63	30	2.9	6.9	MINTIL+PLOUGH
GI	18	67	15	1.2	-	PLOUGH
WI (STR+KEN)	21	56	23	1.3	6.7	MINTIL
WI (JOH)	8	75	18	1.9	6.8	MINTIL
TR	24	31	44	5.9	7.5	MINTIL

RH = Rauischholzhausen, GI = Gießen, WI (STR+KEN) = fields at Wicker-Staßenmühle and Wicker-Kennel; WI (JOH) = Wicker-Johannesfeld, TR = Trebur, MINTIL = minimum tillage using a chisel plough, PLOUGH = mouldboard ploughing, C-org = organic material

3.1.1 Experimental station Wicker

Bayer`s experimental station Wicker is situated between Frankfurt and Wiesbaden (WI-Johannesfeld: latitude 50° 1'55.41´N, longitude 8° 24'25´E, altitude 146 m above sea level; WI-Straßenmühle+Wi-Kennel: latitude 50° 1'34.02´N, longitude 8° 24'1´E, altitude 122 m above sea level). Soil conditions of the fields in Wicker are characterized by loess soil which is formed by the accumulation of wind-blown silt and variable amounts of sand and clay. In general, the soil at the experimental locations in Wicker (Johannesfeld, Straßenmühle and Kennel) are very homogeneous and contain mostly silty clay loam with clay content varying from 18-23% and humus content of 1.3 to 1.9%. The amount of clay was higher at the trial locations Straßenmühle and Kennel compared to Johannesfeld, which contains more silt. The soil parameters are given in Tab. 2. Maintenance applications of growth regulators, fungicides and insecticides were applied according to local agricultural practice. Nitrogen was applied as calcium ammonium nitrate (27% N) on the basis of soil analysis at different rates during the season to meet the nitrogen requirement of the crop.

Climatic conditions

The weather conditions during the growing period (including over wintering phase) of winter wheat (September to August) were characterized by a mean air temperature of 10.7, 10.4 and 10.9 °C, and a sum precipitation of 707.7, 551.3 and 600.2 mm in 2009, 2010 and 2011 respectively (Table 3). In all growing years air temperature reached its maximum in July and August. Lowest temperatures were recorded in the months of December and January. An exception was the month of February in 2012 which was very cold with an average of only -0.9 °C. During the cold period from Feb. 1st until Feb. 12th, the average daily temperatures were between -6°C and-10.9 °C. In general, values of average air temperature of all growing years were higher in comparison with last 53 years (1949-2012). The precipitation during the year 2009/10 was relatively high and in 2010/11 relatively low compared to the long term average. Extremely low precipitation was recorded between March and May of 2011, which led to an early termination of the trials because of severe damage to winter wheat.

Table 3: Air temperature (°C) and precipitation (mm) data during the growth period of winter wheat in 2009-2012 and the last 53 years at Wicker

	Air Temperature °C (monthly mean)				**Precipitation (mm/month)**			
Month	**2009/ 2010***	**2010/ 2011**	**2011/ 2012**	**1949-2012***	**2009/ 2010***	**2010/ 2011**	**2011/ 2012**	**1949-2012***
September	17	14.2	16.8	16	41.3	44.3	34.3	47.8
October	10.5	9.1	10.8	9.8	40.3	25.2	27.8	48.4
November	8.7	6.7	5.4	5.7	74	46	3.2	67.2
December	2.2	-1.9	4.7	1.4	78.5	50.6	106.2	52.6
January	-1.6	2.2	3.5	1.6	43.2	44.2	65.7	41
February	2	2.7	-0.9	3.1	48.4	25.4	5.2	37.8
March	6.8	7.5	8.8	4.9	29	15.8	17.4	41.8
April	10.9	13.8	9.4	9.7	20.2	11.2	47.2	42.5
May	12.3	15.7	15.7	13.8	107.6	31.3	79	57.4
June	18.6	17.9	16.5	16.8	61.3	86.4	117.2	63.7
July	22.3	17.4	19	19.3	60	83.3	55	64.7
August	18.7	19.1	20.6	18.6	103.6	87.6	42	63.4
Mean	10.7	10.4	10.9	10.1	-	-	-	-
SUM	-	-	-	-	707.4	551.3	600.2	628.3

* weather data were used from Frankfurt am Main/Airport, which is located 8-10 km from the trial sites

3.1.2 Experimental station Gießen

The experimental station Gießen is situated in the valley of the Lahn River at a latitude of 50° 36´ North and a longitude of 8° 39´ East and at an altitude of 158 m above sea level. Soil conditions of the fields in Gießen are classified as fluvogenic soil characterized by silty clay with clay content of 28 – 33% (0 – 30 cm). Specific soil analyses which were conducted by the University Gießen directly from the trial site found a clay content of 15% in the top soil (0-15 cm). The soil parameters are given in Tab.2. Furthermore, the soil is characterized by a humus content of 1.2% (0 – 30 cm) and by available field capacity of about 202 mm (0 – 100 cm). Maintenance applications with growth regulators and fungicides were applied as needed according to local agricultural practice. Nitrogen fertilizers were applied on the basis of soil analysis at different rates. The fertilizer calcium ammonium nitrate (27% N) and ammonium sulphate (21% N+24% S) were applied at three different times (70+60+40 kg N/ha) to meet the nitrogen requirements of the crop.

Climatic conditions

The weather conditions during the growing period (including over wintering phase) of winter wheat (September to August) were characterized by a mean air temperature of 8.5, 8.6 and 8.9°C and a sum precipitation of 676.1, 404 and 543 mm in 2009, 2010 and 2011 respectively (Table 4). In all growing years air temperature reached its maximum in July and August. Lowest temperatures were recorded in the months of December and January. The month of February of 2012 was very cold with an average of only -1.2 °C. During the cold period from Feb. 1st until Feb. 12th 2012, the average daily temperatures were between -6.2°C and -11.2 °C, which led to severe frost damage of winter wheat and thus led to the early termination of the field trials in 2011/2012. Generally, values of average air temperature of these growing years were not very different in comparison with the last 20 years (1990-2012). The precipitation during the year 2010/11 and 2011/12 were relatively low compared to the long term average.

Table 4: Air temperature (°C) and precipitation (mm) data during the growth period of winter wheat in 2009-2012 and the last 20 years at Gießen

	Air Temperature °C (monthly mean)				**Precipitation mm/month**			
Month	2009/ 2010	2010/ 2011	2011/ 2012	1990-2010	2009/ 2010	2010/ 2011	2011/ 2012	1990-2010
September	11.4	9.7	11.8	13.7	38.5	66.0	35.4	51.3
October	8.2	8.9	10.1	9.0	48.1	22.1	29.2	50.2
November	6.1	4.7	3.4	4.3	94.0	51.8	0.9	58.3
December	1.8	-2.5	4.3	1.6	67.5	54.2	82.3	64.5
January	-2.2	0.3	1.3	0.3	24.0	17.7	65.7	49.5
February	1.1	2.3	-0.9	0.8	33.1	13.9	3.5	41.4
March	5.4	7.4	7.9	4.4	42.6	6.8	8.9	45.2
April	7.3	9.4	6.8	8.4	11.6	9.6	32.0	42.2
May	12.1	15.1	15.4	12.9	72.9	6.6	55.4	60.2
June	13.3	13.1	11.9	16.0	79.3	50.4	91.1	63.0
July	20.6	16.7	17.9	17.8	59.1	37.0	77.5	68.1
August	16.5	17.7	17.2	17.2	105.4	67.9	61.1	61.1
Mean	8.5	8.6	8.9	8.9	-	-	-	-
SUM	-	-	-	-	676.1	404	543	655

3.1.3 Experimental station Rauischholzhausen

The experimental station Rauischholzhausen is situated nearby Marburg (latitude 50°46´N, longitude 8° 53´E, altitude 220 m above sea level). Soil conditions of Rauischholzhausen are characterized by loess soil which is formed by the accumulation of wind-blown silt and variable amounts of sand and clay that are loosely cemented by calcium carbonate. It is usually homogeneous and highly porous and is traversed by vertical capillaries that permit the sediment to fracture and form vertical bluffs. Generally, the soil of Rauischholzhausen experimental station is characterized by the following parameters: clay content 25% (0–30 cm), humus content 2.4% (0–30 cm) and available field capacity of 130 mm (0–100 cm). Specific soil analyses which were conducted by the University Gießen directly from the trial site found a clay content of 30% in the top soil (0-10 cm) and a humus content of 2.9%. All soil parameters are given in Tab.2. Maintenance applications with growth regulators and fungicides were applied as needed according to local agricultural practice. Nitrogen fertilizers were applied on the basis of soil analysis at different rates. The fertilizer calcium ammonium nitrate (27%N) was applied three different times (80+40+60 kg N/ha) to meet the nitrogen requirements of the crop.

Climatic conditions

The climatic data (air temperature and precipitation) at Rauischholzhausen for the growth period of winter wheat for all three years are presented in Table 5. The weather conditions during the growing period of winter wheat (September to August) were characterized by a mean air temperature of 9.4, 9.3, 9.8°C and a sum precipitation of 676.7; 519.5 and 721.7 mm in 2008, 2009 and 2010 respectively. In all growing years air temperature reached its maximum in July and August. Lowest temperatures were recorded in the months of December and January. The month of February 2012 was especially very cold with an average of only -1.2 °C. During the cold period from Feb. 1st until Feb. 12th the average daily temperatures were between -6°C and-11.9 °C, which killed the winter wheat and thus led to the early termination of field trials in season 2011/12. In general the mean air temperature of Rauischholzhausen was slightly higher than the long term means of the last 20 years (1990-2010) and were warmer than in Gießen during all growing years. The precipitation during the year 2010/11 was relatively low compared to the long term average, whereas the average precipitation in year 2011 was relatively high.

Table 5: Air temperature (°C) and precipitation (mm) data during the growth period of winter wheat in 2009-2012 and the last 20 years at Rauischholzhausen

	Air Temperature °C (monthly mean)				**Precipitation mm/month**			
Month	2009/ 2010	2010/ 2011	2011/ 2012	1990- 2010	2009/ 2010	2010/ 2011	2011/ 2012	1990- 2010
September	14.6	12.4	15.1	13.5	49.3	50.2	61.6	51.8
October	8.8	8.6	9.1	9.0	45.9	24.8	42.3	56.9
November	8.3	6.0	3.9	4.9	110.4	53.2	5.5	51.8
December	1.2	-2.9	4.7	1.2	75.3	80.6	103.9	52.3
January	-2.4	2.3	2.8	0.9	28.5	38.6	84.3	48.8
February	0.7	2.0	-1.2	1.9	54.4	27.8	5.3	43.3
March	5.3	5.7	7.7	5.1	50.3	9.9	10.4	41.4
April	9.2	12.0	9.1	8.9	13.9	30.2	37.9	38.6
May	11.0	14.2	14.8	13.1	68.9	12.4	70.8	62.3
June	17.6	16.8	15.7	15.9	105.4	70.3	118.6	65.3
July	20.1	16.3	17.4	17.8	36.8	42.2	145.2	70.2
August	18.3	17.9	18.5	17.8	37.6	79.4	35.8	61.0
Mean	9.4	9.3	9.8	9.2	-	-	-	-
SUM	-	-	-	-	676.7	519.5	721.7	643.7

3.2 Monitoring the emergence pattern of black-grass (*Alopecurus myosuroides*) during the growing seasons 2009/10, 2010/11 and 2011/12

Using natural weed infestations, the emergence of black-grass (*A. myosuroides*) was observed over three growing seasons (2009-2012) on agricultural fields at different locations in Hessen (Rauischholzhausen, Gießen and Wicker) and within Germany. The soil properties and cultivation methods for each experimental site are listed in Tab. 2.

In all three years (2009/10, 2010/11 and 2011/12) experiments were carried out in Rauischholzhausen (RH), where no ploughing has been practiced for over 20 years and thus ensuring a constant high infestation of *A. myosuroides*. Since the chisel plough is used regularly, for these investigations in 2010 and 2011 the farm field was also partially ploughed using a mouldboard plough. In Gießen (GI), where mouldboard ploughing was practiced, experiments were only carried out in 2009/10, because in the other years there was not sufficient weed pressure. In Wicker (WI), where fields were cultivated with a chisel plough (about 10 cm depth), results are only presented from 2009/10 and 2011/12 when a high and sufficient infestation with *A. myosuroides* was present. In season 2011/12 Bayer CropScience Deutschland GmbH (BCS-D) conducted monitoring studies on agricultural testing fields with winter wheat at multiple locations within Germany. Minimum tillage using a chisel plough was conducted. The locations with silty-clay loam and clay loam were: Möckmühl (Landkreis Heilbronn, Baden-Württemberg), Musdorf (Landkreis Schwäbisch Hall, Baden-Württemberg), Möhnesee-Westrich (Landkreis Soest, Nordrhein-Westfalen) and Grevenkop (Landkreis Steinburg, Schleswig-Holstein). In season 2009/10 two areas with a size of 5-10 m^2 were sprayed within a field with winter wheat using the non-selective and non-residual herbicide Roundup ultramax (glyphosate 450 g/l) in October 2009 and also on March 2010 when herbicides against *A. myosuroides* were applied. Within these plots the natural emergence of black-grass during the growing season was regularly observed in 8 marked plots with the size of 0.1 m^2. The following years (2010/11 and 2011/12) multiple areas (n=3-4) with the size of 1.5-2 m^2 were used for counting, and already emerged individuals of black-grass were clearly labeled with colored plastic rings or sprayed with glyphosate at counting days. In all conducted experiments freshly emerged *A. myosuroides* seedlings in BBCH 11-12 were counted using random squares within the observation plot (0.1 m^2). In monitoring studies conducted by BCS-D in 2011/12, newly emerged seedlings were observed throughout the season using 3 plots with the size of 10 m^2 next to field trials within winter wheat. Already emerged seedlings were sprayed with glyphosate (Roundup ultramax at 2.4 L/ha) at each counting day. Because of severe crop injury due to heavy frost periods in February 2012, some monitoring studies in RH, Musdorf, Grevenkop were cancelled in the spring. Additionally, in Rauischholzhausen in 2011/12 30 already emerged plants of black-grass were dug at the time of the herbicide application (26.10.2011) in order to find out about the emerging depth. The length from the seedling to the mesokotyl was measured. Plants were taken from a field with early sown winter wheat (29.09.2011) where mouldboard ploughing and chisel ploughing cultivation was conducted next to each other.

3.3 Trial set up and herbicide applications of pot experiments

All pot experiments were carried out either within greenhouses or outdoors at the Herbicide Research facilities of Bayer Crop Science AG in Frankfurt am Main, Industrial park Hoechst, Germany. The greenhouses were set with a 14 hour day length to 18-20°C day and 10-12 °C night temperatures and a relative humidity of 50-60%. Round plastic pots, either with a diameter of 10.5 (depth 9 cm) or 13 cm (depth 10 cm) with bottom slots were used for greenhouse experiments. Seeds of *A. myosuroides* were sown with a special measurement spoon with certain volumes, fitting 50-55 seeds per pot for the pot size 10.5 cm and 60-65 seeds for 13 cm pots. In general, seeds of *A. myosuroides* were sown approximately 0.5 cm deep and lightly covered with sand. *Apera spica-venti* was sown by hand with approximately 90-100 seeds per pot and then covered very lightly with sand. The standard soil used in pot experiments was steam sterilized silt loam (sand 15.9%, silt 58.3%, clay 25.8%, pH 7.1, C-org 1.7%) which was filled up to 1 cm from the top of the pots. Field capacity (FC) for the used soil was 44g of water per 100g of dry soil. Pots which were sprayed in pre-emergence were irrigated lightly from above using a hose with a sprinkler. If not described differently applications in post-emergence were mainly watered via sub-irrigation. Herbicides were applied on a track sprayer fitted with a flat fan nozzle (XR Teejet 8001VS) delivering 300 L/ha water at 200 kPa. Each experiment comprised of 4-5 replicates per treatment.

3.4 Trial set up and herbicide applications in field experiments

Weeds were either sprayed in pre-emergence/early post emergence (BBCH 0-11) in autumn or during post-emergence either in autumn (BBCH 12-13) or in the spring (BBCH 11-23). For applications in post-emergence some herbicides were applied with an adjuvant according to manufacturer’s recommendation in order to optimize herbicide efficacy. Herbicides were applied in 300 L water per ha using a hand-held compressed air plot sprayer fitted with flat fan nozzles. The different plot sprayers were equipped with different types of nozzles with different air pressure (Gießen: AIRMIX 110025 at 4.5 bar; Wicker: AIRMIX 110015 at 4 bar; RH: Teejet 2 bar). If not described differently, field experiments were carried out as complete randomized block design and comprised of 3-4 replicates per treatment.

3.5 Herbicides used in experiments

Experiments were conducted in the laboratory, greenhouse and in the field from 2009-2012. An overview about the herbicides used in experiments is given in table 6.

Table 6: Herbicides, active ingredients (a.i.), Concentration (Conc.) and Formulation (Form.) used in experiments

Herbicide ™	a.i. and Safener (S)	Conc. a.i. g L/kg	Form.
Post-emergence herbicides			
ACCase-inhibitors			
Ralon Super	fenoxaprop-P-ethyl	69	EW
	mefenpyr (S)	75	
Topik 100	clodinafop-propagyl	100	EC
	clorquintocet-mexyl (S)	25	
Axial 50 EC	pinoxaden	50	EC
	clorquintocet-mexyl (S)	12.5	
ALS-inhibitors			
Atlantis WG	mesosulfuron-methyl	30	WG
	iodosulfuron-methyl-sodium	6	
	mefenpyr (S)	90	
Broadway	pyroxsulam	68.3	WG
	florasulam	22.8	
	clorquintocet-mexyl (S)	68.3	
Attribut	propoxycarbazone-sodium	700	SG
Lexus	flupyrsulfuron-methyl	500	WG
Alister	diflufenican	150	OD
	iodosulfuron-methyl-natrium	3	
	mesosulfuron-methyl	9	
	mefenpyr-diethyl (S)	27	
EPSP-inhibitors			
Roundup UltraMax	glyphosate	450	SC
Pre-emergence herbicides			
inhibitors of cell division/microtubule assembly/PSII			
Cadou SC	flufenacet	500	SC
Stomp aqua	pendimethalin	455	CS
Malibu	flufenacet	60	EC
	pendimethalin	300	
Bacara Forte	flurtamone	120	SC
	flufenacet	120	
	diflufenican	120	SC
IPU (Arelon Top)	isoproturon	500	
Boxer	prosulfocarb	800	EC
Fenikan	diflufenican	62.5	SC
	isoproturon	500	

TM= trade name, ACCase = Acetyl coenzyme A carboxylase, ALS = Acetolactate synthase, EPSP = 5-enolpyruvylshikimate-3-phosphate synthase, PSII = Photosystem II, a.i. = active ingredient, Conc.= Concentration, Form. = Formulation, S = Safener, WP = Wettable Powder, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, SG = Water soluble granules, OD = dispersible oil, CS = Capsule suspension, SL = Soluble liquid

For post-emergence applications some herbicides were applied with an adjuvant according to manufacturer's recommendation in order to optimize herbicide efficacy. The adjuvants used are listed in Tab. 7.

Table 7: Characterization of additives used in the executed experiments

Trade Name™	Ingredient	Concentration %
Broadway Netzmittel[1]	Rapeseed oil methyl-ester	100
Biopower[2]	sodium laureth sulfate	27
Mero[3]	Rapeseed oil methyl-ester+Ethoxy (7) tridecanol	81+(1-19)
Monfast[4]	Polyoxylated fatty alcohols +propylenglycol	60+25
Trend 90[5]	Isodecyl alcohol ethoxylate	90

[1]with Broadway at 1 L/ha;[2]with Atlantis WG at 0.6-1.0 L/ha; [3]with Attribut at 1L/ha;[4]with Ralon Super at 0.4L/ha; [5]with Lexus at 0.3 L/ha

3.6 Experimental designs of experiments on the soil activity of herbicides against *A. myosuroides*

3.6.1 Pot experiments on the activity of different herbicides against *A. myosuroides* plants via root uptake in aqueous solution

A. myosuroides were sown in plastic pots with silt loam and raised under greenhouse conditions. After 10 days, the emerged seedlings with 2 leaves (BBCH 12) were dug out and put in water for 24 hours. The roots of *A. myosuroides* plants were cut back to the same length (2.5 cm) and then put in glass test tubes (16x160 mm) containing 15 ml water solution with different herbicides. Before, plants were transferred in pipette tips and then fixed at the top of the tube with elastic foil (parafilm) in order to make sure that only the roots of test plants were in contact with the herbicide solution. The tested ACCase-inhibiting herbicides were fenoxaprop-P-ethyl, clodinafop-propagyl and pinoxaden. The formulated ALS-inhibiting herbicides were mesosulfuron-methyl, pyroxsulam, propoxycarbazone and flupyrsulfuron-methyl. Dosages used for each active ingredient were: 0.1, 0.03, 0.01, 0.003 and 0.001 ppm (mg/l). The individual herbicide treatments and products used in the experiment are shown in Table 8 and in result tables. Because of water evaporation the test tubes were refilled with 4-5 ml of water every 3 days. Each treatment was replicated four times. The root growth of treated plants was measured after 14 days and the plant heath (alive/dead) was rated by looking at the upper biomass (shoot). A plant was rated alive when the plant developed 3-4 leaves and the foliage looked healthy and stayed green. A plant was rated as dead, when the plant stopped growing and foliage showed severe toxic symptoms such as chlorosis and necrosis.

Table 8: Herbicides (a.i.s) used at 0.1, 0.03, 0.01, 0.003 and 0.001 ppm (mg/l) in the experiment on root uptake in aqueous solution

No	active ingredient	Formulation/ Conc. a.i.
1	fenoxaprop-P-ethyl	EW 69 g/L
2	clodinafop-propagyl	EC 60 g/L
3	pinoxaden	WP 200 g/kg
4	mesosulfuron-methyl	WG 750 g/kg
5	pyroxsulam	WP 200 g/kg
6	flupyrsulfuron-methyl	WG 500 g/kg (Lexus)
7	propoxycarbazone	SG 700 g/kg (Attribut)

Conc = Concentration; a.i.= active ingredient; WP = Wettable Powder; EC = Emulsifiable Concentrate; WG = Water-Dispersible Granule; SC = Soluble Concentrate; SG = Water soluble granules; EW = Emulsion in water

3.6.2 Effect of herbicide application modes on the efficacy of herbicides

Pot experiments were carried out in a greenhouse using plastic pots (diameter 10.5 cm). Seeds of *A. myosuroides* and *A. spica-venti* were sown in sterilized silt loam. At the 2-3 leaf stage (BBCH 12-13) of the weeds, different post emergence herbicides were applied in three different modes. In the first trial different ACCase- herbicides were applied. In a separate experiment ALS-inhibitors were tested with *A. myosuroides* only. Herbicide applications were conducted as described in chapter 3.2. Direct foliar application without soil contact was achieved by perlite coverage of the soil surface prior to herbicide spray application. This coverage was removed after spraying. Foliar plus soil contact was simulated by a normal spray application without perlite coverage. Soil placement without foliar contact was obtained by distribution of the herbicide solution on the soil surface by means of a pipette. After herbicide application pots were watered by sub-irrigation. Each experiment comprised of 4 replicates per treatment.

3.6.2.1 Effect of different application modes on herbicidal efficacy of ACCase-inhibitors

In this experiment three different ACCase-inhibiting herbicides were applied in three different modes at the 2-3 leaf stage (BBCH 12-13) of the weeds. Herbicides used were: fenoxaprop-P (Ralon Super), clodinafop (Topik) and pinoxaden (Axial). The rates were 80, 60, 40 and 20 g active ingredient per ha. The individual herbicide treatments used in each experiment are shown in Table 9 and in result tables. Efficacy ratings were conducted 21 days after treatment and plants of *A. myosuroides* and *A. spica-venti* were harvested for measurement of fresh shoot weights.

Table 9: ACCase-inhibitors, active ingredients (a.i) and dosages used in the pot experiment on the effect of different application modes on the herbicidal efficacy

No.	Herbicide™ and a.i.	Dosages L/ha (g a.i./ha)			
1-4	Ralon Super fenoxaprop-P	1.2* (80)	0.9 (60)	0.6 (40)	0.3 (20)
5-8	Topik 100 clodinafop	0.8 (80)	0.6* (60)	0.4 (40)	0.2 (20)
9-12	Axial 50EC pinoxaden	1.6 (80)	1.2* (60)	0.8 (40)	0.4 (20)

a.i.= active ingredient, EC = Emulsifiable Concentrate, * full registered field dosage

3.6.2.2 Effect of application modes on herbicidal efficacy of ALS-inhibitors

The pot experiment was conducted in the same way as the pot experiment described above (3.6.2.1). The formulated herbicides, based on recommended field rates were: mesosulfuron at 15, 5 and 1.7 g/ha (WG 750 g a.i./kg); iodosulfuron at 10, 5 and 2.5 g/ha (WG 100 g a.i./kg); pyroxsulam at 15, 5 and 1.7 g/ha (WP 200 g a.i./kg); propoxycarbazone at 70, 35 and 17.5 g/ha (Attribut, SG 700 g a.i./kg) and flupyrsulfuron at 10, 5 and 2.5 g/ha (Lexus, 500 g a.i./kg). Each herbicide was applied with adjuvants according to the manufacturer`s recommendation. The individual herbicide treatments with adjuvants used in each experiment are shown in Table 10 and in result tables. After herbicide application, the pots were watered by sub-irrigation. Because *A. spica venti* did not emerge simultaneously and regularly, herbicide activity was only assessed for *A. myosuroides* 35 days after herbicide spraying by weighing the upper biomass (shoots).

Table 10: ALS-inhibitors, active ingredients (a.i.) and rates used in the pot experiment on the effect of different application modes on the herbicidal efficacy

No.	active ingredient /Formulatiom	Adjuvant (L/ha)	Rates (g a.i./ ha)		
1-3	mesosulfuron (750 WG)	+ Biopower 1.0	15*	5	1.6
4-6	pyroxsulam (200 WP)	+ Broadway Netzmittel 1.0	15*	5	1.6
7-9	flupyrsulfuron (Lexus)	+ Trend90 at 0.3	10*	5	2.5
10-12	propoxycarbazone (Attribut)	+ Mero 1.0	70*	35	17.5
13-15	iodosulfuron (100 WG)	+ Mero 1.0	10*	5	2.5

a.i.= active ingredient, WP = Wettable Powder, WG = Water-Dispersible Granule, * full registered or recommended field rate

3.6.3 Soil activity of herbicides in pot experiments

3.6.3.1 Soil activity of different ACCase-inhibitors in a pot experiment

A pot experiment (pot size diameter. 10.5 cm) was conducted with three different ACCase-inhibitors under greenhouse conditions in August and September 2011. *A. myosuroides* was sown in steam sterilized and also in biologically active silty clay loam. The grass herbicides were applied one day after sowing at pre-emergence. Herbicides used were: fenoxaprop-P at 82,8 g/ha (Ralon Super), clodinafop at 60 g/ha (Topik 100) and pinoxaden at 60 g/ha (Axial EC50) and are presented in Tab. 11. The used rates were equivalent to the maximum registered field rates. The experiment compromised 4 replicates per treatment. The herbicide activity was assessed 35 days after pre-emergence spraying by weighing the upper biomass of the weeds per pot.

Table 11: Herbicides, active ingredients (a.i) and dosages used in a pot experiment on the soil activity of ACCase-inhibitors with steam sterilized biologically active silty clay loam

Herbicide ™	active ingredient (a.i)	Rates (L/ha)	Rates (g.a.i./ha)
Ralon Super	fenoxaprop-P	1.2*	83
Topik100	clodinafop	0.9*	60
Axial 50EC	pinoxaden	1.2*	60

a.i.= active ingredient, EC = Emulsifiable Concentrate, * full registered field dosage

3.6.3.2 Soil activity of post emergence herbicides in an outdoor pot experiment

A pot experiment was carried out from May-June 2010 outdoors in order to investigate the soil activity of different post-emergent herbicides. Seeds of *A. myosuroides* and *A. spica-venti* were sown in plastic pots (diameter 10.5 cm). After sowing a range of ACCase- and ALS-inhibitors were applied pre-emergence. The used dosages were based on recommended field rates. The highest dosages used for clodinafop and pinoxaden were 1/3 higher than the field dosage. Because of high expected soil activity for pyroxsulam and mesosulfuron in pot experiments, the highest rate was 2/3 of the field dosage. The herbicide treatments were: mesosulfuron at 10, 5 and 2.5 g/ha, iodosulfuron at 5, 2.5 and 1.25 g/ha, pyroxsulam at 10, 5 and 2.5 g/ha, propoxycarbazone at 70, 35 and 17.5 g/ha, flupyrsulfuron at 10, 5 and 2.5 g/ha, fenoxaprop-P at 80, 40 and 20 g/ha, clodinafop at 80, 40 and 20 g/ha and pinoxaden at 80, 40 and 20 g/ha. The individual herbicide treatments used in each experiment are shown in Tab. 12 and in the result tables. When the active ingredients were not available as a solo herbicide product, the single a.i. such as mesosulfuron, pyroxsulam and iodosulfuron were applied as formulated active ingredient. After sowing and spraying, the pots were placed outdoors and either irrigated lightly from above using a hose with a sprinkler or watered by natural rainfall to moisten the soil. Each experiment was comprised of 4 replicates per treatment. Herbicide activity was assessed 47 days after pre-emergence spraying by weighing the upper biomass of the weeds.

Table 12: Herbicides, Active ingredients (a.i) and dosages used in an outdoor pot experiment on the soil activity of ACCase- and ALS-inhibitors

No.	active ingredient	Formulation	Rates (g.a.i./ha)		
1-3	mesosulfuron	750 WG	10**	5	2.5
4-6	iodosulfuron	100 WG	5	2.5	1.25
7-9	pyroxsulam	200 WP	10**	5	2.5
10-12	propoxycarbazone	Attribut	70*	35	17.5
13-15	flupyrsulfuron	Lexus	10*	5	2.5
16-18	fenoxaprop-P	Ralon Super	80*	40	20
19-21	clodinafop	Topik100	80***	40	20
22-24	pinoxaden	Axial 50EC	80***	40	20

a.i.=active ingredient, WP= Wettable Powder, EC= Emulsifiable Concentrate, WG= Water-Dispersible Granule, *full registered field dosage, **2/3 of the registered field dosage,***=4/3 the registered field dosage

Climatic conditions during the outdoor pot experiment

The average daily temperature and rainfall during the outdoor experiment are shown in Fig. 4. On the two days after application from May 1st until May 20th the average daily temperatures were exceptionally low, ranging between 6.7-12.9°C. Often, the nights had average temperatures below 8°C with a relatively cold range (4.9-10.5°C) and average daily temperatures usually not exceeding 15°C (ranging between 7-14°C). Except of the period from the 29th-31st of May, after May 21st the average daily temperature usually remained over 15 °C (average daily range 15-25°C), with average night temperatures ranging between 12-18°C. During the experiment there were only a few days without any rainfall. It rained regularly with high amounts of precipitation measured on May 6th (19.8 mm) and June 10th (23.5 mm).

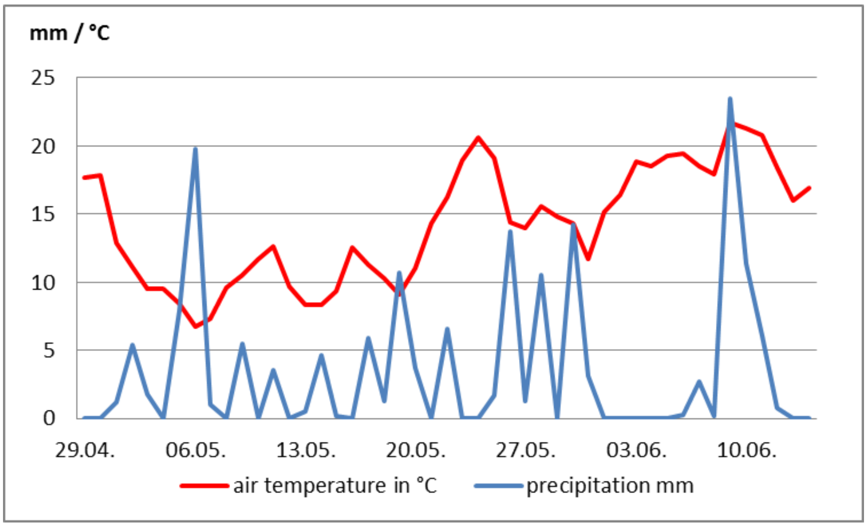

Figure 4: Mean daily air temperature (in °C) and average daily precipitation (in mm) during the outdoor pot experiment from 29.04.-15.06.2010

3.6.3.3 Soil activity of different herbicides at different rates against *A. myosuroides* in a pot experiment under greenhouse conditions

Seeds of *A. myosuroides* were sown in biologically active silt loam and covered lightly with sand. On the same day different herbicides, each at 4-6 different dosages, were applied pre-emergence. Pots (diameter: 13 cm) were watered from above with a sprinkler head. The individual herbicide treatments are shown in Table 13 and in result tables.

Table 13: Herbicide dosages in kg/L/ha in relation to the maximum recommended field rate (F) used in a greenhouse experiment on the soil activity

Herbicide ™	**Dosages in kg/L/ha**					
	F	**3/4F**	**1/2F**	**1/4F**	**1/8F**	**1/16F**
Ralon Super	1.2	0.9	0.6	0.3	-	-
Lexus	0.02	-	0.01	0.005	0.0025	0.00125
Attribut	0.1	-	0.05	0.025	0.0125	0.00625
Cadou SC	-	-	0.125	0.063	0.03125	
	F	**2/3F**	**1/3F**	**1/6F**	**1/12F**	**1/24F**
Topik 100	0.6	0.4	0.2	0.1	-	-
Axial 50 EC	1.2	0.8	0.4	0.2	-	-
Broadway	0.22	0.146	0.073	0.036	0.018	0.009
Atlantis WG	0.5	0.333	0.166	0.083	0.042	0.021

F = Full recommended field dosage; a.i.= active ingredient, Conc = Concentration, a.i.= active ingredient, WP = Wettable Powder, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate

For post emergence herbicides such as ACCase-inhibitors (Ralon Super, Topik and Axial 50EC) and ALS-inhibitors (Atlantis, Broadway and Lexus) the highest dose applied was equivalent to the maximum recommended field rate. For Cadou, half of the maximum field dose was the highest used. The experiment was comprised of 5 replicates per treatment. Herbicide activity was assessed 35 days after pre-emergence spraying by weighing the upper biomass of *A. myosuroides* shoots.

3.6.3.4 Soil activity of different herbicides against *A. myosuroides* in a pot experiment using different watering methods in the greeenhouse

Seeds of *A. myosuroides* were sown in sterilized silt loam and covered with sand. Pots (diameter: 13 cm) were watered from above with a sprinkler head to initiate germination. After 7 days different herbicides were applied pre-emergence. After herbicide application three different irrigation methods were applied. Rainfall was simulated by using a hose with a sprinkler head delivering about 1 mm once a day (9 a.m.) and 1 mm twice a day (9 a.m. and 4 p.m.). Also, sub-irrigation close to field-capacity (FC) was included. The individual herbicide treatments containing ACCase- and ALS-inhibitors used in the experiment are shown in Table 14 and in result tables. These rates were equivalent to the maximum recommended field rates. For Broadway and Atlantis, 1/3 of the recommended field dosage was also included. The experiment was comprised of 5 replicates per treatment. Herbicide activity was assessed 33 days after pre-emergence spraying by weighing the upper biomass (fw) of *A. myosuroides* shoots.

Table 14: Herbicides, active ingredients (a.i) and dosages used in a pot experiment on the soil activity of different herbicides against *A. myosuroides* influenced by different watering methods under greenhouse conditions

Herbicide™	**active ingredient**	**Field rate**	**Dosage L/kg/ha**	**Dosage g a.i./ha**
Ralon Super	fenoxaprop-P	F	1.2	82,8
Topik 100	clodinafop	F	0.6	60
Axial 50EC	pinoxaden	F	1.2	60
Broadway	pyroxsulam+florasulam	F	0.22	15+5
Broadway	pyroxsulam+florasulam	1/3F	0.73	5+1.66
Atlantis WG	mesosulfuron+idosulfuron	F	0.5	15+3
Atlantis WG	mesosulfuron+idosulfuron	1/3F	0.166	5+1
Lexus	flupyrsulfuron	F	0.02	10

F = Full recommended field dosage; a.i. = active ingredient, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule

3.6.3.5 Effect of soil moisture regime, sowing depth and watering type on soil activity of different herbicides against *A. myosuroides* under greenhouse conditions

Different pot experiments were carried out in a greenhouse in which conditions were set with a 14 hour day length to 20/10 °C day/night and a relative humidity of 50%. Seeds of *A. myosuroides* were sown approximately 0.5 cm and 3.5 cm deep in pots (10.5 cm) filled with steam sterilized silt loam. Field capacity (FC) for the used soil was 44g of water per 100g of dry soil. Water regimes were set at 15% FC (6.6g per 100g dry soil), 30% FC (13.2g per 100g dry soil) and 60% FC (26.4g per 100g dry soil) in two ways: either by adding water from above or through sub-irrigation. During the experiments soil moisture levels were controlled regularly by weighing unsown (blank) pots within each water regime. Soil moistures were maintained daily by the addition of water from an automatic irrigation system (NMC-Pro, Netafim), which delivered water ranging from 13 ml to 16 ml per day, depending on the moisture regime and environmental conditions. Water was added to either the soil surface (surface-irrigation) or to the sub-surface zone (sub-irrigation, 7 cm depth) via tube connected plastic drippers (8.5 cm long, 0.7 cm wide) placed in the center of the soil in each pot. Experiments were carried out with 16 herbicide treatments (incl. an untreated check) at two sowing depths (0.5 and 3.5 cm) of *A. myosuroides* and three different water regimes after the herbicide treatment (15% FC, 30% FC and 60% FC) with surface-irrigation, and in another experiment with sub-surface-irrigation at 30% FC. The individual herbicide treatments used in each experiment are shown Tab. 15 and in result tables.The post-emergence herbicides with acetyl Co-A carboxylase (ACCase-) inhibitors at their full recommended field dose were: Ralon Super at 1,2 L/ha (fenoxaprop-P 69 g/l), Topik at 0.6 L/ha (clodinafop 100 g/l) and Axial 50EC at 1.2 L/ha (pinoxaden 50 g/l). Tested acetolactate-syntase (ALS-) inhibitors orientated at full (F) recommended field rates were: Lexus at 0.02 kg/ha (F) (flupyrsulfuron 500 g/kg), Broadway at 0.22 (F), 0.073 (1/3 F) and 0.037 kg/ha (1/6 F) (pyroxsulam 68. 3, florasulam 22.8 g/kg) and Atlantis at 0.5 (F), 0.163 (1/3 F) and 0.083 kg/ha (1/6 F) (mesosulfuron 30 g/kg, iodosulfuron 6 g/kg). Because of their expected high soil activity, Broadway and Atlantis were also applied at 1/3 and a 1/6 of the full rate. For this same reason, Attribut was used with half of the full field dose at 0.05 kg/ha (1/2 F) (propoxycarbazone 700 g/kg). The used pre-emergence herbicides with adjusted rates for the conditions in vessels were: Cadou at 0.25 (1/2 F), 0.125 (1/4 F) and 0.0625 L/ha (1/8 F) (flufenacet 500 g/l) and Stomp aqua at 1098 L/ha (1/4 F) (pendimethalin 455g/l). Herbicide treatments were applied pre-emergence (1 day after sowing). Each experiment comprised of 4 replicates per treatment. Herbicide efficacy was assessed 42 days after application by visual ratings (estimates) and measurement fresh and dry weights of shoot (above ground biomass) and roots.

Table 15: Herbicides, active ingredients (a.i.) and dosages used in a pot experiment in the greenhouse on the soil activity of different herbicides against *A. myosuroides* influenced by different moisture regimes and watering types

Herbicide ™	Active ingredient (a.i)	Rate g a.i./ ha	Rate kg/L/ ha	Field rate
Post-emergence herbicides				
ACCase-inhibitors				
Ralon Super	fenoxaprop-P	82.8	1.2	F
Topik 100	clodinafop	60	0.6	F
Axial 50 EC	pinoxaden	60	1.2	F
ALS-inhibitors				
Broadway	pyroxsulam+florasulam	15+5	0.22	F
Broadway	pyroxsulam+florasulam	5+1.67	0.073	1/3F
Broadway	pyroxsulam+florasulam	2.5+0.84	0.037	1/6F
Atlantis WG	mesosulfuron+iodosulfuron	15+3	0.5	F
Atlantis WG	mesosulfuron+iodosulfuron	5+1	0,163	1/3F
Atlantis WG	mesosulfuron+iodosulfuron	2.5+0.5	0.083	1/6F
Attribut	propoxycarbazone	35	0.05	1/2F
Lexus	flupyrsulfuron	10	0.02	F
Pre-emergence herbicides				
inhibitors of VLCFA-synthase and microtubule assembly				
Cadou SC	flufenacet	62.5	0.25	1/2F
Cadou SC	flufenacet	31.25	0.125	1/4F
Cadou SC	flufenacet	15.63	0.0625	1/8F
Stomp aqua	pendimethalin	500	1099	1/4F

F = Full recommended field dosage; a.i. = active ingredient, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = soluble concentrate, VLCFA = Very-Long-Chain Fatty Acid Synthase,

3.6.4 Soil activity of herbicides in field experiments

3.6.4.1 Experiments on the soil activity of post-emergence herbicides against *A. myosuroides* under field conditions

In order to investigate the soil activity of different post-emergence herbicides against *A. myosuroides* under field conditions, four field experiments were carried out from 2010-2012. Trial locations were 2010/2011 in Wicker-Straßenmühle (STR) and in 2011/2012 in STR, Gießen (GI) and in Wicker-Johannesfeld (JOH). The soil properties and cultivation methods for each experimental site are listed in chapter 3.1 in Tab.2. A regular mechanical sowing machine was used to sow *A. myosuroides* at rate of about 300 seeds/m^2 in Wicker and in GI with about 1000 seeds/m^2 at a sowing depth of 2-2.5 cm. The general info about the trial setup at the different trial locations are summarized in Tab. 16.

