Study Title

Drift deposition pattern of seed treatment particles abraded from Poncho® Beta Plus treated sugar beet pills and emitted by a typical mechanical sowing machine

Amendment No. 1 to Report NAX/SP02-2008

2009-03-20
[Date: yyyy-mm-dd]

Study Director

[redacted]

Author of the Amendment

[redacted]

Study Completion Date

2008-11-14
[Date: yyyy-mm-dd]

Reasons for the Amendment

1.
In the original report, when summarising aggregated values (percentiles, average), drift values have been reported conservatively as < LOQ (limit of quantification) even if the values was actually < LOD (limit of detection). In order to resolve these low levels of residue finding more accurately, a distinction is made in the amended version between <LOQ and <LOD.
Moreover, n the original report, drift values have been calculated on the basis of actually measured values, irrespective whether the actual analytical value was below the limit of quantification or below the limit of detection. However, for a conservative mathematical processing of the dataset, particularly in case of very low values, it is considered more appropriate to replace for mathematical calculations values below the limit of quantification by the limit of quantification itself and to replace the limit of detection by the limit of detection itself. This operation will lead to conservative percentiles and to conservative average values.

Consequence for the integrity of the study: None, as the changes are made in the mathematical processing and not in the analytical raw data.

Summary section of the report:

OLD:

	90th%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID No.	Z
Producer/ Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
3 m	<LOQ
5 m	<LOQ
10 m	<LOQ
20 m	<LOQ
30 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

NEW:

	Conservative[1] 90th%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
3 m	<LOD
5 m	<LOD
10 m	<LOD
20 m	<LOD
30 m	<LOD
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Amendment No. 1

OLD:

	90th%ile Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/ Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
5 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

NEW:

	Conservative[1] 90th%ile Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

OLD:

	90th%ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/ Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of quantification (LOQ) = 0.020 µg a.s./passive PP-mesh dust collector	

NEW:

	Conservative[1] 90th%ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] **For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector**

In the “Discussion and Conclusion” – section of the summary in the original report, it was wrongly stated

OLD:

”… Overall, clothianidin could be quantified in 1.5 % of all ground deposition samples (4 quantifiable values out of 270; “primary” and “secondary” drift combined), with a max. single value of 0.035 g a.s./ha. All 90th%ile values for ground deposition (“primary” and “secondary” drift, respectively) were below the limit of quantification (i.e. = LOQ = 0.014 g a.s./ha)…”

The new and corrected wording is:

NEW:

”… Overall, clothianidin could be quantified in 1.5 % of all ground deposition samples (4 quantifiable values out of ***300****; “primary” and “secondary” drift combined), with a max. single value of 0.035 g a.s./ha. All 90th%ile values for ground deposition (“primary” and “secondary” drift, respectively) were* ***at least*** *below the limit of quantification (i.e. = LOQ = 0.014 g a.s./ha) …”*

Consequence for the integrity of the study: None

Result section of the report:

OLD:

8.1 Ground Deposition

A detailed compilation of all ground deposition results ("primary drift") is presented in Table 9 below.

Table 1 90th%ile ground deposition ("primary drift")

	90th%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
3 m	<LOQ
5 m	<LOQ
10 m	<LOQ
20 m	<LOQ
30 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

Table 2 Average ground deposition ("primary drift")

	Average Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
3 m	<LOQ
5 m	<LOQ
10 m	<LOQ
20 m	<LOQ
30 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

NEW:

8.1 Ground Deposition

A detailed compilation of all ground deposition results ("primary drift") is presented in Table 9 below.

Table 1 90^{th}%ile ground deposition ("primary drift")

	Conservative[1] 90^{th}%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
3 m	<LOD
5 m	<LOD
10 m	<LOD
20 m	<LOD
30 m	<LOD
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Table 2 Average ground deposition ("primary drift")

	Conservative[1] Average Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
3 m	<LOQ
5 m	<LOQ
10 m	<LOD
20 m	<LOQ
30 m	<LOQ
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

OLD:

8.2 Aerial Dislocation of Ground Deposits

A detailed compilation of all ground deposition results ("secondary drift") is presented in Table 10 below.