Table 16: General information about the trial setup at the different trial locations

Location	Plot size m^2	No. rows	Replicates	Sowing date ALOMY	Sowing date Wheat	Application time
GI 2011/12	4.5	8	3	30.09.11	-	05.10.11
STR 2010/11	2.5	10	2	30.09.10	-	04.10.10
STR 2011/12	2.5	10	2	05.10.11	-	05.10.11
JOH 2011/12	1.8	7/10	4	30.09.11	30.09.11	05.10.11
KEN 2011/12	10	10	4	-	30.09.11	14.10.11

GI = Gießen, STR = Wicker-Straßenmühle, JOH = Wicker-Johannesfeld; KEN = Wicker-Kennel, No. = Number, ALOMY = *A. myosuroides*

In Wicker at the location STR plot sizes were 2.5 m^2 (1.26 x 2 m) in both years and consisted of 10 rows of *A. myosuroides*. Plot sizes in GI were 4.5 m^2 (1.5 x 3 m) and consisted of 8 rows. At JOH plot sizes for *A. myosuroides* were 1.8 m^2 (0.9 x 2 m) and consisted of 7 rows at 12 cm row spacing. In JOH there were also other weeds such as *Lolium multiflorum*, *Bromus secalinus* and as crops 10 rows of *triticum aestivum* (var. JB Asano, 350 seeds/m^2), 10 rows of *Hordeum vulgare* (var. Pelican, 250 seeds/m^2) and 3 rows of *Avena sativa* were sown. In Wicker a block design was used with 3 replicates at STR and 4 replicates per treatment at JOH. The experiment in GI was set up using a randomized block design with 3 replicates per treatment. At STR black-grass was sown on the 30.09.10 in the first year and in the second season on 05.10.11. In GI *A. myosuroides* was sown on 30.09.11. In JOH the sowing date for weeds and winter wheat was 30.09.11.

In STR 10 herbicides were applied on 04.10.10 and in the second year 12 treatments were applied on 05.10.11. The 17 different herbicide treatments at GI and JOH were also applied pre-emergence on 05.10.2011. For each post-emergence herbicide (ACCase-inhibitors: Ralon Super, Topik and Axial; ALS-inhibitors: Broadway, Atlantis, Lexus, Attribut) the full registered field rate was used in all experiments. Only at GI and JOH in 2011/2012 was the double of maximum rate included. Moreover, the combination Lexus+Stomp Aqua and the soil herbicides Cadou and Malibu were included with full registered field rates in all trials. Only in 2011/2012 at STR the soil herbicides Stomp aqua and Boxer were included at full dosages. The individual herbicide treatments used in the experiments are shown in Table 17.

Table 17: Herbicides and dosages used in field experiments on the soil activity against *A. myosuroides* conducted in 2010/2011 at Wicker (STR) and in 2011/2012 in Gießen (GI) and Wicker (JOH, STR, KEN)

Herbicide treatment™	**Rate L/kg/ha**	**Field rate**	**10/11**	**11/12**	**11/12**	**11/12**	**11/12**
			STR	**GI**	**JOH**	**STR**	**KEN**
Ralon Super	2.4	X2		x	X		
Ralon Super	1.2	F	x	x	X	x	x
Topik 100	1.2	X2		x	X		
Topik 100	0.6	F	x	x	X	x	x
Axial 50 EC	2.4	X2		x	X		
Axial 50 EC	1.2	F	x	x	X	x	x
Broadway	0.55	X2		x	X		
Broadway	0.275	F	x	x	X	x	x
Atlantis WG	1	X2		x	X		
Atlantis WG	0.5	F	x	x	X	x	x
Lexus	0.04	X2		x	X		
Lexus	0.02	F	x	x	X	x	
Attribut	0.2	X2		x	X		
Attribut	0.1	F	x	x	X	x	
Lexus+Stomp aqua	0.02 +2.5	F+1/2F	x	x	X	x	
Cadou SC	0.5	F	x	x	X	x	
Mailbu	4	F	x	x	X	x	
Stomp aqua	4.4	F				x	
Boxer	5	F				x	

F = full registered field dose, X2 = double the field dose, a.i. = active ingredient, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, STR = Wicker-Straßenmühle, JOH = Wicker-Johannesfeld; KEN = Wicker-Kennel

3.6.4.2 Field experiments in winter wheat on the soil activity of post-emergence herbicides using natural infestations with *A. myosuroides*

In 2011/12 two field experiments were carried out in Wicker at Johannesfeld (JOH) and Kennel (KEN) in order to investigate the soil activity of different post-emergence herbicides against *A. myosuroides* in winter wheat. The wheat fields were chosen because of their naturally heavy infestation with black-grass. After shallow (ca.10 cm) soil tillage using a chisel plough and seed bed preparation with a rotary harrow, winter wheat was sown at JOH on 30.09.11 (cv. JB Asano) and at KEN (cv. Kerobino) on the 08.10.11. At the location JOH plot sizes were 2.5 m^2 (1.25 x 2 m) and consisted 10 rows of winter wheat. A block design was used with 4 replicates per treatment. The experiment in KEN with plot sizes consisting of 10 m^2 (2 x 5 m) was set up using a randomized block design with 4 replicates per treatment. At JOH 17 different herbicide treatments consisting of ACCase- and ALS-inhibitors with full and double the field dosage, the combination Lexus+Stomp Aqua and the soil herbicides Cadou and Malibu were included with full registered field rates and were applied pre-emergence on 05.10.11. At KEN 5 different herbicide treatments consisting of ACCase- and ALS-inhibitors with full registered field rates were applied pre-emergence on 14.10.11. The individual herbicide treatments used in the experiments are shown in Tab. 17 and in result tables.

Weed populations were assessed by counting *A. myosuroides* plants in the fall or inflorescences in May/June within 5 random 0.1 m^2 quadrats per plot. Wheat density in heads/m^2 was assessed in the same way. Further, herbicide efficacy was assessed by visual ratings (estimates) using a scale of 0 (no visual toxic symptoms on plants) to 100% (plant death) regularly. Because of severe lodging in July 2012, the trials were not harvested for grain yield.

3.6.5 Residual soil activity of post-emergence herbicides against *A. myosuroides*

3.6.5.1 Lab study on the Soil-degradation (DT50) of mesosulfuron, pyroxsulam and pinoxaden in different soils at 10 °C

Sample Preparation

About 5 kg of soil was taken from fields of the experimental sites in Gießen, Rauischholzhausen and Wicker (KEN) and stored under field conditions outside. Before the start of the experiment the soil was adapted to room temperature for a few days. Once equilibrated to room temperature, the soil was sieved through a 2 mm sieve and then adjusted to the required maximum water capacity using deionized water. For the experiment the soil moisture was set to 50% of the maximum water capacity by mixing. For the DT50-test the active ingredients were mesosulfuron-methyl, pyroxsulam, pinoxaden and its active metabolite M2 (NOA407854). A stock solution with a concentration of 1 mg a.i./ml acetonitrile was diluted to the test concentration of 0.1 ppm (= 100 ppb or 100 g a.i./ha). For each herbicide 20 glass bottles were labeled with tare weight. 20 g of soil was weighed into each bottle and the test substances were added by means of a pipette with a volume of 200 µl with 50% methanol. The concentration was calculated to dry soil. Then the glasses were placed into a dark chamber which was set at 10°C. Every 2-3 weeks the weight of the test bottles were readjusted by adding deionized water. After different time intervals treated soil from test bottles was transferred to a centrifugation tube (50 ml) and 20 ml of the extraction solution (50% acetonitrile + water). The tubes were put into a laboratory shaker and were vigorously shaken for an hour. After centrifugation for 10 min at 4000 rpm, an aliquot of approximately 1.5 ml was placed into an ultracentrifugation tube and centrifuged for 5 min at 12000 rpm. Then the supernatant was decanted and collected in a separate tube, and 0.5-1 ml of the supernatant was analyzed by UPLC/TQ-MS (see below). This procedure was repeated 3 more times until the extracted amount of substance was less than 1%. A representative bottle containing treated soil was extracted immediately after application (time interval 0) in order to determine to extraction efficiency. The recovery efficiency was always calculated in relation to a standard solution containing diluted soil extract. After various time intervals (0, 2, 10, 14, 17, 21, 28, 35, 52, 65, 80 and 90 days) one bottle of each compound and each soil was selected randomly from the chamber, the soil extracted and analyzed. At each sampling time the amount of parent was calculated in relation to day 0 and expressed as percentage remaining.

UPLC analysis

In order to quantify the amount of chemicals in the soil, the prepared sample was analyzed using an ACQUITY-UPLC from Waters (Ultra Performance Liquid Chromatography) which was equipped with a Xevo-TQ-MS (Triple Quadruple- Mass Spectrometry). An AcQuity-UPLC BEH C18 (2.1 x 50 mm) column containing silica particles with an average size of 1.7 µm were used for separation of substances. The mobile phase containing different compositions of water and acetonitrile was kept at a flow rate of 0.6 ml/min. The gradient profile for the solvent started at 5% acetonitrile (in water) for 0.01 min, followed by a linear change up to 95% acetonitrile after 0.5 min and back to 5% at 1.1 min (Tab. 18).

Table 18: The gradient profile for the solvent consisting of water and acetonitrile used for UPLC-analyses

Time in min	Water in%	Acetonitrile in%
0	95	5
0.01	95	5
0.5	5	95
1	5	95
1.1	95	5
1.4	95	5

The temperature in the column was 50 °C. A sample of 3 µl was injected for each analysis. After components of the mixture moved through the column, they reached a UV-detector and the TQ-MS for identification and quantification of the certain substance. Components of the injected mixture reached the detector at varying times due to differences in the partitioning between mobile and stationary phases. Before the experiment started, the TQ-MS was tuned for each substance and automatically calibrated in order to detect and quantify the test chemical efficiently. The percentage of individual substance was computed from peak areas. Peak area of each substance was proportional to its number of molecules in the sample.

3.6.5.2 Residual soil activity of post-emergence herbicides against *A. myosuroides* in pot experiments under greenhouse conditions

In order to investigate the residual soil activity of post-emergence herbicides, three pot experiments were conducted under greenhouse conditions.

Experiment 1+2

Two experiments were conducted, one from January until March 2011 and the other from April until June 2011. Different herbicides containing 8 (experiment 1) and 10 (experiment 2) treatments were applied weekly on empty pots (diameter 13) with biologically active standard soil silt loam. Treated pots were irrigated twice a week (Mo and Thu) by using a sprinkler head delivering about 2.5 mm. A rainfall gauge was used in order to measure the amount of simulated rainfall. Four weeks after the first herbicide application *A. myosuroides* was sown in pots, then covered with sand and from there on watered regularly from above. Consequently, time intervals from herbicide application until sowing were: 0 days, 7 days, 14 days, 21 days and 28 days. The experiment contained 5 replicates per treatment. The individual herbicide treatments containing ACCase- and ALS-inhibitors used in the experiment are shown in Table 19 and in result tables. The rates were equivalent to the maximum recommended field rates. Because of the expected high soil activity of Broadway and Atlantis, 1/3 of the full rate was also included. In the second experiment (from April – June 2011) Attribut at half the field dosage (0.05 kg/ha) and Atlantis at 1/6 the dosage (at 0.083 kg/ha) were added to the treatment list. Herbicide activity for both trials was assessed 49 days after sowing by weighing the biomass (fw.) of *A. myosuroides* shoots.

Table 19: Herbicides and dosages used in pot experiments on the residual soil activity of cereal herbicides in 2011 under greenhouse conditions

Herbicide treatment ™	Rate kg/L/ha	Field rate	Exp. 1	Exp. 2	Exp. 3
			Jan-Mar	Apr-Jun	Aug-Oct
Ralon Super	1.2	F	X	x	x
Topik 100	0.6	F	X	x	x
Axial 50 EC	1.2	F	X	x	x
Broadway	0.22	F	X	x	x
Broadway	0.07	1/3F	X	x	x
Atlantis WG	0.5	F	X	x	x
Atlantis WG	0.17	1/3F	X	x	x
Atlantis WG	0.083	1/6F		x	
Lexus	0.02	F	X	x	x
Attribut	0.05	1/2F		x	x

F = full registered field dose, X2= double the field dose, a.i.= active ingredient, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, Exp. = Experiment

Climate conditions during the experiments

Average greenhouse temperatures (day and night) during the four weeks after herbicide applications in Experiment 1 (13.01.11-10.02.11) and Experiment 2 (14.04.-12.05.11) are shown in Figure 5. During the whole period of the first Experiment (13.01.11-29.03.11) temperatures in the greenhouse did not vary much and were on average 18.2°C for the day and 12.7°C for the night. Even though the settings for the greenhouse were the same for the Experiment 2 (14.04.-30.06.11), the average daily temperatures during the four weeks after application varied between 19°C – 26C° for the day (average of 22.5°C) and were relatively high. Also, the average night temperature ranged from 13.3-18.6°C and an average of 15.3°C for the period was relatively warm.

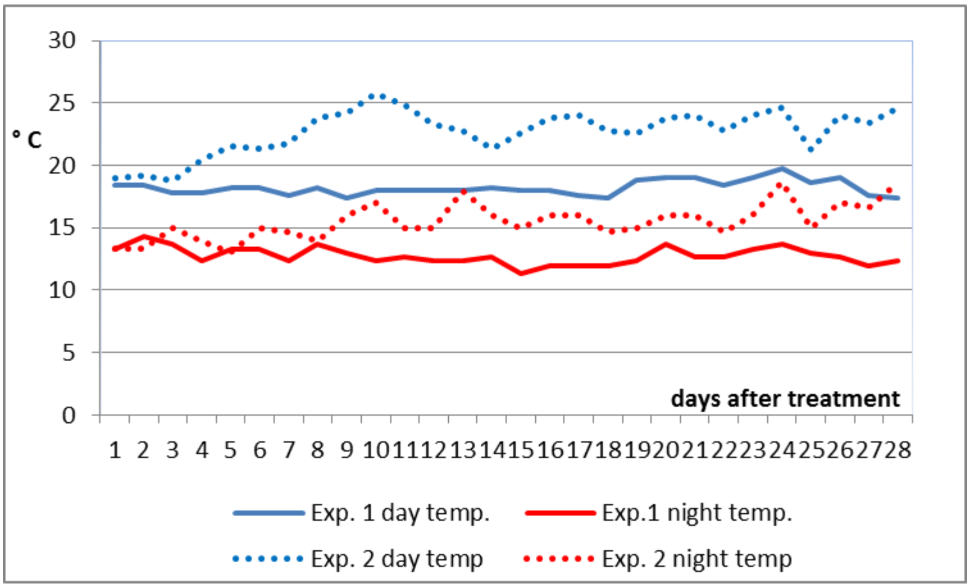

Figure 5: Average greenhouse temperatures (day and night) during the four weeks after herbicide applications in Experiment 1 (13.1.– 10.2.11) and Experiment 2 (14.4.-12.5.11).

Experiment 3

Another greenhouse experiment was conducted from August-October 2011 in order to investigate the influence of irrigation intensity on the residual soil activity of different post-emergence herbicides. Trial set up and proceedings were the same as Experiments 1+2 above. After the application of 9 different herbicides (Tab. 19) on empty pots (diameter 13) with biological active silt loam, treated pots were irrigated differently for a period of four weeks until sowing of *A. myosuroides*. Consequently, the time intervals from herbicide application until sowing were 28 days. According to the experiments above, treated pots were irrigated twice a week (Monday and Thursday) by using a sprinkler head delivering 2.5 mm. Relatively dry soil conditions were achieved by watering 2.5 mm only once a week (Thursday). Relatively wet soil conditions were obtained by watering 1 mm daily. Herbicide activity was assessed 43 days after sowing by weighing the upper biomass of the weeds.

3.6.5.3 Residual soil activity of post-emergence herbicides against *A. myosuroides* under field conditions

In season 2010/2011 and 2011/2012 field experiments were carried out in Wicker at the site Straßenmühle (STR) in order to investigate the residual soil activity of different post-emergence herbicides against *A. myosuroides*. 10 different herbicide treatments in 2010/2011 and 12 different treatments in 2011/2012 were applied on the soil on 04.10.10 and in the second year on 05.10.11. The individual herbicide treatments used in the experiment are shown in Table 20 and in result tables. Treatments consisted of ACCase- and ALS-inhibitors at full registered field dosages, the combination Lexus+Stomp Aqua and the soil herbicides Cadou and Malibu at full registered field rates. In 2011/2012 Boxer and Stomp aqua were also included at full field dosages. In the first year *A. myosuroides* (10 rows) were sown 3 days after application (DAA) (07.10.2010), 28 DAA (01.11.2010), and 168 DAA (21.03.2011) across treated plots using a mechanical sowing machine combined with a shallow rotary harrow. In the second year sowing times *for A. myosuroides* were: 1 day after application (DAA) (06.10.11), 17 DAA (22.10.11), 38 DAA (12.11.11) and 154 DAA (07.03.12). The experiment with effective plot sizes of 2.5 m^2 (2 x 1.25 m) was set up using a block design with 3 replicates per treatment. Herbicide efficacy was assessed by counting *A. myosuroides* plants in the fall and in the spring by using 5 x 0.1m^2 random quadrats per plot and/or by visual ratings (estimates) using a scale of 0 (no visual toxic symptoms on plants) to 100% (plant death).

Table 20: Herbicides and dosages used in field experiments 2010/2011 and 2011/2012 on the residual soil activity against *A. myosuroides* in Wicker (STR)

Herbicide treatment ™	Rate kg//L/ha	Field rate	2010/11	2011/12
Ralon Super	1.2	F	x	x
Topik 100	0.6	F	x	x
Axial 50 EC	1.2	F	x	x
Broadway	0.275	F	x	x
Atlantis WG	0.5	F	x	x
Lexus	0.02	F	x	x
Attribut	0.1	F	x	x
Lexus+Stomp aqua	0.02 +2.5	F+1/2F	x	x
Cadou SC	0.5	F	x	x
Mailbu	4	F	x	x
Cadou SC+Bacara Forte	0.3 +0.75	F	x	
Herold	0.6	F	x	
Stomp aqua	4.4	F		x
Boxer	5	F		x

F = full registered field dose, X2 = double the field dose, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, Wicker-STR = at the site Straßenmühle

3.7 Management of *A. myosuroides*

3.7.1 Greenhouse experiments

3.7.1.1 Effect of application timing on the herbicidal efficacy of ALS- and ACCase-inhibitors against different populations of *A. myosuroides* in pot experiments

A pot experiment was carried out from November 2011 until January 2012 in a greenhouse. Seeds of *A. myosuroides* from the standard sensitive population without known herbicide resistances, the population in Rauischholzhausen with enhanced metabolic resistances for ACCase and ALS-inhibitors and established Targetsite-resistance for ACCase-inhibitors (2041), and a population from England (GB) with very enhanced metabolism for ACCase and ALS-inhibitors and Target site-resistances (ALS 197, ACCase 1781, 2041) were used. Ripe seeds were collected from the field trial site in Rauischholzhausen from untreated plots, then dried, cleaned and stored at room temperature (20-22 °C). The population GB was stored at 5°C at Bayer facilities since harvest in 2010. The results from resistance analyses for the populations in Rauischholzhausen and GB conducted by the Bayer CropScience Resistance laboratory are summarized in Tab. 21 and Tab. 22 and are described below.

The analysis for Target Site Resistance (TSR) of collected *A. myosuroides* was conducted using the Pyrosequencing™ technology (Nordstrom et al. 2000) of known target site gene sequence for ACCase- and ALS-inhibitors as described by Wagner et al. (2007) and Beffa et al. (2012). Results are given as mean percentage of allele frequencies. In order to detect enhanced detoxification of herbicides (Enhanced Metabolic Resistance, EMR) two tillers of each black-grass plant were treated with ^{14}C-radiolabelled compounds following the description by Ruiz-Santaella et al. (2010). Extracts of the plant samples were analyzed for the remaining substances after a certain time using HPLC analysis.

Table 21: Target Site Resistances (TSR) of *A. myosuroides* plants (in %) taken from Rauischholzhausen in the years 2010-2011

Target Site Resistance				**ACCase** I2041N	**ACCase** I1781	**ALS** P197
Location	**Year**	**plot**	**n**			
RH	2010	Ralon Super	8	100%	-	-
RH	2011	Broadway	8	86%	-	-
RH	2012	untreated	24	83%	-	-
GB	2010	-	16	25%	13%	13%

RH = Rauischholzhausen, GB = England (internal standard)

Table 22: Metabolic activity of *A. myosuroides* plants taken from Rauischholzhausen in different years from 2010-2011 compared to an internal standard (GB)

Metabolic activity for				**fenoxaprop-P**			**mesosulfuron**		
Location	**Year**	**plot**	**n**	**no**	**intermediate**	**high**	**no**	**intermediate**	**high**
RH	2010	Ralon Super	8	0%	0%	100%	-	-	-
RH	2011	Broadway	8	0%	29%	71%	0%	86%	14%
RH	2012	untreated	22	-	-	-	63%	32%	5%
RH	2011	untreated	8	-	-	-	36%	50%	14%
GB	2010	-	16	-	-	-	0%	14%	86%

RH = Rauischholzhausen, GB = England (internal standard), n = number of analysed plants

At two different timings (19.12.11 and 02.01.11) seeds of *A. myosuroides* from the three different populations were sown in steam sterilized silt loam and water was added from above to initiate germination. Seeds were sown using a sowing spoon delivering the volume of 50-55 seeds/pot.

The herbicides containing ALS-inhibitors used were: Atlantis, Broadway, Lexus, and Attribut. ACCase-inhibitors used were: Ralon Super, Topik and Axial. Herbicides were applied with their maximum recommended field dose. Because of expected herbicide resistances, Atlantis and Broadway were also applied with double (x2) the field rate and also 3 lower rates (4/5, 3/5, 2/5 of the field rate) were included. The growing stages of *A. myosuroides* at application were at 12 leaf-stage (BBCH 12) and the stage of tillering (BBCH 23). The individual herbicide treatments used in this greenhouse experiment are shown in Table 23 and in result tables. The pot experiment compromised of 4 replicates per treatment. Plants of *A. myosuroides* were harvested for measurement of fresh shoot weights 42 days after application.

Table 23: Herbicides, active ingredients (a.i) used in the experiment on the effect of different application timings (BBCH 12 and 23) on the herbicidal efficacy of ALS- and ACCase-inhibitors against different populations of *A. myosuroides* in pot experiments

Herbicide treatment ™	Active ingredient (a.i.)	Rate kg/L/ha	Rate a.i. g/ha	Field rate
Ralon Super+Monfast	fenoxaprop-P	1,2+0,4	83	F
Topik 100	clodinafop	0,6	60	F
Axial 50 EC	pinoxaden	1,2	60	F
Broadway+NM	pyroxsulam+flo.	0,44+1	30+10	FX2
Broadway+NM	pyroxsulam+flo.	0,22+1	15+5	F
Broadway+NM	pyroxsulam+flo.	0,18+0,8	12+4	4/5 F
Broadway+NM	pyroxsulam+flo.	0,13+0,6	9+3	3/5F
Broadway+NM	pyroxsulam+flo.	0,09+0,4	6+2	2/5F
Atlantis WG+FHS	mesosulfuron+iodo.	1+1	30+6	FX2
Atlantis WG+FHS	mesosulfuron+iodo.	0,5+1	15+3	F
Atlantis WG+FHS	mesosulfuron+iodo.	0,4+0,8	12+2,4	4/5 F
Atlantis WG+FHS	mesosulfuron+iodo.	0,3+0,6	9+1,8	3/5F
Atlantis WG+FHS	mesosulfuron+iodo.	0,2+0,4	6+1,2	2/5F
Attribut+Mero	propoxycarbazone	0,1+1	70	F
Lexus+Trend 90	flupyrsulfuron	0,02+0,3	10	F

EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, flo = florasulam; iodo.= iodosulfuron, F = full field dosage, a.i.= active ingredient, FHS= Biopower, NM = Broadway Netzmittel

3.7.1.2 Effect of application timing on the herbicidal efficacy of pre-emergence herbicides (flufenacet, pendimethalin) against different populations of *A. myosuroides* in pot experiments

A pot experiment was carried out from November 2011 until January 2012 in a greenhouse. Ripe seeds of *A. myosuroides* were collected from the field trial site in Rauischholzhausen from untreated plots, then dried, cleaned and stored at room temperature (20-22 °C). Seeds of *A. myosuroides* from the standard population and the population in Rauischholzhausen were used. The resistance profile for the population from Rauischholzhausen from the laboratory is shown in Tab. 21 and Tab. 22. At three different timings (25.11, 30.11 and 09.12.2011) seeds of *A. myosuroides* from all populations were sown in plastic pots (10.5 cm) and water was added from above to initiate germination. The soil acting herbicides Cadou SC (flufenacet 500 g/l) and Stomp aqua (pendimethalin 455 g/L) were applied at three different rates on the last sowing day. Because of high expected soil activity in pot experiments the highest used rate for Cadou SC was half and Stomp aqua 1/4 of the full field dosage. The growing stages of *A. myosuroides* at application were at 1 leaf-stage (BBCH 11), at seed germination (BBCH 09/10) and pre-emergence (BBCH 00). The individual herbicide treatments used in this greenhouse experiment are shown in Table 24 and in result tables. After herbicide application pots were watered from above for one week and then later by sub-irrigation. The pot experiment comprised of 4 replicates per treatment. Plants of *A. myosuroides* were harvested for measurement of fresh shoot weights 38 days after application.

Table 24: Herbicides, active ingredients (a.i) used in the pot experiment on the effect of three different application timings (BBCH 00, 10 and 11) on the herbicidal efficacy of pre-emergence herbicides against different populations of *A. myosuroides*

Herbicide treatment ™	**a.i. in g/L**	**Rate kg/L/ha**	**Rate a.i. g/ha**	**Field rate**
Cadou SC	flufenacet 500	0,125	31,25	1/2F
Cadou SC	flufenacet 500	0,0625	15,63	1/4F
Cadou SC	flufenacet 500	0,03125	7,81	1/8F
Stomp aqua	pendimethalin 455	1,099	500	1/4F
Stomp aqua	pendimethalin 455	0,549	250	1/8F
Stomp aqua	pendimethalin 455	0,275	125	1/16

a.i.= active ingredient, , SC = soluble Concentrate, F = full field dosage

3.7.2 Field experiments on black-grass control in winter-wheat

From 2009-2012 field experiments with winter wheat were conducted at the research stations in Gießen (GI), Wicker (WI-KEN) and Rauischholzhausen (RH). The locations were chosen because of their regular naturally high levels of infestation with *Alopecurus myosuroides* (300–1000 pl./m^2). In all seasons (2009/2010, 2010/2011 and 2011/2012) trials were carried out in Rauischholzhausen (RH), whereas results from location GI are only presented from 2009/2010 due to the lack of weed pressure in those years. In WI trials were carried out in 2009/10 and 2010/11. Field trial plot sizes were 1.5 m x 9 m at GI and 1.25 m x 8 m at RH and consisted of 8 rows. In WI plot sizes were 2 x 5 m. The experiments were set up using a randomized block design with four replicates. For cultivations and sowing dates individual blocks were used and analysed separately from each other. In order to avoid edge effects, control plots between treatments were used in Wicker and Gießen, whereas in Rauischholzhausen special drift shields were used during application. Dicotyledonous weeds were controlled by applying Starane XL (100 g/L fluroxypyr + 2.5 g/L florasulam) or Primus (50 g/L florasulam) in the spring and appropriate fungicides were applied in April. While the population in GI and WI were still sensitive to herbicides, collected plants from RH showed strong Enhanced Metabolic Resistance (EMR) for fenoxaprop-P-ethyl and Target-Site- Resistance (TSR, I2041N) against ACCase-inhibitors in lab analyses conducted by Bayer CropScience. In 2010 and 2011 a slightly higher metabolism for mesosulfuron-methyl (EMR for ALS-inhibitors) was also detectable. Results are shown in Chapter 3.7.1.1 in Tab. 21 and Tab. 22.

Experiments 2009/11

In 2009/10 experiments were carried out in GI and RH with 11 herbicide treatments (incl. untreated) at an early (20.-30.09) and a late (15.10.-05.11.) sowing timing of winter wheat. In WI with the same treatment list for the fall, was sown at a normal time on 13.10.2009 at 350 seeds/m^2 (cv. Kerobino) after chisel ploughing (approx. 10 cm). In GI winter wheat was sown at 300 seeds/m^2 (cv. Nirvana) after ploughing (25-30 cm) on 30.09.2009 and at 330 seed/m^2 (cv. JB Assano) on 14.10.2009. In RH winter wheat (cv. Tommi) was sown after chisel ploughing (approx. 10 cm) at 250 seeds/m^2 on 22.09.2009 and at 350 seeds/m^2 on 03.11.2009.

The individual herbicide treatments, mixtures and sequences used in each experiment are shown in Table 25 and can also be found in result tables. The used rates are equivalent to field recommended rates. The herbicide Broadway was applied in autumn, although its use is only registered for spring applications in Germany. Nevertheless, there are products containing pyroxsulam which are already registered for autumn in England, which is meanwhile also expected in Germany in 2014. In addition, recommended adjuvants for each herbicide were included.

At the early sowing time, the treatments were sprayed first using the soil acting herbicides Cadou SC and Herold SC during pre/early post emergence (BBCH 09-11) of the wheat (GI: on 21.09.2009 at BBCH 09-11, RH: on 30.09.2009 at BBCH 09/10 and WI: on 20.10.2009). At the 2-3 leaf stage (GI: 27.10.2009, RH: 21.10.2009, 25.11.2009) herbicides containing ACCase- and ALS-inhibitors were applied. The late sown winter wheat in GI and RH was treated in the spring during the beginning of vegetation (GI: 08.04.2010, RH: 24.03.2010). Because of confirmed herbicide resistance in RH, alternative herbicides as shown in Table 25 were used in the spring of 2010 and the following years.

Experiments 2010/2011

In the field experiment at RH in 2010/2011 14 treatments (applications at BBCH 09-11 on 05.10.2010 and at BBCH 11-12 on 14.10.2010) were used for the early sowed wheat and 12 treatments (at BBCH 12-23 on 23.03.2011) for the late sowed wheat (Table 25). Additionally, sequence applications with applied soil-herbicide in autumn (Herold SC 0.6 L/ha) followed by different ALS-Inhibitor applications in the spring were included. The 2010/2011 sowing dates in RH were 22.09.2010 and 28.10.2010. Also, a trial with early sown winter wheat after ploughing cultivation (25-30 cm) was added in the 2010/11 experiments at RH.

The field trial in WI, which was sown on 07.10.10, the pre-emergence herbicides Malibu (4L/ha), Cadou SC+Bacara forte (0.3+0.5 L/ha), Lexus (0.02 kg/ha) and Lexus+Stomp (0.02+2.5 L/ha) complemented the treatment list and were applied on 26.10.10. Post emergence applications were conducted on 16.11.10. Because of severe crop injuries due to long drought periods during April and May, the trial was cancelled.

Field experiments 2011/2012

In 2011/2012, 14 herbicide treatments (at BBCH 09 on 05.10.2011 and at BBCH 11-12 on 25.10.2011) were applied on early sown winter wheat after chisel ploughing and also after ploughing (Table 25). In 2011/12 wheat was sown on 29.09.2011 and 26.10.2011. The trials in RH were terminated in the spring due to severe crop damage during heavy frost periods in the winter (see chapter 3.1).

Table 25: Dosage and timings of herbicide applications in field experiments with winter wheat in Gießen and Rauischholzhausen 2009 – 2011

Herbicides™ and adjuvants	**Rate L/kg/ha**	**2010**					**2011**				**2012**	
		GI		**RH**		**WI**	**RH**			**WI**	**RH**	**WI**
		ES	LS	ES	LS	NS	ES	ES	LS		ES	NS
		(p)	(p)	(cp)	(cp)	(cp)	(p)	(cp)	(cp)	(cp)	(p)	(cp)
Application BBCH 12-13												
Ralon S.+Monfast	1.2+0.4	x	x	x		x				x		
Topik 100	0.6	x	x	x		x				x		
Axial 50EC	0.9/1.2	x	x	x	x	x				x		
Broadway+NM.	0.22+1	x	x	x	x	x	x	x	x	x	x	x
Broadway+NM.	0.275+1				x				x			
Atlantis WG+FHS	0.5+1.0				x				x		x	x
Atlantis WG+FHS	0.4+0,8	x	x	x	x	x	x	x	x	x	x	x
Atlantis WG+FHS	0.3+0.6				x				x			
Alister	1.0	x	x	x	x	x	x	x	x	x	x	x
Ralon S.+Topik	1.0+0.4	x	x	x		x				x		
Ralon S.+Axial	1.0+0.9	x	x	x		x				x		
Lexus+Trend 90	0.02+0.4				x		x	x	x		x	x
Lexus+Stomp aqua	0.02+2.5						x	x			x	x
Atlantis WG+Herold	0.4+0.6						x	x			x	x
Attribut+Mero	0.06+1				x				x			
Attribut+Mero	0.1+1				x				x			
Application BBCH 09-11												
Herold SC	0.6	x	x	x		x	x	x		x	x	x
Cadou SC	0.5	x	x	x		x	x	x		x	x	x
Bacara F.+Cadou	0.75+0.3						x	x			x	x
Malibu	4.0						x	x			x	x
Stomp aqua+IPU	2.5+2.5						x	x			x	x
Fenikan+IPU	2+1						x	x				
Malibu+Lexus+ Boxer	4+0.02+2										x	x

ES = early sowing, LS=late sowing, NS= normal sowing time p = mouldboard ploughing, cp = chisel ploughing, GI= Gießen, RH= Rauischholzhausen, NM = Broadway Netzmittel, FHS = Biopower, IPU= isoproturon, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate

3.8 Study parameters

Parameters in pot experiments

Herbicide efficacy was assessed 4-7 weeks after application by visual ratings (estimates) using a scale of 0 (no visual toxic symptoms on plants) to 100% (plant death). At the time of assessments plants in untreated pots had 3-5 tillers (BBCH 23-25). Further, plants were harvested for measurement of fresh shoot (above ground biomass) 21-49 days after application (DAA). For one pot experiment root weights were additionally taken. Before, the roots were washed using a sprinkler head and then dried off with a paper towel. Before the measurement of dry weights harvested plant material was dried at 60 °C for at least 5 days.

Parameters in field

Herbicide efficacy was assessed regularly (autumn and spring) by visual ratings (estimates) using a scale of 0 (no visual toxic symptoms on plants) to 100% (plant death) comparing to the untreated plots. Moreover, herbicidal efficacy was assessed by counting surviving *A. myosuroides* plants in the fall or early spring using 5 x 0.1m^2 random quadrats per plot. For experiments conducted in winter wheat, inflorescences (heads) of *A. myosuroides* and winter wheat were counted in May or June. Wheat yields were determined by harvesting with a small-plot combine (grain moisture: 14%).

3.9 Statistical Analysis

For field data the statistical software package PIAF Stat (Schmidtke and Voit 2001) (Programm Information Auswertung Feldversuche), a Program for Statistical Evaluation of Field Trials, was used to perform analyses of variance for checking the significance of different treatments (p = 5%). Multiple t-tests (LSD = Least significance difference) were used to differentiate between means at $p < 0.05$. For all received data from pot experiments an ANOVA was conducted. Multiple t-tests (LSD) or Tukey's HSD (Honestly Significant Difference) test were conducted using the p = 0.05 level of significance. Statistical analyses were conducted using SPSS 19.0. A for Bayer customized Program called “Activity Base” was used to analyze received data from lab analyses (UPLC) for the DT50 values. Different graphs (half-life curves) were automatically generated by the program and the curve with the best kinetic fit, based on Chi-square and r^2, were chosen.

4 Results

4.1 Emergence pattern of black-grass (*Alopercurus myosuroides*) during the growing seasons 2009/10, 2010/11 and 2011/12

Growing season 2009/10

In Rauischholzhausen (RH), with minimum tillage cultivation (chisel ploughing), *A. myosuroides* emerged to about 75% of the total emergence during the four week period between sowing (22.09.09) and the application of post-emergence herbicides, including glyphosate (Tab. 26). Further, slight emergence of black-grass individuals occurred in November and had its peak in December of 2009 (11%), followed by continuous emergence of *A. myosuroides* individuals from March until the beginning of May 2010. The site with the late sowing of winter wheat (03.11.2009) showed a similar emergence pattern for *A. myosuroides*, but less extensive. About 78% of the total *A. myosuroides* emergence was completed by the spring application (24.03.10) and newly emerged seedlings (54, 20 and 23 plants/m^2) were still observed until the end of May. The total infestation was reduced by over 90% with a later sowing time. On the field in Gießen (GI), where conventional mouldboard ploughing was used, weed infestation with *A. myosuroides* was similar to RH. In contrast, almost 100% of the total emergence of *A. myosuroides* occurred during the 3 week period from sowing (30.09.2009) until the application of post-emergence herbicides (27.10.2009). There were no later flushes of emergence in the fall and practically no freshly emerged black-grass individuals observed in the spring. Also, the late sowing of winter wheat, which reduced the total infestation by over 90% compared to the early sowing, did not show any late emerging *A. myosuroides* after the spring application. At Wicker (Wi-JOH), where winter wheat was sown on 13.10.2009 after minimum tillage cultivation (chisel ploughing), most plants were emerged by the time of the fall application (25.11.09). Moreover, while no *A. myosuroides* emerged during the fall, another peak emergence (30 plants/m^2, 13%) was observed in the spring.

Table 26: Germination of *A. myosuroides* individuals (per m^2) in early and late sown winter wheat during the field season 2009/10 in Rauischholzhausen (RH), Gießen and Wicker-Johannesfeld (WI-JOH)

Location	**ALOMY**	**Dates**							
		21.10	27.11.	14.12.	24.03.	07.04.	05.05.	25.05.	28.06.
RH (ES,MIN)	pl/m^2	**990***	25	140	58	50	50	0	0
	% of toal	75%	2%	11%	4%	4%	4%	0%	0%
RH (LS, MIN)	pl/m^2				**336***	54	20	23	0
	% of toal				78%	12%	5%	5%	0%
		27.10	27.11.	14.12.	24.03.	23.04.	05.05.	25.05.	28.06.
Gießen (ES, PL)	pl/m^2	**975***	0	0	0	1	4	0	0
	% of toal	99%	0%	0%	0%	0%	0%	0%	0%
Gießen (LS, PL)	pl/m^2				**39***	0	0	0	0
	% of toal				100%	0%	0%	0%	0%
			25.11	15.12.	26.03.	20.04	11.05.	15.06	
Wicker (NS,MIN)	pl/m^2		**199***	0	2	30	3	0	
	% of toal		85%	0%	1%	13%	1%	0%	

Counted individuals were newly emerged (BBCH 11-12) * = application of post-emergence herbicides incl.Round up ultramax (2.4 L/ha), pl = plants, ALOMY = *A. myosuroides*, ES = early sowing of winter wheat, LS = late sowing time of winter wheat, MIN= chisel ploughing, PL = mouldboard ploughing,

Growing season 2010/11

In the second year (2010/11) the emergence of *A. myosuroides* in the wheat field in RH followed a similar, though less extensive pattern (Tab. 27). Peak emergence (by about 80%) occurred after sowing (22.09.10) followed by continuous emergence with a peak in November 2010 (14%), zero emergence until March and another peak (5%) in April through the beginning of May. In comparison, total emerged individuals of *A. myosuroides* were reduced by around 90% by using the plough. Further, there was close to zero emergence in late autumn and no emergence observable in the spring of 2011. The late sowing of winter wheat (28.10.10) combined with a herbicide application in the spring did not result in high weed pressure and thus not many late emerging weeds.

Table 27: Germination of *A. myosuroides* individuals (per m^2) in early and late sown winter wheat during the field season 2010/11 in Rauischholzhausen (RH)

Location	ALOMY	Dates							
		14.10.	05.11.	17.11.	25.1.	23.3.	20.4.	3.5.	30.5.
RH (ES, MIN)	pl/m^2	**347***	28	37	**0***	0	23	3	0
	% of toal	79%	6%	8%	0%	0%	5%	1%	0%
RH (ES, PL)	pl/m^2	**37***	1	6	**0***	0	0	0	0
	% of toal	84%	2%	14%	0%	0%	0%	0%	0%
RH (LS, MIN)	pl/m^2					**46***	4	1	0
	% of toal					90%	8%	2%	0%

Counted individuals were newly emerged (BBCH 11-12),*= application of Round up ultramax (2.4 L/ha), ALOMY = *A. myosuroides*, pl = plants, ES = early sowing of winter wheat, LS = late sowing time of winter wheat, MIN = chisel ploughing, PL = mouldboard ploughing

Growing season 2011/12

In the third year (2011/12) the emergence of *A. myosuroides* at RH followed a similar emergence pattern as in the previous years (Tab. 28). Peak emergence occurred (by about 90%) during the period after sowing (29.09.11), followed by continuous emergence through November 2011 (9%), zero emergence until the end of February and another relatively small number in April. In comparison, total emerged individuals of *A. myosuroides* were reduced by over 80% by ploughing on the same field. During the period from herbicide application (25.10.11) until the end of November 2011 there was still continuous emergence observable and little emergence April 2012. The late sowing of winter wheat (26.10.10) after chisel ploughing reduced the black-grass infestation by over 90% compared to early sowing, resulting in relatively low black-grass pressure in April. There was no observation data available beyond that.

Observations on other regular farm fields in Rauischholzhausen (RH-farm), Trebur, and Wicker (KEN and JOH), with early sown winter wheat after chisel ploughing, showed that the peak emergence of black-grass occurred after sowing (78-93%). At all locations continuous emerging of *A. myosuroides* occurred for 5-6 weeks after post emergence herbicide application resulting in freshly emerged seedlings ranging from 6 (Wicker-KEN) to 64 plants/m^2 (RH-farm). At every location, *A. myosuroides* plants emerged again in the spring resulting in 2 (Wicker-KEN) to 39 (Wicker-JOH) freshly emerged black-grass individuals per m^2 in April.

Table 28: Germination of *A. myosuroides* individuals (per m^2;% of total emergence) in early and late sown winter wheat during the field season 2011/12 in Rauischholzhausen (RH) and other farm fields in Rauischholzhausen (RH-farm), Trebur and Wicker-Kennel and Wicker-Johannesfeld (WI-KEN and WI-JOH)

Location	ALOMY	Dates			
RH (ES, MIN)		25.10.	29.11	28.02.	19.04.
	plants/ m^2	**779***	77	0	6
	% of total	90%	9%	0%	1%
RH (ES, PL)		25.10.	29,11	28.02.	19.04.
	plants/ m^2	**136***	44	0	5
	% of total	74%	24%	0%	3%
RH (LS, MIN)		25.10.	29.11	28.02.	19.04.
	plants/ m^2	-	-	-	58
	% of total				100%
RH-farm (ES, MIN)		25.10.	29.11	28.02.	19.04.
	plants/ m^2	**648***	64	0	10
	% of total	90%	9%	0%	1%
Trebur (ES, MIN)		05.10.	28.11	13.12.	24.04.
	plants/ m^2	**620***	0	33	17
	% of total	93%	0%	5%	3%
Wicker-KEN (ES, MIN)		28.10.	15.11	13.12.	24.04.
	plants/ m^2	**36***	2	6	2
	% of total	78%	4%	13%	4%
Wicker-JOH (ES, MIN)		05.10.	28.11	13.12.	24.04.
	plants/ m^2	**314***	0	12	39
	% of total	86%	0%	3%	11%

Counted individuals were newly emerged (BBCH 11-12) *= application of Roundup ultramax at 2.4 L/ha, ALOMY = *A. myosuroides*, pl = plants, ES = early sowing, LS = late sowing of winter wheat, MIN = chisel ploughing, sowing dates for winter wheat at RH-farm on 30.09.2011, Trebur on 06.10.2011, Wicker-KEN on 08.10.11, Wicker-JOH on 30.09.11

External data in 2011/2012

The data received from Bayer CropScience Deutschland GmbH (BCS-D) (Tab. 29) confirm that peak emergence of *A. myosuroides* occurs during the 3-5 week period from sowing until the application of post-emergence herbicides in the fall (88-99%). At three locations continuous emergence of black-grass occurred through November, resulting in a large number of freshly emerged seedlings 3 weeks after application ranging from 4 (Grevenkop) to 34 plants/m^2 (Musdorf). While no further data are available for Grevenkop and Musdorf, little emergence was observed continuously through June at Möhnesee-Westrich. At Möckmühl no further emergence occurred during the winter, whereas a relatively large number of plants were counted in June 2012.

Table 29: Germination of black-grass individuals (per m^2;% of total emergence) in monitoring studies conducted by BCS-D in winter wheat during the field season 2011/2012 on different farm fields within Germany after chisel ploughing (MINTIL)

Location	**ALOMY**	**Dates**			
Möckmühl/Heilbronn		27.10.	28,11	15.03.	6.06.
	plants/ m^2	408*	0	0	55
	% of total emergence	88%	0%	0%	12%
Musdorf/ Schwäbisch Hall		28.10.	18.11.	-	-
	plants/ m^2	437*	34	-	-
	% of total emergence	93%	7%		
Möhnesee Westrich/Soest		2.11.	21.11.	2.04.	8.06.
	plants/ m^2	156*	16	2	2
	% of total emergence	89%	9%	1%	1%
Grevenkop/Marsch		2.11.	01.12.	-	-
	plants/ m^2	287*	4	-	-
	% of total emergence	99%	1%		

Counted were newly emerged: Individuals in BBCH 11-12, * = application of Roundup ultramax (2,4 L/ha), ALOMY = *A. myosuroides*, Sowing dates for winter wheat: Möckmühl on 03.10.11; Musdorf on 30.09.11, Möhnesee on 29.09.11, Grevenkop on 29.09.11.

Soil depth of emerged black-grass plants after ploughing and chisel ploughing

In Rauischholzhausen in 2011/12 a total 20plants were dug out in order to measure the length from the seedling to the mesokotyl. Plants were taken from a field with early sown winter wheat (29.09.2011) where ploughing and chisel ploughing cultivation was conducted next to each other. The average emerging depth of black-grass was 4.0 cm after chisel ploughing compared to 4.5 cm after mouldboard ploughing (Table 30). Comparing the emergence depths of 20 already emerged plants of black-grass from ploughed and tilled soil, the percentage of plants emerged in the top soil layers were the same (< 2.5 cm). Differences occurred mainly in the depth of 2.5 to 5 cm, where a much higher proportion of seedlings emerged after chisel ploughing compared to ploughing. Further, after ploughing cultivation a relatively high percentage of over 45% of black-grass plants emerged from a depth below 5 cm.

Table 30: Depth of emerged plants of *A. myosuroides* (number and %) after chisel ploughing and mouldboard ploughing cultivation in Rauischholzhausen in 2011/2012 at the time of the herbicide application

	ploughing cultivation		**chisel ploughing**	
mean (n=20)	**4.5 cm**		**4.0 cm**	
Depth in cm	**No.**	**%**	**No.**	**%**
< 2.5	5	25	5	25
2.5 to 5.0	6	30	11	55
5 to 7.5	6	30	4	20
>7.5	3	15	-	-
total	20	100	20	100

No.: number of plants

4.2 Experiments on the soil activity of herbicides against *A. myosuroides*

4.2.1 Effect of different herbicides in aqueous solution on root growth and plant vitality of *A. myosuroides* plants

The effect of different herbicides at different dosages on *A. myosuroides* plants (root growth and vitality) whose roots were exposed to herbicides in aqueous solution are shown in Table 31. Roots of *A. myosuorides* plants which were exposed to plain water (untreated) grew on average 10.9 cm after 14 days. All herbicide treatments inhibited root growth of *A. myosuroides*, significantly ranging from 55% (fenoxaprop 0.001) to 100% (fenoxaprop, clodinafop, pinoxaden, pyroxsulam and flupyrsulfuron at 0.1 and 0.03 mg/L and propoxycarbazone at 0.1, 0.03 and 0.01 mg/L). Roots exposed to fenoxaprop (0.003-0.1 mg/L, clodinafop (0.01-0.1 mg/L), pinoxaden (0.001-0.1 mg/L), mesosulfuron (0.003-0.1 mg/l, pyroxsulam (0.001-0.1 mg/L), flupyrsulfuron (0.003-0.1 mg/L) and propoxycarbazone (0.001-0.1 mg/L) showed the highest root growth inhibition ranging from 78%-100% and did not differ significantly from each other.

Further, these treatments often resulted in visible plant death. At the lowest concentration (0.001 mg/L) fenoxaprop, clodinafop, mesosulfuron and flupyrsulfuron showed significantly lower root growth inhibitions compared to the higher concentration (0.001 mg/L). In contrast, growth inhibitions caused by propoxycarbazone and pyroxsulam were statistically the same at all concentrations. Healthy looking shoots were observed with fenoxaprop (0.001-0.03), clodinafop (0.001-0.01), pinoxaden (0.001-0.01), mesosulfuron (0.001-0.01), pyroxsulam (0.001), flupyrsulfuron (0.001), and propoxycarbazone (0.001) even though significant root inhibitions of up to 100% (fenoxaprop 0.03) were observed.

Table 31: Effect of different herbicides in aqueous solution on root growth and plant vitality of *A. myosuroides* after 14 days exposure

Active ingredient	Conc. ppm (mg/L)	Root growth (cm)	Root growth Inhibition (%)	Shoot rating
fenoxaprop-P	0.1	0 a	100	dead
	0.03	0 a	100	alive
	0.01	0,4 ab	97	alive
	0.003	1.7 abcd	85	alive
	0.001	4.9 e	55	alive
clodinafop	0.1	0 a	100	dead
	0.03	0 a	100	dead
	0.01	2.3 abcd	78	alive
	0.003	2.9 cde	73	alive
	0.001	3 de	73	alive
pinoxaden	0.1	0 a	100	dead
	0.03	0 a	100	dead
	0.01	0.3 a	97	alive
	0.003	1.4 abcd	87	alive
	0.001	1.7 abcd	85	alive
mesosulfuron	0.1	0.4 ab	96	dead
	0.03	0.6 abc	95	dead
	0.01	0.8 abcd	92	dead/alive
	0.003	2.4 abcd	79	dead/alive
	0.001	2.9 cde	74	alive
pyroxsulam	0.1	0 a	100	dead
	0.03	0 a	100	dead
	0.01	0.15 a	99	dead/alive
	0.003	0.2 a	98	dead
	0.001	1.4 abcd	87	alive
flupyrsulfuron	0.1	0 a	100	dead
	0.03	0 a	100	dead
	0.01	0.2 a	98	dead
	0.003	0.1 a	99	dead
	0.001	2.7 bcde	75	alive
propoxycarbazone	0.1	0 a	100	dead
	0.03	0 a	100	dead
	0.01	0 a	100	dead
	0.003	0.3 a	98	dead
	0.001	1.9 abcd	83	alive
untreated		10.9 f	0	alive

Values in column followed by different letters are significantly different ($p = 0.05$) according to Tukey's HSD test, HSD = 2.39 cm; alive = green and healthy looking shoot, dead = leaves showing toxic symptoms (chlorosis, necrosis), Conc. = Concentration, ppm = parts per million

4.2.2 Effect of herbicide application modes on the efficacy of herbicides

4.2.2.1 Effect of different application modes (foliar, soil and foliar+soil) on herbicidal efficacy of ACCase-inhibitors against *A. myosuroides* and *A. spica-venti*

Herbicide treatment and application mode had highly significant effects (P < 0.001) on biomass reductions of *A. myosuroides* and *A. spica-venti*. Further, there were highly significant two-way interactions for herbicide treatment x application mode (Tab. 32).

Table 32: Results of ANOVA analysis of weed biomass reductions (fw shoot) by the factors herbicide treatment (ACCase-inhibitors) and application mode

Fixed effects	**parameter**	**df**	**p-value**
Alopecurus myosuroides			
application mode	shoot fw red.	2	< 0.001
herbicide	shoot fw red.	12	< 0.001
application mode x herbicide	shoot fw red.	24	< 0.001
Apera spica-venti			
application mode	shoot fw red.	2	< 0.001
herbicide	shoot fw red.	12	< 0.001
application mode x herbicide	shoot fw red.	24	< 0.001

p = 0.05, fw = fresh weight, red.= reduction, df = degree of freedom

The effect of herbicide treatments which were applied with three different application modes on *A. myosuroides* are shown in Table 33 as average shoot weight (fw.) reductions (in %) relative to untreated plants. At the time of the final assessments the fresh weights of shoots of untreated plants were on average 15.6-26.3g per pot. Comparing the different application methods with each other, the regular application method (foliar+soil) showed the highest level of efficacy for all herbicides within the same dosage.

After regular post-emergence application (foliar+soil) all herbicides showed a similar high activity at 40, 60 and 80 g/ha ranging from 81-96% biomass reductions which did not differ significantly from each other. At 20 g/ha pinoxaden and clodinafop still showed about 70% shoot biomass reductions, whereas fenoxaprop showed a low level of activity (40%).

Using only foliar application (without soil), all grass herbicides fenoxaprop, clodinafop and pinoxaden at 80 and 60 g/ha showed high levels of *A. myosuroides* control ranging from 78-96% biomass reductions, which did not differ significantly from the regular application (foliar+soil). At 40 g/ha only pinoxaden showed a high level of activity with 92%. Clodinafop and fenoxaprop showed significantly lower efficacies ranging from 41-47%, which are also significantly lower than the reductions caused by regular herbicide application (foliar+soil). With 20 g/ha no herbicide reached a decent level of weed control.

With herbicide placement to the soil only clodinafop (80g, 60g) and pinoxaden (80g, 60g and 40g) showed high biomass reductions (72-86%), whereas fenoxaprop showed levels of weed control ranging from 28-47%, which were significantly lower compared to the regular (foliar+soil) and foliar applications with fenoxaprop.

Clodinafop at 40 g/ha showed a significantly higher shoot reduction after soil application than the foliar placement, but did not reach the efficacy of a regular application (soil+foliar). Similarly, pinoxaden applied at 20g showed low biomass reductions after soil placement (45%) which were not significantly different from the foliar application but significantly lower compared to the regular application (70%).

Table 33: Influence of application mode on herbicidal efficacy (shoot fw. red. in %) of different ACCase-inhibitors against *Alopecurus myosuroides* 21 days after application.

Herbicide	Dosage (g a.i./ha)	Application mode		
		foliar	soil	foliar+soil
		fw red (%)	fw red (%)	fw red (%)
fenoxaprop-P	80	84 fghij	47 cde	93 hij
	60	78 fghij	32 bc	92 ghij
	40	47 cde	31 bc	81 fghij
	20	-3 a	28 bc	41 c
clodinafop	80	90 fghij	84 fghij	94 ij
	60	78 fghij	72 fghi	94 ij
	40	41 c	67 def	93 hij
	20	33 bc	16 ab	69 efg
pinoxaden	80	91 ghij	82 fghij	96 j
	60	92 ghij	87 fghij	96 j
	40	92 ghij	86 fghij	94 ij
	20	39 bc	45 cd	70 efgh
untreated		0 a	0 a	0 a
untreated (in g)		15.6	24.2	26.3

values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red.= reduction relative to the untreated, a.i.= active ingredient

The effect of herbicide treatments which were applied with three different application modes on *A. spica-venti* are shown in Table 34 as average shoot weight (fw.) reductions (in %) relative to untreated plants. At the time of final assessments the fresh weights of shoots of untreated plants were on average 4.63-5.26g per pot. Comparing the different application modes with each other, the regular application (foliar+soil) showed the highest level of efficacy against *Apera-spica venti* for all herbicides at the same dosage. After normal post-emergence application (foliar+soil), all grass herbicides showed the highest level of *A. spica-venti* control at all rates 80, 60 and 40 and 20g (except for fenoxaprop at 20g) ranging from 73-97% biomass reduction, and did not differ significantly from each other. With herbicide placement to the soil only, clodinafop (80, 60 and 40g) and pinoxaden (all rates) showed high biomass reductions (78-94%), whereas fenoxaprop showed low levels of biomass reductions ranging from 10-63%, which were often significantly lower in comparison to regular and foliar applications with fenoxaprop. With herbicide placement to the foliage only, fenoxaprop and pinoxaden at 80, 60 and 40g showed high biomass reductions (80-97%), whereas clodinafop showed low levels of biomass reductions ranging from 7-43%, which were significantly lower compared to regular (soil+foliar) and foliar applications with clodinafop. pinoxaden and clodinafop at 20 g/ha using only soil application showed significantly higher activities than with foliar application, and a similarly high activity to the normal application.

Table 34: Influence of application mode on herbicidal efficacy (shoot fw. red.in %) of different ACCase-inhibitors against *Apera spica-venti* 21 days after application

		Application mode					
	Dosage	**foliar**		**soil**		**foliar+soil**	
Herbicide	**(g a.i./ha)**	**fw red (%)**		**fw red (%)**		**fw red (%)**	
fenoxaprop-P	80	94	g	63	defg	94	g
	60	82	fg	62	defg	91	g
	40	80	fg	32	abcd	88	g
	20	4	a	10	ab	50	cdef
clodinafop	80	43	bcde	78	efg	96	g
	60	21	abc	79	efg	93	g
	40	10	ab	86	fg	89	g
	20	7	ab	50	cdef	73	efg
pinoxaden	80	97	g	94	g	96	g
	60	96	g	93	g	97	g
	40	93	g	92	g	95	g
	20	25	abc	83	fg	90	g
untreated		0	a	0	a	0	a
untreated (in g)		4.63		5.26		5.25	

values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red. = reduction relative to the untreated, a.i.= active ingredient

4.2.2.2 Effect of application modes (foliar, soil and foliar+soil) on herbicidal efficacy of ALS-inhibitors against *A. myosuroides*

Herbicide treatment and the application mode had highly significant effects ($p<0.001$) on biomass reductions of *A. myosuroides*. Moreover, there were highly significant two-way interactions for herbicide treatment x application mode (Tab. 35).

Table 35: Results of ANOVA analysis of weed biomass reductions (fw shoot in %) of *A. myosuroides* by the factors herbicide (ALS-inhibitors) and application mode

Fixed effects	parameter	df	p-value
application mode	shoot fw. red.	2	< 0.001
herbicide	shoot fw. red.	15	< 0.001
application mode x herbicide	shoot fw. red.	30	< 0.001

p = 0.05, fw = fresh weight, red.= reduction, df = degree of freedom

The effect of herbicide treatments which were applied with three different application modes on *A. myosuroides* are shown in Table 36 as average shoot weight (fw.) reductions (in %) relative to untreated plants. Fresh weights of shoots of untreated plants were on average 44.8-45.6g per pot. Mesosulfuron (15 g/ha), pyroxsulam (5 and 15 g/ha), flupyrsulfuron (5 and 10 g/ha) and propoxycarbazone (17.5, 35 and 70 g/ha) showed high levels of *A. myosuorides* control ranging from 84-99% biomass reduction with all application methods and did not differ significantly from each other. Lower dosages of pyroxsulam (1.7 g/ha) and flupyrsulfuron (2.5 g/ha) also showed very high shoot weight reductions (93-99%) after soil and normal application (soil+foliage), whereas just foliar application had significantly lower levels of weed control ranging from 45-60%. Also, mesosulfuron (5 g/ha) showed very high shoot reductions (97%) after normal post emergence application, whereas soil and foliar application showed reduced biomass reductions ranging from 56-73%. Differences were significant between regular application and soil placement at 5 and 1.7 g/ha of mesosulfuron. Iodosulfuron (5 and 10 g/ha) showed high levels of weed control after normal application and soil placement ranging from 67-88% shoot reduction, which did not differ significantly. In contrast iodosulfuron did not lead to significant biomass reductions with only foliar application, ranging from 4-19%.

Table 36: Influence of application mode on herbicidal efficacy (shoot fw. red.%) of different ALS-inhibitors against *Alopecurus myosuroides* 35 days after application

		Application mode		
	Dosage	**foliar**	**soil**	**foliar+soil**
Herbicide	**(g a.i./ha)**	**fw red (%)**	**fw red (%)**	**fw red (%)**
mesosulfuron	15	99 k	98 k	99 k
	5	73 efghi	56 cdef	97 jk
	1.7	54 cde	36 bc	66 defgh
pyroxsulam	15	96 jk	99 k	99 k
	5	91 ijk	99 k	99 k
	1.7	45 cd	98 k	99 k
flupyrsulfuron	10	98 k	99 k	99 k
	5	94 jk	98 k	99 k
	2.5	60 defg	93 jk	93 jk
propoxycarbazone	70	98 k	99 k	99 k
	35	91 ijk	99 k	99 k
	17.5	84 ghijk	99 k	99 k
iodosulfuron	10	19 ab	88 hijk	79 fghijk
	5	10 a	76 efghijk	67 defghi
	2.5	4 a	55 cdef	36 bc
untreated		0 a	0 a	0 a
untreated (in g)		45.6	44.8	45.2

values in column and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red.= reduction relative to the untreated, a.i.= active ingredient

4.2.3 Soil activity of herbicides in pot experiments

4.2.3.1 Soil activity of different ACCase-inhibitors in pot experiments

Significant interactions did not occur, nor was the soil activity of the herbicides significantly affected by the factor soil. The effect of herbicide treatments which were applied at two soils (biological active and sterilized soil) on *A. myosuroides* are shown in Table 37 as average shoot weight (fw.) reductions (in %) relative to untreated plants after 35 days.

The applied ACCase-inhibitors showed very different levels of soil efficacy at their maximum recommended field dose. While clodinafop and pinoxaden showed a highly significant (p = 0.05) pre-emergence activity against *A. myosuroides* (88% and 85%) in sterilized soil, the fresh weights of plants treated with fenoxaprop showed no significant difference to the untreated control (p = 0.05). With biologically active soil, the pre-emergence activity was reduced for every herbicide ranging from 8-23%, but not significantly.

Table 37: The effect of different pre-emergence applications of ACCase-inhibitors on shoot biomass reductions (fw red. in %) of *Alopecurus myosuroides* in a pot experiment 35 days after application.

Herbicide	**Active ingredient**	**Rate kg/L/ha**	**Rate g.ai/ha**	**active soil**		**sterilized soil**	
				fw red%		**fw red%**	
Ralon Super	fenoxaprop	1.2	82.8	11	A	19	A
Topik	clodinafop	0.6	60	66	B	88	B
Axial	pinoxaden	1.2	60	72	B	85	B
Untreated				0	A	0	A
Untreated (in g)				6.0		7.4	

mean values within a given column followed with the same latter are not significantly different according to Tukey-test (p = 0.05), fw = fresh weight, red.= reduction relative to the untreated, a.i.= active ingredient

4.2.3.2 Soil activity of post emergence herbicides in an outdoor pot experiment

In this experiment there were 16-20 *A. myosuroides* plants and 20-25 *A. spica-venti* plants in untreated pots with an average weight of 2.94g per pot (*A. myosuroides*) and 2.65g per pot (*A. spica-venti*). Treatment effects are shown in Table 38 as average shoot fresh weight reductions (fw.) after 47 days in % relative to the untreated pots. All used post emergence herbicides showed a certain level of pre-emergence weed control even at low dosages, resulting in mostly significant fresh weight reductions (p = 0.05). The ALS-inhibitors mesosulfuron (at 10 g/ha), pyroxsulam (2.5, 5 and 10 g/ha), propoxycarbazone (17.5, 35 and 70g) showed a high level of weed control, ranging for *A. myosuroides* from 85–95% and for *A. spica-venti* from 82-100%, which did not differ significantly. Flupyrsulfuron (10 g/ha) and mesosulfuron (5 g/ha) showed relatively good weed control ranging from 72-74%. Iodosulfuron (at 2.5 and 5 g/ha) showed very high pre-emergence activity (85-100%) against *A. spica-venti* compared to medium activity (57-59%) against *A. myosuroides*. At lower dosages the level of weed control decreased partially significantly for most herbicides, except for propoxycarbazone and pyroxsulam. Even at low rates these herbicides demonstrated a high level (82-92%) of soil activity against both weed species. The ACCase-inhibitors pinoxaden and clodinafop at the highest dose showed a relatively high level of pre-emergence weed control, ranging for *A. myosuroides* from 68-88% and for *A. spica-venti* from 64-83%, which did not differ significantly. In contrast, the ACCase-inhibitor fenoxaprop provided a low level of soil activity in this experiment.

Table 38: The effect of different pre-emergence applications of different ACCase- and ALS-inhibitors on Shoot biomass reductions (fw red. in %) of *Alopecurus myosuroides* and *Apera-spica venti* in an outdoor pot experiment 47 days after application.

Herbicide	Rate g.ai/ha	Shoot (fw) reduction %			
		A. myosuroides		***A. spica-venti***	
mesosulfuron	10	92	AB	100	A
	5	72	CDEF	73	ABCD
	2.5	60	FG	66	ABCD
iodosulfuron	5	59	FG	100	A
	2.5	57	FGH	85	ABC
	1.25	50	GHI	54	CDEF
pyroxsulam	10	93	A	100	A
	5	91	ABC	99	A
	2.5	85	ABCDE	92	AB
propoxycarbazone	70	95	A	98	A
	35	94	A	90	AB
	17.5	88	ABCD	82	ABCD
flupyrsulfuron	10	73	BCDEF	74	ABCD
	5	53	FGH	51	DEF
	2.5	48	GHI	28	FGH
fenoxaprop-P	80	8	KL	7	GH
	40	22	JK	-2	H
	20	31	IJ	11	GH
clodinafop	80	88	ABCD	64	BCDE
	40	67	EFG	71	ABCD
	20	27	J	34	EFG
pinoxaden	80	68	DEFG	83	ABCD
	40	58	FG	60	BCDE
	20	37	HIJ	25	FGH
untreated		0	L	0	H
untreated (in g)		2,94		2,65	

mean values within a given column followed with the same latter are not significantly different according to Tukey`HSD test (p = 0.05), fw= fresh weight, red.= reduction rel. to the untreated, ai.= active ingredient

4.2.3.3 Soil activity of different herbicides at different rates against *A. myosuroides* in a pot experiment under greenhouse conditions

The average shoot biomasses (fw) of *A. myosuroides* plants after 35 days was 11.8g per pot. The effect of herbicide treatments on *A. myosuroides* are shown in Tab. 39 as average shoot weight (fw.) reductions (in %) relative to untreated plants

Almost all herbicides, except for Ralon Super, which were applied pre-emergence showed significant shoot biomass reductions ranging from 38% (Topik) to 100% (Broadway, Atlantis and Attribut) at the full field dosage. Axial (1.2 and 0.8 L/ha), Broadway (0.22, 0.146, 0.07, 0.035 and 0.0175 kg/ha), Atlantis (0.5, 0.33, 0.166 and 0.0833 kg/ha), Lexus (0.02 kg/ha), Attribut (0.1, 0.05, 0.025, 0.0125 and 0.00625 kg/ha) and Cadou (0.125, 0.0625 L/ha) showed high levels of *A. myosuroides* control resulting in fresh weight reductions ranging from 67-100%. There were no significant differences within these treatments. With lower dosages, the level of weed control decreased partially significantly for most herbicides, except for Attribut which even showed reductions of 87% at 1/12 of the field rate. Also, Broadway at 1/12 of field dose, Axial at the 2/3 of field dose, Atlantis 1/6 and Cadou at 1/4 of the maximum field dosage did not show a significant decline in the level of weed control compared to the full field rate. Even at 1/24 of the full recommended field dosage, Broadway showed significant shoot reductions of 59%.

Table 39: The effect of pre-emergence applications of ACCase- and ALS-inhibitors at different dosages on shoot biomass reductions (fw red. in %) of *Alopecurus myosuroides* in a pot experiment 35 days after application

Herbicide	**Active ingredient**	**Rate kg/L/ha**	**Field Rate**	**Rate g a.i./ha**	**Shoot (fw) red. (%)**
Ralon Super	**fenoxaprop-P**	1.2	F	83	**20** abcd
		0.9	3/4F	62	**15** abcd
		0.6	1/2F	41	**-12** a
		0.3	1/6F	21	**-7** ab
Topik	**clodinafop**	0.6	F	60	**38** defg
		0.4	2/3F	40	**14** abcd
		0.2	1/3F	20	**0** abc
		0.1	1/6F	10	**-6** ab
Axial	**pinoxaden**	1.2	F	60	**84** hi
		0.8	2/3F	40	**80** hi
		0.4	1/3F	20	**34** defg
		0.2	1/6F	10	**-7** ab
Broadway	**pyroxsulam**+ florasulam	0.22	F	**15**+5	**100** i
		0.146	2/3F	**10**+3.3	**100** i
		0.07	1/3F	**5**+1.67	**99** i
		0.035	1/6F	**2.5**+0.84	**95** i
		0.018	1/12F	**1.25**+0.42	**75** hi
		0.009	1/24F	**0.63**+0.21	**59** fgh
Atlantis	**mesosulfuron**+ iodosulfuron	0.5	F	**15**+3	**100** i
		0.333	2/3F	**10**+2	**98** i
		0.166	1/3F	**5**+1	**82** hi
		0.0833	1/6F	**2.5**+0.5	**67** ghi
		0.042	1/12F	**1.25**+0.25	**30** cdef
		0.021	1/24F	**0.63**+0.13	**18** abcd
Lexus	**flupyrsulfuron**	0.02	F	10	**68** ghi
		0.01	1/2F	5	**36** defg
		0.005	1/4F	2.5	**25** bcde
		0.0025	1/8F	1.25	**9** abcd
		0.00125	1/12F	0.63	**-10** a
Attribut	**propoxycarbazone**	0.1	F	70	**100** i
		0.05	1/2F	35	**100** i
		0.025	1/4F	17.5	**100** i
		0.0125	1/8F	8.75	**99** i
		0.00625	1/12F	4.38	**87** hi
Cadou	**flufenacet**	0.125	1/2F	62,5	**95** i
		0.0625	1/4F	31.25	**85** hi
		0.03125	1/8F	15.63	**55** efgh
untreated					**0** abc
untreated (g)					**11.8**

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to Tukey`s HSG tests, fw. = fresh weight, red.= reduction relative to the untreated, a.i.= active ingredient, F = full recommended field dosage

4.2.3.4 Soil activity of different herbicides against *A. myosuroides* in a pot experiment using different watering methods in the greenhouse

The factor of irrigation method had no significant effect while herbicides showed highly significant effects ($p < 0.001$) on the biomass reductions of *A. myosuroides* plants. Further, a significant interaction between herbicide and irrigation method was found (Tab. 40).

Table 40: Results of ANOVA analyses for shoot biomass reductions (fw) for the factors irrigation method and herbicides

Fixed effects	**parameter**	**df**	**p-value ≤**
irrigation method	Shoot fw red	2	0.880
herbicide	Shoot fw red	8	< 0.001
irrigation method x herbicide	Shoot fw red	16	< 0.001

p = 0.05 , fw = fresh weight, red.= reduction, df =degree of freedom

The average shoot biomasses (fw) of *A. myosuroides* plants grown with top-soil irrigation were 8.4g (1x1 mm) and 8.9g (2x1 mm) per pot. Biomasses of untreated plants grown under sub-irrigation were significantly lighter, weighing 6.8g per pot (statistics not shown). The effect of herbicide treatments on *A. myosuroides* grown with 3 different irrigation methods are shown in Tab. 41 as average shoot weight (fw.) reductions (in %) relative to untreated plants.

Almost all herbicide treatments applied in pre-emergence, except for Ralon Super and a lower rate of Atlantis (0.166 kg/ha), showed very high levels of *A. myosuroides* control resulting in fresh weight reductions ranging from 88% (Axial, sub-irrigation)-100% (Broadway 0.22 kg/ha, 1x1 mm) with all irrigation methods. There were no significant differences neither within treatments nor within irrigation methods. The lower rate of Atlantis (0.166 kg/ha) showed significantly higher fw reductions with sub-irrigation and top-irrigation (2x1 mm) ranging from 97-99% compared to the less intensive top-irrigation (1x1 mm) showing 82%. Ralon Super showed significant shoot reductions in the range of 29-40% with sub-irrigation and top-irrigation (1x1 mm), whereas the more intensive watering (2x1 mm) did not result in significant pre-emergence activity.

Table 41: Effect of different herbicides applied in pre-emergence on the shoot biomass reduction (fw) of *A. myosuroides* plants grown under 3 different irrigation methods (sub-irrigation, top-irrigation with a sprinkler head delivering 1x1 mm and 2x1 mm) for 33 days after application

Product	Active ingredient	Rate L/kg/ha	Rate g a.i./ha	Field Rate	sub-irrigation fw red (%)		top-irr. 1x1 mm fw red (%)		top-irr. 2x1 mm fw red (%)	
Ralon Super	fenoxaprop-P	1.2	83	F	**29**	b	**40**	b	**11**	a
Topik	clodinafop	0.6	60	F	**92**	cde	**94**	de	**97**	de
Axial	pinoxaden	1.2	60	F	**88**	cd	**99**	de	**97**	de
Broadway	pyroxsulam+ florasulam	0.22	15+5	F	**99**	de	**100**	e	**99**	de
Broadway	pyroxsulam+ florasulam	0.073	5+1.66	1/3F	**99**	de	**99**	de	**99**	de
Atlantis	mesosulfuron +idosulfuron	0.5	15+3	F	**99**	de	**99**	de	**99**	de
Atlantis	mesosulfuron + idosulfuron	0.166	5+1	1/3F	**97**	de	**82**	c	**99**	de
Lexus	flupyrsulfuron	0.02	10	F	**98**	de	**95**	de	**99**	de
untreated					**0**	a	**0**	a	**0**	a
untreated (g)					6.8		8.9		8.4	

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, LSD5% = 11.3%; fw = fresh weight, red. = reduction relative to the untreated, a.i.= active ingredient, top-irr. = top soil irrigation with a sprinkler head, F= full recommended field dosage

4.2.3.5 Effect of soil moisture and sowing depth on the soil activity of different herbicides against *A. myosuroides* in the greenhouse

Overview of statistical analyses

All main effect factors, soil moisture, herbicide treatments and sowing depth had highly significant effects ($p < 0.001$) on the biomass reductions of *A. myosuroides* plants (Tab. 42). While no significant 3-way interactions occurred, there were highly significant two-way interactions for herbicide x sowing depth and herbicide x soil moisture for biomass reductions. No interaction was observed for soil moisture x sowing depth.

Table 42: Results of ANOVA analysis for weed biomass reductions (root and shoot) by the factors soil moisture regime, sowing depth and herbicides

Fixed effects	parameter	df	p-value
soil moisture regime	shoot fw red.	2	< 0.001
	shoot dw red.	2	< 0.001
	root fw red.	2	< 0.001
	root dw red.	2	< 0.001
sowing depth	shoot fw red.	1	< 0.001
	shoot dw red.	1	0.001
	root fw red.	1	< 0.001
	root dw red.	1	< 0.001
herbicide	shoot fw red.	15	< 0.001
	shoot dw red.	15	< 0.001
	root fw red.	15	< 0.001
	root dw red.	15	< 0.001
soil moisture regime x herbicide	shoot fw red.	30	< 0.001
	shoot dw red.	30	< 0.001
	root fw red.	30	< 0.001
	root dw red.	30	< 0.001
sowing depth x herbicide	shoot fw red.	15	< 0.001
	shoot dw red.	15	< 0.001
	root fw red.	15	< 0.001
	root dw red.	15	< 0.001
soil moisture regime x sowing depth	shoot fw red.	2	0.556
	shoot dw red.	2	0.578
	root fw red.	2	0.256
	root dw red.	2	0.086
soil moisture regime x sowing depth x herbicide	shoot fw red.	30	0.120
	shoot dw red.	30	0.069
	root fw red.	30	0.093
	root dw red.	30	0.063

p = 0.05, fw = fresh weight, dw = dry weight, red.= reduction df =degree of freedom

Effect of soil moisture on the soil activity of different herbicides against *A. myosuroides* (including 2 sowing depths)

Soil moisture had a significant effect on the biomass of *A. myosuroides* in untreated pots. The shoot and root biomasses (fw and dw) of *A. myosuroides* plants grown at 60% FC were significantly higher (by more than twice the size) than those grown under 15% FC and 30% FC (Tab. 43 and 44), which did not differ significantly from each other (statistics not shown in result tables). This effect was stronger at the deeper sowing depth of 3.5 cm.

Because there was no significant interaction between the factors soil moisture and sowing depth, the average effect of herbicide treatments on the fresh weights (fw) and dry weights (dw) of *A. myosuroides* plants grown under 3 different soil moistures are shown as average reductions (in %) over two sowing depths (0.5 and 3.5 cm) in Tab. 43 (shoots) and Tab. 44 (roots).

All applied herbicides showed pre-emergence activity for at least one soil moisture regime leading to either significant shoot or root biomass reduction of A. myosuroides plants. Most herbicide treatments reached the highest weed control level at relatively wet or moist soil conditions (30% FC and 60% FC). At the same time, pre-emergence activity by most herbicides decreased partially significantly with lower levels of soil moisture.

Cadou at the highest rate (0.25 L/ha) showed high levels of weed control at 60% FC and 30% FC (82-96% shoot red., 73-95% root red.) only, whereas the reductions declined significantly (by 39-59%) to 37-43% shoot red. and 44-56% root red. at 15% FC. Also, the lower rates of Cadou (0.0625, 0.125 L/ha) showed an enormous loss of herbicide efficacy (by 31-60% red.) with decreased soil moisture, resulting in significant weed biomass reductions at 30% FC and 60% FC ranging from 57-71% (shoot red.) and 45-78% (root red.), and almost no significant soil activity with relativly dry soil conditions (FC 15%).

Table 43: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (shoots) grown under 3 different water regimes (top-irrigation)* 42 days after application

			FC 15%	**FC 30%**	**FC 60%**	**FC 15%**	**FC 30%**	**FC 60%**
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1.2	F	**14**abc	**5**ab	**1**a	**13**abcd	**10**ab	**11**abc
Topik	0.6	F	**23**abcde	**63**efghijkl	**59**defghijkl	**18**abcdef	**68**ghijklmn	**59**fghijklmn
Axial	1.2	F	**18**abcd	**35**abcdef	**13**abc	**17**abcde	**44**bcdefghi	**9**ab
Broadway	0.22	F	**95**kl	**99**kl	**100**l	**96**klmn	**99**mn	**100**n
Broadway	0.073	1/3F	**90**ijkl	**96**kl	**99**kl	**92**jklmn	**96**klmn	**98**lmn
Broadway	0.037	1/6F	**62**efghijkl	**91**jkl	**97**kl	**64**ghijklmn	**88**jklmn	**96**klmn
Atlantis	0.5	F	**75**fghijkl	**94**kl	**90**ijkl	**70**ghijklmn	**91**jklmn	**86**jklmn
Atlantis	0.167	1/3F	**49**cdefghij	**44**bcdefgh	**84**hijkl	**53**cdefghij	**35**abcdefg	**78**hijklmn
Atlantis	0.083	1/6F	**1**a	**29**abcde	**25**abcde	**3**ab	**14**abcd	**13**abcd
Attribut	0.05	1/2F	**82**ghijkl	**97**kl	**97**kl	**81**ijklmn	**96**klmn	**96**klmn
Lexus	0.02	F	**49**cdefghi	**75**fghijkl	**90**ijkl	**55**defghijk	**66**ghijklmn	**87**jklmn
Cadou	0.25	1/2F	**37**abcdef	**83**hijkl	**96**kl	**43**bcdefghi	**82**ijklmn	**95**klmn
Cadou	0.125	1/4F	**28**abcde	**62**efghijkl	**71**fghijkl	**28**abcdefg	**58**efghijklm	**66**ghijklmn
Cadou	0.063	1/8F	**6**ab	**57**defghijk	**41**abcdefg	**8**ab	**57**efghijkl	**38**abcdefgh
Stomp a.	1.098	1/4F	**41**abcdefg	**0**a	**12**abc	**38**abcdefgh	**2**ab	**-1**a
untreated			**0**a	**0**a	**0**a	**0**a	**0**a	**0**a
untreated (in g)			6.44	6.87	15.82	0.90	0.92	2.01

Values in columns and lines followed by different letters are significantly different (p=0.05) according to LSD-tests, FC.= field capacity of the soil, fw.= fresh weight, dw.= dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a = Stomp aqua, *average over two sowing depths (0.5 and 3.5 cm) of *A. myosuroides*, LSD 5% = 42.5% fw red; LSD 5% = 42.2% dw red.

Table 44: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (roots) grown under 3 different water regimes (top-irrigation)* 42 days after application

			FC 15%	FC 30%	FC 60%	FC 15%	FC 30%	FC 60%
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1.2	F	**19**abcde	**56**efghijklm	**9**ab	**22**abc	**52**bcdefghi	**19**ab
Topik	0.6	F	**46**bcdefghij	**84**ijklmno	**69**fghijklmno	**46**bcdefgh	**83**ijklm	**71**fghijklm
Axial	1.2	F	**42**bcdefgh	**76**ghijklmno	**39**abcdefg	**36**bcdef	**70**fghijklm	**38**bcdefg
Broadway	0.22	F	**93**lmno	**99**no	**100**o	**95**klm	**99**m	**100**m
Broadway	0.073	1/3F	**90**klmno	**94**lmno	**99**no	**90**jklm	**93**klm	**99**m
Broadway	0.037	1/6F	**56**efghijklm	**92**klmno	**96**mno	**66**efghijklm	**91**klm	**95**klm
Atlantis	0.5	F	**87**jklmno	**93**lmno	**95**mno	**88**jklm	**92**klm	**94**klm
Atlantis	0.167	1/3F	**58**efghijklmn	**69**fghijklmno	**94**lmno	**62**efghijk	**62**efghijkl	**93**klm
Atlantis	0.083	1/6F	**14**abcd	**75**ghijklmno	**59**efghijklmno	**22**abc	**66**efghijklm	**55**cdefghij
Attribut	0.05	1/2F	**81**hijklmno	**95**mno	**97**no	**85**ijklm	**95**klm	**97**lm
Lexus	0.02	F	**54**defghijkl	**77**ghijklmno	**95**lmno	**63**efghijkl	**71**fghijklm	**93**klm
Cadou	0.25	1/2F	**44**bcdefghi	**79**ghijklmno	**95**mno	**56**cdefghij	**73**ghijklm	**95**klm
Cadou	0.125	1/4F	**32**abcdef	**62**fghijklmno	**78**ghijklmno	**42**bcdefgh	**43**bcdefgh	**74**hijklm
Cadou	0.063	1/8F	**12**abc	**72**fghijklmno	**40**abcdefg	**27**abcd	**62**defghijk	**45**bcdefgh
Stomp a.	1.098	1/4F	**33**abcdef	**51**cdefghijk	**34**abcdef	**42**bcdefgh	**34**abcde	**34**abcde
untreated			**0**a	**0**a	**0**a	**0**a	**0**a	**0**a
untreated (in g)			1.70	1.63	4.16	0.28	0.22	0.49

Values in column and lines followed by different letters are significantly different (p=0.05) according to LSD-tests, FC.= field capacity of the soil, fw.= fresh weight, dw.= dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a. = Stomp aqua, *average over two sowing depths (0.5 and 3.5 cm) of *A. myosuroides,* LSD 5% = 40.5% fw red; LSD 5% = 34.9% dw red.

In contrast, Stomp aqua caused the highest shoot (41% fw. red) and root reduction (42% dw red.) at 15% FC, which was significant for the root reductions.

Broadway (0.22, 0.073 kg/ha) showed a high level of *A. myosuroides* control at all soil moisture regimes, leading to shoot dw red. ranging from 92-100% and root dw red. ranging from 90-100%. The lowest dose of Broadway (0.037 kg/ha) showed a similar high soil activity at 30% FC and 60% FC, whereas the biomass reductions dropped to 64% (shoot dw red.) and 66% (root dw red.) at 15% FC. These losses of soil activity at dry soil conditions were not significant, however.

Attribut and the full rate of Atlantis (0.5 kg/ha) showed just a slight loss of pre-emergence activity from moist to dry soil conditions, resulting high biomass reductions ranging from 70-97% shoot reductions and 81-97% root reductions.

Atlantis (0.167 kg/ha) and Lexus resulted in a significantly high soil activity at 60% FC, resulting high shoot (78-90%) and root (93-95%) reductions, whereas the activity decreased partially significantly (by over 26-41%) to 49-53% shoot reductions and 54-63% root reductions at 15% FC.

Atlantis (0.083 kg/ha) led to significant root reductions at 60% FC and 30% FC (55-75%) but neither root biomasses at 15% FC nor shoot biomasses at any soil moisture showed significant soil activity.

For the used ACCase-inhibitors, only Topik led to a high level of *A. myosuroides* control at 60% FC and 30% FC, with significant shoot and root reductions (shoot 59-68%, root 69-83%). The soil activity of Topik dropped partially significantly at relativly dry soil conditions (FC 15%) to 18-23% shoot reductions and 46% root reductions. Ralon Super did not exhibit any activity on the above shoot biomass and Axial showed moderate but significant shoot reductions (44%) at only 30% FC. On the other hand, root biomasses showed significant dw reductions for Axial for all moisture regimes ranging from 36-70% reductions. Ralon Super application resulted in significant pre-emergence activity with 56% root (dw) reductions only at 30% FC.

Effect of weed sowing depth on herbicide activity against *A. myosuroides* including three different soil moisture levels (15, 30 and 60% FC) with top soil irrigation

Sowing depth had a significant reductive effect on the biomass of shoots and roots of *A. myosuroides* plants in untreated pots. Especially at 30% FC and 15% FC, where biomasses were about half the size compared to those at 60% FC (data not shown in tables).