Table 3 90th%ile aerial dislocation of ground deposits ("secondary drift")

	90th%ile Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
5 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

Table 4 Average aerial dislocation of ground deposits ("secondary drift")

	Average Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
5 m	<LOQ
50 m	<LOQ
Limit of quantification (LOQ) = 0.014 g a.s./ha	

NEW:

8.2 Aerial Dislocation of Ground Deposits

A detailed compilation of all ground deposition results ("secondary drift") is presented in Table 10 below.

Table 3 90th%ile aerial dislocation of ground deposits ("secondary drift")

	Conservative[1] 90th%ile Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Table 4 Average aerial dislocation of ground deposits ("secondary drift")

	Conservative[1] Average Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Amendment No. 1

OLD:

8.3 Atmospheric Drift

A detailed compilation of all in-flight dust measurements ("atmospheric drift") is presented in Table 11 below.

Table 5 90th%ile measurements of in-flight dust ("atmospheric drift")

	90th%ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of quantification (LOQ) = 0.020 µg a.s./passive PP-mesh dust collector	

Table 6 Average measurements of in-flight dust ("atmospheric drift")

	Average Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.056
2 m	0.092
3 m	0.069
4 m	0.039
5 m	0.049
	30 m distance from the "zero-line"
1 m	0.053
2 m	0.081
3 m	0.037
4 m	0.087
5 m	0.133
Limit of quantification (LOQ) = 0.020 µg a.s./passive PP-mesh dust collector	

NEW:

8.3 Atmospheric Drift

A detailed compilation of all in-flight dust measurements ("atmospheric drift") is presented in Table 11 below.

Table 5 90th %ile measurements of in-flight dust ("atmospheric drift")

	Conservative[1] 90th %ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector

Table 6 Average measurements of in-flight dust ("atmospheric drift")

	Conservative[1] Average Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	Z
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.056
2 m	0.093
3 m	0.069
4 m	0.041
5 m	0.052
	30 m distance from the "zero-line"
1 m	0.056
2 m	0.082
3 m	0.040
4 m	0.090
5 m	0.135
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector

CERTIFICATION OF AUTHENTICITY

Signature of the Study Director

[redacted]

Study Director 2009-03-20
[Date: yyyy-mm-dd]

Signature of Management

[redacted]

Manager Test Facility and Representative of the Sponsor 2009-03-20
[Date: yyyy-mm-dd]

Checked by QA-Unit

[redacted]

Quality Assurance Unit 2009-3-20
[Date: yyyy-mm-dd]

Inquiries should be directed to:

Bayer CropScience AG
Institute for Ecotoxicology
D-40789 Monheim, GERMANY
Phone No.: [redacted]

M-309580-02-1

Final Report

Drift deposition pattern of seed treatment particles abraded from Poncho® Beta Plus treated sugar beet pills and emitted by a typical mechanical sowing machine

Test Guideline

Special designed study following principally the BBA Drift Guideline Part VII, 2-1.1, 1992

Study Director

[redacted]

Authors

[redacted]

Test Facility

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GLP-Study Number

E 308 3545-8

Report No:

NAX/SP02-2008

Report Date:

2008-11-14
[yyyy-mm-dd]

DATA CONFIDENTIALITY STATEMENT

This page is intentionally left blank for the purpose of submitting administrative information that is required by regulations promulgated by various countries.

STATEMENT OF COMPLIANCE

[redacted]

CERTIFICATION OF AUTHENTICITY (see original report)

[redacted]

INQUIRIES

Inquiries should be directed to:

Bayer CropScience AG
Institute for Ecotoxicology
D-40789 Monheim, GERMANY
Phone No.: [redacted]

QUALITY ASSURANCE STATEMENT (see original report)

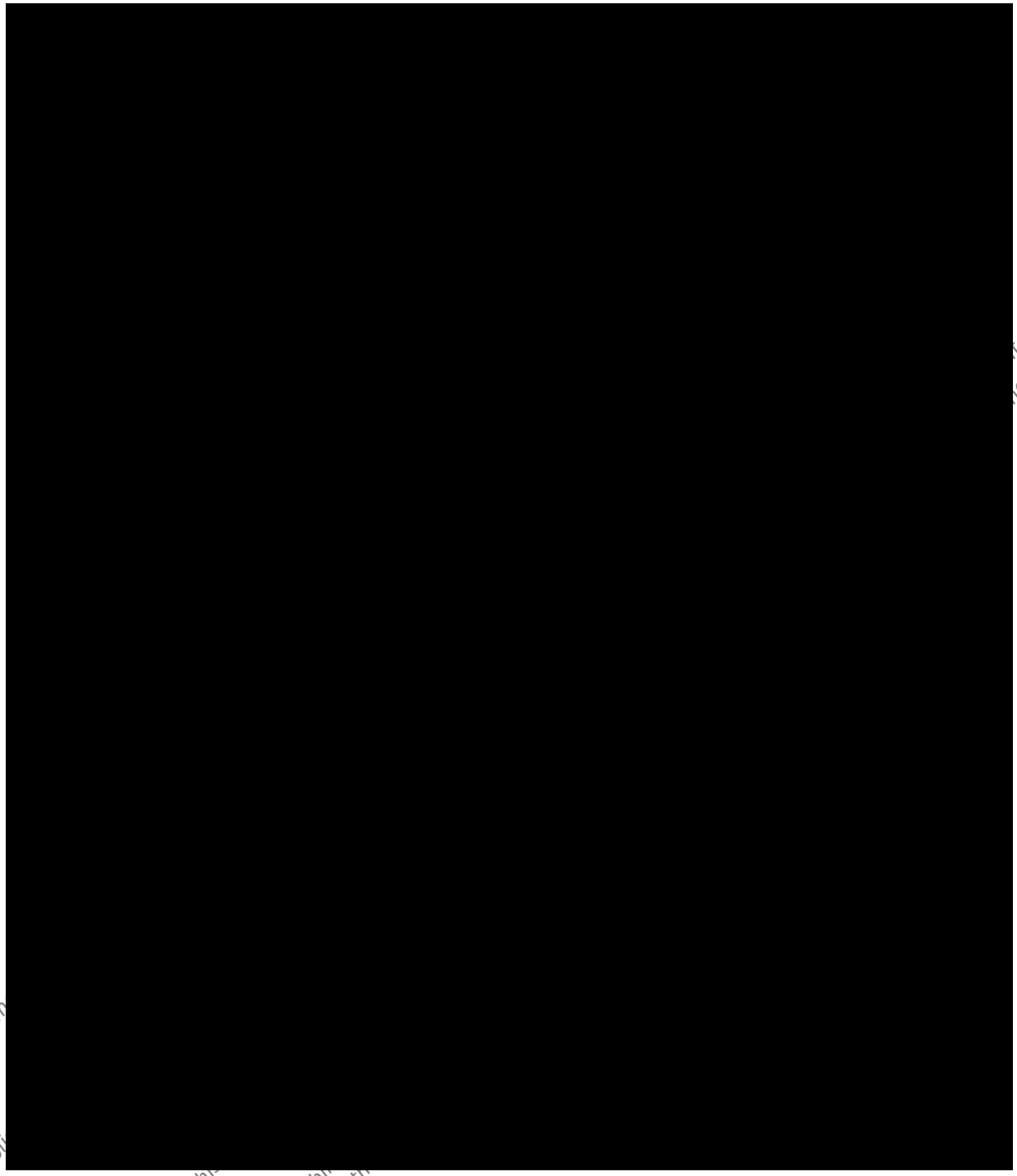


TABLE OF CONTENTS

1 SUMMARY

Report: [redacted] (2008): Drift deposition pattern of seed treatment particles abraded from Poncho® Beta Plus treated sugar beet pills and emitted by a typical mechanical sowing machine.
Bayer CropScience AG, unpublished Report No.: NAX/SP02-2008, Date: November 14, 2008

Guidelines: Special designed study, following principally the BBA Drift Guideline Part VII, 2-1.1, 1992

GLP: The field part was not performed under GLP, only the residue analysis part of the study was conducted under GLP

Experimental starting and completion date:
Start and end of field activities: August 20, 2008
Start and end of analytical activities: August 21, 2008 - August 26, 2008

Material and methods:
Test item: Commercially treated sugar beet pills, treated with Poncho® Beta Plus, which contains the neonicotinoid active substances clothianidin and imidacloprid (analysed neonicotinoid seed loading: 0.589 mg clothianidin a.s./pill, 0.325 mg imidacloprid a.s./pill). Test location: Leverkusen - Hitdorf, North Rhine-Westphalia, Germany. Seed density: 1.17 Units/ha, corresponding to 117,000 pills/ha, which is equivalent based on the actually measured pill loading to 68.9 g clothianidin and 38.0 g imidacloprid a.s./ha.