Because there was no significant interaction between the factors sowing depth and soil moisture, the average effect of herbicide treatments on the fresh weights (fw) and dry weights (dw) of *A. myosuroides* plants grown at two different sowing depths are shown as average reductions (in %) over the three soil moisture regimes (15, 30 and 60% FC) in Tab. 45 (shoots) and Tab. 46 (roots).

All herbicide treatments caused significant root and/or shoot biomass reductions either at the shallow or deeper sowing depth of *A. myosuroides*. Furthermore, most herbicide treatments, especially Topik, Broadway (0.037 kg/ha), Lexus and Cadou (all rates) indicated a partially significant decrease in the level of weed control, ranging from 17 to 33% shoot reduction and 15 to 42% root reduction, with an increased sowing depth for *A. myosuroides.*

Table 45: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (shoots) grown at two sowing depths (0.5 and 3.5 cm)* 42 days after application

			Shoot reduction (fw)		**Shoot reduction (dw)**	
sowing depth (cm)			**0.5**	**3.5**	**0.5**	**3.5**
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1.2	F	**5**ab	**9**abc	**1**ab	**14**abc
Topik	0.6	F	**59**efgh	**38**cdef	**57**defghijk	**40**cdefgh
Axial	1.2	F	**20**abcd	**23**abcd	**20**abc	**27**abcdef
Broadway	0.22	F	**99**k	**97**k	**99**n	**98**n
Broadway	0.073	1/3F	**97**k	**93**jk	**97**n	**93**mn
Broadway	0.037	1/6F	**93**jk	**74**ghijk	**93**lmn	**73**hijklmn
Atlantis	0.5	F	**91**ijk	**82**hijk	**88**jklmn	**77**ijklmn
Atlantis	0.167	1/3F	**60**efghi	**58**efgh	**56**defghijk	**55**defghij
Atlantis	0.083	1/6F	**4**ab	**32**bcde	**-5**a	**26**abcde
Attribut	0.05	1/2F	**94**k	**91**ijk	**93**lmn	**90**klmn
Lexus	0.02	F	**80**hijk	**62**efghij	**79**jklmn	**59**efghijkl
Cadou	0.25	1/2F	**86**hijk	**58**efgh	**86**jklmn	**61**fghijklm
Cadou	0.125	1/4 F	**68**fghijk	**39**cdef	**67**ghijklmn	**34**bcdefg
Cadou	0.063	1/8F	**45**defg	**24**abcd	**44**cdefghi	**25**abcd
Stomp a.	1.098	1/4F	**16**abcd	**19**abcd	**17**abc	**17**abc
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			11.53	7.89	1.53	1.02

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over 3 different soil moisture regimes with surface-irrigation; LSD 5% = 31.3% fw red; LSD 5% = 33.9% dw red.

At the shallow sowing depth, Lexus, Cadou (0.25 L/ha) and Broadway (0.037 kg/ha) reached high levels of *A. myosuroides* control (79-93% shoot reduction and 81-92% root reduction) compared to 58-73% and 57-77% root red. in the deeper sowing depth. Topik, which also did not show a high soil activity at the shallow sowing depth, led to even lower levels of black-grass control (shoot reductions 40%, root reductions 59%) at the deeper sowing depth.

In contrast, the high pre-emergence activities of Broadway (0.22 and 0.073 kg/ha), Attribut and Atlantis (0.5 kg/ha) were not clearly affected by the deeper sowing depth. Consequently only these herbicides showed high levels of average weed control at both sowing depths, resulting in significant shoot reductions ranging from 77-99% and root red. 89-99%. Also, the soil activity by Atlantis (0.167 kg/ha) was similar at both sowing depths, showing a low to medium level of *A. myosuroides* control (55% shoot dw red., 73% root dw red.). The lowest rate of Atlantis (0.083 kg/ha), which showed an overall low level of pre-emergence activity at the shallow sowing depth, showed increased soil activity at the lower sowing depth. Furthermore, the low levels of pre-emergence activity of Axial, Ralon Super, and Stomp aqua were not greatly affected by sowing depth.

Table 46: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (roots) grown at two sowing depths (0.5 and 3.5 cm)* 42 days after application

			Root reduction (fw)		**Root reduction (dw)**	
	sowing depth (cm)		**0.5**	**3.5**	**0.5**	**3.5**
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1,2	F	**19**ab	**22**ab	**22**ab	**41**bcde
Topik	0.6	F	**74**fghij	**74**efghijk	**74**efghijk	**59**cdefghij
Axial	1,2	F	**53**cdefg	**47**bcdefg	**47**bcdefg	**49**bcdefg
Broadway	0.22	F	**99**j	**98**k	**98**k	**97**k
Broadway	0.073	1/3F	**97**j	**96**k	**96**k	**92**ijk
Broadway	0.037	1/6F	**92**j	**92**ijk	**92**ijk	**77**fghijk
Atlantis	0.5	F	**94**j	**94**jk	**94**jk	**89**hijk
Atlantis	0.167	1/3F	**74**fghij	**71**efghijk	**71**efghijk	**73**efghijk
Atlantis	0.083	1/6F	**35**abcd	**43**bcdef	**35**abcd	**60**cdefghij
Attribut	0.05	1/2F	**93**j	**93**ijk	**93**ijk	**91**ijk
Lexus	0.02	F	**82**ghij	**81**ghijk	**81**ghijk	**70**defghijk
Cadou	0.25	1/2F	**88**hij	**91**ijk	**91**ijk	**58**cdefghi
Cadou	0.125	1/4 F	**75**fghij	**74**efghijk	**74**efghijk	**32**abc
Cadou	0.063	1/8F	**56**cdefg	**54**bcdefgh	**54**bcdefgh	**35**abcd
Stomp a.	1.098	1/4F	**45**bcdef	**42**bcdef	**42**bcdef	**32**abc
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			3.17	1.83	0.43	0.27

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over 3 different soil moisture regimes with surface-irrigation; LSD 5% = 31.6% fw red; LSD5% = 35.5% dw red.

4.2.3.6 Effect of watering type (sub- and top-irrigation) and sowing depth on the herbicide activity against *A. myosuroides* in the greenhouse

Overview of statistical analyses

The main factors watering type, herbicide treatment and sowing depths had highly significant effects ($p < 0.001$) on the biomass reductions of *A. myosuroides* plants (Tab. 47). While no significant 3-way interactions occurred, there were highly significant two-way interactions for herbicide x sowing depth and herbicide x watering type for biomass reductions.

Table 47: Results of ANOVA analyses for weed biomass reductions (root and shoot) by the factors watering type, sowing depth and herbicides

Fixed effects	parameter	df	p-value
watering type at 30%FC	shoot fw red.	1	< 0.001
	shoot dw red.	1	< 0.001
	root fw red.	1	< 0.001
	root dw red.	1	< 0.001
sowing depth	shoot fw red.	1	< 0.001
	shoot dw red.	1	< 0.001
	root fw red.	1	< 0.001
	root dw red.	1	< 0.001
herbicide	shoot fw red.	15	< 0.001
	shoot dw red.	15	< 0.001
	root fw red.	15	< 0.001
	root dw red.	15	< 0.001
Watering type x herbicide	shoot fw red.	15	< 0.001
	shoot dw red.	15	< 0.001
	root fw red.	15	< 0.001
	root dw red.	15	< 0.001
sowing depth x herbicide	shoot fw red.	15	0.002
	shoot dw red.	15	0.003
	root fw red.	15	0.023
	root dw red.	15	< 0.001
watering type x sowing depth	shoot fw red.	1	0.904
	shoot dw red.	1	0.672
	root fw red.	1	0.093
	root dw red.	1	0.136
sowing depth x herbicidex watering type	shoot fw red.	15	0.429
	shoot dw red.	15	0.792
	root fw red.	15	0.471
	root dw red.	15	0.183

p = 0.05, fw = fresh weight, dw = dry weight, red.= reduction df = degree of freedom, FC = field capacity of the soil

Effect of watering type (top- versus sub-irrigation) on the herbicide activity against *A. myosuroides* at the medium soil moisture regime (30% FC)

Changing the method of watering at 30% FC from top-irrigation to sub-irrigation showed an increase in biomass of shoots and roots of *A. myosuroides* plants in untreated pots (dw) (Tab. 48 and Tab. 49). Especially at the deeper sowing depth, where root biomass (dw) was significantly increased (by more than 3 times) using sub-irrigation compared to surface-irrigation (data not shown in tables). Since there was no significant interaction between the factors of watering type and sowing depth, the average effect of sub- and top-irrigation at the medium moisture regime (30% FC) on the pre-emergence activity of different herbicide treatments on the fresh weights (fw) and dry weights (dw) of *A. myosuroides* plants, are shown as average reductions (in %) over two sowing depths (0.5 and 3.5 cm) in Tab. 48 (shoots) and Tab. 49 (roots).

Table 48: Effect of different herbicides applied in pre-emergence on the biomasses (fw and dw) of *A. myosuroides* plants (shoot) grown under different irrigation methods (top- and sub-irrigation)* 42 days after application

			Shoot reduction (fw)		**Shoot reduction (dw)**	
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**top-irrigation (%)**	**sub-irrigation (%)**	**top-irrigation (%)**	**sub-irrigation (%)**
Ralon S.	1.2	F	**5**ab	**6**ab	**10**abc	**2**ab
Topik	0.6	F	**63**defghij	**25**abcd	**68**defgh	**24**abcd
Axial	1.2	F	**35**abcdef	**13**abc	**44**abcdefg	**12**abc
Broadway	0.22	F	**99**j	**48**abcdefgh	**99**h	**52**bcdefgh
Broadway	0.073	1/3F	**96**ij	**39**abcdef	**96**h	**34**abcde
Broadway	0.037	1/6F	**91**ghij	**21**abcd	**88**fgh	**28**abcd
Atlantis	0.5	F	**94**hij	**47**abcdefgh	**91**gh	**38**abcdef
Atlantis	0.167	1/3F	**44**abcdefg	**7**ab	**35**abcde	**8**abc
Atlantis	0.083	1/6F	**29**abcde	**4**ab	**14**abc	**5**ab
Attribut	0.05	1/2F	**97**j	**33**abcde	**96**h	**25**abcd
Lexus	0.02	F	**75**efghij	**49**bcdefghi	**66**defgh	**31**abcde
Cadou	0.25	1/2F	**83**fghij	**63**defghij	**82**efgh	**51**abcdefgh
Cadou	0.125	1/4 F	**62**defghij	**21**abcd	**58**cdefgh	**30**abcd
Cadou	0.063	1/8F	**57**cdefghij	**7**ab	**57**cdefgh	**21**abcd
Stomp a.	1.098	1/4F	**0**a	**1**ab	**2**ab	**19**abcd
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			6.87	8.16	0.92	1.44

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over two sowing depths (0.5 and 3.5 cm); LSD 5% = 48.2% fw red; LSD 5% = 50.1% dw red.

Following top-irrigation, all applied herbicides in pre-emergence (except for Stomp Aqua) showed significant root dw reductions (52-99%), and most herbicides (except for Ralon Super, Axial, Stomp Aqua, Atlantis at 0.167 and 0.083 kg/ha) showed significant shoot dw reductions (57-99%). Broadway (all rates), Atlantis (0.5 kg/ha) and Attribut showed the highest levels of *A. myosuroides* control resulting in 88-99% shoot red. and 91-99% root red. Also, Cadou (0.25 kg/ha), Lexus and Topik showed relatively high levels of soil activity leading to 66-83% shoot red. and 71-83% root red..

Changing the watering method from top-irrigation to sub-irrigation at 30% FC decreased pre-emergence activity for all herbicide treatments often by over 35% root and/or shoot reduction losses.

Table 49: Effect of different herbicides applied in pre-emergence on the biomasses (fw and dw) of A. myosuroides plants (root) grown under different irrigation methods (top- and sub-irrigation)* 42 days after application

Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**Root reduction (fw)**		**Root reduction (dw)**	
			top-irrigation (%)	**sub-irrigation (%)**	**top-irrigation (%)**	**sub-irrigation (%)**
Ralon S.	1.2	F	**56**defghij	**20**abcd	**52**bcdefghi	**8**ab
Topik	0.6	F	**84**hijk	**50**cdefgh	**83**ghij	**48**bcdefgh
Axial	1.2	F	**76**efghijk	**38**abcdef	**70**efghij	**32**abcdef
Broadway	0.22	F	**99**k	**55**defghij	**99**j	**57**cdefghij
Broadway	0.073	1/3F	**94**jk	**36**abcde	**93**hij	**48**bcdefgh
Broadway	0.037	1/6F	**92**ijk	**35**abcde	**91**hij	**45**abcdefg
Atlantis	0.5	F	**93**jk	**45**bcdefgh	**92**hij	**29**abcdef
Atlantis	0.167	1/3F	**69**efghijk	**13**abc	**62**cdefghij	**19**abc
Atlantis	0.083	1/6F	**75**efghijk	**21**abcd	**66**defghij	**7**ab
Attribut	0.05	1/2F	**95**jk	**42**bcdefg	**95**ij	**26**abcde
Lexus	0.02	F	**77**fghijk	**49**cdefgh	**71**efghij	**32**abcdef
Cadou	0.25	1/2F	**79**ghijk	**70**efghijk	**73**fghij	**66**defghij
Cadou	0.125	1/4 F	**62**efghijk	**55**defghij	**53**bcdefghi	**45**abcdefg
Cadou	0.063	1/8F	**72**efghijk	**41**bcdefg	**62**cdefghij	**33**abcdef
Stomp a.	1.098	1/4F	**51**cdefghi	**5**ab	**34**abcdef	**21**abcd
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			1.63	1.66	0.22	0.45

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over two sowing depths (0.5 and 3.5 cm); LSD 5% = 40.6% fw red; LSD 5% = 45.9% dw red.

The highest significant decreases in shoot reduction, ranging from 50-71%, were shown by Atlantis (0.5 kg/ha), Attribut, Broadway (all rates), and Cadou (0.063 L/ha). Atlantis (all rates), Broadway (all rates), Attribut and Stomp Aqua showed significant losses in root reduction ranging from 43-68% after sub-irrigation. Changing the method of watering also led to great declines in the level of weed control for Lexus, Axial and Topik 100 (32-35% dw shoot red., 35-39% root dw red.).

Consequently, no herbicide treatment showed a high level of weed control after sub-irrigation leading to a maximum weed control of 52% shoot (dw) reduction (Broadway 0.22 kg/ha) and 66% root (dw) reduction (Cadou 0.25 L/ha). The only herbicides showing any significant shoot or root biomass reduction after sub-irrigation were Broadway (0.22 kg/ha), Cadou (0.25 kg/ha), Lexus and Topik.

Effect of sowing depth on the soil activity of different herbicides against *A. myosuroides* at medium soil moisture level (30% FC including top- and sub-irrigation)

Similar to the other trial shown above (4.2.3.5), sowing depth had a significant reductive effect on the biomass of shoots and roots of *A. myosuroides* plants in untreated pots (Tab. 50 and Tab. 51).

Because there was no significant interaction between the factors of watering type and sowing depth, the average effect of herbicide treatments on the fresh weights (fw) and dry weights (dw) of *A. myosuroides* grown at two different sowing depths are shown as average biomass reductions (in %) over two watering types at 30% FC (top and sub-irrigation) in Tab. 50 (shoots) and Tab. 51 (roots).

Table 50: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (shoots) grown at two sowing depths (0.5 and 3.5 cm)* 42 days after application

			Shoot reduction (fw)		**Shoot reduction (dw)**	
	sowing depth (cm)		**0.5**	**3.5**	**0.5**	**3.5**
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1.2	F	**9**abc	**3**ab	**8**abc	**4**ab
Topik	0.6	F	**56**efghi	**33**cdef	**57**hijklmn	**35**cdefghij
Axial	1.2	F	**23**abcd	**25**abcd	**27**abcdefg	**29**abcdefgh
Broadway	0.22	F	**74**hij	**73**hij	**76**mn	**75**lmn
Broadway	0.073	1/3F	**69**ghij	**67**ghij	**70**lmn	**60**ijklmn
Broadway	0.037	1/6F	**67**ghij	**45**defgh	**68**klmn	**48**efghijklm
Atlantis	0.5	F	**75**ij	**66**ghij	**70**lmn	**59**hijklmn
Atlantis	0.167	1/3F	**21**abcd	**30**bcde	**20**abcde	**24**abcdef
Atlantis	0.083	1/6F	**10**abc	**23**abcd	**1**a	**18**abcde
Attribut	0.05	1/2F	**69**ghij	**61**fghij	**66**klmn	**55**ghijklmn
Lexus	0.02	F	**67**ghij	**57**efghi	**59**hijklmn	**39**defghijk
Cadou	0.25	1/2F	**86**j	**60**fghij	**81**n	**52**fghijklmn
Cadou	0.125	1/4 F	**59**fghij	**24**abcd	**63**jklmn	**24**abcdef
Cadou	0.063	1/8F	**42**defg	**22**abcd	**45**efghijkl	**33**bcdefghi
Stomp a.	1.098	1/4F	**3**ab	**-1**a	**13**abcd	**8**abc
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			**9.33**	**5.70**	**1.44**	**0.92**

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over two watering types (top- and sub-irrigation) at 30% FC; LSD 5% = 28.4% fw red; LSD 5% = 29.9% dw red.

Table 51: Effect of different herbicides applied in pre-emergence on the biomasses (fw+dw) of *A. myosuroides* plants (roots) grown at two sowing depths (0.5 and 3.5 cm)* 42 days after application.

			Root reduction (fw)		**Root reduction (dw)**	
	sowing depth (cm)		**0.5**	**3.5**	**0.5**	**3.5**
Herbicide treatment	**Rate kg/L/ha**	**Field Rate**	**fw red. (%)**	**fw red. (%)**	**dw red. (%)**	**dw red. (%)**
Ralon S.	1.2	F	**39**bcd	**37**bcd	**33**abcdef	**27**abcd
Topik	0.6	F	**78**fghij	**56**bcdefghi	**76**gh	**54**cdefgh
Axial	1.2	F	**56**bcdefghi	**58**cdefghi	**50**bcdefgh	**52**bcdefgh
Broadway	0.22	F	**66**defghij	**68**defghij	**76**gh	**80**gh
Broadway	0.073	1/3F	**79**ghij	**72**efghij	**77**gh	**71**efgh
Broadway	0.037	1/6F	**78**fghij	**49**bcdefgh	**76**gh	**60**defgh
Atlantis	0.5	F	**78**fghij	**60**cdefghij	**73**fgh	**47**bcdefg
Atlantis	0.167	1/3F	**44**bcde	**38**bcd	**39**abcdefg	**42**bcdefg
Atlantis	0.083	1/6F	**47**bcdef	**48**bcdefg	**30**abcde	**42**bcdefg
Attribut	0.05	1/2F	**80**hij	**57**cdefghi	**76**gh	**45**bcdefg
Lexus	0.02	F	**71**efghij	**55**bcdefghi	**63**defgh	**40**abcdefg
Cadou	0.25	1/2F	**91**j	**58**cdefghi	**91**h	**48**bcdefg
Cadou	0.125	1/4F	**83**ij	**34**bc	**75**fgh	**13**ab
Cadou	0.063	1/8F	**68**defghij	**46**bcde	**61**defgh	**45**bcdefg
Stomp a.	1.098	1/4F	**31**abc	**26**ab	**42**bcdefg	**13**abc
untreated			**0**a	**0**a	**0**a	**0**a
untreated (in g)			**2.09**	**1.20**	**0.39**	**0.28**

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, FC = field capacity of the soil, fw = fresh weight, dw = dry weight, red.= reduction, Ralon S.= Ralon Super, Stomp a.= Stomp aqua, *average over two watering types (top and sub-irrigation) at 30% FC; LSD 5% = 31.5% fw red; LSD 5% = 41.2% dw red.

All herbicide treatments showed significant root and/or shoot biomass reductions at either the shallow and deeper sowing depth. Topik, Broadway (all rates), Atlantis (0.5 kg/ha), Attribut, Lexus, and Cadou (0.25 and 0.125 L/ha) showed medium to high levels of weed control at the shallow sowing depth, resulting in significant shoot dw red. ranging from 57-81% and significant root dw reductions ranging from 63-91%. Similar to the trial above, most herbicide treatments, especially Topik, Broadway (0.037 kg/ha), Lexus and Cadou (0.25 and 0.125 L/ha) showed a decrease in the level of weed control with an increased sowing depth for *A. myosuroides* (20-40% shoot red. and 16-62% root red.), which were mostly significant for Cadou. In addition, Attribut and Atlantis (0.5 kg/ha) showed a decline in root dw reductions ranging from 26-30%, while shoot biomasses were not greatly affected.

The high pre-emergence activity of Broadway (0.22 and 0.073 kg/ha) as well as the low pre-emergence activities of Axial, Ralon Super, Stomp aqua, and Atlantis (0.167 and 0.083 kg/ha) were not greatly affected by the deeper sowing depth.

4.2.4 Soil activity of herbicides in field experiments

4.2.4.1 Experiments on the soil activity of post-emergence herbicides under field conditions using artificial infestations with *A. myosuroides*

Experimental station Wicker-Staßenmühle (STR) in 2010/11 and 2011/12

In the experiment in 2010/11 with an average infestation of 98 plants/m^2 of *A. myosuroides* at the time of the assessment, the applied ACCase-inhibitors (Ralon Super, Topik, Axial) did not show much pre-emergence black-grass control (Tab. 52). In contrast, ALS-inhibitors led to medium to high levels of weed control ranging from 65% (Atlantis) to 96% (Attribut). Nevertheless, the highest soil activity was achieved with soil acting herbicides such as Cadou, Malibu and the combination Lexus+Stomp aqua resulting in efficacies ranging from 98-100%.

Table 52: Herbicidal efficacy (in % reduction in plant numbers relative to untreated and/or visual ratings) of pre-emergence herbicide treatments at the full field rate against *A. myosuroides* in field experiments in Wicker (STR) conducted in 2010/2011 and 2011/2012

Herbicide treatment	Rate L/kg/ha	Season 2010/11	Season 2011/12						
		18.04.11	10.12.11			23.04.12			
		HE%	Plants/m^2		red%	Plants/m^2		red%	HE%
Ralon Super	1.2	0	100	e	3	69	ef	9	0
Topik	0.6	7	76	de	26	57	de	25	0
Axial	1.2	15	70	cd	32	50	d	34	17
Broadway	0.275	80	6	a	94	15	ab	80	90
Atlantis	0.5	65	23	ab	77	19	abc	75	92
Lexus	0.02	93	22	ab	79	19	abc	75	91
Attribut	0.1	96	10	a	90	13	ab	83	94
Lexus + Stomp aqua	0.02 +2.5	98	9	a	91	11	ab	86	95
Cadou	0.5	99	41	bc	60	11	ab	85	95
Mailbu	4	100	35	ab	66	9	a	88	96
Stomp aqua	4.4	-	42	bc	59	25	bc	67	63
Boxer	5	-	33	ab	68	34	c	55	43
untreated			103	e	-	76	f	-	0

Values in columns and lines followed by different letters are significantly different (p = 0.05) accor-ding to LSD-tests, red. = reduction, HE = herbicidal efficacy in % control according to visual ratings

In 2011/12 there were on average 103 *A. myosuroides* plants/m^2 in untreated pots in autumn (10.12.11), whereas the weed density was reduced to 76 plants/m^2 in the spring (23.04.12). Treatment effects are shown in Table 52 as average weed density (plants/m^2) per plot and in % relative to the untreated plots. Also, herbicidal efficacy ratings (HE) in the spring are shown in Table 52.

Almost all used post emergence herbicides showed a certain level of pre-emergence weed control, which are in most cases significant ($p = 0.05$) to the untreated control.

All ALS-inhibitors (Atlantis, Broadway, Lexus, Attribut) at the highest recommended field dose and the combination of Lexus+Stomp aqua showed good to very good weed control ranging from 77-91% in autumn and 75-86% in the spring. The ACCase-inhibitors (Ralon Super, Topik and Axial) showed a relatively low level of pre-emergence weed control in autumn and in the spring ranging from 3% (Ralon Super) to 34% (Axial). However, Topik and Axial showed significant differences in plant numbers to the untreated control.

The soil herbicides (Cadou, Herold, Stomp Aqua and Boxer) showed significant weed control ranging from 59 -68% in autumn. In the spring, Cadou and Malibu showed higher levels of weed control (85-88%), whereas weed control by Boxer and Stomp aqua remained on a relatively low level (55-67%).

Experimental station Gießen (GI) in 2011/12

In 2011/12 at Gießen there were on average 47 plants/m^2 of *A. myosuroides* in untreated plots in autumn (05.12.11), whereas the weed density was reduced in the spring to 36 plants/m^2 (19.04.12). Treatment effects are shown in Table 53 as average weed density (plants/m^2) per plot and in % relative to the untreated plots. Efficacy ratings (HE) conducted on April 8th 2012 are also shown in the Table 53 and support the data presented below.

The ACCase-inhibitors (Ralon Super, Topik and Axial) at the highest recommended field dose showed relatively low levels of pre-emergence weed control in autumn and in spring ranging from 1% (Topik) to 26% (Axial), which were not significantly different to the untreated control. Axial at double the field rate increased the level of soil activity significantly, resulting in significant pre-emergence black-grass control (red. 66-77%), whereas Topik and Ralon Super at X2 dose remained on a low level.

Almost all used post-emergence herbicides showed a certain level of pre-emergence weed control, which are in almost all cases significant.

Table 53: Herbicidal efficacy (in % reduction in plant numbers relative to untreated and visual ratings) of pre-emergence herbicide treatments at the full and double field dosage applied on *A. myosuroides* in a field experiment in Gießen (GI) 2011/2012

Herbicide treatment	Rate L/kg/ha	Field rate	05.12.2011		19.04.2012		
			Plants/m^2	red.%	Plants/m^2	red.%	HE%
Ralon Super	2.4	X2	54 f	-16	32 b	11	0
Ralon Super	1.2	F	41 ef	11	27 b	26	10
Topik	1.2	X2	48 ef	-3	30 b	18	0
Topik	0.6	F	37 e	20	36 b	1	8
Axial	2.4	X2	16 abc	66	13 a	65	77
Axial	1.2	F	35 de	26	27 b	27	40
Broadway	0.55	X2	1 a	97	3 a	91	97
Broadway	0.275	F	10 ab	79	10 a	71	93
Atlantis	1	X2	11 ab	77	4 a	90	98
Atlantis	0.5	F	19 bcd	60	8 a	78	91
Lexus	0.04	X2	32 cde	31	27 b	25	57
Lexus	0.02	F	33 de	30	29 b	17	58
Attribut	0.2	X2	8 ab	83	3 a	91	99
Attribut	0.1	F	11 ab	77	11 a	69	92
Lexus+ Stomp aqua	0.02 +2.5	F+ 1/2F	20 bcd	57	7 a	82	91
Cadou	0.5	F	12 ab	74	7 a	82	95
Mailbu	4	F	5 ab	89	2 a	95	98
untreated			47 ef	-	36 b	-	0
LSD$_{0.05}$			11.3		16.5		

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, red. = reduction, HE = herbicidal efficacy in % control according to visual ratings, F = full field dose, X2 = double of the field dose

The ALS-inhibitors Atlantis, Broadway and Attribut at the highest recommended field dose and Lexus+Stomp showed decent weed control ranging from 57-79% in autumn, which were significant to the untreated control but not to each other (p=0.05). Countings conducted in the spring showed similar soil activity, resulting in weed reductions from 69-82%. In contrast, Lexus resulted in low levels of weed control (17-30%), and also doubling the field rate did not increase soil activity. In general, there was no significant dosage effect, although double the field dose led to relatively high levels of weed control by all ALS-inhibitors, except for Lexus, which ranged from 77% (Atlantis) – 97% (Broadway) in autumn and over 90% in the spring.

As expected, the soil herbicides (Cadou, Herold) showed high levels of significant weed control ranging from 74 -89% in autumn and even higher levels of weed control in the spring (82-95%), and they did not differ significantly.

Experimental station Wicker (Johannesfeld) in 2011/12

In 2011/12 at Wicker- Johannesfeld (JOH) there were on average 315 plants/m^2 of *A. myosuroides* in untreated pots in autumn (10.12.11), whereas the weed density was reduced in the spring to 213 plants/m^2 (24.04.12). Treatment effects are shown in Table 54 as average weed density (plants/m^2) per plot and in % relative to the untreated plots. Efficacy ratings (HE) conducted on 28.11.11 are also shown in the table and support the data presented below.

Table 54: Herbicidal efficacy (in % reduction in plant numbers relative to untreated and visual ratings) of pre-emergence herbicide treatments at full and double field dosage against *A. myosuroides* in a field experiment in Wicker (JOH) 2011/2012

Herbicide treatment	Rate L/kg/ha	Field rate	10.12.12		28.11.12	24.04.12	
			Plants/m^2	red.%	HE%	Plants/m^2	red.%
Ralon Super	2.4	X2	259 ef	18	5	173 e	19
Ralon Super	1.2	F	250 def	21	0	184 e	14
Topik	1.2	X2	84 abc	73	48	53 bcd	75
Topik	0.6	F	146 cde	53	18	91 d	57
Axial	2.4	X2	33 abc	90	88	40 abc	81
Axial	1.2	F	133 bcd	58	45	69 cd	67
Broadway	0.55	X2	0 a	100	100	1 a	100
Broadway	0.275	F	1 a	100	100	9 ab	96
Atlantis	1	X2	3 a	99	99	2 a	99
Atlantis	0.5	F	36 abc	88	90	25 abc	88
Lexus	0.04	X2	6 a	98	98	10 ab	95
Lexus	0.02	F	24 ab	92	91	24 abc	89
Attribut	0.2	X2	13 a	98	99	14 ab	97
Attribut	0.1	F	5 a	96	96	6 ab	94
Lexus+ Stomp aqua	0.02+ 2.5	F+ 1/2F	11 a	96	86	15 ab	93
Cadou	0.5	F	73 abc	77	80	36 abc	83
Mailbu	4	F	71 abc	77	92	39 abc	82
untreated			315 f	-	0	213 e	-
LSD.$_{0.05}$			117			50	

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, red. = reduction, HE = herbicidal efficacy in % control according to visual ratings, F= full field dose, X2 = double of the field dose, JOH= Johannesfeld

All herbicide treatments showed a certain level of pre-emergence black-grass control, which are in almost all cases, except for Ralon Super, significant to the untreated control (p = 0.05). In contrast to Ralon Super, the ACCase-inhibitors Topik and Axial at the full recommended field dose showed a decent level of pre-emergence weed control in autumn and spring ranging from 53-67% reduction in black-grass infestation. Double the field rate of Axial and Topik increased the level of soil activity (73-90%), whereas Ralon Super at X2 dose remained on a low level. The ALS-inhibitors Atlantis, Broadway, Lexus and Attribut at the highest recommended field dose and the combination Lexus+Stomp showed the highest soil activities against *A. myosuroides* in autumn and spring ranging from 88-100%, which were not significantly different to each other (p = 0.05). Those ALS-herbicides applied at double the field dosage increased soil activity and gave nearly complete weed control (95-100%). The soil herbicides (Cadou, Herold) reduced the number of black-grass individuals by 77% in autumn and by 82-83% in spring respectively, but did not differ significantly from each other.

4.2.4.2 Field experiments in winter wheat on the soil activity of post-emergence herbicides using natural infestations with A. myosuroides

Experimental station Wicker (Johannesfeld) in 2011/12

In the field sown with winter wheat at Wicker- Johannesfeld (JOH) there were on average 334 heads/m^2 (inflorescences) of *A. myosuroides* in untreated plots counted on May 25th. Treatment effects are shown in Table 55 as average weed density (heads/m^2) per plot, in % relative to the untreated plots. Efficacy ratings (HE) conducted on 24.04.12 are also shown in tables and support the data presented below.

All herbicide treatments showed a certain level of pre-emergence black-grass control, which are in most cases, except for Ralon Super (1.2 L/ha), significant to the untreated control (p = 0.05). At the highest recommended field dosage, Axial provided the best pre-emergence black-grass control (77%) within the ACCase-inhibitors, whereas Ralon Super and Topik did not show much soil activity (19-35%). At double the field rate, increased levels of soil activity were observed with Axial (93%) and Topik 100 (71%), whereas Ralon Super at the X2 dose remained on a lower level.

The ALS-inhibitors Atlantis, Broadway, Lexus and Attribut at the recommended and double field dose, as well as the combination Lexus+Stomp gave very high levels pre-emergence control of *A. myosuroides* ranging from 98-100%, which were not significantly different from each other (p = 0.05). As expected, the soil herbicides Cadou and Herold showed high soil activity and reduced the number of black-grass individuals by 92-93%.

In untreated plots, the average density of wheat was 434 heads/m^2 (inflorescences). While applied ACCase-inhibitors did not have a significant effect on the winter wheat density, almost all other herbicides increased the numbers of heads/m^2 significantly up to 27% compared to the untreated control. Broadway at double the field rate was the only treatment which led to significantly lower wheat densities compared to the untreated control.

Table 55: Effect of different herbicides applied in pre-emergence at the full and double field dosage on the infestation with *A. myosuroides* (heads/m^2), Herbicidal efficacy (in % reduction relative to untreated) and wheat density (heads/m^2) in a field experiment in winter wheat in Wicker (JOH) 2011/2012

Herbicide treatment	Rate L/kg/ha	Field rate	***A. myosuroides***			**Winter wheat**	
			heads/m^2	**red.%**	**HE%**	**heads/m^2**	**red.%**
Ralon Super	2.4	X2	234 c	30	36	456 bcd	-5
Ralon Super	1.2	F	270 cd	19	14	440 bcd	-1
Topik	1.2	X2	98 b	71	56	437 bcd	-1
Topik	0.6	F	218 c	35	35	432 bc	0
Axial	2,4	X2	24 ab	93	89	461 bcde	-6
Axial	1.2	F	78 ab	77	51	422 b	3
Broadway	0.55	X2	0 a	100	99	323 a	26
Broadway	0.275	F	0 a	100	99	484 bcdef	-11
Atlantis	1	X2	0 a	100	99	528 ef	-22
Atlantis	0.5	F	6 a	98	99	503 def	-16
Lexus	0.04	X2	0 a	100	99	454 bcd	-5
Lexus	0.02	F	0 a	100	99	549 f	-26
Attribut	0.2	X2	0 a	100	99	542 f	-25
Attribut	0.1	F	3 a	99	97	552 f	-27
Lexus+ Stomp aqua	0.02+ 2.5	F+ 1/2	3 a	99	98	547 f	-26
Cadou	0.5	F	24 ab	93	96	552 f	-27
Mailbu	4	F	25 ab	92	97	497 cdef	-14
unbehandelt			334 d	-	0	434 bc	-
LSD$_{.0.05}$			79.6			69	

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, red. = reduction, HE = herbicidal efficacy in % control according to visual ratings, F = full field dose, X2 = double of the field dose

Experimental station Wicker (Kennel) in 2011/12

The agricultural field grown with winter wheat at Wicker-Kennel (KEN) was naturally infested with *A. myosuroides*. In untreated plots there were on average 56 plants/m^2 in autumn (8.12.11) and 201 heads/m^2 in early summer (12.06.12). Treatment effects are shown in Table 56 as average weed density (plants and heads/m^2) per plot, in % relative to the untreated plots. Efficacy ratings (HE) conducted on 19.04.12 are also shown in tables and support the data presented below. At the highest recommended field dosage, Axial provided a medium level of pre-emergence black-grass control (51-57%) within the ACCase-inhibitors, whereas Ralon Super and Topik did not show significant soil activity. Weed countings in autumn showed that Broadway gave the highest pre-emergence black-grass control (90%), compared to Atlantis with a relatively low level of soil activity. In the spring both ALS-inhibitors Atlantis and Broadway at the recommended field dose gave very high levels pre-emergence *A. myosuroides* control ranging from 95-96%, which were not significantly different to each other (p = 0.05). Countings of the wheat heads on 15.06.11 showed that herbicide treatments cleary increased the density of heads per m^2, but not significantly.

Table 56: Effect of different herbicides applied in pre-emergence at the full registered field dosage on the infestation with *A. myosuroides* (plants and heads/m^2) and wheat density (heads/m^2) in a field experiment with winter wheat in Wicker (KEN) 2011/2012

Herbicide treatment	**Rate kg/L/ha**	***A. myosuroides***					**Wheat**
		plants/m^2	**red.%**	**heads/m^2**	**red.%**	**HE%**	**heads/m^2**
Ralon Super	1.2	48 cd	16	155 c	23	0	764 a
Topik	0.6	58 d	-4	183 cd	9	10	770 a
Axial	1.2	28 b	51	87 b	57	38	770 a
Broadway	0.275	6 a	90	9 a	96	96	772 a
Atlantis	0.5	31 bc	45	11 a	95	98	807 a
untreated		56 d		201 d			761 a
LSD$_{0.05}$		18		41			55

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, red. = reduction, HE = herbicidal efficacy in % control according to visual ratings, KEN = Wicker-Kennel

4.2.5 Residual soil activity of post-emergence herbicides against *A. myosuroides*

4.2.5.1 Soil-degradation (DT50) of mesosulfuron, pyroxsulam and pinoxaden in different soils under laboratory conditions

The degradation times (DT50) for mesosulfuron, pyroxsulam and pinoxaden and its major active metabolite (M2 = NOA407854) are given in Tab. 56. The half-life times for mesosulfuron were the longest in every soil showing relatively high DT50 values from 36 for Rauischholzhausen (RH), 41 for Wicker (WI) and 58 days for Gießen (GI). The DT50 for pyroxsulam was on a similarly high level (43 days) in the soil from GI, whereas at WI (18 days) and RH (19 days), the degradation of pyroxsulam was much faster. pinoxaden degraded in all soils very quickly (0-1 days), but the metabolite M2 showed in all soils a relatively high DT50 ranging from 24 days (WI and RH) and 26 days (GI). In general the soil in GI degraded slower than the soil from WI and RH.

Table 57: The half-lives (DT50) for mesosulfuron, pyroxsulam and pinoxaden and its major active metabolite (M2 = NOA407854) in different agricultural soils (Gießen, Wicker and Rauischholzhausen) in a lab-study with soil moisture of 50% (of max. water capacity) and temperature of 10 °C

Active ingredient	Gießen (GI) DT50	Wicker (WI) DT50	Rauischh. (RH) DT50
mesosulfuron	58 d	41 d	36 d
pyroxsulam	39 d	19 d	18 d
pinoxaden	<1 d	1 d	<1 d
M2 (NOA407854)	26 d	24 d	24 d

d = days, M2= major active metabolite from pinoxaden, DT50 = amount of degradation time (half-life) in days it takes for 50 percent of the parent compound to degrade from soil

4.2.5.2 Residual soil activity of post-emergence herbicides against *Alopecurus myosuroides* in pot experiments under greenhouse conditions

Experiment 1

At the time of the final assessments (49 days after sowing) of the experiment there were 20-25 *A. myosuroides* plants in untreated pots and they had an average shoot weight ranging from 10.25 to 12.57g per pot. Differences were not significant (p=0.05) (statistics not shown). The residual soil activity of applied herbicides is shown in Table 58 as shoot fresh weight reductions of *A. myosuroides* (% relative to untreated) which were sown in herbicide treated pots after different periods of time (0, 7, 14, 21 and 28 days). All ALS-herbicides (Broadway 0.22 and 0.07 kg/ha, Atlantis 0.5 and 0.17 kg/ha, Lexus 0.02 kg/ha) exhibited a high level of soil activity for at least 14 days resulting in significant biomass reductions ranging from 75% (Broadway 0.07 kg) to 100% (Atlantis). Shoot biomasses of *A. myosuroides* which was sown 21d and 28d after treatment were not significantly reduced by applications with Lexus and Broadway (0.07 kg/ha), whereas Atlantis (0.5 and 0.17 kg/ha) as well as Broadway at the higher dose (0.22 kg/ha) resulted in biomass reductions ranging from 65% (Broadway 0.22 kg/ha) to 100% (Atlantis 0.5 kg/ha), and were not significantly different from each other (p = 0.05). All applied ACCase-inhibitors (Axial, Ralon Super and Topik) did not show any significant residual soil activity. Axial showed soil activity only on *A. myosuroides* which was sown on the same day of the treatment leading to significant fw reductions of 76%, whereas Topik and Ralon Super did not show significant weed control. The decline in pre-emergence soil activity with a delay of the sowing time of *A. myosuroides* from 0d to 28d was significant for Lexus, Axial and Broadway (0.073 kg/ha), whereas the soil activity by Atlantis remained on the same high level at all sowing dates, which demonstrates a relatively long residual soil activity.

Table 58: Residual soil activity of herbicides as shoot fresh weight reductions of *A. myosuroides* (% relative to untreated) which was sown in herbicide treated pots after 0, 7, 14, 21 and 28 days after application (January-March 2011)

		0d		7d		14d		21d		28d	
Herbicide treatment	**Rate kg/L/ha**	**fw red. (%)**		**fw red. (%)**		**fw red. (%)**		**fw red (%)**		**fw red (%)**	
Ralon Super	1.2*	7	ab	3	ab	3	ab	0	ab	3	ab
Topik	0.6*	47	bcde	13	abc	-6	a	-4	a	-1	a
Axial 50EC	1.2*	76	de	-7	a	-1	a	-1	a	9	ab
Broadway	0.22*	98	e	98	e	87	e	76	de	65	cde
Broadway	0.07	94	e	83	e	73	de	14	abc	4	ab
Atlantis WG	0.5*	100	e	99	e	100	e	100	e	100	e
Atlantis WG	0.17	99	e	98	e	98	e	93	e	86	e
Lexus	0.02*	84	e	83	de	75	e	25	abcd	14	abc
untreated		0	ab	0	ab	0	ab	0	ab	0	ab
untreated (g)		12.26		11.20		10.25		10.82		12.57	

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red.= reduction, d=days , final assessments were conducted 49 days after sowing; * = maximum recommended/registered field rate

Experiment 2

At the time of the final assessments (49 days after sowing) there were 21-25 *A. myosuroides* plants in untreated pots and they had an average shoot weight ranging from 12.6 to 13.9g per pot which did not differ significantly (p = 0.05) (statistics not shown). The residual soil activity of applied herbicides are shown in Tab. 59 as shoot fresh weight reductions of *A. myosuroides* plants (% relative to untreated) which were sown in treated pots after different time periods (0, 7, 14, 21 and 28 days). Similar to the experiment above, no ACCase-inhibitor showed any residual soil activity. Like above, Axial showed a high level of significant weed control leading to significant shoot weight reductions on *A. myosuroides* (72%) which were sown on the day of the herbicide treatment. ALS-inhibitors such as Atlantis (0.5 and 0.17 kg), Broadway and Attribut showed a high level of residual soil activity of up to 28 days, resulting in significant fresh weight reductions of sown *A. myosuroides* ranging from 98-100%, and differences were not significant (p = 0.05). Most the time there were no significant dosage effects observed for Atlantis and Broadway. In this experiment Broadway showed fw reductions of 100% on *A. myosuroides* which was sown 4 weeks after treatment also at lower dosages (0.073 kg/ha). Atlantis at the lowest rate (0.083 kg/ha) demonstrated significant pre-emergence activity for up to 28 days resulting in shoot reductions ranging from 56-89%. Lexus only exhibited significant biomass reductions (82-87%) on *A. myosuroides* which was sown 0-7 days after treatment, whereas no significant soil activity was observed beyond that. The decline in pre-emergence weed control with a delay in sowing time of *A. myosuroides* from 0d to 28d was significant for Lexus and Axial and (0.073 kg/ha), whereas the soil activity caused by other herbicides such as Atlantis, Broadway and Attribut remained on the same high level of activity at all replanting dates. Thus Atlantis, Broadway and Attribut demontrated long residual soil activity in this experiment.