The aims of the study were to quantify (i) the ground deposition rate of clothianidin-containing seed treatment particles [g a.s./ha] at various distances from the field margin during the sowing operation of Poncho® Beta Plus treated sugar beet pills (nominally 0.60 mg clothianidin a.s./pill, 0.30 mg imidacloprid a.s./pill) with a typical mechanical sowing machine ("primary drift"); in addition (ii) measurements were made to determine the quantity of clothianidin-containing seed treatment particles [g a.s./ha] transported downwind post-sowing (over a period of 24 h; "secondary drift") and finally to determine (iii) the in-flight clothianidin-containing dust as measured by passive dust-drift collectors [µg a.s./collector] at various distances downwind from the drilling area in different elevations above the ground.

The actual machine tested was a Kverneland Accord Monopill SE, a 12-row mechanical precision sugar beet planter (12 hoppers).

Commercially treated sugar beet pills (Cultivar "Berenika") were produced and packed by KWS Saat AG, D-37555 Einbeck, Germany. The pills were treated with the insecticidal active substances clothianidin, imidacloprid and beta-cyfluthrin and with the fungicidal active substances thiram and hymexazol. The sugar beet pills were delivered in the originally sealed card-boxes, each containing one single Sugar Beet Unit (= 100,000 pills).

Before drilling, the hoppers of the Kverneland Accord Monopill SE were filled on the yard of the machine-hall of Bayer CropScience's Application Technology Unit, Building 5912, D-40789 Monheim, approximately 2 - 3 km away from the trial site (access to the trial site was *via* paved roads [≈50%] and field paths [≈50%]). Each hopper was filled with 1 originally sealed card-box (= 1 Sugar Beet Unit = 1 × 100,000 pills). Particular care was taken to quantitatively transfer the entire content of each card-box into the hoppers, including any contained dust and transport-related seed treatment abrasion. Thus, in total 12 card-boxes were used (= 12 × 100,000 pills = 1,200,000 pills, sufficient for ≈ 9 - 12 ha).

The size of each drilling plot was about 1.0 ha with an orientation of the sampling devices 180° ± 30° to the prevailing wind direction. An average wind speed of 2 - 5 m/s and a deviation of wind direction of maximum ± 30° to the perpendicular wind direction (*i.e.*, 180° to the sampling devices) were the target conditions during drilling.

All clothianidin-containing dust and abrasion particles which deposited at 1, 3, 5, 10, 20, 30 and 50 metres distance from the drilling area during sugar beet sowing ("primary drift") were sampled in polystyrene Petri-dishes (Ø 13.7 cm, 147.41 cm²), filled with an acetonitrile-water mixture (2/8, v/v). For each sampling distance, three arrays of 10 Petri-dishes each were installed with a distance of 1 metre between the dishes and 50 m between the arrays. Accordingly, a total of 30 samples were taken for each sampling distance. After drilling was completed, an additional waiting period of *ca.*15 minutes was left before the beginning of sampling to allow those dust particles which had not yet been deposited to settle on the sampling area. Sowing started directly adjacent to the sampling area. After the additional waiting period of 15 minutes elapsed, the content of each Petri-dish was quantitatively transferred into individually labelled polyethylene flasks by means of a polyethylene funnel and each polyethylene flask was tightly closed with its corresponding polypropylene screw cap. Sampling always started at the 50 metre distance and proceeded towards the drilling area in order to avoid any downwind cross-contamination.

Passive dust-drift collectors were installed at 1 m, 2 m, 3 m, 4 m and 5 m above the soil surface. The dust collectors were made of a polypropylene fabric mesh, built up of filaments with a 0.80 × 0.18 mm cross-section. This type of collector has a slightly oval shape with a length of ≈ 85 mm and a diameter of ≈ 65 mm; at its poles, the diameter is ≈ 50 mm. The polypropylene fabric mesh collectors were pinned on each end of horizontal metal rods, which in turn were mounted at the respective height on a vertical tripod-pylon (height ≈ 6 m), giving in total 10 collectors per pylon (2 at each height). In all arrays, a pylon was installed at 5 and 30 m distance from the drilling area, respectively, resulting in 6 collectors per height per distance. Once the 30 m line and later on, the 5 m line have been reached during the sampling process of the Petri-dishes ("primary drift", see above), also the passive collectors were sampled and placed in individually labelled plastic containers. In the laboratory, each individual passive collector was extracted with an appropriate volume of acetonitrile/water (2/8, v/v) in an ultrasonic bath to enhance the extraction process.

After the Petri-dishes filled with acetonitrile/water (2/8, v/v) and the passive dust-drift collectors had been sampled ("primary drift" and "atmospheric drift", see above), ten new polystyrene Petri-dishes (Ø 13.7 cm, height 1.7 cm, 147.41 cm² surface) were installed on the field ground of the sampling area in all arrays at three distances from the "zero line" (1, 5 and 50 m), respectively. The Petri-dishes were filled with about 100 mL of an glycerol/(Millipore-)water mixture (1/1, v/v) and were exposed for a period of 24 h in order to quantify the amount of clothianidin which may be dislodged from the sowing area *via* secondary processes ("secondary drift"). After 24 h, the content of each Petri-dish was quantitatively transferred into individually labelled polyethylene flasks by means of a polyethylene funnel and each polyethylene flask was tightly closed with its corresponding polypropylene screw cap.

At the day of drilling/sampling (August 20, 2008), all polyethylene flasks filled with acetonitrile/water ("primary drift") together with all passive polypropylene-mesh dust-drift collectors ("atmospheric drift") were transported by car to Bayer CropScience AG's Institute for Residues, Operator and Consumer Safety (BCS-D-ROCS, D-40789 Monheim, Germany). The following day, the polyethylene flasks filled with glycerol/water ("secondary drift") were also transported by car to BCS-D-ROCS. The samples were stored at ambient temperature in the laboratory until analysis. All samples were analysed for their clothianidin content according to method 00554/M001.

Findings:

	Conservative[1] 90th%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
3 m	<LOD
5 m	<LOD
10 m	<LOD
20 m	<LOD
30 m	<LOD
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] **For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha**

Overall, in one out of 210 measurements concerning "primary drift" (7 distances × 30 Petri-dishes per distance), the LOQ of 0.014 g clothianidin a.s./ha was exceeded and in five further cases, the residues were between the LOQ (i.e. 0.014 g clothianidin a.s./ha) and the LOD (i.e. 0.004 g clothianidin a.s./ha).

	Conservative[1] 90th%ile Aerial Dislocation of Ground Deposits ("Secondary Drift") [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Overall, twenty-four hours post-sowing, clothianidin residues were quantifiable in three out of 90 samples (3 distances × 30 Petri-dishes per distance); seven further samples revealed residues between the LOQ (i.e. 0.014 g clothianidin a.s./ha) and the LOD (i.e. 0.004 g clothianidin a.s./ha).

	Conservative[1] 90th%ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector

Overall, in ≈75% of all samples, quantifiable clothianidin residues were found; in ≈25% of all samples, clothianidin residues were between the LOQ (i.e. 0.020 g clothianidin a.s./sampler) and the LOD (i.e. 0.006 g clothianidin a.s./sampler).

The grand average wind-speed (mean of means of all drilling paths) during the drilling operation was 6 m/s and therefore in good agreement with the target conditions (*i.e.*, 2 - 5 m/s). The average deviation of the actual wind direction from the perpendicular direction relative to the sampling devices was at all drilling paths strictly in-line to the target conditions (*i.e.*, deviation < ± 30°).