Table 59: Residual soil activity of herbicides as shoot fresh weight reductions of *A. myosuroides* (% relative to untreated) which was sown in herbicide treated pots after 0, 7, 14, 21 and 28 days after application (April-June 2011)

		0d	**7d**	**14d**	**21d**	**28d**
Herbicide treatment	**Rate kg/L/ha**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**fw red (%)**	**fw red (%)**
Ralon Super	1.2*	15 abc	6 ab	2 ab	11 ab	-5 ab
Topik	0.6*	26 abc	9 ab	9 ab	3 ab	1 ab
Axial 50EC	1.2*	72 def	-2 ab	-7 ab	7 ab	16 abc
Broadway	0.22*	100 f	100 f	100 f	100 f	100 f
Broadway	0.073	100 f	99 ef	100 f	100 f	100 f
Atlantis WG	0.5*	100 f	100 f	100 f	100 f	100 f
Atlantis WG	0.167	100 f	99 ef	99 ef	98 ef	100 ef
Atlantis WG	0.083	89 ef	86 ef	86 ef	76 def	56 cde
Lexus	0.02*	97 ef	82 ef	33 bcd	15 abc	12 ab
Attribut	0.05**	100 f	100 f	100 f	99 ef	100 f
untreated		0 ab	0 ab	0 ab	0 ab	0 ab
untreated (g)		13.6	12.6	13.7	13.0	13.9

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red.= reduction, d=days, final assessments were conducted 49 days after sowing, * = maximum recommended/registered field rate, ** = half of maximum field rate

Experiment 3

Shoot fresh weights of *A. myosuroides* in untreated pots which were intensively watered (1 mm daily) before sowing were with 19.5g significantly (p = 0.05) heavier than those watered only once a week (15.5g) and twice a week (16.7g) (statistics not shown). The effect of different watering intensities (dry, normal, wet) on the persistence of residual soil activity of herbicides are shown in Table 60 as shoot fresh weight reductions of *A. myosuroides* which was sown in pots 28 days after application. Atlantis (0.5 and 0.17 kg/ha) and Attribut (0.05 kg/ha) showed long residual soil activity with all irrigation methods, resulting in significant fw reductions of *A. myosuroides* ranging from 92-100%, which did not differ significantly neither within treatment nor within watering intensity. Broadway (0.22 and 0.07 kg/ha) showed the same level of residual soil activity only under relatively dry soil conditions (97-98%), whereas more intense watering for the four week period after herbicide treatment resulted in significant decreases of soil activity, with biomass reductions of *A. myosuroides* ranging from 15-45%. There were no significant differences neither within rates of Broadway nor between the normal and wet watering method. Atlantis did not exhibit a significant dosage effect either. Similar to the other trials above, Lexus as well as ACCase-inhibitors such as Ralon Super, Topik and Axial did not show significant soil activity against *A. myosuroides* which was sown 4 weeks after application, independent of the watering method.

Table 60: The effect of different watering intensities (dry, normal, wet) on the residual soil activity of herbicides as shoot fresh weight reductions of *A. myosuroides* (% relative to untreated) which was sown in herbicide treated pots 28 days after application (August-October 2011)

Herbicide treatment	**Rate kg/L/ha**	**Dry** fw red. (%)		**Normal** fw red. (%)		**Wet** fw red. (%)	
Ralon Super	1.2*	3	a	-2	a	1	a
Topik	0.6*	-4	a	0	a	4	a
Axial 50EC	1.2*	0	a	13	a	1	a
Broadway	0.22*	98	c	45	ab	19	a
Broadway	0.07	97	bc	26	a	15	a
Atlantis WG	0.5*	99	c	98	c	99	c
Atlantis WG	0.17	94	bc	95	bc	92	bc
Lexus	0.02*	7	a	-5	a	3	a
Attribut	0.05**	100	c	100	c	100	c
untreated		0	a	0	a	0	a
untreated (g)		15.5		16.7		19.5	

Irrigation interval: dry = 2.5 mm once in a week, normal = 2.5 mm twice a week, wet = 1 mm daily; Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, fw = fresh weight, red.= reduction, final assessments were conducted 43 days after sowing, * = maximum recommended/registered field rate, ** = half of maximum field rate

4.2.5.3 Residual soil activity of post-emergence herbicides against *A. myosuroides* under field conditions

Experimental station Wicker-Staßenmühle (STR) in 2010/11

The residual soil activity of herbicides at the Experimental station Wicker-Staßenmühle is shown in Tab. 61 as reductions of *A. myosuroides* infestation (% relative to untreated) which was sown in treated plots after different intervals following application (3, 28 and 168 days). The ACCase- inhibitors did not show residual soil activity.

The ALS-inhibitors (Atlantis, Broadway, Lexus and Attribut) showed decent to high soil activity on *A. myosuroides* which were sown only 3 days after application (DAA) ranging from 75% (Lexus)-95% (Attribut). Also, the sowing 28 days after application (DAA) demonstrated relatively long residual soil activity ranging from 40% (Broadway) – 75% (Attribut). In the spring only Attribut showed a low level of soil activity.

The soil acting herbicides (Cadou and Malibu) as well as Lexus+Stomp aqua showed the high residual soil activities 3 DAA and 28 DAA (90-99%) which lasted until the spring (168 DAA), showing soil activities ranging from 50-55%.

Table 61: Residual soil activity (in % reduction relative to untreated) of different herbicides against *A. myosuroides* which was sown in treated soil after 3, 28 and 168 days after application (DAA) in a field experiment in Wicker-Straßenmühle in 2010/2011

	Rate	HE in% rel. to untreated		
Herbicide treatment	**L/kg/ha**	**3 DAA**	**28 DAA**	**168 DAA**
Ralon Super	1,2*	0	0	0
Topik 100	0,6*	0	0	0
Axial 50 EC	1,2*	10	3	0
Broadway	0,275*	90	40	0
Atlantis WG	0,5*	85	60	0
Lexus	0,02*	75	45	0
Attribut	0,1*	95	75	15
Lexus+Stomp aqua	0,02*+2,5	90	96	50
Cadou SC	0,5*	95	95	55
Mailbu	4*	95	99	50
untreated		0	0	0
untreated (plants/m^2)		104	124	137

HE = herbicidal efficacy in % control according to visual ratings, DAA= time period in days after application until *A. myosuroides* was sown, * = maximum registered field rate

Experimental station Wicker-Staßenmühle (STR) in 2011/12

At the time of the final countings in the spring (23.04.12) there were different levels of infestations of *A. myosuroides* in untreated plots within the 4 different sowing times ranging from 24 (17 DAA) to 112 (138 DAA) plants/m^2. The residual soil activity of herbicides are shown in Tab. 62 as reductions in infestations with *A. myosuroides* (% relative to the appropriate untreated) which was sown in treated plots 1, 17, 38 and 138 days after application (DAA). Efficacy ratings (HE) on the soil activity conducted on 04.05.12 are also shown in the result tables and support the data presented.

The ACCase- inhibitors at full recommended field rates did not show any residual soil activity. However, Axial led to little soil activity in the beginning shortly after application (1 DAA), which was significant. The ALS-inhibitors (Atlantis, Broadway, Lexus and Attribut) showed decent to relatively high soil activity on *A. myosuroides* which was sown 1 DAA ranging from 54% (Atlantis WG) to 73% (Attribut). While Atlantis, Lexus and Attribut demonstrated residual soil activity ranging from 38% (Lexus, Attribut) to 55% (Atlantis) also 17 DAA, Broadway did not lead to significant soil activity anymore. Even 38 DAA Atlantis and Attribut exhibited significant reductions of sown black-grass infestation compared to untreated plots. Even though there were no significant reductions in plant numbers at the sowing time conducted 138 DAA, herbicidal efficacies, visible as stunting symptoms, clearly demonstrated a long residual activity by Atlantis until the spring (138 DAA) .

The emergence herbicides (Cadou, Malibu, Stomp) as well as Lexus+Stomp showed significant soil activities from the beginning until the spring ranging from 51% (Stomp 1 DAA) to 97% (Malibu 17 DAA). Especially pendimethalin containing treatments such as Malibu, Stomp and Lexus+Stomp showed the highest soil activities 17 DAA and 38 DAA with at least 90% or more. Cadou demonstrated a similar high level of persistent soil activity in autumn (1-38 DAA) ranging from 76-80%. In the spring (138 DAA) the highest black-grass reductions were again from pendimethalin containing herbicides with 69% (Malibu) to 88% (Lexus+Stomp), whereas Cadou led to 52%, which was partially significantly different. Visual efficacy ratings (HE) clearly support these asseesments and observations made on residual soil activity.

Table 62: Residual soil activity (in % reduction relative to untreated and efficacy ratings) of different herbicides against *A. myosuroides* which was sown in treated soil after 1, 17, 38 and 138 days after application in a field experiment in Wicker- Straßenmühle in 2011/2012

Herbicide treatment	**Rate L/kg/ha**	**% reduction in plant numbers rel. to untreated**				**HE in% rel. to untreated**			
		1 D	**17 D**	**38 D**	**138 D**	**1 D**	**17 D**	**38 D**	**138 D**
Ralon Super	1.2	-9 g	1 de	23 bcd	-10 de	0	0	0	0
Topik 100	0.6	6 fg	4 de	5 cd	-14 e	0	0	0	0
Axial 50 EC	1.2	21 ef	-2 e	5 cd	-1 cde	17	0	0	0
Broadway	0.275	62 bcd	13 de	3 cd	12 cde	82	22	0	0
Atlantis WG	0.5	54 cd	55 bc	45 b	18 cd	88	67	70	58
Lexus	0.02	63 bcd	38 cd	5 cd	20 c	86	33	14	22
Attribut	0.1	73 ab	38 cd	29 bc	22 c	92	33	23	17
Lexus+ Stomp aqua	0.02+ 2.5	71 bc	97 a	90 a	88 a	88	99	92	93
Cadou SC	0.5	77 ab	80 ab	76 a	52 b	93	91	89	66
Mailbu	4	88 a	97 a	90 a	69 ab	96	95	92	77
Stomp aqua	4.4	51 d	94 ab	94 a	84 a	60	97	95	94
Boxer	5	33 e	55 bc	14 cd	-10 de	30	58	7	8
untreated		0 g	0 de	0 d	0 cde	0	0	0	0
untreated (plants/ m^2)		65	24	46	112				

Values in columns followed by different letters are significantly different (p = 0.05) according to LSD-tests,rel.= relative, HE= herbicidal efficacy in% control according to visual ratings; D = time period in days after application until *A. myosuroides* was sown

4.3 Management of *A. myosuroides*

4.3.1 Greenhouse experiments

4.3.1.1 Effect of application timing on the herbicidal efficacy of ALS- and ACCase-inhibitors against different populations of *A. myosuroides* in pot experiments

Herbicide treatment, growth stage of black-grass at application and the *A. myosuroide*s population had highly significant effects (p < 0.001) on the biomass reductions of *A. myosuroides* (Tab. 63). There were highly significant two-way interactions for herbicide treatment*growth stage, growth stage*population and herbicide treatment*population for biomass reductions. Furthermore, significant three-way interactions also occurred for these factors.

Table 63: Results of ANOVA analysis for biomass reductions (fw. shoot) of *A. myosuroides* by the factors Growth stage, Population and Herbicides

Fixed effects	**parameter**	**df**	**p-value**
Growth stage	shoot fw red.	1	< 0.001
Population	shoot fw red.	2	< 0.001
Herbicide treatment	shoot fw red.	15	< 0.001
Growth stage * Population	shoot fw red.	2	< 0.001
Growth stage * Herbicide treatment	shoot fw red.	15	< 0.001
Population * Herbicide treament	shoot fw red.	30	< 0.001
Growth stage * Population * Herbicide	shoot fw red.	30	< 0.001

p = 0.05, fw = fresh weight, red.= reduction, df = degree of freedom

The average shoot biomasses (fw) of untreated *A. myosuroides* plants 42 days after application were in the range from 16.2 (GB) to 33.1g (Standard) per pot for the early post application (at BBCH 12). The biomasses of *A. myosuroides* which as sown earlier for the later application timing (BBCH 23) had higher fresh weights ranging from 20.4 (RH) to 52.0g per pot (standard). The effect of herbicide treatments on the fresh weights (fw) of *A. myosuroides* shoots are shown in Tab. 64 as fw red.% compared to the referring untreated.

All herbicides showed a high level of control against the sensitive *A. myosuroides* population at all rates, independent from the growth stage, resulting in biomass reductions ranging from 91 to 99%, which did not differ significantly from each other. The efficacy against *A. myosuroides* at growth stage 12 (BBCH 12) was often better than the activity at BBCH 23, but not significantly.

The ACCase-inhibitors did not show any significant herbicidal activity against the black-grass population from RH at neither the early nor the late application timing. When applied at the early growth stage (BBCH 12) of black-grass, Atlantis and Broadway at high and low dosages showed very high levels of black-grass control

(90-97%). Relatively good weed control was achieved by Attribut (79%), whereas Lexus showed poor efficacy (50%). Altantis was often slightly better than Broadway and Attribut, but differences were not significant. Clearer differences occurred when herbicides were applied at the later growing stage, because then Attribut and Lexus did not show any significant weed control any more. At the tillering-stage of the target weed (BBCH 23), not even Broadway and Atlantis led to acceptable levels of black-grass reductions, ranging from 35 to 70%. Even though there were no significant differences on the same dosage level, Atlantis showed superior efficacies than Broadway, especially when lower rates such as 3/5 or 2/5 of recommended field rates were applied. Comparing the two application timings, the level of weed control achieved by ALS-inhibitors decreased by 25 to 60% with a later application timing (BBCH 23), which was often significant.

The population GB was not acceptably controlled by any applied herbicide neither at the early nor at the later timing. While practically no differences between treated and control plants were detectable after all other treatments, Atlantis at double the recommended field rate (X2) showed the highest significant and visible activity of 67% at the early application timing.

Table 64: Effect of application timing on the herbicidal efficacy ALS- and ACCase-inhibitors against different populations of *A. myosuroides* (sensitive standard, Rauischholzhausen, GB10089) in a pot experiment

			Standard		**RH**		**GB**	
			GS 12	**GS 23**	**GS 12**	**GS 23**	**GS 12**	**GS 23**
Herbicide treatment	**Rate kg/L/ha**	**Field rate**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**	**fw red. (%)**
Ralon Super	1.2	F	91lmno	91mno	14abcdef	11abcde	1abc	4abc
Topik	0.6	F	99o	95no	-2ab	24abcdefg	0abc	5abcde
Axial EC50	1.2	F	99o	95no	4abcd	24abcdefg	7abcde	6abcde
Broadway	0.44	X2	99o	95no	97no	70jklmno	15abcdef	5abcde
	0.22	F	99o	95no	95no	57hijk	2abc	-2ab
	0.18	4/5F	99o	95no	96no	60ijkl	-4a	0abc
	0.13	3/5F	99o	95no	95no	35efghi	0abc	-1abc
	0.09	2/5F	99o	95no	90lmno	44fghij	3abc	-1abc
Atlantis WG	1	X2	99o	95no	96no	70jklmno	67jklmn	27bcdefgh
	0.5	X	99o	95no	97no	72jklmno	35defghi	8abcde
	0.4	4/5F	99o	95no	97no	71jklmno	22abcdefg	15abcdef
	0.3	3/5F	99o	95no	96no	64ijklm	11abcde	12abcde
	0.2	2/5F	99o	95no	95no	57hijk	18abcdef	13abcde
Attribut	0.1	F	99o	96no	79klmno	29cdefgh	0abc	6abcde
Lexus	0.02	F	99o	95no	50ghijk	27bcdefgh	8abcde	7abcde
untreated			0abc	0abc	0abc	0abc	0abc	0abc
untreated (in g)			33.1	52	16.5	20.4	16.2	21.4

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, GD5%:30.7%; fw = fresh weight, red.=reduction, GS = Growth stage according to BBCH, RH.= Rauischholzhausen, GB = English population; F = full field dose, X2 = double of the field dose; final assessments 42 DAA, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule

4.3.1.2 Effect of application timing on the herbicidal efficacy of pre-emergence herbicides against different populations of *A. myosuroides* in pot experiments

Herbicide treatment, growth stage of black-grass at application and the A. myosuroides population had highly significant effects ($p < 0.001$) on the biomass reductions of *A. myosuroides* (Tab. 65). There were highly significant two-way interactions for herbicide treatment*growth stage and herbicide treatment x population for biomass reductions. Significant three-way interactions also occurred for the factors.

Table 65: Results of ANOVA analysis for biomass reductions (fw. shoot) of *A. myosuroides* by the factors Growth stage, Population and pre-emergence Herbicides treatments

Fixed effects	parameter	**df**	**p-value**
Growth stage	shoot fw red.	2	<0.001
Population	shoot fw red.	1	<0.001
Herbicide treatment	shoot fw red.	6	<0.001
Growth stage x Population	shoot fw red.	2	0.157
Growth stage x Herbicide treatment	shoot fw red.	12	<0.001
Population x Herbicide treatment	shoot fw red.	6	<0.001
Growth stage x Population x Herbicide	shoot fw red.	12	0.011

p = 0.05%, fw = fresh weight, red.= reduction, df = degree of freedom

The average shoot biomasses (fw) of untreated *A. myosuroides* plants were 14.8 to 16g per pot 38 days after herbicide application. The biomasses of untreated *A. myosuroides* which was sown earlier for the application timing at BBCH 10 were on average 24.9 to 27.7g per pot. Plants which were sprayed at BBCH 11 reached biomasses from 31.7 to 34.2g per pot. The effect of herbicide treatments on the fresh weights (fw) of *A. myosuroides* shoots are shown in Tab. 66 as fw red% compared to untreated.

Cadou showed a high level of weed control against the standard population of *A. myosuroides* at all rates, independent from growth stage, resulting in biomass reductions ranging from 88-100%, which did not differ significantly from each other. A relatively high level of weed control was also observed with Stomp aqua at 1.099 L/ha (91%) and at 0.549 L/ha (70%) applied in pre-emergence (BBCH 00), whereas the lowest dosage (0.275 L/ha) and later applications at BBCH 10 and 11 of the target weed often did not result in significant shoot reductions.

The population from Rauischholzhausen was well controlled (90-96%) with all dosages of Cadou which were applied pre-emergence. A significant dosage effect was observed when the application was conducted at BBCH 10, when the lowest dosage of Cadou showed significantly lower efficacies (74%) than the higher dosages. This effect was even stronger with treatments at BBCH 11, when only the highest dosage led to a high activity of 96%, whereas the lower dosages of Cadou (0.063, 0.031 L/ha) showed significantly lower herbicidal efficacies (55% and 68%). Similar to above, Stomp aqua showed a decent level of significant weed control at 1.099 L/ha and 0.549 L/ha (62-64%) when applied in pre-emergence (BBCH 00), whereas the lowest dosage (0.275 L/ha) as well as later applications at BBCH 10 and 11 resulted in low herbicidal activities, which were often not significant.

Cadou and Stomp aqua reached the highest weed control when they were applied pre-emergence. Both herbicides showed decreases of fw reductions with increasing growth stages of *A. myosuroides*, which were partially significant. Furthermore, Cadou and Stomp showed generally higher levels of weed control against the standard population compared to the black-grass population from Rauischholzhausen. Differences in sensitivity of the two populations against the two soil herbicides were partially significant, especially with Stomp aqua (1.099 L/ha), as well as for Cadou at lower dosages with later application timings.

Table 66: Effect of application timing on the herbicidal efficacy (shoot fw reduction in % relative to untreated) of pre-emergence herbicides against *A. myosuroides* from two different populations of in pot experiments

Herbicide treatment	Rate kg/L/ha	Field rate	BBCH 00 fw red. (%)		BBCH 10 fw red. (%)		BBCH 11 fw red. (%)	
Population: Standard								
Cadou SC	0.125	1/2F	99	k	100	k	99	k
Cadou SC	0.063	1/4F	100	k	100	k	98	k
Cadou SC	0.031	1/8F	96	jk	97	k	88	hijk
Stomp aqua	1.099	1/4F	91	ijk	29	de	20	abcde
Stomp aqua	0.549	1/8F	70	ghi	22	abcde	6	abc
Stomp aqua	0.275	1/16F	25	cde	13	abcd	10	abcd
untreated			0	a	0	a	0	a
untreated (in g)			14.8		24.9		34.2	
Population: Rauischholzhausen								
Cadou SC	0.125	1/2F	96	jk	98	k	96	jk
Cadou SC	0.063	1/4F	98	k	88	hijk	68	gh
Cadou SC	0.031	1/8F	90	hijk	74	ghij	55	fg
Stomp aqua	1.099	1/4F	64	g	23	bcde	23	bcde
Stomp aqua	0.549	1/8F	62	g	27	cde	1	ab
Stomp aqua	0.275	1/16F	38	ef	27	cde	10	abcd
untreated			0	a	0	a	0	a
untreated (in g)			16		27.6		31.7	

Values in columns and lines followed by different letters are significantly different (p = 0.05) according to LSD-tests, GD5%: 22.7%; fw = fresh weight, red.= reduction, F= full field dose; final assessments were conducted 42 DAA, SC = Soluble Concentrate

4.3.2 Field experiments

The effect of sowing time and cultivation on the infestation with *A. myosuroides* in the different field experiments from 2009-2011 are summarized in Tab. 67. The reductive effect by a delayed sowing time for winter wheat is presented in the following paragraphs in more detail, and the results of field experiments are shown for each trial location. Furthermore, the effect by using mouldboard ploughing as cultivation is described in more detail below.

Table 67: Effect of sowing time and cultivation on the infestation with *A. myosuroides* in the different field experiments from 2009-2011

	Early sowing time		**Late sowing time**	
Location	**plants/ m^2**	**Heads/m^2**	**plants/ m^2**	**Heads/m^2**
RH (2009)	990 (21.10)	3340	336*(24.03)	775
GI (2009)	975 (27.10.)	718	39 (24.03)	244
WI (2009)	199 (25.11)	133		
RH (2010)	347 (14.10)	1039	46 (23.03.)	318
RH (2011)	630 (25.10.)	-	58 (19.04)	-

	Chisel ploughing		**Ploughing**	
Location	**plants/ m^2**	**Heads/m^2**	**plants/ m^2**	**Heads/m^2**
RH (2010)	347 (14.10)	1039	37 (14.10.)	88
RH (2011)	630 (25.10.)	-	192 (25.10.)	-

Plants were counted at the herbicde application in autumn or spring, heads (inflorescens) of *A. myosuroides* were counted in May/June, ES = early sowing of winter wheat, LS = late sowing time of winter wheat, RH = Rauischholzhausen, WI = Wicker, GI = Gießen

4.3.2.1 Field experiments in Gießen 2009/2010

The performance of different herbicide applications in early and late sown winter wheat are summarized in Table 68. The early sowing of winter wheat (30.09.2009) resulted in a high infestation with *A. myosuroides* in untreated plots, consisting of 975 plants/m^2 in autumn and 718 heads/m^2 in the spring (Tab. 67). Most herbicide treatments gave high levels of control of *A. myosuroides* ranging from 88% (Cadou SC) to 99% (Broadway). The countings of inflorecences of *A. myosuroides* were generally in line with the efficacy ratings.

The yield of winter wheat in untreated plots was 35.5 dt/ha. Due to the effective control of black-grass, all herbicides significantly increased the yield to over 100 dt/ha. Applications with Broadway, Atlantis, Alister, Ralon Super+Topik and Ralon Super+Axial showed the highest yields ranging from 115.9–120.4 dt/ha but did not differ significantly from each other.

Delaying the sowing time substantially reduced black-grass infestation to 39 plants/m^2 and 244 heads/m^2 (Tab. 67 and Tab. 68) compared with the earlier sowing time and resulted in a high yield (108.3 dt/ha) in control plots. Herbicide treatments applied in the spring showed nearly 100% weed control and resulted in yields ranging from 112.5 dt/ha (Topik) to 115.5 dt/ha (Atlantis). Applications in autumn with soil-herbicides also gave high levels of weed control and yields ranging from 106.7 dt/ha (Herold) to 112.7 dt/ha (Cadou), which did not significantly differ from each other.

Table 68: Effect of herbicides on density of *A. myosuroides* and grain yield of wheat in early and late sown winter wheat in Gießen (mouldboard ploughing, no herbicide resistances) in season 2009/2010.

		Early sowing (ES)					**Late sowing (LS)**				
Herbicide Treatment	**Dose (L/kg/ha)**	**BBCH**	**ALOMY Heads**		**HE (%)**	**GY (dt ha)**	**BBCH**	**ALOMY Heads**		**HE (%)**	**GY (dt/ha)**
			(m^2)	**(red%)**				**(m^2)**	**(red%)**		
Ralon Super	1.2	12	98_a	86	90	103.9_{bc}	12−22	0_a	100	99	114.0_b
Topik	0.6	12	58_a	92	92	110.6_{bcde}	12−22	3_a	99	99	112.5_{ab}
Axial 50EC	0,9	12	29_a	96	93	111.1_{cdef}	12−22	3_a	99	99	112.6_{ab}
Broadway+NM	0.22+1	12	3_a	100	99	120.4_g	12−22	1_a	100	99	112.9_{ab}
Atlantis WG+FH	0.4+0.8	12	5_a	99	96	119.6_{fg}	12−22	4_a	98	99	115.5_b
Alister	1	12	33_a	95	96	118.7_{efg}	12−22	0_a	100	99	113.4_{ab}
Ralon S.+Topik	1+0.4	12	5_a	99	96	115.9_{defg}	12−22	3_a	99	99	114.0_{ab}
Ralon S.+Axial	1+0.9	12	18_a	97	96	116.0_{defg}	12−22	1_a	100	99	113.1_{ab}
Herold SC	0.6	9−11	34_a	95	96	109.5_{bcd}	12−22	24_a	90	98	106.7_a
Cadou SC	0.5	9−11	60_a	92	88	102.5_b	12−22	13_a	95	97	112.7_{ab}
untreated			718_b	−	0	35.5_a	12−22	244_b	−	0	108.3_{ab}
LSD $_{0.05}$			221			8.5		45			7.3

Values in a column followed by different letters are significantly different (p = 0.05), ALOMY= *Alopecurus myosuroides*, FH = Biopower, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, GY = grain yield of wheat (dt/ha), HE = Herbicide efficacy (%) was assessed on 08.04.11 (ES) and 25.05.11 (LS); heads (inflorecenses) of *A. myosuroides* were counted on 10.05.11 (ES) and 07.06.11 (LS)

4.3.2.2 Field experiments in Wicker 2009/2010 and 2010/2011

The performance of different herbicide applications in the field experiments in winter wheat which were conducted 2009/10 and in 2010/11 in Wicker are summarized in Table 69. The spreading of seeds just before sowing of winter wheat resulted in infestations at the time of post emergence herbicide application (25.11.09) with *Alopecurus. myosuroides* and *Bromus secalinus* consisting of 199 plants/m^2 and 189 plants/m^2 respectively. All herbicides showed a high efficacy against *A. myosuroides*, ranging from 95-97%, which did not differ much from each other. In the spring there where 133 heads of *A. myosuroides* per m^2 present in untreated plots. All herbicides offered almost 100% control. Looking at the efficacy against *B. secalinus*, only the herbicides Broadway and Attribut demonstrated a high level of control. In the season 2010/2011, there was only a low natural infestation of *A. myosuroides* (43 plants/m^2).

All herbicide treatments showed nearly 100% control. Because of the severe drought period in May 2011, the trial had to be cancelled.

Table 69: Effect of herbicides on density of *A. myosuroides* in winter wheat in Wicker (chisel ploughing, no herbicide resistances) in season 2009/2010 and 2010/2011

			2009/2010			**2010/2011**
Herbicide Treatment	**Dose (L/kg ha)**	**BBCH**	**ALOMY Heads (m^2)**	**ALOMY HE (%)**	**BROSE HE (%)**	**ALOMY HE (%)**
Ralon Super	1,2	12	0	95	3	99
Topik100	0,6	12	0	95	0	99
Axial 50EC	0,9	12	0	95	0	99
Broadway+NM	0.22+1	12	0	97	97	99
Atlantis WG+FHS	0.4+0.8	12	0	97	35	99
Alister	1	12	0	95	44	99
Ralon S.+Topik	1+0.4	12	0	95	3	99
Ralon S.+Axial	1+0.9	12	0	95	3	99
Attribut	0.1	12	0	97	99	99
Herold SC	0.6	9−11	0	97	24	99
Cadou SC	0,5	9−11	0	97	18	98
Malibu	4	9−11	-	-	-	99
Cadou SC+ Bacara forte	0.3+ 0.75	9−11	-	-	-	99
Lexus	0.02	9−11	-	-	-	99
Lexus+Stomp	0.02+2.5	9−11	-	-	-	99
untreated			133	0	0	0

Weed Infestation on 25.11.09 ALOMY: 199 Plants/ m^2, BROSE: 189 plants/m^2, Weed infestation on 16.11.10 ALOMY 43 Plants/ m^2, NM= Broadway Netzmittel, FHS=Biopower, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, ALOMY = *Alopecurus myosuroides*, BROSE = *Bromus secalinus*, HE = Herbicide efficacy (%) was assesed on 15.04.10 and 08.04.11; heads (inflorescences) of *A. myosuroides* were counted 10.05.10

4.3.2.3 Field experiments in Rauischholzhausen 2009/2010

The performance of herbicide applications in early and late sown winter wheat are shown in Tab. 70. The early sowing (22.09.2009) resulted in high infestations with *A. myosuroides*, consisting of 990 plants/m^2 (Tab. 67 and Tab. 70) in October and 3340 heads/m^2 in May, which led to a very low wheat yield of 17 dt/ha in untreated plots. Single applications with ACCase-inhibitors (Ralon Super, Topik, Axial) were more or less ineffective (12-20% weed control, 26.1–36.9 dt/ha). The mixture of different ACCase-inhibitors increased the efficacy to a maximum of 30% (Ralon Super+Axial). In contrast, the used ALS-inhibitors were more effective against *A. myosuroides* and showed a level of weed control ranging from 80% (Broadway) to 97% (Atlantis) which caused yields ranging from 62.9 dt/ha to 78 dt/ha. Applications during post-emergence with herbicides containing mesosulfuron (Atlantis WG, Alister) showed the lowest number of heads/m^2 of black-grass in the spring (Atlantis 15 heads/m^2, Alister 84 heads/m^2) and resulted in the highest yields (75.1-78 dt/ha). Weed control with pre-emergence herbicides was less effective (55–60%) and produced a yield of 58 dt/ha (Cadou) and 56.7 dt/ha (Herold) respectively.

A pre-emergence treatment with Cadou (0.5 L/ha) in autumn followed by Atlantis (0.4 kg/ha) in spring showed a high level of weed control (85%) and increased the yield to 67.3 dt/ha (Tab. 72). Applications with fairly ineffective ACCase-inhibitors in autumn followed by applications with ALS-inhibitors (Attribut, Atlantis, Lexus, Broadway) at recommended field rates led to yields ranging from 43.3 – 45.1 dt/ha, but the differences were not significant.

Delaying the sowing time (03.11.2009) substantially reduced black-grass infestation by 66% (336 plants/m^2 in April, 775 heads/m^2 in May) compared with the earlier sowing time with a yield of 58.9 dt/ha (Tab. 67 and 70). The application with Axial showed a low herbicidal efficacy of 38% but increased the yield significantly (72.7 dt/ha). All herbicides containing ALS-inhibitors showed a high level of weed control ranging from 89% (Lexus) to 98% (Atlantis) and increased the yield ranging from 83 to 86.3 dt/ha. Differences between the untreated control and Axial were statistically significant. The presence of fewer than 5 heads/m^2 of *A. myosuroides* showed effective weed control by Atlantis even at low rates.

Table 70: Effect of herbicides on density of *A. myosuroides* and grain yield of wheat in early and late sown winter wheat in Rauischholzhausen (chisel ploughing, herbicide resistances) in season 2009/2010

Early sowing (ES)						
Herbicide Treatment	**Dose (L/kg ha)**	**BBCH**	**ALOMY Heads (m^2)**	**ALOMY Heads (red.%)**	**HE (%)**	**GY (dt ha)**
Ralon Super	1.2	12	—		12	30.0_b
Topik100	0.6	12	—		12	26.1_b
Axial 50EC	0,9	12	—		20	36.9_c
Broadway+NM	0.22+1	12	409_b	88	80	62.9_e
Atlantis WG+FHS	0.4+0.8	12	15_a	100	97	78.0_f
Alister	1	12	84_a	97	95	75.1f
Ralon S.+Topik 100	1+0.4	12	—		18	31.2_b
Ralon S.+Axial 50EC	1+0.9	12	—		30	40.4_c
Herold SC	0.6	9−11	508_b	85	60	56.6_d
Cadou SC	0.5	9−11	594_b	82	55	58.0_{de}
Untreated		—	3340_c	—	0	17.0_a
LSD $_{0.05}$			268			5.4
Late sowing (LS)						
Atlantis WG+FHS	0.3+0.6	12−22	4_a	99	97	83.2_c
Atlantis WG+FHS	0.5+1	12−22	1_a	100	98	84.8_c
Axial 50EC	1.2	12−22	450_d	42	38	72.7_b
Broadway+NM	0.22+1.0	12−22	3_a	100	95	82.3_c
Atlantis WG+FHS	0.4+0.8	12−22	15_{ab}	98	96	84.7_c
Alister	1	12−22	9_{ab}	99	95	86.3_c
Broadway+NM	0.275+1	12−22	15_{ab}	98	94	83.5_c
Attribut+Mero	0.06+1	12−22	40_b	95	92	83.5_c
Attribut+Mero	0.1+1	12−22	10_{ab}	99	95	84.9_c
Lexus+Trend 90	0.02+0.3	12−22	83_c	89	89	84.7_c
untreated			775_e	—	0	58.9_a
LSD $_{0.05}$			32			4.1

Values in a column followed by different letters are significantly different (p = 0.05), ALOMY = *Alopecurus myosuroides*,NM= Broadway Netzmittel, FHS=Biopower, IPU = isoproturon, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate,GY=grain yield of wheat (dt/ha), HE = Herbicide efficacy (%) was assessed on on 07.04.10 (ES) and 05.05.10 (LS), heads (inflorescences) of *A. myosuroides* were counted in 25.05.2010

4.3.2.4 Field experiments Rauischholzhausen 2010/2011

The performance of herbicide applications in early sown winter wheat after chisel ploughing and conventional ploughing are shown in Table 71. The early sowing of winter wheat (22.09.2010) resulted in a high infestation with *A. myosuroides* in unsprayed plots with 347 plants/m^2 in October 2010 and 1039 heads/m^2 in May 2011 (Tab. 67 and Tab. 71), which resulted in an average yield of 40 dt/ha. Most post-emergence herbicides (ALS-inhibitors) showed a moderate to high level of weed control ranging from 81% (Stomp aqua+Lexus) to 98% (Atlantis) and produced yields ranging from 62.2 to 82.1 dt/ha. In contrast, weed control was poorer for all herbicides treatments applied during pre-emergence, resulting in yields ranging from 48.3 dt/ha (Herold) to 54.7 dt/ha (Fenikan+IPU), but the differences were not statistically significant. Treatments with Herold (0.6 L/ha) in autumn followed by different ALS-inhibitors in the spring showed high levels of weed control (91–97%) and yields ranging from 68.2 (Herold+Lexus) to 74.4 dt/ha (Herold+Atlantis) (Table 72).

Weed populations with *A. myosuroides* were 89% lower on ploughed plots compared with shallow chisel ploughed ones, counting 37 plants/m^2 in October (Tab. 67 and Tab. 71) and 88 heads/m^2 in May, which resulted in a yield of 73.1 dt/ha in untreated plots. The combination of ploughing with herbicide treatment either in pre- or post-emergence resulted in high levels of weed control ranging from 88% (Bacara Forte+Cadou) to 98% (Atlantis, Lexus) and significantly higher yields ranging from 85.2 dt/ha (Cadou) to 92.5 dt/ha (Herold+Atlantis). Whereas many herbicide treatments reduced the weed pressure to lower than 25 heads/m^2 on ploughed land, the best single herbicide treatment (Atlantis) on chisel ploughed land still resulted in the presence of over 100 heads/m^2 and reduction of just 90% compared to the untreated control. In general, the reductions in the number of inflorescences of *A. myosuroides* were lower compared to the efficacy ratings which were conducted in early spring.

Table 71: Effect of herbicides on density of *A. myosuroides* and grain yield of wheat in early sown winter wheat after mouldboard ploughing and chisel ploughing cultivation in Rauischholzhausen (herbicide resistances) in season 2010/2011

Herbicide Treatment	**Dose (L/kg ha)**	**BBCH**	**Chisel ploughing**					**Ploughing**				
			ALOMY Heads		**HE (%)**	**GY (dt/ha)**	**WH (m^2)**	**ALOMY Heads**		**HE (%)**	**GY (dt ha)**	**WH (m^2)**
			(m^2)	**red(%)**				**(m^2)**	**red(%)**			
Broadway+ NM	0.22 +1	12	440_{bc}	58	91	64.8_{de}	355_{c}	23_{bc}	74	94	89.2_{de}	361_{bcd}
AtlantisWG+ FHS	0.4+ 0.8	12	100_{a}	90	98	82.1_{g}	365_{c}	4_{a}	95	98	91.3_{de}	368_{cd}
Alister	1	12	251_{ab}	76	94	74.2_{f}	362_{c}	9_{ab}	90	97	91.1_{de}	369_{cd}
Lexus + Trend 90	0.02+ 0.3	12	462_{bc}	56	93	68.0_{def}	340_{c}	11_{ab}	88	98	90.3_{de}	366_{cd}
Stomp Aqua +Lexus	2.5+ 0.02	12	740_{d}	29	81	62.2_{cd}	314_{bc}	21_{abc}	76	93	89.8_{de}	364_{cd}
Herold SC+ AtlantisWG+ FHS	0.6+ 0.4+ 0.8	12	269_{ab}	74	93	71.8_{ef}	353_{c}	6_{a}	93	97	92.5_{e}	374_{d}
Cadou SC	0.5	9−11	813_{de}	22	39	50.2_{b}	231_{a}	21_{abc}	76	89	85.2_{b}	345_{ab}
Herold SC	0.6	9−11	765_{d}	26	44	48.3_{b}	262_{ab}	15_{abc}	83	89	88.2_{bcd}	357_{bc}
Baccara F.+ Cadou SC	0.75+ 0.3	9−11	718_{d}	31	45	52.4_{b}	236_{a}	28_{bc}	68	88	87.9_{bcd}	356_{bc}
Malibu	4	9−11	895_{de}	14	45	51.7_{b}	248_{a}	14_{abc}	84	95	89.8_{de}	364_{cd}
Stomp A.+ IPU	2.5+ 2.5	9−11	853_{de}	18	41	52.8_{b}	240_{a}	19_{abc}	78	93	88.8_{cd}	360_{bcd}
Fenikan+ IPU	2+ 1	9−11	675_{cd}	35	50	54.7_{bc}	240_{a}	33_{c}	63	91	88.2_{bcd}	357_{bc}
untreated			1039_{e}	–	–	40.0_{a}	231_{a}	88_{d}	–	–	73.1_{a}	336_{a}
LSD $_{0.05}$			248			7.5	60	21			3.5	16

Values in a column followed by different letters are significantly different ($p = 0.05$), ALOMY = *Alopecurus myosuroides*, NM = Broadway Netzmittel, FHS = Biopower, IPU = isoproturon, WG = Water-Dispersible Granule, SC = Soluble Concentrate GY = grain yield of wheat (dt/ha), WH = heads of wheat, HE = Herbicide efficacy (%) was assesd on 23.03.11; heads (inflorescences) of *A. myosuroides* were counted in 10.05.2011, heads of wheat on 12.07.11

Table 72: Effect of sequencial herbicide applications (autumn+spring) on density of *A. myosuroides* and grain yield of wheat in early sown winter wheat after chisel ploughing cultivation in Rauischholzhausen (herbicide resistances) in season 2009/2010 and 2010/2011.

1st application (autumn)			**2nd application (spring)**						
Herbicide Treatment	**Dose (L/kg/ha)**	**BBCH**	**Herbicide Treatments**	**Dose (L/kg/ha)**	**ALOMY Heads (m^2)**	**ALOMY Heads red(%)**	**HE (%)**	**GY (dt ha)**	**WH (m^2)**
Season 2009/2010									
Ralon Super	1.2	12	Broadway+NM	0.22+1	–		25	43.6_b	–
Topik100	0.6	12	Atlantis WG+FH	0.4+0.8	–		35	45.1_b	–
Axial 50EC	0.9	12	Attribut+Mero	0.1+1.0	–		27	43.3_b	–
Ralon S.+Topik	1+0.4	12	Lexus+Trend 90	0.02+0.3	–		18	43.1_b	–
Cadou SC	0.5	9–11	Atlantis WG+FH	0.4+0.8	–		85	67.3_c	–
Untreated	–	–	–	–	–		–	30.5_a	–
LSD $_{0.05}$								8.7	
Season 2010/2011									
Herold SC	0.6	9–11	Atlantis WG+FH	0.4+0.8	0_a	100	97	74.4_c	322_c
Herold SC	0.6	9–11	Broadway+NM	0.22+1.0	118_a	81	94	69.9_b	298_{bc}
Herold SC	0.6	9–11	Attribut+Mero	0.1+1.0	198_a	68	94	70.1_{bc}	326_c
Herold SC	0.6	9–11	Lexus+Trend 90	0.02+0.3	243_a	60	91	68.2_b	276_b
untreated	–	–	–	–	613_b			48.7_a	205_a
LSD $_{0.05}$					296			4.2	44

Values in a column followed by different letters are significantly different (p = 0.05), ALOMY= *Alopecurus myosuroides*, NM = Broadway Netzmittel, FH = Biopower, IPU = isoproturon, EC = Emulsifiable Concentrate, WG = Water-Dispersible Granule, SC = Soluble Concentrate, GY = grain yield of wheat (dt/ha), WH = heads of wheat, HE = Herbicide efficacy (%) was assessed on 05.05.10 and 09.05.11; heads (inflorescences) of *A. myosuroides* were counted on 25.05.10 and 30.05.2011.