Discussion and Conclusion:

Overall, clothianidin could be quantified in 1.5 % of all ground deposition samples (4 quantifiable values out of 300; "primary" and "secondary" drift combined), with a max. single value of 0.035 g a.s./ha. All 90^{th}%ile values for ground deposition ("primary" and "secondary" drift, respectively) were at least below the limit of quantification (i.e. = LOQ = 0.014 g a.s./ha).

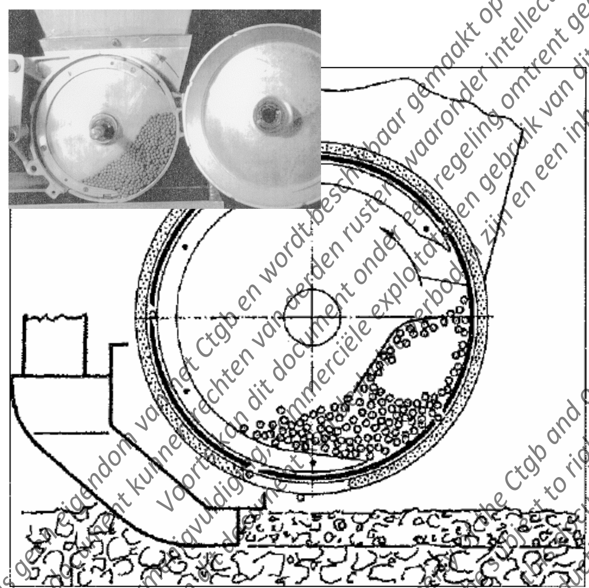
Considering atmospheric drift, clothianidin was measured in 75% of the passive polypropylene-mesh-collectors which were set up in different heights at 5 and 30 m distance from the sowing area. However, in contrast to ground deposition data, which are direct, area-related exposure figures [g a.s./ha], the airborne residues determined in passive samplers of an unknown collection efficiency only allow for a derivation of qualitative conclusions.

The consistent overall lack of quantifiable deposition within the off-field area suggests that airborne particles, trapped by passive polypropylene-mesh-collectors in the same area, are mainly subject to further dispersion and dilution.

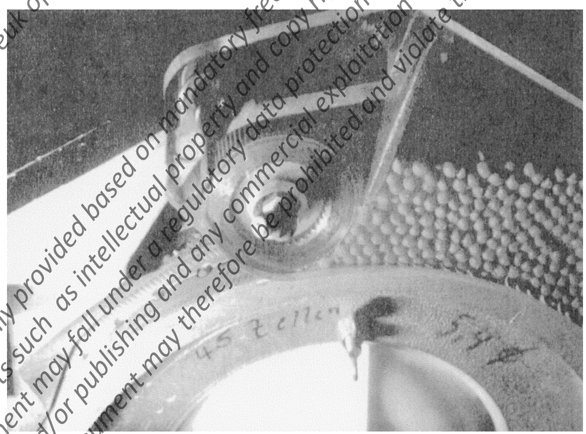
2 INTRODUCTION

Mechanical sugar beet drilling does not operate with any air assistance. In order to accomplish a single-kernel pill deposition (“individualisation of pills”), rotating disks or drums with pre-defined perforations (“cells”) transport single pills from the storage (internal or external, see below) to the pill outlet, where individual pills drop via gravitation only into the seed furrow. Thus, potentially abraded particles which may contain active substance(s) are released by gravitation only directly above the ground to be immediately incorporated, as directly after deposition of the pill (and the potentially abraded dust) the furrow is properly closed. The rotation of the individual disk/drums per sowing element (generally, one independently operation sowing element per row) is correlated with the driving speed of the tractor to assure a homogenous intra-row spacing.

Mechanical, non air-assisted sugar beet pill separation principle (to achieve a homogenous intra-row spacing of individual sugar beet pills)



Principle of mechanical sugar beet drilling (“internal-filling”)



Principle of mechanical sugar beet drilling (“external-filling”)

The purpose of the study was to quantify the deposited clothianidin rate (g a.s./ha) at various distances downwind from the drilled area during and after mechanical drilling of sugar beet pills, treated with Poncho® Beta Plus. In addition, also the in-flight clothianidin content was measured via passive dust drift collectors, placed at various distances from the drilling area in different elevations above the ground.