The performance of herbicide applications in late sown winter wheat are shown in Table 73. Delaying the sowing time (28.10.2010) substantially reduced the blackgrass infestation by 87% (46 plants/m^2 in October and 318 heads/m^2 in May) compared with the earlier sowing time (Tab. 67), and resulted in a high yield of 84.2 dt/ha. Herbicide treatments showed a low to high efficacy range from 25% (IPU) to 95% (Atlantis WG) and increased the yield range from 85.6 to 89.1 dt/ha significantly compared with the control, but had significant differences among each other.

Table 73: Effect of herbicides on density of *A. myosuroides* and grain yield of wheat in late sown winter wheat after chisel ploughing cultivation in Rauischholzhausen (herbicide resistances) in season 2010/2011.

Herbicide Treatments	**Dose (L/kg ha)**	**BBCH**	**ALOMY Heads**		**HE (%)**	**GY (dt ha)**
			(m^2)	**red (%)**		
Atlantis WG+FHS	0.3+0.6	12−22	58_{ab}	82	92	87.5_{bc}
Atlantis WG+FHS	0.4+0.8	12−22	23_{a}	93	95	89.1_{c}
Atlantis WG+FHS	0.5+1.0	12−22	38_{a}	88	95	88.3_{bc}
Broadway+NM	0.22+1.0	12−22	223_{cde}	30	70	88.2_{bc}
Broadway+NM	0.275+1	12−22	225_{cde}	29	68	86.4_{ab}
Attribut+Mero	0.06+1.0	12−22	195_{cde}	39	77	86.6_{ab}
Attribut+Mero	0.1+1.0	12−22	198_{cde}	38	78	85.8_{ab}
Lexus+Trend 90	0.02+0.3	12−22	175_{bcd}	45	73	87.8_{bc}
Alister	1.0	12−22	128_{abc}	60	82	88.1_{bc}
IPU	3.0	12−22	263_{de}	17	25	85.6_{ab}
Stomp aqua+IPU	2.5+2.5	12−22	300_{de}	6	25	85.8_{ab}
untreated	−	12−22	318_{e}		0	84.2_{a}
LSD $_{0.05}$			**132**			**3.3**

Values in a column followed by different letters are significantly different (p = 0.05), ALOMY = *Alopecurus myosuroides* Huds., NM= Broadway Netzmittel, FHS = Biopower, IPU = isoproturon, WG = Water -Dispersible Granule, GY = grain yield of wheat (dt/ha), HE = Herbicide efficacy (%) was assesd on 09.05.11; heads (inflorescences) of *A. myosuroides* were counted on 30.05.11

4.3.2.5 Field experiment Rauischholzhausen 2011/2012

The performance of herbicide applications in early sown winter wheat after chisel ploughing and mouldboard ploughing are shown in Table 67 and Table 74. The early sowing of winter wheat after ploughing cultivation resulted in a 70% reduction of infestation with *A. myosuroides* in untreated plots (192 plants/m^2) compared with plots after chisel ploughing cultivation (630 plants/m^2) in the autumn (Tab. 67 and Tab. 74). All post-emergence herbicides (ALS-inhibitors) showed high levels of weed control ranging from 88% (Stomp aqua+Lexus) to 98% (Atlantis, Atlantis+Herold) after chisel cultivation and 86% (Stomp aqua+Lexus) to 99% (Atlantis, Atlantis+Herold) after ploughing, resulting in significantly reduced weed densities (chisel ploughing: 1 to 35 plants/m^2, ploughing: 1 to 25 plants/m^2) in the spring.

After chisel cultivation the applications with Malibu+Lexus+Boxer and Stomp Aqua+IPU during pre-emergence showed a high level of weed control (91-93%) and very low weed densities of 23 plants/m^2, which did not differ statistically from ALS-inhibitors. Plots applied during pre-emergence with different herbicides showed higher weed densities ranging from 78 plants/m^2 (Baccara Forte+Cadou) to 206 plants/m^2 (Cadou) and moderate efficacies (56-68%). After ploughing cultivation, most pre-emergence herbicides showed high weed control efficacy and weed densities ranging from 4–24 plants/m^2 and did not differ significantly from post-emergence herbicides. Delaying the sowing time from 29.09.2011 to 26.10.2011 substantially reduced black-grass infestation in the spring by over 90% to 58 plants/m^2, compared with 630 plants/m^2 at the earlier sowing time (Tab. 67).

Table 74: Effect of herbicides on density of *A. myosuroides* in early sown winter wheat after mouldboard ploughing and chisel ploughing cultivation in Rauischholzhausen (herbicide resistances) in season 2011/2012.

Herbicide Treatment	**Dose (L/kg ha)**	**BBCH**	**Chisel ploughing**			**Ploughing**		
			ALOMY Plants		**HE**	**ALOMY Plants**		**HE**
			(m^2)	**(red%)**	**(%)**	**(m^2)**	**(red%)**	**(%)**
Broadway+NM	0.22+1	12	22_{ab}	97	94	14_{ab}	93	95
Atlantis WG+FHS	0.4+0.8	12	3_{a}	100	97	3_{a}	98	98
Atlantis WG+FHS	0.5+1	12	3_{a}	100	98	2_{a}	99	99
Alister	1	12	8_{ab}	99	97	1_{a}	99	98
Lexus +Trend90	0.02+0.3	12	35_{abc}	94	92	10_{ab}	95	95
Stomp Aqua + Lexus	2.5+ 0.02	12	—	-	88	25_{ab}	87	86
Herold SC+ Atlantis WG+FHS	0.6+ 0.4+0.8	12	1_{a}	100	98	2_{a}	99	99
Cadou SC	0.5	9−10	206_{d}	67	56	43_{b}	78	75
Herold SC	0.6	9−10	108_{c}	83	61	34_{ab}	82	86
Baccara F.+ Cadou SC	0.75+ 0.3	9−10	78_{bc}	88	64	31_{ab}	84	88
Malibu	4	9−10	81_{bc}	87	68	22_{ab}	89	80
Malibu+ Lexus+ Boxer	4+ 0.02+ 2	9−10	23_{ab}	96	93	4_{a}	98	98
Stomp Aqua+IPU	2.5+2.5	9−10	23_{ab}	96	91	13_{ab}	93	94
untreated	—		630_{d}	-	—	192_{c}	–	—
LSD $_{0.05}$			75			35		

Values in a column followed by different letters are significantly different ($p = 0.05$), ALOMY = *Alopecurus myosuroides*, NM = Broadway Netzmittel, FHS = Biopower, IPU = isoproturon, WG = Water-Dispersible Granule, SC = Soluble Concentrate, HE = Herbicide efficacy (%) was assessed on 21.03.12, plants of *A. myosuroides* were counted in 19.04.12

5 Discussion

5.1 Emergence pattern of black-grass (*Alopercurus myosuroides*) during the growing seasons 2009-2012

An important characteristic of most agricultural weeds is the ability to emerge at a time that ensures the best conditions possible for seedling growth and competition with crops. The persistence of primary dormancy and, in many species like *A. myosuroides*, the seasonal variation of secondary dormancy are essential factors that inhibit germination during unfavorable periods and enable germination over several years (Froud-Williams et al. 1984).

For germination to take place, the level of dormancy must be low and certain environmental conditions (e.g. temperature, moisture, light, oxygen) are required. Most black-grass (*Alopecurus myosuroides*) seeds are shed prior to harvest of winter cereal crops (Moss 1983) and most seeds have a degree of primary dormancy which prevents them from germinating immediately (Moss 1980b, Colbach et al. 2002a, Colbach et al. 2002b). When seeds are produced under cool and wet periods during maturation, the primary dormancy can last up to 6-8 weeks (Moss et al. 2006). If seeds are incorporated in the soil via ploughing or chisel ploughing, they might go into secondary dormancy. Generally, *A. myosuroides* has a low seed persistence in the soil (Jensen 2009). Survival of buried seeds in the soil is about 20-30% per year, so after 3 to 4 years burial, only about 1-3% of seeds will still be viable (Moss 2010, Moss 2013). Nevertheless, Moss (1985c) has shown that a small proportion of seeds are likely to persist for many years and possibly act as a source for future weed infestations which can build-up very quickly. Menck (1968) has shown that seeds are of persisting in soil for at least for 9 years.

However, with the initial primary dormancy of *A. myosuroides* seeds of short duration and given favorable environmental conditions such as soil temperatures of 10-15°C with adequate soil moisture, many seeds are capable of germinating from August to November from the first few centimeters down the soil profile (Menck 1968, Naylor 1970, Moss 1980a, Moss 1985c). This period coincides with the sowing of winter cereals in Germany, so in the majority of cases black-grass emerges prior or along with the crop (Moss 1980b). Seedlings emerging prior to sowing can easily be destroyed by cultivations and/or use of a non-selective herbicide, while those emerging within the crop require the application of selective herbicides. Provided there is sufficient moisture, about 80% of black-grass germination and emergence occurs in the autumn (Thurston 1972, Moss 2010, Moss 2013). Kampe (1975) investigated the emergence dynamics of black-grass and Apera spica-venti in winter-sown cereals over three years in the Pfalz region. He found that in early sown winter wheat, on average 52% of black-grass was emerged by mid November and 79% by

December, with a yearly variation of 67-91%. In his experiments the emergence of black-grass occurred continually in January and February and reached 100% either in March or April.

In contrast, the emergence of black-grass in late sown winter wheat was more variable according to Kampe (1975) and reached in December 2-42%, in January 26-77% and in March over 95% on average. In Germany, the main germination period for black-grass is from September to November. By December, 80% of black-grass has emerged and from February to April another small flush of black-grass is possible (Bayer CropScience 2004)(compare chapter 4.1). Received monitoring data from field trials of this research demonstrate a similar emergence pattern and confirm the publications listed above. After early sown winter wheat, peak emergence of *A. myosuroides* (74 to 99%) mainly occured during the 4-6 week period from sowing until the application of herbicides, which reveals that the primary dormancy was generally of short duration. Nevertheless, in most experiments a protracted germination period of black-grass was observed after post-emergence herbicide application followed by another flush of relatively small extent in the spring. However, should be noted that these observations were conducted in plots which were sprayed with glyphosate, thus ensuring no competition by neither crop nor other weeds and full light exposure. Therefore, received results could only be seen as a potential emergence of black-grass.

Anderson and Espeby (2009) concluded from their experiments that the exposure to light interacting with low-level seed dormancy regulates the germination of *A. myosuroides*. They concluded that *A. myosuroides* emergence is largely restricted to the autumn, because seeds are more sensitive to light during this period. As a consequence, soil cultivation triggers successful germination and is more effective in autumn than in the spring. Light is known to be an important trigger of seed germination and is one of the main factors explaining the stimulating effect of soil cultivation on weed seedling emergence (Hartmann and Nezadal 1990, Anderson and Espeby 2009**).**

Light exposure helps to break dormancy and stimulates germination (Colbach et al. 2002a). On the contrary, Müllverstedt (1963b) investigated different factors which might be responsible for increased emergence of weeds such as *A. myosuroides* following mechanical weed control. In his experiments neither light nor the possible displacement of weed seeds by cultivating at soil levels favorable for germination had any influence. He concluded that better air diffusion in soil which was loosened by cultivation was the responsible factor for seed emergence. Müllverstedt (1963a) also demonstrated that the germination of different weed species is highly influenced by the oxygen pressure.

Previous research has shown, that at seed maturity, the proportion of dormant seeds depends on the conditions in which seeds were produced (Colbach and Dürr 2003, Moss et al. 2006). Seeds originating from black-grass plants from winter crops (long germination-maturity duration) are much more dormant that those produced in spring crops.

In addition, the natural seed dormancy tends to be highly influenced by the weather conditions during maturation (Fenner 1991, Moss et al. 2006) and is different every year (Froud-Williams et al. 1984), which may cause variable emergence patterns for black-grass in field trials as also demonstrated by Kampe (1975). In pot studies seeds of *A. myosuroides* were less dormant when produced under warm and dry conditions, rather than under cool and wet conditions (Moss et al. 2006). When seeds are produced under cool conditions and with a lot of precipitation, the primary dormancy can last up to 6-8 weeks. Most black-grass seeds are then emerging from the first few centimeters down the soil profile (Menck 1968, Naylor 1970, Moss 1985c). It seems obvious that a protracted germination period and late flushes of *A. myosuroides* especially occur in years with a high level of seed dormancy, and this might be responsible for the observed emergence pattern in experiments. In Rauischholzhausen for instance, late flushes of black-grass by 9-24% of the total emergence occurred before winter in November and December. According to Thursten (1972), later germinating black-grass tends to tiller less, are less competitive and shed fewer seeds than those germinating in the early autumn. Under extreme drought or wet conditions, germination can be delayed until the following spring. When this occurs the actual number of plants emerging is frequently lower than what would have emerged in a more favorable autumn (Thursten 1972).

Moreover, the proportion of non-dormant seeds are highly dependent on the conditions after seeds shed. As for many species, the deeper the black-grass seeds are located in the soil, the less they germinate (Colbach et al. 2006). In addition, Colbach et al. (2002b) showed that the alternation of dry and moist periods during summer and the stimulation by light influences the dormancy of seeds after shed. While short and dry periods break dormancy, the longer the dryness persists the more seeds acquire secondary dormancy.

Ecological factors have been shown to play a major role, not only by influencing secondary dormancy (Baskin and Baskin 1985) but also by either inducing or inhibiting germination. The occurrence of the long lasting emergence of *A. myosuroides* up to May which occurred especially after chisel ploughing cultivation over the experimental period of three growing seasons is also reported by Menck (1968).

He even demonstrated that sown *A. myosuroides* can emerge throughout the whole year. However, he concluded that the percentage of the emerged seeds is highly dependent on the seed dormancy. He suggested that a secondary dormancy, according to its natural endogenous germination rhythm, is induced by cool and wet conditions during the winter which will be reversed as soon as temperatures pick up and the soil starts drying up in the spring. Similarly, Colbach et al. (2006) states that secondary dormancy sets in gradually from mid-autumn onwards, because of cold and humid conditions. In spring it is steadily broken and disappears at the beginning of summer. This induced secondary dormancy seemed to be more likely be the reason for late flushes which were observed until May rather than a high primary dormancy. More research needs to be conducted in order to understand the emergence pattern in correlation to the dormancy levels.

In Rauischholzhausen, ploughing cultivation not only had a reductive effect on the total black-grass infestation, but it also significantly reduced the number of late emerging *A. myosuroides*, which was highly likely due to the burial of fresh seeds. Nevertheless, the difference in the emergence pattern was observed in Rauischholzhausen in 2011/12, where after ploughing cultivation relatively more (24%) of the total black-grass emerged after herbicide application compared to chisel ploughing (9%). The reason might be that seeds were brought up to the surface again by ploughing and lead to a high weed population. But it is more likely that differences in soil properties which were observed during the herbicide application in autumn were responsible for the different emergence pattern. After ploughing the seedbed was cloddy and relatively dry compared to the minimum tilled land. Therefore soil cultivation probably had an impact on the protracted emergence pattern of black-grass. According to Wolber (2009) a fine crumbling seedbed ensures an even emergence of the crop and also black-grass. Especially when cultivation takes place in unfavorable soil conditions, like high moisture, cloddy soil aggregates are usually produced which then naturally break up over time through the influence of weather. As a result, the emergence of black-grass might be stimulated when seeds are then left on the surface or due to light exposure (Anderson and Akerblom 2009).

During the years of observation it was noticed that populations of black-grass were highly influenced by climate conditions as well (drought, frost) and sometimes were reduced. Because of frost periods during February in 2012, all of the black-grass individuals in the 1-2 leaf stage were eliminated, while already tillering plants survived. Similar observations were made after long drought periods which can also suppress and reduce black-grass infestations.

The observed emergence pattern over the three growing seasons explains why black-grass is an increasing problem, with more autumn sown crops, earlier sowing and more minimum tillage acting as the critical factors. Consequently, a good prediction of the current year`s initial dormancy status of the local black-grass seeds could help farmers identify the correct black-grass management and reduce *A. myosuroides* infestations by cultural means. As already conducted by Moss et al. (2006), the dormancy level of black-grass could be tested on seed samples or estimated by climate conditions during black-grass seed maturation. Provided there is enough soil moisture to permit germination, a low dormancy year should allow maximum germination to take place before sowing, hence a lower infestation in the crop. When seed dormancy is predicted to be high, such as in a cool, wet year (Moss et al. 2006), a more protracted germination period would be expected even with adequate soil moisture. The farmer would be able to optimise the cultivation (potentially ploughing cultivation) and sowing strategy (possibly delay sowing time) in order to maximize the proportion of black-grass emerging prior to sowing. This way seedlings emerging prior to sowing can be destroyed easily by cultivations and/or use of a non-selective herbicide. Landschreiber (2014) concluded from field experiments in Northern Germany that repeated shallow cultivations (1-2 cm) might be a promising approach to maximize the germination of fresh seeds prior to sowing, while those which emerge within the crop require the application of selective herbicides. Moreover, the farmer is able to optimise herbicide timing and choice. Pre- and post-emergence herbicide applications would have to be more robust, containing herbicides with a long lasting residual component to cover the protracted period of black-grass emergence. With protracted flushes throughout the season, the timing of post-emergence herbicide application is critical for effective black-grass control.

Conclusions

In conclusion, black-grass emergence pattern is dependent on the dormancy level and varies between location and within years, and is therefore difficult to predict. It was confirmed that the peak emergence of black-grass appears in autumn, sometimes with a protracted period of emergence until winter. Another flush of some extent usually occurs in the spring. It can be concluded that protracted flushes of black-grass over the season occur especially in fields where minimum tillage is practiced and a high infestation is present. On such locations the application timing, in particular of post-emergence herbicides, is critical for effective black-grass control. Moreover, it is very important that herbicide treatments (pre- and post-emergence) are more robust and contain active ingredients with a long lasting residual component in order to cover the protracted period of black-grass emergence, which would be expected particularly in years with a high dormancy level. Seed-bed preparation, in particular after ploughing cultivation (avoidance of cloddy seed-beds), is critical in order to avoid late flushes of black-grass and maximize herbicide performance. Obtained results gave indications that black-grass populations in the field were highly affected by environmental conditions such as frost and drought, which could change their density.

5.2 Soil activity of post-emergence herbicides

5.2.1 The effect of root uptake of different herbicides in aqueous solution on root growth and plant vitality of *A. myosuroides* plants

ACCase-inhibitors, so called grass herbicides, are known to be taken up by green plant tissues of the weed (leaves and stems) and generally not via roots (AgrEvo 1, AgrEvo 2, Hoechst 1988, Raffel and Fluh 1992, Raffel et al. 2006, Syngenta 2013a). Hofer et al. (2006) reported that pinoxaden is taken up via leaves and distributed by the phloem and xylem within a plant, whereas the uptake by roots is very limited (Syngenta 2013a). Nevertheless, obtained results show that ACCase-inhibitors such as fenoxaprop-P, clodinafop and pinoxaden can be taken up by the roots of *A. myosuroides* plants and then cause phytotoxic effects such as root growth inhibition, which often leads to plant death. Even though these experiments were conducted under unrealistic circumstances, with herbicides being in aqueous solution and without influence and interaction with the soil matrix, these results gave a general idea of potential soil activity.

ALS-inhibitors are systematic, phloem and xylem mobile herbicides that are adsorbed via leaves, shoots and roots. According to technical information brochures and publications, leaves as well as roots are the primary uptake sites for ALS-inhibitors in plants (Tröltzsch 1998, Bayer 2002, Bayer 2005, Köcher 2005, Dow AgroSciences 2007). The label from products containing ALS-inhibitors such as Atlantis, Attribut and Lexus indicate that propoxycarbazone, flupyrsulfuron and mesosulfuron can be taken up by the roots and have the ability to control germinating and emerging grasses like black-grass (Teaney et al. 1995, Bayer 2013, DuPont 2013). As expected, all ALS-inhibitors (mesosulfuron, pyroxsulam, flupyrsulfuron, propoxycarbazone) showed root growth inhibition just after root uptake at very low concentrations in the aqueous solution.

Conclusions

In conclusion, all tested herbicides (ACCase- and ALS-inhibitors) which are generally used as post-emergence herbicides only, can be taken up via roots by *A. myosuroides*. Thus, many post-emergence herbicides potentially provide a certain level of soil activity against grass weeds via root uptake when they are applied to the soil.

5.2.2 Effect of different application modes (foliar, soil and foliar+soil) on herbicidal efficacy of ACCase- and ALS-inhibitors

For fenoxaprop-P and clodinafop, it is reported that they are active only through the foliage uptake (AgrEvo1, Hoechst 1988, Raffel and Fluh 1992, Hofer et al. 2006). Product brochures of Axial 50EC and Topik 100 state that the application can be done independently from soil type and soil moisture, because their activity relies exclusively on foliage activity (Syngenta 2013a, Syngenta 2013b). Nevertheless, special uptake studies were conducted by Syngenta (2013) which showed that the uptake of pinoxaden into plants is mainly through leaves, whereas the uptake by roots is very limited (Hofer et al. 2005, Syngenta 2013a). After soil application an activity of about 30% was assessed against *Avena arvensis* and *Lolium mulitflorum*. In contrast to those findings, placement studies presented in this research demonstrated that pinoxaden can achieve high and equivalent levels of weed control after uptake from a soil application only, compared to regular post-emergence spraying. clodinafop showed similar results and is also active through soil uptake. These findings are contrary to the published technical information (Raffel and Fluh 1992, Syngenta 2013b). In conducted studies, fenoxaprop-P has clear predominance for the foliar component of action which confirms previous research (AgrEvo1).

For all ACCase-inhibitors, especially for clodinafop and pinoxaden, it was demonstrated that a normal application (foliage + soil) showed the highest activity compared to solely foliage uptake, which proves that a certain uptake proportion comes from the soil even for ACCase-inhibitors. In contrast to the field, greenhouse plants in pot experiments usually have optimal growth conditions due to controlled temperature and water regimes. Soil moisture has been shown to affect the uptake and translocation of foliar-applied herbicides. Several researchers have found a reduction in efficacy of post-emergence herbicides when plants were grown under conditions of low soil moisture levels (Kidder and Behrens 1988, Reynolds et al. 1988, Boydston 1990, Olsen et al. 1999).

Drought stress can initiate morphological responses such as leaf rolling or the formation of thick cuticles which can reduce the amount of herbicide that enters the plant. In addition, cell membrane permeability and water movement within plants may be reduced, thus limiting translocation (Price 1983). On the other hand, green foxtail (*Setaria viridis*) control with fenoxaprop, fluazifop-P, haloxyfop and sethoxydim was greater when plants were grown under wet conditions compared with dry conditions before and after herbicide application (Boydston 1990). Kochia susceptibility to imazethapyr (Nalewaja et al. 1990) and chlorsulfuron (Nalewaja and Woznica 1985) decreased as available soil water became limited. Johnson grass (*Sorghum halepense*) control with glyphosate increased as soil moisture increased from 12 to 20% (McWhorter and Azlin 1978).

In contrast to grass-herbicides, sulfonylureas generally enter plants through foliar and soil uptake and are trans located acropetally and basipetally within the plant and often can be applied flexibly (pre- or post-emergent). The relative uptake proportion of each active ingredient is generally dependent on the application conditions, such as soil moisture, temperature and the thickness of the foliage wax layer. An example of a sulfonylurea which is taken up mainly by the roots is imazosulfuron (Drobny et al. 2012), whereas thiameturon-methyl can be regarded as a mainly foliar-active herbicide because of its rapid degradation (Kudsk et al. 1989). Also, the corn herbicide foramsulfuron (Collins et al. 2001) provides weed control primary through foliar activity after penetration into the leaves of grasses and broad-leaved weeds. Special placement studies conducted by the manufacturer in the greenhouse have shown that some ALS-compounds, such as mesosulfuron, act on the target weeds both via the foliage and the soil, usually with predominantly foliar action (Bayer 2002, Köcher 2005). There were a few grass weed species which showed significant efficacy of mesosulfuron via soil, e.g. *Alopecurus myosuroides* and *Apera spica-venti*. In their experiments mesosulfuron at 5 g/ha applied to foliar and foliar+soil application showed high levels of *A. myosuroides* control, whereas 35% plant damage resulted in soil placement (Köcher 2005). Received results for mesosulfuron confirm these findings in principle, even though an activity of 56% after soil-placement was a little higher. Further, results of this research show that iodosulfuron was much more active against *A. myosuroides* via the soil than via the foliage. In contrast, the manufacturer (Köcher 2005) showed in their experiments with *Lolium multiflorum* that iodosulfuron-methyl-sodium at a rate of 10g a.i./ha acts via foliar uptake (90% damage) and also offers a significant soil activity (75% damage). Similarly, pyroxsulam, flupyrsulfuron and propoxycarbazone showed that black-grass can be controlled via soil application only. For flupyrsulfuron it is known that the active ingredient is active through both foliar and root uptake (Teaney et al. 1995), which was confirmed by this research.

Tröltzsch (1998) reported that in special uptake experiments carried out within climate chambers, they were able to demonstrate that with adequate soil moisture, the activity of flupyrsulfuron against A*. myosuroides* came 60% through soil uptake and 40% through the foliage. Results of current research also confirm studies from Dow AgroSciences (2007), which showed that for pyroxsulam the leaves and roots are the primary uptake site in plants. Similarly, metosulam, also a triazolopyrimidine sulfonanilide herbicide for weed control in winter cereals and corn, achieves equivalent levels of weed control after uptake from the soil after pre-emergence application or after foliar uptake from post-emergence spraying (Downard et al. 1993).

The post-emergence herbicide propoxycarbazone-sodium (Attribut) is absorbed via leaves and roots and offers contact and residual weed control (Bayer 2005).

According to the manufacturer, root uptake is predominant under normal growing conditions with adequate soil moisture. Obtained results confirm that roots and foliage are uptake sites for propoxycarbazone. Similarly, thiencarbazone-methyl, a relatively new corn herbicide belonging to the same chemical class (thesulfonly-amino-carbonyltriazolinones), can be used as pre- and post-emergent herbicide and provides foliar and soil activity (Santel 2012).

Conclusions

Against expectations, the conducted trials showed that all herbicides provide herbicidal activity after only a soil application. All ACCase- and most ALS-inhibitors showed higher activities after a normal herbicide application (foliage and soil) than after just foliage uptake, which leads to the conclusion that most post-emergence herbicides more or less also rely on the activity via soil uptake in order to achieve their maximum level of activity.

5.2.3 Soil activity of herbicides under controlled controlled greenhouse and field conditions

In general, the soil activity of herbicides can only exist when the active ingredient is able to be taken up by either the roots or the hypocotyls of the weed. As shown in laboratory studies and discussed above, most investigated post-emergence herbicides containing ACCase- and ALS-inhibitors fulfill this criteria and can potentially provide soil activity against weeds via root uptake. Furthermore, it is essential that there is availability of the herbicide in the root zone of the weed. On the one hand this is highly dependent on characteristics of the chemical itself like the mobility, soil absorption, water solubility (logKoW) and the degradation stability (DT50). On the other hand, many environmental factors such as the soil (organic matter, biological activity, temperature, clay content, structure), precipitation and plant growth have a large impact (Devine et al. 1993, Aldrich and Kremer 1997).

Received results show that herbicides, including soil acting herbicides (flufenacet and pendimethalin) as well as post-emergence herbicides (ACCase- and ALS-inhibitors) can have a certain soil activity against *A. myosuroides*. Conducted pot experiments showed that all tested herbicides had mostly significant reductive effects on either shoot and/or root biomasses *of A. myosuroides* plants.

For most herbicides it was possible to evaluate soil activity on shoot biomass reductions of *A. myosuroides* plants either by visual estimates or shoot weights. Taking the weights of roots might be time consuming, but it appears to be a very good and sometimes better parameter to recognize soil activity. The experiments of this research showed that root biomasses showed similar or often more biomass reductions than shoots. In the case of some herbicides (e.g. Axial 50EC) shoots showed a relatively low level of soil activity while root reductions showed a higher soil activity. Bioassay procedures, based on the root growth of maize (*Zea mays* L.), sunflower (*Helianthus annus* L.), lentil (*Lens esculenta* M.) and sugar beet (*Beta vulgaris* L.) were used to study the phytotoxicity and persistence of four different sulfonylureas in three soils (Kotoula-Syka et al. 1993). Anderson (1988) used oat roots to characterize the pre-emergence bioactivity of diclofop. Furthermore, the response of *A. myosuroides* roots to different soil moisture regimes has previously been shown (Blair 1985). A positive advantage of root evaluation was also that damages to the root system caused by ALS-inhibitors (such as Atlantis WG, Lexus) were noticible in addition to the biomass reductions. In pot experiments the roots were stunned, less structured and less fine than those from untreated plants, which is most likely due to the ALS-inhibition causing suppression of developmental growing and differentiation of treated roots. Also, flufenacet caused visual damage on the roots which looked like the formation of root knots due to cell division inhibition.

5.2.3.1 Soil activity of ACCase-inhibitors in pot experiments

Even though it was shown above that root uptake is potentially possible for fenoxaprop-P, the soil activity in pot and field experiments was very low and confirmed previous cited publications (AgrEvo1, Hoechst 1988 Fortmeier et al. 2006) which report that its efficacy is relying on foliage uptake only.

While received results for fenoxaprop-P showed consistently low soil activity, obtained results for pinoxaden and clodinafop were variable which was probably due to the effect of different growing conditions in experiments, which can occur in greenhouse experiments as reported by Krähmer et al. (1994).

In contrast and against expectations, results from pot and field experiments indicate that pinoxaden and clodinafop can have relatively high soil activity against grasses such as *A. myosuroides* and *A. spica-venti*. Previously, it has been shown that ACCase-inhibitors such clodinafop and pinoxaden are entering the plant entirely or mainly through the foliage (Amreim et al. 1989, Cornes et al. 1989, Raffel et al. 2006, Hofer et al. 2006, Syngenta 2013a). Technical product labels and brochures for Axial 50EC (pinoxaden) and Topik 100 (clodinafop) state that the application can be done independently from soil type and soil moisture, because their activity relys exclusivley on foliage activity (Syngenta 2013b). Further, no risk of carry-over damage on succeeding crops (rotational crops) or on crops which are planted shortly after application in the case of crop failure is given in product labels or technical brochures of these ACCase-inhibitors in Germany. In contrast, the product label of Topik from the United Kingdom states that in the event of crop failure cereals maybe sown only after an interval of 3 weeks after the application. According to Syngenta UK, even 4 weeks should elapse after application of Axial before rye-grass, maize, oats or broad-leaved crops are safe to be planted as replacement crops in the event of a crop failure (Syngenta 2013c). This indicates that these active ingredients have a certain soil activity which was confirmed by various pot and field experiments in this research.

In contrast to the field, greenhouse plants usually find optimal growth conditions due to controlled temperature and water regimes. Higher temperatures for instance, when soil moisture is not limited, will result in more active plant growth and probably increased herbicide entry as previous concluded by Lundkvist (1997). Generally, growing conditions that favour weed growth will favour herbicide uptake, translocation and overall activity (Strek and Green 2001). This can be used as an explanation for different results received for pinoxaden in pot experiments. The pot experiment outdoors and the pot experiment under different moisture regimes (compare chapter 4.2.3.2 and 4.2.3.5) were both conducted under relatively cold and wet climate conditions when there was a relatively low level of soil activity observed. Whereas under relatively warm greenhouse conditions the soil activity was high for pinoxaden. On the contrary, clodinafop showed a high soil activity independent from the temperature. Cornes et al. (1989) also reported that the activity of clodinafop was not dependent on temperature, although symptoms were slower to develop in cool temperatures.

In addition, soil moisture has been shown to be important for isoproturon activity on *A. myosuroides* in pot experiments in a greenhouse (Blair 1985). Hewson and Read (1985) concluded from their series of field trials carried out on many farms over 12 years that good control of larger plants by isoproturon generally coincided with moist soil conditions. On the other hand, excessive watering or rainfall after spray application could cause leaching below the rooting zone of small target plants which could lead to their survival (Aldrich and Kremer 1997). Furthermore, plants also show a higher susceptibly to residual herbicides in pot experiments, because the root growth is limited and the chemical is highly concentrated in the root zone in a pot. This was shown in two experiments which were conducted at the same time and in the same manner. Clodinafop showed a much higher soil activity in the experiment (compare chapter 3.6.3.1 and 4.2.3.1) when a smaller pot size (10.5 cm) was used compared to relatively low pre-emergence activity in the experiment (compare chapter 3.6.3.3 and 4.2.3.3) with bigger pot sizes (13 cm). This shows that some herbicides, such as clodinafop, are highly influenced by translocation processes and thus result in lower pre-emergence activity. This is probably a reason why the soil activity from ACCase-inhibitors, such as clodinafop, were generally lower or often not detectable in field experiments.

In addition, sown *A. myosuroides* in pot experiments emerge simultaneously from the same depth, while continuous emerging can be observed in the field as shown in chapter 4.1 (Meiners et al. 2011). Krähmer et al. (1994) reported that several factors such as the crop and pest genome, the environmental conditions, physiochemical properties of the applied compounds and crop-pest interactions are causing variability in data generated from greenhouse and field trials. Their received data with chlortoluron and isoproturon showed a lot of variability in greenhouse and field trials. The effect of weather factors on the performance of isoproturon and clodinafop-progagyl to control *A. myosuroides* were investigated in field experiments by Collings et al. (2003). They concluded that the weather conditions affected the herbicide performance more than the growth stage of the weed. This may explain variable results as well.

Also, possible reasons why herbicidal activities of tested herbicides are generally higher in the greenhouse is that the soil is less biologically active. As shown in a pot experiment conducted with the same soil but two different levels of soil activity (compare 4.2.3.1), the herbicidal efficacy of pinoxaden and clodinafop after pre-emergence application was much higher in steam treated soil compared to biologically active soil (untreated). Even though the biological activity was not measured in pot experiments, it can be concluded that the steam treated soil is less active, which might have led to a longer persistence of chemical compounds because of decreased biological degradation. Microbial activity in soils has been reported to be an important factor for herbicide decomposition in soil (Zimsahl et al. 1982, Walker et at. 1989, Mueller et al. 1992).

Obtained results for ACCase-inhibitors were inconsistent, which is probably also due to the effect of the different application timings after sowing *A. myosuroides* in experiments. Comparing results obtained from pot experiments when pots of bigger sizes were used (diameter 13 cm), like in the pot experiment in the greenhouse using different watering methods (chapter 4.2.3.4), clodinafop and pinoxaden showed their highest soil activity. In these experiments the pre-emergence applications were conducted one week after sowing, at a timing when black-grass seeds already germinated below the soil surface but had not yet emerged. The treated soil was moist at the time of the application and all herbicides were probably able to quickly move downwards to the germinated black-grass seeds in a high concentration. On the contrary, relatively low efficacies from ACCase-inhibitors in other pot experiments like in trial on effect of soil moisture and sowing depth on the soil activity (compare 4.2.3.5) might have been caused by the relatively long period from application until the germination of black-grass seeds. By the time seeds germinated, herbicides could have already been degraded or leached out of the root zone. As reported by Carter (2000), the amount of the herbicide which moves away from the area of application depends on the physico-chemical properties of the chemical and the agro-climatic conditions of the target site. The soil sorption coefficient of ACCase-inhibitors (Koc) and the half-life (DT50) which are properties of importance (Carter

2000) and indicate a relatively high mobility and low persistence of ACCase-inhibitors.

The variability of soil activity from ACCase-inhibitors could not be explained by the physico-chemical properties such as water solubility or DT50, which do not differ much according to the PPDB (2012). Instead, differences in soil activity of the three ACCase-inhibitors is probably associated with differences in the soil absorption properties. Pinoxaden and its active metabolite M2, which showed the highest and most consistent soil activity in most experiments, has the lowest soil absorption (Koc 323 ml/g, metabolite M2: 10 ml/g), followed by clodinafop-propagyl (Koc 1466 ml/g, clodinafop: 46.3 ml/g) and fenoxaprop-P-ethyl (Koc 11354 ml/g, fenoxaprop-P: 282 ml/g) (DAR 2004a, DAR 2006a, DAR 2006b, PPDB 2012).

Conclusions

It was shown above that all tested ACCase-inhibitors are able to be taken up by the roots of *A. myosuroides* and thus potentially provide activity when applied to the soil.

However, it can be concluded that soil activity most likely depends on the interaction between properties of herbicides and soil characteristics which thereby influences the availability of the herbicides in the root zone.

Differences in soil activity are probably due to differences in physico-chemical properties of the herbicides and not due to the ability to be taken up via roots.

pinoxaden and clodinafop are able to provide soil activity against grass weeds such as *Alopecurus. myosuroides* and *Apera-spica-venti*, however this is highly dependent on the environmental conditions (soil moisture, temperature, biologically activity of the soil), growing conditions, and as well as the right application timing.

5.2.3.2 Soil activity of ACCase-inhibitors in field experiments

Conducted field experiments generally confirm observations which were made in pot experiments. As expected fenoxaprop-P did not show any soil activity, whereas clodinafop and especially pinoxaden showed relatively high soil activity in some experiments. However, as already observed in pot experiments, the activity was very inconsistent and varied within the years and trial locations, which confirm conclusions made by Krähmer et al. (1994) who report that several factors like the crop and pest genome, the environmental conditions, physiochemical properties of the applied compounds and crop-pest interactions are causing variability in data generated from greenhouse and field trials.

Inconsistency could be explained with the influence of the application timing in relation to the seedlings emergence of black-grass, which was already observed in pot experiments. Soil activity achieved by pinoxaden and clodinafop was very high in experiments conducted in 2011/2012 at Wicker-Johannesfeld when pre-emergence application of herbicides took place a few days after sowing when sown black-grass seeds were already emerging. The precipitation of about 17 mm within 72 hours after application most likely supported the herbicide uptake and consequently led to high soil activity by pinoxaden and clodinafop. Rain is known to increase the activity of soil-applied herbicides by dissolving and moving the herbicide into the top few centimenter of the soil where most weeds germinate (Kudsk 2002). Lundkvist (1997) concluded from his studies on dichlorprop-P/MCPA and tribenuron-methyl that the efficacy is highly influenced by weather conditions close to time of application. The author identified rain to be important for herbicide uptake and soil temperature to promote plant growth.

Furthermore, sown *A. myosuroides* in experiments usually have low dormancy and emerge simultaneously from the same depth, while a protracted germination period can be observed in the field (Meiners et al. 2011). Because of natural emergence of black-grass after application, efficacies were probably not complete (100%), which shows there is not a long lasting soil activity present from ACCase-inhibitors. These findings agree with the studies mentioned above. The received results confirm observations which were made in a field trial conducted by Bayer CropScience in 2010 (not published). Pinoxaden and mesosulfuron achieved almost complete black-grass control after a single pre-emergence application, which was linked to the ideal application timing and intensive precipitation directly after spaying. Also in that experiment, at the time of the application most sown black-grass seeds were already emerging.

Even though the sowing time for black-grass, the pre-emergence application timing and the weather was the same or very similar in Gießen in 2011, efficacies from ACCase-inhibitors were much lower than in Wi-Johannesfield. Only double of the field dosage of Axial showed significant pre-emergence activity. Because no black-grass had emerged within 4 weeks after sowing, a delayed/extracted emerging occurred at a low rate, highly likely because of the dry soil conditions.

The lack of pre-emergence efficacies from ACCase-inhibitors in Wicker-Straßenmühle in 2010 and 2011 is probably also due to the fact that most black grass plants survived because they were emerging sometime after the application when the concentration was too low for the chemicals to work. In 2011, herbicides were applied on the same day as the weeds were sown. Duefer and Heitefuss (1992) demonstrated in their studies on chlortoluron that a sufficient efficacy in controlling *A. myosuroides* is highly dependent on the minimum phytotoxic concentration in the soil, so called “Grenzkonzentration”. In their field experiments all plants, which germinated during the short inactivation time after application were controlled, whereas plants which germinated after this time were not controlled due to high degradation rates of chlortoluron. As reported by Aldrich and Kremer (1997), there are several factors such as adsorption, leaching and degradation which can prevent fully satisfactory weed control with a soil-applied herbicide.

Nevertheless, the trials in winter wheat with a natural occurring infestation of *A. myosuroides* in Wicker 2011/2012 (Kennel and Johannesfeld) demonstrated that pre-emergence applications of ACCase-inhibitors, especially pinoxaden, are capable of reducing natural black-grass infestations significantly by over 50%.

Conclusions

In conclusion, the ACCase-inhibitors, in particular pinoxaden and clodinafop, potentially have a relatively high soil activity when they are applied on grass weeds which are in the process of germination. When this is not the case, then usually the concentration of the active ingredient in the root zone would not be high enough to control late emerging black-grass individuals. Thus, no durable residual control can be expected by any of the ACCase-inhibitors fenoxaprop-P, clodinafop and pinoxaden.

Immediate rainfall after application enhances soil activity, because the active ingredients are more likely to reach the emerging seedlings right away with an effective concentration.

In most cases ACCase-inhibitors such as pinoxaden and clodinafop showed weed control when applied pre-emergence in both, pot- and field experiments, suggesting that when these herbicides are applied post-emergence under field conditions, some not yet emerged grasses may be controlled to some extent.

5.2.3.3 Soil activity ALS-inhibitors in pot experiments

As expected all tested herbicides containing ALS-inhibitors showed more or less a high level of soil activity against *A. myosuroides* in experiments which were carried out in the greenhouse and in the field. In contrast to the ACCase-inhibitors discussed above, which work mostly through leaf uptake, ALS-inhibitors such as pyroxsulam, flupyrsulfuron, propoxycarbazone and mesosulfuron are also able to be taken up by the roots to be active as cited above (Tröltzsch 1998, Bayer 2005, Köcher 2005, Dow AgroSciences 2007, Dow AgroSciences 2012).

Many sulfonylureas have been attributed with a relatively high soil activity, and were subsequently developed for pre- and post-emergence control of a number of broad-leaved weeds and grasses in cereals, such as the herbicide triasulfuron (Amrein and Gerber 1985) and rimsulfuron (Onofri 1996). On the other hand, some ALS-inhibitors have a very short half-life in soil, e.g. thiameturon-methyl (Rahman et al. 1988, Kusdk 1989) and foramsulfuron (Collins et al. 2001), and are mainly used as foliar-active herbicides. Because of the high residual soil activity afforded by some of the chemicals in this class of sulfonylureas, many product labels contain re-cropping restrictions in order to avoid carry over injuries on succeeding crops (Blair and Martin 1988, Russell et al. 2002). The persistence and mobility of flupyrsulfuron has been measured in the soil of several winter wheat crops after autumn or spring applications by Rouchaud et al. (1999). In their experiments, flupyrsulfuron did not move to deeper soil layers and remained concentrated within a thin layer near the soil surface, where a high concentration could provide high herbicide efficacy.

The chemical company DuPont marketed their herbicide flupyrsulfuron-methyl in the mid 1990s as the first herbicide with post-emergence and residual activity for selective control of black-grass in cereals for use as an alternative to isoproturon. The German product labels from ALS-inhibitors such as Atlantis WG, Attribut and Lexus indicate that the main active ingredients propoxycarbazone, flupyrsulfuron and mesosulfuron respectively, have a soil activity and the ability to control germinating and emerging grasses like black-grass (Bayer 2013a, DuPont 2013). Further, the product labels from Attribut and Lexus state that root uptake of the herbicide via moist soils is necessary in order to achieve optimal herbicidal activity against grasses such as *A. myosuroides* (DuPont 2013, Bayer 2013a).

Atlantis WG is also supposed to work through root uptake at higher dosages, even though no long-lasting residual activity can be attributed to the product (Bayer 2013a). According to the label in the UK, the weeds germinating after application will not be controlled (Bayer 2013b).

Moreover, carry-over risks on rotational crops and/or on replacement crops in the case of crop failure of most products such Atlantis, Lexus and Attribut demonstrate their high residual soil activity (Bayer 2013a, DuPont 2013). Risk of damage on succeeding crops such as rapeseed, dicotyledonous catch crops or sugar beets is given in product labels or technical brochures of the ALS-inhibitors mentioned above. In contrast, label information of Broadway claims that the herbicide containing pyroxsulam is working through foliage uptake. The manufacturer points out that there are no restrictions on following crops (Dow AgroSciences 2013). Nevertheless, the German product label states that in case of crop failure only corn and cereals should be replanted after a ploughing cultivation of 20 cm depth, which is a sign of soil activity.

The efficacies after pre-emergence application received from pot experiments were more consistent for ALS-inhibitors such as propoxycarbazone, mesosulfuron, pyroxsulam and flupyrsulfuron than for the ACCase-inhibitors discussed above. Comparing ALS-inhibitors, high levels of soil activities by flupyrsulfuron were highly dependent on adequate soil moisture which explains inconsistent results in experiments. These observations confirm the manufacturer information on the active ingredient (Tröltzsch 1998, DuPont 2013). Soil activity from sulfonylureas generally require moisture (from rainfall or irrigation) for the best results (Strek and Green 2001, Russell et al. 2002). The impact of soil moisture shall be discussed later on.

Similarly to the ACCase-inhibitors above, the highest soil activity by all ALS-inhibitors including flupyrsulfuron was observed when herbicides were applied sometime after sowing, when black-grass seedlings had already started germination. The applied herbicides were probably able to quickly move downwards through the moist soil profile in a high concentration directly to the already germinated black-grass seeds and killed the seedlings via root uptake.

Further, it was demonstrated for herbicides containing pyroxsulam, propoxycarbazone or mesosulfuron that only a small portion of the full field dosage is needed to achieve high efficacies after a pre-emergence application in conducted pot experiments. Particularly for Broadway and Attribut, only small amount of active ingredient, less than 1/6 of the field dosage, was needed to achieve adequate soil activities and high levels of control against *A. myosuroides* in pot experiments. This flexibility in the minimum effective dosage shows the high potential of soil activity even at different environmental conditions. It also shows a very high affinity from the chemical to the target site in the weed species. In contrast to the field, greenhouse plants usually find optimal growth conditions due to controlled temperature and moist water regimes, which is known to promote soil activity in pot experiments (Blair, 1985, Kudsk 2002). As discussed above, plants might also show higher susceptibility to residual herbicides in pot experiments since the root growth is limited, and this is because the chemical is highly concentrated in the root zone in a pot. This happens especially with weeds, which are sown close to the soil surface and therefore the root zone is located close to herbicide. Moreover, the biotic degrading of the chemical might be decreased by the usage of steam-treated soil in most experiments, which causes a longer persistence. For these reasons the impact of soil moisture, watering method and sowing depth on the soil activity were tested in special trials and are discussed below.

Conclusion

In conclusion, herbicides containing ALS-inhibitors such as propoxycarbazone, mesosulfuron, pyroxsulam and to some extent flupyrsulfuron, are able to provide very high levels of soil activity against grass weeds such as *A. spica-venti* and *A. myosuroides*.

Showing very high soil activities even at very low application rates, pyroxsulam, mesosulfuron and propoxycarbazone should be very effective under field conditions.

Soil activity can be expected to be the highest when grass weeds are already germinating and when rainfall occurs after application.

5.2.3.4 Soil activity of ALS-inhibitors in field experiments

High pre-emergence activities by ALS-inhibitors such as pyroxsulam, flupyrsulfuron, mesosulfuron and propoxycarbazone were observed in field experiments and confirm observations which were made in the greenhouse experiments. Generally, there were little differences within the different field experiments and often little differences within the used herbicides. In field experiments, soil activity achieved by ALS-inhibitors was often on a similar level as regular used pre-emergence herbicides containing flufenacet or pendimethalin, which demonstrates their potentially high soil activity. The only low pre-emergence efficacy achieved by flupyrsulfuron (Lexus) in Gießen 2011/2012 could be explained by the dry soil conditions during the emerging period of sown black-grass. The high dependency of flupyrsulfuron on soil moisture confirms previous research (DuPont 2013) and observations made in pot experiments conducted at different soil moisture levels. In field trials conducted over 5 years with the herbicide Lexus-Class containing flupyrsulfuron, a very good efficacy against *A. myosuroides* was achieved, as long as a decent soil moisture was present at least 2 weeks after application in order to ensure uptake trough the roots (Tröltzsch 1998).

Received results for Broadway are contrary to the company`s technical information. The manufacturer claims that the herbicide containing pyroxsulam is working through foliage uptake only, and points out that there are no after cropping restrictions (Dow AgroSciences 2007, Dow AgroSciences 2013).

Results of this research support and enhance the knowledge already available for mesosulfuron. According to the manufacturer label in the UK, Atlantis WG has a moderate residual life in soil under normal conditions. Residual efficacy will be enhanced where seedbeds are fine and moist, whereas high soil temperatures and cloddy seedbeds may reduce the residual efficacy of Atlantis WG (Bayer 2013b). Against the manufacturer information in Germany (Bayer 2013a), presented field experiments showed that Atlantis WG obtained a long residual soil activity which even controlled late emerging black-grass, as observed in Wicker-Johannesfeld and Gießen 2011/2012.

According to the German product label, the application of Atlantis has to be done post-emergent to the grasses (Bayer 2013a). Results of this research showed the contrary. The two field experiments in sown winter wheat in 2011/2012 in Wicker demonstrated that pre-emergence applications of ALS-inhibitors such as Atlantis and Broadway at field dosages can lead to very high reductions, up to 95%, of naturally occurring black-grass infestation, and lead to high yield potential of the crop.

Conclusions

Received results conclude that post-emergence herbicides containing ALS-inhibitors, such as mesosulfuron, pyroxsulam, propoxycarbazone and flupyrsulfuron can provide high soil activity against *A. myosuroides* under field conditions.

Their level of weed control after pre-emergence application often matches the activity of soil-herbicides like flufenacet or pendimethalin which are regularly used as pre- and early post emergence herbicides. This means that ALS-inhibitors usually persist long enough to be available in a high enough concentration in the upper soil layers in order to control later emerging grasses to a certain extent, however, this is highly dependent on environmental conditions.

5.2.3.5 The effect of soil moisture on the soil activity of herbicides

Soil moisture may affect herbicide efficacy by altering adsorption, translocation or metabolism (Boydston 1990, Olsen et al. 1999, Olson et al. 2000). When ALS-inhibitors are applied to the foliage, soil moisture ensures uptake and translocation throughout the weed and subsequent weed control (Olson et al. 2000). On the other hand, soil moisture deficit can affect the uptake and translocation of foliar-applied herbicides. Several researchers have found a reduction in the efficacy of post-emergence herbicides when plants were grown under conditions of low soil moisture (Kidder and Behrens 1988, Reynolds et al. 1988, Boydston 1990). Also, in previous research, adequate soil moisture has been shown to improve herbicide efficacy of foliar applied herbicides such as fenoxaprop, fluazifop-P, haloxyfop imazethapyr, sethoxydim (Boydston 1990), imazethapyr (Nalewaja et al. 1990), glyphosate (McWhorter and Azlin 1978) and chlorsulfuron (Nalewaja and Woznica 1985). Soil moisture also determines the uptake of organophosphate insecticides which are commonly used in-furrow in maize and thus affects their interaction with ALS-inhibiting herbicides which often results in crop damage (Bailey and Kapusta 1994).

Soil moisture has previously been demonstrated to be important for isoproturon activity for *A. myosuroides* control in pot experiments in a greenhouse (Blair 1985). Malfyt and Quakenbush (1991) found similar results for imazamethabenz-methyl. Anderson (1988) showed that damage caused by diclofop on oat (*Avena sativa*) sown at different sowing depths increased when the amount of simulated rainfall increased. Furthermore, Walker (1971) also demonstrated that crop damages of soil acting herbicides such as atrazine, aimazine, linuron, lenacil and aziprotryne were increased as moisture content of the soil increased. In contrast low soil moisture levels caused *T. aestivum* injury by terbutryn (Wu et al. 1974).

Received results from the pot experiment with different soil moisture levels (compare 4.2.3.5) show that a high level of soil moisture is required to achieve high levels of weed control through pre-emergence application by most herbicides. The herbicides

Cadou SC and Lexus, containing flufenacet and flupyrsulfuron respectively, were most affected. Obtained results demonstrated that intensive watering and a relatively high level of soil moisture increased the activity of soil-applied herbicides and led to an overall high level of soil activity. Also, in field trials conducted in 2011, high levels of soil activity by ACCase- and ALS-inhibitors were observed when a rain period occurred after the pre-emergence application. ALS-inhibiting herbicides have been reported to require significant rainfall to activate and are required to be in soil solution for roots to absorb (Strek and Green 2001). Adequate rainfall is necessary to maintain a high soil moisture content in the surface layers, as this is important for maximizing the activity of soil-applied herbicides by dissolving and moving the herbicide into the top few centimeters of the soil where weed seeds germinate. This would be also be the most logical explanation for the obtained results. Nalewayja and Woznica (1985) demonstrated that high moisture after chlorsulfuron application was more phytotoxic to *Kochia scoparia* and *Setaria viridis* compared to high soil moisture before treatment. Further, the authors conclude form the results that their soil activity is generally increased as soil moisture increases because adequate soil moisture ensures that herbicides are solubilized and available for root uptake.

In field studies with flupyrsulfuron, a very good efficacy against *A. myosuroides* was achieved, as long as a decent soil moisture was present at least two weeks after application in order to ensure uptake through the roots (Tröltzsch 1998). Furthermore, the product labels of Attribut and Lexus even state that adequate soil moisture is required in order to achieve optimal herbicidal efficacy through root uptake and thus high levels of black-grass control (Bayer 2013a, DuPont 2013). Also, the product label from the UK states that the residual efficacy of Atlantis will be enhanced where seedbeds are fine and moist (Bayer 2013b).

Moreover, there is evidence that watering from the top seemed to be essential for a high level of soil activity. With watering from the top, it was highly likely that herbicides were able to move downwards into the soil profile in order to be more available for deeper located roots.

Additionally, with adequate soil moisture, very low dosages and thus small amounts of active ingredients like pyroxsulam, propoxycarbazone, mesosulfuron or flufenacet were needed to achieve high levels of soil activity against *A. myosuroides* in pot experiments. On the other hand, the minimum effective dosage in order to achieve high levels of black-grass control increased with dryer soil conditions. This was shown for mesosulfuron, pyroxsulam and flufenacet. Moreover, results received from pot experiments suggest that ALS-inhibitors such as Broadway and Attribut were less affected by the soil moisture content because of their high water solubility (PPDB 2012). In contrast, the soil activity from flufenacet was more affected by reduced soil moisture, which was due to its relatively low water solubility compared to ALS-inhibitors.

Soil-applied herbicides, particularly the ones which are absorbed primarily via the roots, such as flufenacet, may fail totally if the soil is dry around the time of the application. According to Kudsk (2002) this is because the herbicide does not move into the upper centimetres of the soil where most weed seeds germinate and because the roots of weed plants do not explore dry soil.

In support of obtained results, Blair (1985) observed that increased soil moisture not only affects the availability of isoproturon in the root zone but also leads to adventitious root development of *A. myosuroides* in the appropriate surface zone in order to take up herbicides. On the contrary, overabundant soil moisture can also reduce activity with soil applied herbicides by promoting leaching out of the effective zone of weeds (near the soil surface) or enhancing degradation (Strek and Green 2001, Strek 2012). Degradation in the soil generally increases as soil moisture increases, reducing the herbicide available for weed control (Dinelli et al. 1998). Degradation processes as well as herbicide translocation within the pot and out of the pot probably occurred in pot experiments, especially when watering was conducted via a hose daily. Nevertheless, in the pot experiment which included the different soil moisture regimes (compare 4.2.3.5), the water regimes were controlled via dripping irrigation system, which is why leaching out of pots probably did not occur during this particular experiment. Also, no water leaked out of the pots visually. For this reason poor weed control because of inadequate herbicide movement is much more the reason for weed control failure than movement of the herbicide out of effective root zone. Results received from the pot experiment with a deeper sowing depth for *A. myosuroides* support these conclusions.

Conclusions

Results conclude that intensive watering and a relatively high level of soil moisture increase the activity of soil-applied herbicides such as ACCase-inhibitors (pinoxaden, clodinafop), ALS-inhibitors (mesosulfuron, pyroxsulam, propoxycarbazone and flupyrsulfuron) and flufenacet against *A. myosuroides*. This leads to the conclusion that herbicides require significant soil moisture in order to activate and enable the herbicides to be in soil solution.

As the soil activity decreases significantly with decreasing soil moisture levels and increasing sowing depths by most herbicides, it can be concluded that adequate herbicide movement and availability of the active ingredient in the root zone is the main reason for high levels of soil activity after irrigation.

Especially herbicides with a very high water solubility such as propoxycarbazone, mesosulfuron and pyroxsulam can provide high soil activity even at dryer soil conditions, whereas flufenacet and flupyrsulfuron might fail to effectively control weeds, particularly when they are germinating from deeper soil zones.

5.2.3.6 The effect of sub-irrigation on the soil activity of herbicides

As the main route for herbicide uptake appears to be via roots, it would seem that herbicides applied in pre-emergence have to move down into the soil to be very effective. Obtained results demonstrated that watering from the top is essential for a successful weed control with herbicides applied on pre-emergence, while sub-irrigation led to an overall low level of soil activity. Similar to obtained results, Blair (1985) showed in his experiments with isoproturon that water applied to the soil surface caused more damage on potted *A. myosuroides* plants compared with sub-irrigation. The method of watering probably affected herbicide activity in these trials by its influence on both location of the herbicide within the pot and distribution of soil moisture. During the experiments, pots after sub-irrigation at 30% FC resulted in a relatively dry soil surface and uneven distribution of moisture in the profile. This suggests that after sub-irrigation less water was available to solubilize herbicides at the surface, and thus the herbicides remained mostly in the surface layers of the soil. Further, observed salt accumulation on the surface of sub-irrigated pots showed that water mainly moved upwards and not downwards. This probably limited the root uptake by *A. myosuroides* seedlings, especially when seeds were sown deeper at a depth of 3.5 cm. Received results confirm those from Blair (1978) Prendeville et al. (1967) and Prendeville (1968) who found that urea herbicides must move into the soil to control grassy weeds like *A. fatua* and *A. myosuroides* and that the degree of weed control obtained is likely to depend on the development of the seedling roots in relation to the position of the herbicide. Principles, pathways and processes of herbicide movements in soils which support this conclusion are well described by Carter (2000).

Conclusions

In conclusion, irrigation from the top or rainfall is essential for successful pre-emergence weed control with herbicides applied on the soil. It can be concluded that water on the soil surface is needed to solubilize the applied herbicides and to move the active ingredients downwards into the top few centimeters of the soil where weed seeds germinate.

5.2.3.7 The effect of sowing depth on the emergence rate of *A. myosuroides* and its control with herbicides

Each weed species is known to have a characteristic emergence response to burial depth and models have been developed that describe this for a range of species (Benvenuti et al. 2001). Successful emergence is dependent on a combination of suitable soil structural properties and the presence of sufficient reserves in the seed to sustain growth. It is generally accepted that larger seeds are able to emerge successfully from greater burial depths because of their greater food reserves. Germination of annual weeds is known to be inhibited by soil depth (Stoller and Wax 1973) but the biological reason for depth inhibition has not yet been fully clarified. It is well known that light (Anderson and Espeby 2009), temperature (Benvenuti and Macchia 1993), soil water content (Roberts and Potter 1985), gas exchange (Benvenuti and Macchia 1993), oxygen pressure (Müllverstedt 1963a) and the degree of soil compaction (Pereja and Stanifoth 1985) represent factors limiting buried seed germination.

In general, the majority *A. myosuroides* seedlings in the field germinate from seeds close to soil surface (Naylor 1970, Moss 1985c) and its emergence decreases with increasing soil depth (Colbach et al. 2005). In pot experiments of this research, the emergence rate of *A. myosuroides* was strongly dependent on sowing depth in interaction with the amount and the application method of water, which explains differences in measured root and shoot biomasses in pot experiments. At the shallow sowing depth (0.5 cm) the average number of *A. myosuroides* seedlings per pot ranged from 18 (30% FC) to 21 (15% FC), compared to 9 (15%FC) to 14 (60% FC) plants at the sowing depth of 3.5 cm (data not shown in result tables). Increased sowing depth from 0.5 to 3.5 cm decreased the number of plants depending on the moisture regime by 26% (60% FC) to 57% (15% FC) with top-irrigation, whereas after sub-irrigation the number of plants was similar at both sowing depths.

Other work has shown that the emergence rate of *A. myosuroides* seedlings of around 90% were not affected by a sowing depth of 4 cm, but a significant decrease in emergence rate by about 60% was observed at a burial depth of 6 cm (Benvenuti et al. 2001). Cussans et al. (1996) showed that the reductive effect of sowing depth on *A. myosuroides* seedling emergence is also influenced by soil aggregate sizes. In his experiments seed emergence was enhanced when seeds were covered by coarser aggregates. Menck (1968) showed in his pot experiments that the effect of sowing depth on *A. myosuroides* emergence is highly dependent on the soil fabric. While he found high emergence rates (around 80%) at 1 cm sowing depth for all soil types, he observed decreased emergence rates at a depth of 4 cm ranging from 0-10% for organic and sandy soils and a decrease of 20-35% for heavier soils with a high content of silt and clay, which is similar to the findings of this research. Although

A. myosuroides is able to emerge from a depth of 15 cm in loose organic soil (Menck 1968), these findings would have no practical implementations since *A. myosuroides* is favored by water retentive soils, so it tends to be more of a problem on heavier clay or silt rather than on lighter sandy soils (Moss 2010). But even for silt loam soils, low emergence rates (2-8%) can be expected at depths of 7-8 cm (Menck 1968, Benvenuti et al. 2001). Previously, conducted pot experiments for the soil (silt loam) used in experiments of this research confirm that *A. myosuroides* is able to emerge from up to 10 cm at low rates (data not shown).

In a field experiment in Rauischholzhausen, black-grass plants emerged from an average depth of 4 cm after chisel ploughing and 4.5 cm after moulboard ploughing. Individual seedlings had emerged after both cultivation systems from 8 cm depth. Even though the averages were similar, the proportion of seedlings which were close to the soil surface after chisel ploughing was higher compared to moulboard ploughing, and it can be suggested that black-grass control with soil-applied herbicides would be expected to be higher after minimum tillage. Moss (1985b, 1985c) found in his studies that on direct drilled land all seedlings were derived from seeds within 3 cm of the soil surface. On tine cultivated and ploughed land, some seedlings emerged from greater depths, but the majority of seedlings were derived from seeds within 5 cm of the soil surface which confirms findings of this research. In contrast, the mean emerging depth for tine was 2.1 cm after tine cultivation and 2.5 cm after using the plough, which were a little shallower compared to obtained findings.

In general the degree of pre-emergence weed control by herbicides likely depends upon the depth of the emerging seedling within the soil profile in relation to the position of the herbicide in the soil. Carter (2000) showed that the amount of herbicide that moves away from the area of application is dependent on the physical and chemical properties such as soil degradation (DT50), organic-carbon sorption coefficient (Koc), and the agroclimatic characteristics of the target site such as soil structure, soil hydrology and climatic effects. Especially the soil sorption coefficient (Koc) and the half-life (DT50) of the herbicide determine the mobility and the persistence of the herbicide. Opping and Sagar (1992) showed that the movement of triasulfuron down the soil profile is inversely related to the organic matter of the soil, and the amount and frequency of rain influenced the extent of leaching. According to low level organic matter (1.7%) and a medium level of clay (26%) of the used soil in pot experiments, the used soil is expected be little to moderately adsorptive for herbicides. This is why the Koc as well as the soil moisture and sowing depth are important factors for the presented results. Since *A. myosuroides* usually germinates close to the soil surface, results of this research confirm that it is usually no problem for most herbicides (e.g. flufenacet, flupyrsulfuron, mesosulfuron) to reach the root zone of the target weeds, unless conditions are exceptionally dry or the soil it strongly absorptive.

The observations made during the pot experiments, that water did not leak out of pots even at the high soil moisture regime during the experiment, at least for the first 4 weeks, lead to the assumption that herbicides remained mostly in the pot and did not leach out the effective zone of weeds. For this reason poor weed control because of inadequate herbicide movement is much more the reason for weed control failure than movement of the herbicide beyond the effective root zone.

Based on the obtained results from pot experiments, current soil-herbicides containing flufenacet (e.g. Cadou SC) applied in pre-emergence alone do not seem to be able to consistently prevent seed return of *A. myosuroides* plants emerging from a deeper soil depth, especially when constant rainfall and high soil moisture levels are missing. Because of the loss of soil activity by herbicides such as flufenacet with increasing sowing depth in pot experiments, it can be assumed that those herbicides were not able to move down deep enough into the soil profile to control *A. myosuroides* seedlings, especially at the low soil moisture regime. The relative high Koc (401 ml/g) and low water solubility (56 mg/l) support these assumptions (PPDB 2012).

The results obtained agree with previous results by Blair (1978), who suggested that herbicides like isoproturon, chlortoluron and metoxuron must move into the soil in order to control *A. myosurorides* and that the level of weed control obtained is likely dependent on the location of the seedling roots in relation to the position of the herbicide in the soil. Blair et al. (1991) demonstrated that deeper sowing (5 cm) protected different wheat cultivars from damage by isoproturon and partially chlortoluron compared to shallower sowing depths (0.6 cm). Also, Blair and Martin (1987) have reported a threefold difference in isoproturon activity on wheat, depending on sowing depth and soil moisture. While the response of *A. myosuroides* to isoproturon has been previously shown to be unaffected by planting depth (Blair and Martin 1987), Blair et al. (1991) showed that *B. sterilis* and *A. myosuroides* plants are less damaged when planted at a depth of 5 cm. Anderson (1988) showed that deep (2.5 cm) compared to shallow (0.6 cm) sowing of oat *(Avena sativa)* resulted in less damage from diclofop, which was less in a sandy clay loam compared to a sandy soil. Similar to experiments of this research, damage was also greater when the amount of simulated rainfall was increased, but due to diclofop`s immobility in sandy clay loam, no damage on deeper sown oats was observed even after intense simulated rainfall (Anderson 1988).

ALS-inhibitors showed relatively high soil activity at both the shallow and deep sowing depth of *A. myosuroides*. The adsorption and desorption of a chemical compound is an important factor for its movement in the soil and affects the availability of the chemical for plant uptake or soil microbial degradation. The adsorption coefficient (Kd) and the values normalized to the organic carbon content (Koc) describe the degree of binding of the active ingredient to the soil. The average Koc values for the used ALS-herbicides are in a range of 28 (propoxycarbazone) to 94 (mesosulfuron) (Bayer 2002, PPDB 2012) and signalize low soil absorption properties. Using the mobility index for chemical compounds proposed by McCall et al. (1980), all ALS-herbicides are in the mobility class high to very high. Gildemeister and Schäfer (2005) observed in their experiments that the adsorption for mesosulfuron is not strongly correlated to the organic carbon content nor to other soil properties like the pH and therefore concluded that the binding of the active ingredient in soil is partially irreversible.

Also, due to their acidic nature and relatively high water solubility, all used ALS-inhibitors, mesosulfuron, pyroxsulam, flupyrsulfuron and propoxycarbazone are very mobile and consequently showed weed control at deeper sowing depths at adequate soil moisture levels. Only the activity of flupyrsulfuron (Lexus) was greatly affected by the deeper sowing depth compared to the other ALS-inhibitors, which indicates that the chemical did not move downwards enough to control the seeds at the deeper sowing depth.

Even though many sulfonylureas have very mobile properties which can be confirmed in lab studies, field experiments over different locations showed that flupyrsulfuron (Rouchaud et al. 1999) and chlorsulfuron (Strek 1998a, Strek 1998b) did not move to deeper soil layers and remained concentrated in a thin layer near the soil surface, which supports the observations of this research. As discussed above the herbicides pyroxsulam, mesosulfuron and propoxycarbazone are highly active via the soil and potent in pot experiments, and thus only a small proportion of these active ingredients is needed to achieve high activity. Also, pyroxsulam followed a similar trend as flupyrsulfuron but only at 1/6 of the field dosage. Because the effective dosage of flupyrsulfuron is not as flexible, which was also shown in another pot experiment, the deeper sowing depth had a large impact on the efficacy.

Because of the relatively short duration of the experiments, it can be concluded that the degradation of the chemicals (DT50) were not the main factor responsible for differences in soil activity. Pyroxsulam, for instance, has according to the PPDB (2012) a much faster soil degradation (DT50 lab: 3 days, DT50 field: 13 days) than mesosulfuron (DT50 lab: 45 days, DT50 field: 78 days) and showed the highest weed control level in presented experiments. Furthermore the microbial metabolism of the herbicides was not expected to be very high during the period of weed germination, due to steam treatment of the soil which likely reduced biological activity. Because of the assumption that the herbicides remained in the root zone, there must have been another reason involved in the showing of soil activity.

Previous studies have shown that the degree of herbicidal activity of ALS-inhibitors such as triazolpyrimidine and sulfonanilide are influenced by the potency of the herbicide on the target enzyme, ALS, and also by the rate of herbicide uptake and metabolism in the plant (Hodges et al. 1990). An increased root uptake of pyroxsulam in *A. myosuroides* has been recently reported by deBoer et al. (2011). They suggest that this possibly caused activity enhancement in black-grass owing to the root uptake. Thus, observed differences in soil activity between used ALS-inhibitors might have been due to different uptake, translocation or metabolism of ALS-inhibitors by *A. myosuroides*. It has been previously suggested that some herbicides are also be able to be absorbed by the mesokotyl or shoot roots (Krähmer 2012) which may have also caused different adsorption characteristics by the herbicides.

Conclusion

Findings of this research support conclusions made by former researchers that black-grass mainly germinates within the first few centimeters (< 5 cm) of the soil profile and that its seedling emergence decreases with higher soil depths.

As the proportion of seedlings which emerge close to the soil surface after chisel ploughing is higher compared to ploughing, it can be assumed that black-grass control with soil-applied herbicides should be higher after minimum tillage.

The degree of weed control after soil application is highly dependent upon the depth of the seedling roots within the soil profile in relation to the position of the herbicide in the soil. Since *A. myosuroides* germinates close to the soil surface, it should be no problem for most herbicides to reach the seed level and perform soil activity, unless conditions are exceptionally dry or the soil it strongly absorptive.

In the absence of adequate soil moisture, black-grass seedlings which are emerging from deeper soil depths are more difficult to control, especially with herbicides with a lower water solubility and higher soil adsorption (Koc), e.g. flufenacet.

It can be concluded that due to their acidic nature and relatively high water solubility most ALS-inhibitors, such as mesosulfuron, pyroxsulam, flupyrsulfuron and propoxycarbazone, are very mobile and consequently are more capable to perform weed control at deeper sowing depths.

As *A. myosuroides* emerges from up to 8-10 cm soil depth, it can be concluded that herbicides such as flufenacet might not be able to reach the root zone of the deeper emerging seeds, and therefore are suspected not to show sufficient weed control in the absence of adequate rainfall in autumn.

5.2.4 Residual soil activity of post-emergence herbicides against *A. myosuroides*

5.2.4.1 Soil-degradation (DT50) of mesosulfuron, pyroxsulam and pinoxaden in different soils under laboratory conditions

In the presented experiment on the dissipation of herbicides in 3 different agricultural soils, Gießen, Rauischholzhausen and Wicker, under laboratory conditions (10°C, 50% of the maximum water capacity) mesosulfuron showed the longest soil persistence (highest DT50) compared to pyroxsulam and pinoxaden. The obtained results generally confirm previous research on the chemicals` degradation behaviors in soils. According to the European registration dossier, mesosulfuron degrades under laboratory conditions at 20°C with DT50 values ranging from 6-91 days (9 soils, pH 5.2-7.5) with a mean of 45 days (PPDB 2012). The manufacturer (Bayer 2002, Gildemeister and Schäfer 2005) investigated the route of degradation of mesosulfuron-methyl in laboratory studies with 8 contrasting soils from Europe and North America under standard conditions of 20 °C and 50% maximum water holding capacity. The calculated first-order DT50 values for mesosulfuron-methyl ranged from 8 to 74 days with a mean of 32 days. The relatively wide range of received DT50 values could not be explained by differences in physico-chemical properties (such as soil texture, pH values or cation exchange capacity) or by differences in soil microbiological biomass. Instead, Gildemeister and Schäfer (2005) suggested that the variability of DT50 values could be associated with differences in the composition in the soil microbial biomass, which was supported by the fact that the soils also showed different metabolite patterns in the degradation.

The degradation of chemical compounds is generally slower under cooler temperatures (Brown 1990, Drobny et al. 2012), which explains that values are not totally comparable to regular studies, which are usually conduced at 20 °C instead of 10 °C, which were used in the experiment. However, two laboratory studies on the degradation of mesosulfuron-methyl were performed at 10°C with 2 soils (pH 5.2-6.8) with similar conditions to the conducted experiment of this research. The DT50 values were between 2-3 times longer at low temperatures (107-212 days) compared to warmer conditions (vs 38.8-41.2 days at 20°C) (Bayer 2002), which was much longer than the DT50 values found in own experiments.

The degradation rate of mesosulfuron and also for pyroxsulam differed among soils, being the fastest in Rauischholzhausen, followed by Wicker and slowest in Gießen. The high degradation rate in soil from Rauischholzhausen might be due to the higher organic carbon content (C-org) and thus a higher microbial biomass. Most herbicides such as sulfonylurea herbicides degrade in soils through a combination of bridge hydrolysis and microbial degradation (Brown 1990). As hydrolysis is significantly faster under acidic (pH 5) rather than alkaline (pH 8) conditions, the persistence of sulfonylureas such as mesosulfuron can be expected be similar in all used soils. As all used soils had almost neutral pH (pH 6.7-6.9) values, neither long nor short persistence was observed in conducted experiments. Because the soils used also had very similar soil textures, differences in the composition in the soil microbial biomass might have also been caused by different DT50 values in the three different soils as reported by Gildemeister and Schäfer (2005).

In contrast to reviewed literature, the received DT50 values for pyroxsulam, with DT50 values ranging from 18-39 days were persistently relatively long. According to the manufacturer`s technical Bulletin (Dow AgroSciences 2007), aerobic microbial degradation is the primary route of breakdown of pyroxsulam in the soil. Pyroxsulam undergoes rapid aerobal microbial degradation with an average laboratory soil half-life of three days at 20 °C (Dow AgroSciences 2007). In the EU dossier pyroxsulam also degrades very quickly under laboratory conditions with DT50 values ranging from 1-15.2 days (PPDB 2012). The discrepancy to received results might be explained by the low temperatures which slowed down the microbial degradation of pyroxsulam, which was already reported by Brown (1990) and Drobny et al. (2012). The observations also confirm findings by the manufacturer, who found that aerobic microbial degradation is the primary route of breakdown of pyroxsulam in soil, which requires adequate soil moisture and temperature for breakdown to occur (Dow 2007).

In the presented laboratory experiment, pinoxaden degraded quickly within one day and was practically undetectable anymore in soil samples on any later extraction day. These findings confirm previous publications on pinoxaden. According to the European DAR (DAR 2006a), pinoxaden degrades very quickly under laboratory conditions at 20°C with DT50 values ranging from 0.086-1.46 days. PPDB (2012) give for pinoxaden a typical DT50 value of 0.5 days. The major transformation products formed are generally the metabolits M2 and M3, whereas metabolized free acid M2 is still active (DAR 2006a). Aerobic soil transformation studies found M2 to be non-persistent with DT50 values of 0.893-56.4 days. PPDB (2012) list for M2 a mean half-time of 23.2 days under laboratory conditions at 20°C with a range of 2.15-44.2 days, which is similar to the values we found in all three soils, but at a lower temperature of 10 °C**.** However, since the M2 is still active a certain residual soil activity can be expected according to its relatively long persistence in the soil.

Conclusions

As the laboratory soil degradation rates at temperatures of 10 °C for all herbicides were relatively slow in all soils, a longer field persistence can be expected after application in autumn when temperatures are generally lower than in the spring. Thus, it can be concluded that an autumn application of pinoxaden, pyroxsulam and in particular mesosulfuron would potentially provide a relatively long residual soil activity against later germinating black-grass.

In pot experiments it was shown that even very small application rates of pyroxsulam and mesosulfuron can provide a high level of soil activity. This leads to the conclusion that a slow degradation rate would keep the concentration of the herbicide in the root zone high enough in order to provide residual soil activity against *A. myosuroides* to some extent.

The persistence and thus potential soil activity of herbicides is highly variable within the location. While the herbicide's chemical properties influence the soil degradation behavior in general, the environmental conditions such as soil properties (e.g. organic biomass) have an impact on the degradation rate.

5.2.4.2 Residual soil activity of post-emergence herbicides against black-grass in pot experiments under greenhouse conditions

In pot experiments under greenhouse conditions all ACCase-inhibitor herbicides Ralon Super, Topik 100 and Axial 50EC did not show any significant residual soil activity, which confirms the findings discussed above and previous cited publications. The DT50 values of the parent compounds such as fenoxaprop-P-ethyl, clodinafop-propagyl and pinoxaden are generally very short with a DT50 of 0.2-1.5 days, whereas the metabolized free acids usually persist a little longer (DAR 2004a, DAR 2006a, DAR 2006b, PPDB 2012).

These free acids such as clodinafop and the M2, metabolized from pinoxaden, can be attributed to have some residual soil activity. In the experiments black-grass was sown right after herbicide application. Since 5-7 days passed by until black-grass seeds germinated, clodinafop and in particular pinoxaden must have been present with a high enough concentration in order to show activity on the first sowing time. The DT50 of the different active acids support these observations. For fenoxaprop-P half-lives of 4-20 days were reported with a mean of 7.5 days (DAR 2006b, PPDB 2012), which was generally the shortest among them. In laboratory studies on clodinafop which were submitted for the European registration, the free acid degrades with DT50 values of 7.4-18 days and a mean of 12 days (DAR 2004, PPDB 2012). Aerobic soil transformation studies on pinoxaden found for the free acid, the metabolite M2, DT50 values of 0.9-56 days (DAR 2006a), while PPDB (2012) report an average DT50 value of about 23 days for the M2.

While the results for the ACCase-inhibitors did not differ much between the 2 presented pot experiments, results obtained for the ALS-inhibitors showed variable results, in particular for Broadway and Lexus. The variability of residual soil activity could be explained by different climate conditions during experiments. As shown in section 3.6.5.2, the average temperature in the same greenhouse was over 4 °C warmer in the 2nd experiment, which was conducted from April to June, compared to the first experiment which was done from January-March 2011. Without changing the intensity of watering, the soil in pots in the 2nd experiment was much dryer during the experimental period because of the higher temperature and also additional hours of sun light hitting the soil surface.

The major mechanisms of degradation in soils are aqueous hydrolysis and microbial degradation (Walker et al 1989, Brown 1990). Indirect photolysis is generally a minor mechanism and does not contribute to the degradation of sulfonylureas such as flupyrsulfuron and mesosulfuron (Bayer 2002, Strek 2005) and also pyroxsulam and propoxycarbazone (Bayer 2005, Dow 2007) are not degraded through photolysis. According to Strek (2005) and Brown (1990) the break down processes of sulfonylureas are highly dependent on the soil properties, climate conditions and the compound itself. While hydrolytic breakdown is often dominant at acidic soil conditions, the microbial degradation is more important in neutral to alkalic soils, whose processes are highly dependent on optimal temperature and soil moisture conditions (Strek 2005). Dry conditions for instance decrease microbial breakdown, whereas cold temperatures can slow down degradation (Brown 1990). Generally hydrolysis is only relevant under acidic conditions for most herbicides.

For mesosulfuron (Bayer 2002) it has been reported that it degrades relatively quickly under acidic conditions (e.g. mesosulfuron half-live 3.5 days at pH 4) whereas it is hydrolyticly stable under neutral and basic conditions (pH≥7). Also, pyroxsulam and propoxycarbazone are known to be stable to hydrolysis at neutral soil conditions (Drobny et al. 2012). As a consequence it can be concluded that compounds were primarily degraded via microbial processes in pot experiments, because the standard soil used in pot experiments was a silt loam (sand 15.9%, silt 58.3%, clay 25.8%,C-org 1.6%) with a pH of 7.1. On the contrary, flupyrsulfuron is degraded by hydrolysis at 20 °C and pH 7 with a DT50 of 12 days, which is why degradation processes in the soil were probably chemically and microbially.

The differences in the duration of residual soil activity of ALS-inhibitors observed in the pot experiments can generally be explained by the compounds different degradation behavior in soils (DT50 values). Atlantis WG demonstrated high residual soil activity (> 4 weeks) independently from the climate conditions in all 3 presented pot experiments. The degradation of mesosulfuron was not very affected by soil moisture conditions, resulting in relatively long persistence and thus long activity. PPDB (2012) list a relatively slow degradation for mesosulfuron with a typical laboratory DT50 (20 °C) of 45 days, with values ranging from 6-91 days. Also the high DT50 values of mesosulfuron from field studies for on average 78 days (PPDB 2012) support current findings of a relatively long residual activity in soils. Iodosulfuron, the other active ingredient contained in the used herbicide Atlantis, is generally not very active against *A. myosuroides* and has low DT50 values of 2 days under laboratory conditions (PPDB 2012). Further, Trabold et al. (2000) report that iodosulfuron is primarily degraded by microbes. In field dissipation studies idosulfuron degraded fast with DT50 values ranging from 1-5 days, which might be reduced with dry soil conditions. In the PPDB (2012) an average half-life of 8 days is given for iodosulfuron.

In addition, received results showed that propoxycarbazone also has a high residual soil activity which lasts for at least 28 days independently of the moisture condition. In laboratory soil degradation studies it was shown that propoxycarbazone-sodium was degraded slowly to moderately ranging from 23-99 days (mean=61 days) depending on the soil type investigated (Bayer 2005, PPDB 2012). In field dissipation studies conducted in Europe, propoxycarbazone-sodium was rapidly degraded in soils with DT50 values ranging from 6-56 days and a mean of 23 days.

Flupyrsulfuron is degraded chemically and microbialy in soils. In laboratory aerobic soil studies (at a temperature of 20°C) flupyrsulfuron degraded relatively fast with DT50 ranging from 6-26 days and a mean of 13 days (PPDB 2012), which supports results of this research. In field dissipation studies conducted in France and the UK, flupyrsulfuron degraded quickly with DT50 values from 6-11 days and a mean of 8 days. The rate of degradation is not as much affected by the pH and increases in warmer conditions (Rouchaud et al. 1999), which can be used as an explanation for the longer residual soil activity of flupyrsulfuron in the first experiment, which was conducted under cooler temperatures.

In contrast to the other ALS-inhibitors discussed before, Broadway showed a much longer residual soil activity (> 4 weeks) in the 2nd experiment, when warmer and thus dryer soil conditions were present. The 3rd experiment, which was conducted with different watering intensities supports these observations. It was demonstrated that under dry soil conditions Broadway showed a long residual activity for at least 28 days, whereas at moist and wet soil conditions no persistent soil activity was detectable. It can be concluded that pyroxsulam degrades relatively fast under moist soil conditions, whereas dry soil conditions lead to a longer persistence and thus longer residual activity. The observations confirm findings by the manufacturer, who found that aerobic microbial degradation is the primary route of breakdown of pyroxsulam in soils, which requires adequate soil moisture and temperature for breakdown to occur (Dow AgroScience 2007). According to the manufacturer, pyroxsulam degrades rapidly with an average laboratory soil half-life of three days at 20 °C. pyroxsulam is more tightly bound to organic matter than to clay and its solubility increases with pH value. Consequently, degradation rates are generally faster with higher pH and lower organic matter (Dow AgroScience 2007).

Strek (2005) who did intensive research on soil residual herbicides reports that the effect of drought on the degradation of sulfonylureas depends on the relative importance of the microbial degradation versus aqueous hydrolysis, on soil parameters and on the particular herbicide itself. Under conditions where soil temperatures and moisture are adequate for normal plant growth, the relative importance of each degradation mechanism may be theoretically where microbiological degradation is major, hydrolysis is secondary and indirect photolysis is minor. In contrast, under drought conditions degradation attributed to soil microbes becomes much smaller due to negative effects on the microbes. This can cause a decrease in the overall rate of degradation of those compounds which degrade only or mostly through microbial activity. This is most likely the explanation for the variable results for pyroxsulam under different soil moisture regimes which were presented. However, Strek (2005) explains that an increased hydrolysis due to a decrease of the pH near the soil constituent surfaces can be observed as the soil water decreases. This increased degradation via hydrolysis may then compensate partially or entirely for the loss in microbial degradation. This could be the reason why propoxycarbazone and mesosulfuron in particular showed long residual activity in all experiments independently from the soil moisture regime.

Laboratory experiments of pyroxsulam yielded an average Koc of 30 ml/g (range 2-129), indicating that pyroxsulam was weakly to moderately adsorbed (Dow AgroScience 2007). Consequently, it is also possible that high watering intensity could have also led the chemical compound to leach out of the root zone or even out of the pot, which could explain differences in soil activity at different watering intensities. Nevertheless, this is questionable, because the physico-chemical properties of the compounds such as water solubility or soil adsorption coefficients (Koc) do not differ much between the ALS-inhibitors used according to the PPDB (2012). Using the mobility index proposed by McCall et al. (1980), all used ALS-inhibitors are according to their relatively low soil adsorption in the mobility class high and very high. As a consequence, the water mobility of these chemical compounds is highly likely not the reason for differences in the soil activity between ALS-inhibitors.

Conclusions

In can be concluded that ACCase-inhibitors do not provide any or very short residual soil activity against *A. myosuroides*. On the contrary, herbicides containing ALS-inhibitors, in particular mesosulfuron, propoxycarbazone, pyroxsulam can provide a relatively long residual soil activity against *A. myosuroides*.

Furthermore, it can be concluded that the duration of soil activity is generally dependent on the degradation behavior of the compound itself and is highly influenced by climate conditions, especially by soil moisture.

A relatively long residual soil activity can be expected by mesosulfuron and propoxycabazone independently from soil moisture after application (precipitation intensity). On the other hand, it can be concluded that pyroxsulam`s dissipation rate is relatively quick under moist soil conditions, whereas dry soil conditions lead to a longer persistence and thus longer residual soil activity.

5.2.4.3 Residual soil activity of post-emergence herbicides against *A. myosuroides* under field conditions

The complex network of factors operating in field studies may result in very different results than those in controlled laboratory experiments (Strek 2005). The dynamics of soil drying and rewetting, temperature fluctuations and the presence of light are a few factors absent in laboratory tests and which can contribute to the faster degradation generally observed in field soils. Also, pot experiments in the greenhouse have been reported to increase herbicidal activities as discussed above.

The field studies which were conducted on the duration of residual soil activity of post-emergence herbicides over two years in Wicker confirm observations which were in greenhouse experiments for ACCase-inhibitors. As in the conducted pot experiments in the greenhouse, pinoxaden showed significant soil activity only in the beginning of the field trial in both years of testing. However, all ACCase-inhibitors did not show any residual soil activity against sown black-grass, which can be explained with the generally short persistence of this chemical group in soils as discussed above.

For the European registration of pinoxaden, field dissipation studies on bare soil were performed by the manufacturer on a range of soil types in various locations in spring and autumn. Studies generally confirmed the degradation routes observed in laboratory studies, with pinoxaden degrading very quickly (half-lives < 1 day). Also, the active metabolite M2 degraded relatively rapidly, with half-lives of between 0.69 and 9.73 days (DAR 2006a, PPDB 2012).

In 4 field dissipation trials conducted in Germany for the EU-registration, the parent clodinafop-propagyl degraded immediately and was not detectable in any samples taken on the day of the application. DT50s values of the acid metabolite (clodinafop) were 4.6-13 days with a mean of 8 days (DAR 2003). In field studies conducted in the UK, clodinafop degraded a bit slower, with the DT50 values of 19-33 days (PBDB 2012).

Field degradation studies in various soils resulted in half-lives for hydrolyses of fenoxaprop-P-ethyl to the free acid of under 1 day (Agrevo1, DAR 2005, PPDB 2012). According to the manufacturer, the half-lives for degradation of fenoxaprop-P were 2-17 days (Agrevo 1). The technical brochure for PUMA containing fenoxaprop-P-ethyl states, that the dissipation time for the herbicidal active acid fenoxaprop depends on the microbiological activity of the soil, but is generally with 1-2 weeks of relatively short duration (Hoechst 1988). In support of the obtained results in field experiments, no risk of damage on succeeding crops or early after cropping is given in product labels or technical brochures of the ACCase-inhibitors as mentioned above.

The herbicides flupyrsulfuron, mesosulfuron, and pyroxsulam are weak acids with pKa of 4.94, 4.35 and 4.67 respectively (PPDB 2012). Propoxycarbazone is according to PPDB (2012) with a pKa of 2.1 a strong acid. They exist primarily in the neutral form at a pH below pKa and in the anionic form at a pH above pKa (Nicholls and Evens 1985). Therefore, in more alkaline soils, these herbicides will be present in the anionic form. Low sorption properties of sulfonylureas and higher pH values (Walker et al. 1989) is likely to cause greater leaching after heavy rainfall (Nicholls and Evens 1985). At high pH levels in soils those herbicides as anions are very mobile. This could lead to increased persistence of residues over a long period of time as the microbial activity would be expected to be much lower with increasing depth. Further, the long-term sulfonylurea degradation mostly depends on chemical hydrolysis, controlled by soil pH. Thus, in alkaline soils where chemical hydrolysis is minimized, herbicides such as chlorsulfuron (Walker and Welch 1989, Kotoula-Syka et al. 1993, Strek 1998a, Strek 1998b) can persist long enough to injure certain rotational crops. Because the soils at Wicker where the experiments were carried out had a pH of 6.8, a low to moderate of risk of leaching was expected and a balanced degradation rate.

In contrast to ACCase-inhibitors, there are usually carry-over risks on rotational crops and/or on replacement crops in case of crop failure of most ALS-inhibiting products such Atlantis, Lexus and Attribut which demonstrate their long residual soil activity (Bayer 2013a, DuPont 2013). Generally the re-cropping intervals for sensitive crops after the application of ALS-inhibitors which are registered in the autumn and spring such as Atlantis WG and Lexus are dependent on the dosage, application timing and soil cultivation. For instance after the application of max. 300 g/ha of Atlantis WG in autumn, only cereals and maize are considered safe to be planted in the spring in the event of crop failure and only after tillage cultivation (Bayer 2013a). Further, according to the German label, injuries on succeeding crops such as rapeseed, dicotyledonous catch-crops, sunflower and sugar beets might occur after the application of 400 g/ha in the spring, which enables the awareness of long residual activity of the mesosulfuron. Similar re-cropping intervals and carryover risks are reported for Lexus and Attribut. Neither winter oilseed rape nor dicotyledonous catch-crops may be sown to succeed a winter wheat treated with Lexus or Attribut in the spring. Even after an application of Lexus in autumn there might be a risk of crop damage on rapeseed or on dicotyledonous catch-crops in the following year.

The received results from ALS-inhibitors confirm observations which were made in pot experiments in the greenhouse. Differences in the persistence of soil activity between the ALS-herbicides can generally be explained by the different degradation periods (DT50) in soils of the active compounds. As expected, the residual soil activity among the ALS-inhibitors was the longest for Atlantis in both years, which can be explained by the relatively long degradation period of mesosulfuron in soils.

The degradation of mesosulfuron in agricultural soils was investigated by the manufacturer in 6 field dissipation studies at different locations in Northern and Southern Europe. DT50 values calculated for the trials with spring application were between 44 and 76 days for mesosulfuron with a mean value of 64 days (Gildemeister and Schäfer 2005). In 4 field soil dissipation studies on mesosulfuron which were submitted for the European registration, the parent degraded relatively slowly after autumn application with DT50 values ranging from 77-114 days and a mean of 99 days. Under field conditions, the degradation of a compound is strongly influenced by the variable soil temperature and soil moisture. According to Gildemeister and Schäfer (2005) this can result in a slow-down of the degradation during colder or dryer periods or in lag-phases after autumn application due to low temperatures in the winter, which can be used as an explanation for presented results in this research. The manufacturer concluded from the results that the compound is considered moderately degradable in the field soils and is unlikely to be detected in soil a year after application of the recommended application rate (Gildemeister and Schäfer 2005, Bayer 2002).

Similar to the other ALS-inhibitors, flupyrsulfuron-methyl sodium is weakly adsorbed to the soil, resulting in Kocs ranging from 15-55 ml/g and a mean of 28 ml/g (PPDB 2012). However, the persistence and mobility of flupyrsulfuron has been measured in the soil of several winter wheat crops after autumn or spring applications by Rouchaud et al. (1999). In their experiments flupyrsulfuron did not move to deeper into the soil layers and remained concentrated in a thin layer near the soil surface, where a high concentration could provide high herbicide efficacy. Flupyrsulfuron is degraded chemically and microbialy in soil. In various field dissipation studies conducted in France and the UK, flupyrsulfuron degraded relatively fast with DT50 values ranging from 6-11 days and a mean of 8 days (PPDB 2012). The sulfonylurea herbicides chlorsulfuron and triasulfuron have uses in winter wheat similar to that of flupyrsulfuron. chlorsulfuron has a longer soil half-life between 11.2-185 days (mean 36 days) (PPDB 2012), and it results in low levels of persistent residues in soil (Anderson and Barrett 1985). Triasulfuron has a soil persistence similar to that of chlorsulfuron (Kotoula-Syka et al. 1993).

Several other sulfonylureas used in cereals such as chlorsulfuron and metsulfuron are known to persist relatively long in the soil which may result in residual injuries on several succeeding crops (Anderson and Barrett 1985, Peterson and Arnold 1985, Nicholls et al. 1987). The residual phytotoxicity and persistence of the herbicides chlorsulfuron, tribenuron-methyl, triasulfuron and metsulfuron-methyl was demonstrated on bioassays with different sensitive crops by Kotoula-Syka et al. (1993). The results indicated that phytotoxicity of all herbicides was not affected by soil texture but was increased with increasing herbicide concentration and soil pH. The behavior and persistence of sulfonylureas in the soil and its influencing factors has been investigated by many researchers in the past 30 years and is summarized by Blair and Martin (1988), Russell et al. (2002), Strek (2005), and Grey and McCullough (2012).

As expected, obtained results showed that propoxycarbazone also has a long residual soil activity which can last for up to 28 days independently of the moisture conditions, which confirms observations made in pot experiments. In field dissipation studies conducted in Europe, propoxycarbazone-sodium was rapidly degraded in the soil with DT50 values ranging from 6-56 days and a mean of 23 days (PPDB 2012). Also, carryover risks of rotational crops such as winter oilseed rape in product labels of Attribut support observations which were made in the field.

On the contrary, label information of Broadway claims that the herbicide containing pyroxsulam works only through foliage uptake and the company points out that there are no after cropping restrictions (Dow AgroSciences 2013). In general pyroxsulam degrades rapidly and residues in the soil generally do not persist long enough to damage crops in the following season. Field dissipation studies for pyroxsulam, conducted in-season in western Canada, resulted in an average soil half-life of 13 days (Dow AgroSciences 2007). Pyroxsulam is more tightly bound to organic matter than to clay and solubility increases with pH. Consequently, degradation rates are generally faster with higher pH and lower organic matter. Like many other compounds which are microbialy degraded, it requires adequate soil moisture and temperature for breakdown to occur (Dow AgroSciences 2007). This can be used as an explanation for variable results for Broadway in the two trialing years. In the first experiment in 2010, when Broadway showed residual soil activity up to 28 days, pyroxsulam probably persisted longer in the soil because of relative dry soil conditions after herbicide application. In 2010 there was practically no rainfall measured for 11 days after application, compared to regular rainfall events right after application in 2011. As a consequence, there was persistent soil activity observed. Dry soils have previously been reported to slow down chemical and microbial breakdown processes by researchers (Strek and Green 2001, Drobny et al. 2012). Further, these conclusions are supported by the observations which were made in presented greenhouse experiments, which proved that Broadway can have a long residual activity under dry soil conditions as discussed above.

As expected, the longest residual soil activity which lasted until the following spring in both years was observed by herbicides which are usually applied pre-/early post emergent to the crop such as flufenacet (Cadou SC, Malibu) and pendimethalin (Stomp aqua, Malibu). Differences in the residual soil activity between these herbicides can generally be explained by the different degradation periods (DT50) in soils of the active compounds.

The rate of degradation of flufenacet in soils has been investigated in a range of laboratory and field studies (Bayer 2000). In 16 field studies conducted at 10 different sites in Europe, flufenacet degraded rapidly in soil with and without plant cover. No mobility of flufenacet was observed and residues remained in the upper soil layer throughout the duration of all field tests. In field dissipation studies conducted for the European registration, flufenacet was moderately persistent with DT50 values ranging from 38-43 days after autumn application and a mean of 40 days (PPDB 2012).

The long residual activity against black-grass of products containing pendimethalin (Malibu, Stomp aqua) was observed in field experiments and can be explained by the relatively slow degradation and thus long persistence in soils. In field dissipation studies conducted for the European registration, pendimethalin was moderate persistent with DT50 values ranging from 27-186 days with a mean of 90 days (PPDB 2012). Also, laboratory studies results generally showed high DT50 values of 72-172 days (mean 123 days). The herbicide Boxer (prosulfocarb) generally has a relatively short persistence in soils, resulting in DT50 values in the field of 6.5- 13 days and a mean of 9.8 days (PPDB 2012). The obtained results did not show any long residual soil activity of prosulfocarb which is in agreement to previous knowledge.

Conclusions

In can be concluded that ACCase-inhibitors do not provide any or very short residual soil activity against *A. myosuroides* under field conditions.

After application of ALS-herbicides in autumn, the concentration of the active compounds such as pyroxsulam, mesosulfuron, propoxycarbazone, flupyrsulfuron in the upper soil layers is often high enough to control later emerging *A. myosuroides* to some extent.

Generally, mesosulfuron and propoxycarbazone have a tendency to provide longer residual soil activity than flupyrsulfuron and pyroxsulam. Nevertheless, herbicides based on flufenacet and pendimethalin are much more suited to provide residual soil activity against black-grass, which can last until the following spring.

In conclusion, residual soil activity is variable within the years and is highly dependent on environmental conditions, e.g. soil moisture. In particular, pyroxsulam seems to persist longer under dry soil conditions and thus provides longer residual soil activity.

5.3 Management of *A. myosuroides*

5.3.1 Effect of sowing date on populations of *A. myosuroides*

Generally, an earlier sowing date of winter wheat gives the opportunity for a longer growing season for crop development and thus potentially creates higher yields. Furthermore, farmers are often under enormous time pressure to complete their field work in favorable weather and soil conditions. In particular, farms with heavier soils and more or less high natural infestations of *A. myosuroides*, have a narrow time window for optimal soil conditions and are so called "Minutenböden". Farmers are highly dependent on good weather and often cannot risk potentially poor weather in late autumn and consequently use early sowing times.

As shown above, *A. myosuroides* is able to emerge over a long period of time from September until May the following year (Meiners et al., 2012), but the probability of its emergence is higher in early sown winter wheat compared to later drillings. This finding agrees with former investigations of Kemmer and Koch (1980). All conducted field trials showed that delaying the sowing date of winter wheat by 2-6 weeks reduced the weed density of *A. myosuroides* significantly by 66-87%. Other studies have also shown that later sowing of winter cereals can reduce *A. myosuroides* seedling emergence compared with 3 to 4 weeks earlier sowing time (Moss 1985a, Amann 1991, Amann et al. 1992, Melander 1995).

Hurle (1993) reported that in an experiment, delaying the sowing from September to October decreased the plant number of black-grass by 72%. In contrast, Cussans (2009) only found in the UK a clear reduction in black-grass population density when sowing was delayed until November, as September and October sowings resulted in similar densities with black-grass. The conducted meta-analyses of 19 field experiments in the UK since 1985 showed that delaying sowing from September to the end of October decreased *A. myosuroides* infestations by approximately 50% (Moss et al. 2013). A delayed drilling in autumn by about 3 weeks reduced plant numbers in the UK by 31% on average (Moss 2013), although a high variability occurred in the data from September to mid-October. Sowing wheat in the spring achieved an 88% reduction in *A. myosuroides* plant densities compared with autumn sowing (Moss et al. 2013).

Studies od this research confirm studies by Moss (2013) that the later the drilling the higher the effect. These results confirm that the delaying of sowing time as well as control of emerged plants by burn-down applications with glyphosate and seedbed preparation can be an effective tool to reduce infestations with black-grass. Other factors such as less favorable conditions for germination as well as higher susceptibility to freezing among later emerging seedlings may also be responsible for reduced weed numbers when sowing is delayed. In the field experiment 2011/2012 it was observed that early emerged and already tillering plants were able to survive heavy frost periods during the winter, however, later emerged seedlings (by 5 weeks) with 2-3 leaves were not able to survive (data not shown).

In these field experiments yield reductions by *A. myosuroides* at the earlier sowing date with reduced tillage were markedly higher (42-85 dt/ha or 5.9 dt/100 black-grass heads on average of all experiments) than yield losses at later sowing times (5-27 dt/ha or 2.6 dt/100 black-grass heads on average). Moss (1985a), Kötter (1991) and Melander (1995) also observed that *A. myosuroides* caused greater yield loss the earlier the sowing date. In addition, the relative time of emergence strongly influences the outcome of competition between crop and weed (Cousens et al. 1987). Therefore it was concluded that grasses at the later drilling date have a reduced competitive ability (Melander 1995). Grass weed individuals which still emerge in the spring after herbicide applications usually do not affect the yield as drastically as in the autumn, most likely because of crop competition. In experiments with high infestations of black-grass, it was shown that best yield responses come from herbicide treatments applied in the autumn or early winter, rather than in the spring (Wilson et al. 1985). Especially the late sown winter wheat in Rauischholzhausen led to high yields in all years, even though the black-grass control was not very effective. According to Thursten (1972), later germinating black-grass tends to tiller less, is less competitive and sheds fewer seeds than those germinating in the early autumn. However, these plants still produce seeds and might cause weed problems in future growing seasons, especially if these weeds are resistant. Therefore, independent from the sowing date, it must be a goal that close to a hundred per cent of the infestation should be controlled for effective and long-term black-grass management. Nevertheless, in field trials it was demonstrated that an early sowing time can still work and give high yields of winter wheat as long an effective chemical control of *A. myosuroides* is possible. In field trials conducted by Kötter (1991), the yield of early sown wheat was reduced by 4-5 dt/ha per 100 heads of *A. myosuroides* while Machefer et al. (1998) found an average reduction by 1.3 dt/ha per 100 black-grass heads on average in various field trials. Conclusively, Kötter (1991) and Blair et al. (1999) demonstrated that the yield losses from *A. myosuroides* in winter wheat are highly variable within years and sites and are very dependent on factors like crop, crop density, sowing time and nitrogen level (Hurle 1993).

Landschreiber (2014) concluded from field experiments in Northern Germany that it would be most beneficial to maximize germination of fresh seeds prior to sowing as much as possible, with repeated and very shallow cultivations (1-2 cm). Delayed sowing time would be the most effective way to reduce black-grass pressure by agricultural means, in order to allow enough time for the maximum number of black-grass to germinate. Also, the wheat variety might have positive effects.

Conclusions

It can be concluded that a later sowing date is a very effective management tool, as current research showed that delaying the sowing of winter cereals can reduce the infestation of black-grass. Delaying the sowing time gives the opportunity to maximize the proportion of black-grass emerging prior to sowing, which can then be controlled mechanically or by use of non-selective herbicides. Also, late emerging weeds are usually not as far developed in the spring and are therefore more susceptible to herbicide applications. In addition, black-grass individuals which emerge later or in the spring usually do not affect the yield as drastically as in the autumn, as they are usually less competitive.

5.3.2 Effect of the cultivation system on populations of *A. myosuroides*

Normally the germination of *A*. *myosuroides* is limited to the first 2.5 centimeters of soil (Naylor 1970, Moss 1985c) and its emergence decreases with a larger soil depth (Colbach et al. 2005). In various trials the emergence of *A. myosuroides* decreased by about 70% at a soil depth of 6 cm, while no emergence was recorded at 10 cm by Benvenuti et al. (2001). In experiments by Menck (1968), it was stated that the maximum depth of emergence for black-grass was 15 cm. Recently, Moss (2013) published that black-grass plants can only emerge successfully from seeds retained within the 5 cm of surface soil, which is why minimum tillage encourages black-grass. As reported by Swanton et al. (1993), many scientists have proven that conservation tillage is leading to an increase in monocot and perennial weed species. Results of this research indicate that mouldboard ploughing has a major reductive effect on the density of *A. myosuroides* in early sown winter wheat. These results support those from other experiments (Pollard et al. 1982. Moss 1985b, Knab and Hurle 1988, Amann et al. 1992), which showed that infestations of *A. myosuroides* are higher after reduced tillage than after ploughing. Amann (1991) showed that reduced tillage results in at least a threefold increase of infestations compared to tillage cultivation. Hurle and colleagues concluded in three experiments that mouldboard ploughing would reduce black-grass populations by 85% compared with tine cultivation (Hurle 1993, Knab and Hurle 1988). The implications of long-term French cropping system experiments are that mouldboard ploughing achieved a 25-52% reduction in *A. myosuroides* density (Chauvel et al. 2009). The conducted meta-analyses of 25 field experiments in the UK since 1979 showed that mouldboard ploughing reduced the number of *A. myosuroides* populations in the subsequent cereal crop by an average of 69% compared to non-inversion tillage (Moss 2013, Moss et al. 2013). So these results above broadly support the conclusions from this research that mouldboard ploughing is a very effective cultivation practice in order to reduce *A. myosuroides* populations. In comparison, these field trials showed that infestations with *A. myosuroides* were reduced by 90% in 2010 and by 70% in 2011 due to onetime ploughing. This is most likely because most fresh seeds are buried to depths of more than 5 cm after ploughing cultivation (Cussans and Moss 1990) and have a reduced chance of emergence, while with shallow tine cultivation they remain in the upper layer and contribute to the infestation immediately (Naylor 1970, Knab and Hurle 1988, Hurle 1993).

Moss (1980) calculated that 80-90% of the plants in direct drilled crops were derived from seeds shed in the previous crop. Previous work has shown that seed numbers of buried *A. myosuroides* decline rapidly in the soil annually by 72-83% (Moss 1985c). But only a small proportion, approximately 1% of the seeds, are likely to persist and stay viable for many years and can act as a source for future weed infestations if no control measures are used (Moss 1980, Pollard et al. 1982, Moss 1985c). Recent publications state that the survival of buried seeds in the soil is about 20-30% per year, so after 3 to 4 years burial, only about 1-3% of seeds will still be viable (Moss 2010). Menck (1968) considers that seeds can survive up to 9-11 years. In a study on the longevity of seeds related to placement in the soil, Jensen (2009) demonstrated that leaving the seeds of *A. myosuroides* directly on the soil surface after an interval of 1-2 months or also approximately 1 year, reduced the persistence of surviving seeds to very low levels compared with incorporated seeds.

It is known that the beneficial effect of the first cultivation is the largest, while further cultivations bring seeds up to the surface again and lead to a constant low weed population without weed control measures (Cussans and Moss 1990). Nevertheless, rotational ploughing once every 4-5 years has been proposed as one method of improving the overall level of weed control in reduced tillage systems (Cousans and Moss 1982, Froud-Williams et al. 1983, Cousans and Moss 1990). Since previously buried viable seeds would regularly be brought up again and create fresh infestations, it can be assumed that the effects from ploughing at the location in Rauischholzhausen would be most effective in the beginning when populations are high. On the contrary, Landschreiber (2014) concluded from their recent experiments in Northern Germany that the most efficient way order to eradicate black-grass would be to leave seeds close to the surface where non-dormant seeds can germinate prior to sowing, and not to bury fresh *A. myosuroides* seeds to the deeper soil level via mouldboard ploughing or chisel ploughing cultivation. She concluded that it would be most beneficial to maximize germination of fresh seeds prior to sowing as much as possible with repeated, very shallow, cultivations (1-2 cm). In order to allow more time to maximize the proportion of black-grass to germinate, a delayed sowing time would be the most effective way, as already discussed above.

Conclusions

It can be concluded that ploughing cultivation is very effective in reducing high densities of black-grass populations and should be considered for an immediate management tool. In contrast to minimum tillage, ploughing buries most freshly shed seeds to a depth where emergence is unlikely. It can be concluded, that ploughing cultivation is usually most effective in the beginning when populations are high, because the amount of fresh viable seeds buried will be greater than the amount of old seeds which might be brought up to the surface again.

5.3.3 Effect of different herbicide treatments in field experiments

The field experiments carried out at two research stations demonstrated that the performance of herbicides varied strongly within the two locations. In GI, with no recorded herbicide resistances, all herbicide treatments had the potential to give high levels of control of *A. myosuroides* in early and late sown winter wheat.

Only the pre-emergence herbicides Cadou SC and Herold SC did not give as good of a weed control as post-emergent herbicides with ACCase- and ALS-inhibitors, most likely because a lot of weeds were already emerged and too far developed for optimal control. An experiment in the greenhouse has shown that the best efficacy of soil acting herbicides such as Cadou SC can be achieved when it is applied pre-emergence (see chapter 4.3.1.2), whereas a decrease of herbicidal activity can be observed with increasing growth stages of *A. myosuroides*. Further, there was almost no precipitation recorded 14 days after herbicide application in the fall. This possibly led to unfavorable soil conditions (dry) for optimal herbicide efficacy by soil acting herbicides (data not shown). A pot experiment under controlled conditions demonstrated that the herbicidal activity of flufenacet is highly dependent on adequate soil moisture which was discussed above (see chapter 5.2.3.5).

For the post-emergence herbicides, Ralon Super seemed to be less efficient than Axial50EC and Topik100, and worked best in combination. The highest herbicidal efficacy in the fall was achieved by ALS-inhibitors such as Broadway, Alister and Atlantis WG resulting in the highest grain yield.

Similarly, all herbicides containing ACCase and ALS-inhibitors showed high herbicidal efficacies against sensitive black-grass at the location Wicker. Also, a high efficacy received from soil herbicides containing flufenacet and pendimethalin was probably due to the relatively high soil moisture conditions around the time of the herbicide application in both years, promoting residual activity, as proven and confirmed in greenhouse experiments.

In contrast, most herbicide treatments gave variable and often inadequate weed control in RH in early and late sown winter wheat due to the herbicide resistances which were found for the black-grass population in lab-analyses (see also 4.3.1). Collected plants showed enhanced metabolism resistance (EMR) for fenoxaprop-P-ethyl and mesosulfuron-methyl and the well-established ACCase-Target-Site-resistance (I2041N). Therefore an effective weed control in field experiments conducted in Rauischholzhausen was not possible anymore with applied ACCase-inhibitors in early and late sown winter wheat. A greenhouse experiment presented in this research (see chapter 4.3.1.1) also confirmed that the black-grass population from Rauischholzhausen was not sensitive to ACCase-inhibitors. However, there was some evidence that pinoxaden (Axial 50EC) gave better weed control than the other ACCase-inhibitors in the field experiment, which supports recent work by Petit et al. (2010).

The best control of the local black-grass population, where collected plants also showed little enhanced metabolism of the ALS-inhibitor mesosulfuron-methyl in laboratory tests, was given by applications with Atlantis WG (mesosulfuron-methyl). Other investigations have also shown that Atlantis WG appeared to be the most effective post-emergence applied product, especially on biotypes with metabolic resistances (Menne and Hogrefe, 2012). Obtained greenhouse results support these observations and showed that Atlantis is the most effective herbicide against the resistant black-grass population from RH compared to Lexus, Attribut and Broadway, especially at later growth stages (see chapter 4.3.1.1). While all herbicides showed a high level of control against the sensitive population, Atlantis showed as the only herbicide with some control against the highly resistant population from the UK.

The very effective weed control of ALS-inhibitors in autumn on small black-grass individuals at Rauischholzhausen are in support with observations which were made in the greenhouse. It was demonstrated that ALS-herbicides such as Atlantis, Broadway, Lexus and Attribut showed a significant/much higher level of black-grass control when applied at the early growth stage (BBCH 12) of black-grass compared to applications at later growing stages (BBCH 23). This might explain the low level of weed control in percentage terms which was often achieved in the spring by ALS-herbicides.

In most cases enhanced metabolism of resistant weeds has been associated with enhanced cytochrome P450 mono-oxygenase (Hall et al.1994, Hyde et al. 1996, Hall et al. 1997) or glutathione-S-transferase (GST) activity (Cummings et al. 1997, Cobb et al., 2001, Reade et al. 2004). It has been shown that the GST activity increases with the development of black-grass, suggesting that younger plants are more susceptible to herbicide applications compared to older plants (Cobb et al. 2001, Shim et al. 2003).

Although several other ALS-herbicides such as Broadway or Lexus gave moderate to very good levels of control in percentage terms visually like in 2010/2011, the actual number of black-grass inflorescences present in the spring was often substantial and inefficient for sustainable weed control.

In field experiments it was often observed that even small black-grass plants close to the ground surface and often not visible at first sight are able to produce seeds in the wheat field. It is reported that farmers usually see no need to change their production system and that they accept inefficient weed control caused by resistances, as long as high yields can still be produced. Conducted trials in RH as well as in GI prove that especially in late sown winter wheat all ALS-inhibitors applied in the spring achieved, independently of the level of weed control, high yields which did not differ much from each other. The question arises in such situations of how quickly resistance will continue to evolve if there are weeds escaping herbicide applications and producing seeds. Over time, it can be assumed that the problem will increase if the farmer is not able to achieve a high level of weed control. Models dealing with general predictions of black-grass infestations (Cussans and Moss 1990, Colbach et al. 2005, Colbach et al. 2006) as well as models on the development of resistances after different cultivation and herbicide strategies (Gressel and Segel 1990, Cavan et al. 2000, Chauvel et al. 2009) are available.

The moderate control achieved by soil herbicides based on flufenacet and pendimethalin in RH in 2009 and 2010 is most likely due to herbicide resistance in combination with a lack of soil moisture, which is very important for herbicide uptake by the roots in order to achieve high levels of weed control (Mitnacht and Kemmer 1990, Wolber 2011). In 2009 there was almost no rainfall recorded 5 days after herbicide application. Also, in 2010 there was no precipitation recorded for 10 days after herbicide treatments. In 2011 the weed control by soil herbicides was slightly better, most likely because of the precipitation recorded 3 days after application (10.7 mm) and seven days beyond that (25.6 mm) (data not shown). The field trials at Rauischholzhausen confirm obtained results from pot experiments, which demonstrated that current soil-herbicides containing flufenacet (e.g. Cadou SC) are highly dependent on sufficient soil moisture and constant rainfall in order to achieve high level of black-grass control. Results from pot experiments showed further that flufenacet, applied in pre-emergence, does not seem to be able consistently control *A. myosuroides* plants emerging from a deeper soil depth, which partially explains the field results.

The moderate control achieved by soil herbicides in RH in all years could be explained by the enhanced herbicide metabolism of the black grass population. It is reported that populations with enhanced metabolism resistance also reduce the efficacy of different pre-emergence herbicides belonging to different chemical families to a certain degree (Moss and Hull 1999). In most cases enhanced metabolism of resistant weeds has been associated with enhanced cytochrome P450 mono-oxygenase (Hyde et al. 1996, Hall et al. 1997) or glutathione-S-transferase activity (Cummings et al. 1997, Reade et al. 2004).

It has been reported that resistant *A. myosuroides* and *L. rigidum* biotypes showed non target-site cross-resistance across several herbicide modes of action. In vivo studies on herbicide metabolism and P450 inhibitors in resistant biotypes showed that P450 enzymes catalyzed enhanced rates of metabolism of several herbicides of different modes of action (Hall et al 1994, Moss and Cussans 1985, Powles and Yu 2010). Petersen et al. (2012) found in their studies with different black-grass populations, that Non-target-Site-Resistances (NTSR) against mesosulfuron in plants can also result in the loss of efficacy of flufenacet. Additionally, Klingenhagen (2012) observed a lack of herbicidal efficacy by pre-emergence herbicides, including Cadou SC, Herold SC, Stomp aqua, and IPU in his field experiments against black-grass populations which were resistant to Atlantis WG.

The greenhouse experiment (chapter 4.3.1.2) confirmed these observations which were made in the field experiments. Obtained results showed that the *A. myosuroides* population from Rauischholzhausen showed less sensitivity against soil herbicides such as flufenacet and pendimethalin compared to a standard sensitive population, especially when herbicides were applied at lower dosages and/or later application timings when black-grass seedling were already emerged (BBCH 10 and 11).

In addition, Menne et al. (2012) concluded from their research that reduced sensitivity of black-grass to flufenacet in not necessarily due to resistance, but is more likely from the seasonal variation in soil moisture, amount of rainfall, temperature conditions and application timing which all influence the efficacy level of flufenacet in the field.

The poorer performance, especially of most soil acting herbicides in long-term non-ploughed soils in the field experiments conducted at RH, could also be due to the more adsorptive nature of the soil induced by a higher content of organic matter. Previous work has shown that a high content of humus (e.g. straw residues, mulch) is highly adsorptive to herbicides (Embling et al. 1983) and is capable of reducing herbicide performance (Nyffeler and Blair 1978, Moss 1985b). In contrast, it is probable that ploughing buries a lot of high adsorptive organic matter below 2.5 cm deep and provides more favourable conditions for soil-acting herbicides. Governmental institutions and public services recommend that for locations such as RH, with high infestations of *A. myosuroides* and early sowing dates, should use sequential herbicide applications. These usually include pre-emergence/early post-emergence herbicide applications based on flufenacet or pendimethalin in the autumn followed by a spring application with an ALS-inhibitor containing mesosulfuron+iodosulfuron, pyroxsulam or propoxycarbazone (Wolber 2011, Gehring et al. 2012). Results received from RH show that applications with soil acting herbicides (Cadou SC, Herold SC) in autumn followed by ALS-applications in the spring gave higher levels of weed control and higher yields than single applications of soil-acting herbicides and most ALS-herbicides in the fall. Nevertheless, sequence applications were not as successful as single post-emergence applications in the fall by Atlantis or Alister. This result can be explained by the non-efficient weed control of soil acting herbicides and the high (yield affecting) weed competition in autumn. Conclusively the most efficient way to control black-grass in early sown winter wheat can be achieved by a single herbicide treatment containing mesosulfuron. However, the soil-acting herbicides should still be considered for the avoidance and management of herbicide resistant weed populations as reported by Wolber (2011). As it is already recommended in Great Britain, it might be a successful approach to apply soil-acting herbicides containing flufenacet in pre-emergence, possibly followed in the autumn by a residual herbicide such as chlortoluron (mixed with diflufenican or pendimethalin) followed by mesosulfuron based products either in the same autumn or the next spring.

Conclusions

It is concluded that the presence of enhanced metabolic resistance to ACCase-inhibitors (EMR) in weed populations can confer to resistance to herbicides with different mode of actions which further complicates the weed management strategies.

In order to achieve maximal control by soil-acting herbicides containing flufenacet or pendimethalin, applications should be conducted at optimal conditions. From greenhouse experiments it can be concluded that applications should be conducted at pre-emergence and at moist soil conditions in order to maximize efficacy.

It can also be concluded that sites with higher densities of *A. myosuroides* require earlier treatments and usually result in greater yield reductions even after the application of soil active herbicides. Consequently, at such sites weed control should be focused in autumn rather than with spring treatments.

In order to achieve maximal levels of control against problematic sites with metabolic resistant black-grass, applications with ALS-herbicides containing mesosulfuron, pyroxsulam, flupyrsulfuron, should be conducted at early weed development stages, preferably in autumn.

The results clearly showed that early sowing favors grass weeds such as *A. myosuroides* resulting in higher weed biomass, earlier competition and thus higher potential yield losses. Further, non-chemical practices such as ploughing or late sowing are very effective supporting black-grass control by reducing the weed pressure before chemical treatments.

It was found that ploughing combined with an application of effective herbicides, such as single or sequence applications including ALS-inhibitors, can improve weed control and provide reliable solutions in early sown cereals independent from sites and even at such locations as RH with resistance problems. Herbicide programs containing ALS-inhibitors can still provide commercially acceptable solutions when ACCase-inhibitor resistance is widely present within a black-grass population, but not in a sustainable manner.

At the same time there is evidence that a later sowing time with herbicides applied in the spring can be very successful and lead to high yields in winter wheat

A farmer affected by high level of metabolic resistant black-grass should make use of ploughing and later sowing, in combination with crop rotation and a suitable herbicide program in order to give consistantly high levels of control.

6 Summary

The emergence of black-grass (*Alopecurus myosuroides*) was observed over three growing seasons (2009-2012) on agricultural fields at different locations within Hessen (Rauischholzhausen, Gießen and Wicker) and Germany. The emergence pattern generally varied within years, locations and cultivation systems. At the experimental station Rauischholzhausen, under low tillage conditions, *A. myosuroides* emerged steadily between September and May in all years. Peak emergence of about 75-90% occurred after sowing until the middle of October, followed by continuous emergence until winter by 9-24% and another peak of 1-12% in the spring. Observations confirm that a prolonged emergence with late flushes of black-grass usually occur before winter and/or in the spring. It was concluded that herbicide treatments (pre- and post-emergence) would have to be more robust, containing active ingredients with a long lasting residual component in order to cover the protracted period of black-grass emergence, especially in years with a high dormancy level.

Multiple pot experiments using silt loam were carried out either in greenhouses or outdoors in order to characterize the soil activity of herbicides. The most common post-emergence herbicides (ACCase- and ALS-inhibitors) for grass control in cereals were applied pre-emergence to sown black-grass, and herbicide activity was assessed 5-9 weeks after treatment by weighing the upper biomass of the weeds per pot. The results were variable for ACCase-inhibitors, which were highly dependent on environmental conditions (application timing, soil moisture, biological activity of the soil). However, ACCase-inhibitors, in particular pinoxaden and clodinafop, demonstrated very high soil activity (up to 97% control) when they were applied on grass weeds which were in the process of germination. The ALS-inhibitor herbicides propoxycarbazone, pyroxsulam and mesosulfuron consistently demonstrated very high soil activity against *Alopecurus myosuroides* in all pot experiments, even at very low doses. This was comparable to herbicides containing flufenacet and pendimethalin, which are used regularly for pre/early post emergence weed control. Also, the ALS-herbicide flupyrsulfuron demonstrated high soil activity in pot experiments. However, the results were inconsistent and seemed more dependent on environmental conditions.

Different pot experiments were conducted to evaluate the effect of soil moisture, sowing depth and irrigation method on the soil activity of post-emergence herbicides. Notably, for the herbicides flupyrsulfuron and flufenacet, soil activity decreased significantly at dry soil moisture conditions and increasing sowing depths for black-grass. Results received from pot experiments under different water regimes

concluded that intensive watering from above and a relatively high level of soil moisture after application increased the activity of soil-applied herbicides (ACCase-inhibitors: pinoxaden and clodinafop, ALS-inhibitors: mesosulfuron, pyroxsulam, propoxycarbazone and flupyrsulfuron) and flufenacet against *A. myosuroides*. A medium soil moisture level (30% WC) that was maintained through sub-irrigation, led to a large decrease in the soil activity of all herbicides. This suggests that herbicides applied to the soil surface need to be solubilized and moved downwards into the top few centimeters of the soil where the weed seeds germinate.
Field experiments were carried out at the experimental stations in Gießen (silt clay) and Wicker (silty clay loam) from 2010 to 2012 in order to confirm soil activity of post-emergence herbicides under field conditions. ACCase-inhibitors showed variable results and the achieved soil activity was generally lower than in pot experiments. Even with immediate rainfall after application and favorable conditions, the maximum activity assessed for pinoxaden and clodinafop was 56-67%. On the contrary, very high levels of soil activity were confirmed for ALS-inhibitors in most field experiments, which were on a similar level to the herbicides containing flufenacet and pendimethalin.

A lab study on the soil-degradation (DT50) of mesosulfuron, pyroxsulam and pinoxaden (incl. M2) in different soils at 10 °C was conducted using UPLC/TQ-MS. A relatively long persistence was demonstrated for all active ingredients: pyroxsulam (18-39 days), mesosulfuron (36-58 days) and M2 derived from pinoxaden (24-26 days). In order to understand the ability of post-emergence herbicides to provide residual soil activity, multiple pot and field experiments were conducted. Black-grass seeds were sown at different time intervals after herbicide application on the soil surface. It was concluded that ACCase-inhibitors do not provide any or very short residual soil activity against A. myosuroides. On the contrary, herbicides containing ALS-inhibitors, in particular mesosulfuron, propoxycarbazone and pyroxsulam may provide a relatively long residual activity against *A. myosuroides*. Their persistence generally depends on the environmental conditions. Pyroxsulam seems to degrade relatively quickly under moist soil conditions, whereas dry soil conditions lead to longer residual soil activity.

Field experiments were conducted from 2009 to 2011 at two locations to examine the efficacy of different herbicides for *A. myosuroides* control in winter wheat in combination with different cropping systems. Two different sowing dates for winter wheat (early and late) and two tillage systems (shallow chisel ploughing and deep ploughing) were chosen. The results showed that early sowing favors grass weeds such as *A. myosuroides*, resulting in higher weed biomass, earlier competition and thus higher potential yield losses of wheat. Further non-chemical practices such as

ploughing or late sowing are very effective supporting black-grass control by reducing the weed pressure before chemical treatments. It was concluded that the presence of enhanced metabolism resistance to ACCase-inhibitors in weed populations can usually confer to resistance to herbicides with different modes of action which further complicates the weed management strategies. The results clearly demonstrate that ploughing combined with an application of effective herbicides including ALS-inhibitors, can improve weed control and provide reliable solutions in early sown wheat independent from sites and even at locations with resistance problems with ACCase-inhibitors. It can be concluded that sites with higher densities of *A. myosuroides* require earlier treatments and usually result in greater yield reductions even after the application of soil active herbicides. Consequently, at such sites weed control should be applied in autumn rather than with spring treatments, which was supported by additional greenhouse experiments. At the same time there is evidence that late sowing time with applied herbicides in the spring can be very successful and lead to high yields in winter wheat. In conclusion, high infestations with grass weeds and multiple resistance mechanisms can be effectively managed with integrated weed management strategies, including effective herbicide applications combined with non-chemical practices such as delaying the sowing date or ploughing cultivation.

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Eidesstattliche Erklärung

Ich versichere, dass ich die vorliegende Dissertation selbstständig verfasst und keine anderen als die angegebenen Quellen und Hilfsmittel verwendet habe. Alle Ausführungen, die anderen Arbeiten wörtlich oder sinngemäß entnommen sind, sind kenntlich gemacht. Die Arbeit wurde in gleicher oder ähnlicher Form noch in keinem anderen Studiengang als Prüfungsleistung verwendet. Ich stimme zu, dass die vorliegende Arbeit mit einer Anti-Plagiatssoftware überprüft werden darf.

Gießen, 28.10.2014

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