3 RESPONSIBILITIES

Sponsor:	Bayer CropScience AG Portfolio Management – Seed Treatment Alfred-Nobel-Str. 50 D-40789 Monheim, Germany
Test Facility:	Bayer CropScience AG Development-Ecotoxicology (BCS-D-ETX), Building 6620 Alfred-Nobel-Str. 50, D-40789 Monheim, Germany
Test Site Analytical Part	Bayer CropScience AG Development-Residues, Operator and Consumer Safety (BCS-D-ROCS) Building 6610 Alfred-Nobel-Str. 50, D-40789 Monheim, Germany
Study Director:	[redacted]
Responsible persons Field Part & Drilling equipment (Non - GLP):	[redacted]
Principal investigator Residue Analysis (GLP):	[redacted]
Responsible person Seed Material and Seed Treatment Quality Testing (Non - GLP):	[redacted]

4 MATERIAL AND METHOD NON-GLP FIELD PART

4.1 Test Item

Trade name:	Poncho® Beta Plus
Active substances (a.s.):	**1.** Clothianidin **2.** Imidacloprid **3.** Beta-Cyfluthrin
Chemical code:	**1.** TI-435 **2.** NTN 33893 **3.** FCR 4545
Formulation:	Poncho® Beta FS 453.34 (400 g TI-435/L + 53.34 g FCR 4545/L) & Gaucho® WS 70 (700 g/kg NTN 33893)
Empirical formulas:	**1.** $C_6H_8ClN_5O_2S$ **2.** $C_9H_{10}ClN_5O_2$ **3.** $C_{22}H_{18}Cl_2FNO_3$
CAS-No.'s.:	**1.** 210880-92-5 **2.** 138261-41-3 **3.** 68359-37-5
CAS-Names:	**1.** [*C*(*E*)]-*N*-[(2-chloro-5-thiazolyl)methyl]-*N*'-methyl-*N*'-nitroguanidine **2.** (2*E*)-1-[(6-chloro-3-pyridinyl)methyl]-*N*-nitro-2-imidazolidinimine **3.** Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloro-ethenyl)-2,2-dimethylcyclopropanecarboxylate
Indication:	Insecticides

4.2 Treated Sugar Beet Pills

Origin of commercial seeds:	KWS Saat AG, D-37555 Einbeck, Germany
Species:	*Beta vulgaris*
Cultivar:	Berenika
Registration-number:	16-8023A
Thousand Pill Weight (TPW):	29.7 g
Date of packaging:	19/08/2008
Appearance:	Bright orange sugar beet pills
Seed dressing constituents:	Insecticides: Clothianidin, imidacloprid, beta-cyfluthrin Fungicides: Thiram, hymexazol
Target content of clothianidin per individual beet pill (per Unit):	0.600 mg a.s./sugar beet pill (60 g a.s./Unit)
Target content of imidacloprid per individual beet pill (per Unit):	0.300 mg a.s./sugar beet pill (30 g a.s./Unit)
Analytically verified content of clothianidin per individual beet pill (per Unit):	0.589 mg a.s./sugar beet pill 58.94 g a.s./Unit
Analytically verified content of imidacloprid per individual beet pill (per Unit):	0.325 mg a.s./sugar beet pill 32.50 g a.s./Unit
Degree of loading clothianidin (analysed/target):	98.2 %
Degree of loading imidacloprid (analysed/target):	108.3 %

4.3 Sowing Machine Parameters, Drilling Parameters During Dust Drift Measurement and Soil/Field Identification

Machine ID-No.	**Z**
Name	Kverneland Accord Monopill SE
Seed separation principle	Mechanical (sub-type: “external-filling”), no air assistance
Deflection system	No particular deflection system (un-modified)
Number of drilling rows	12
Completed paths	9
Row-distance	0.45 m
Total No. of rows drilled during drift measurement	12 × 9 = 108
Working width	5.40 m
Width of drilled area (length of drilled area)	108 × 0.45 m = 9 × 5.40 m = 48.6 m (210 m)
Total plot size area drilled	1.02 ha (210 m length × 48.6 m width = 10,200 m^2)
Intra-row pill spacing	19 cm
Drilling depth	2.5 cm
Time to drill total area	27 minutes
Filling of each individual hopper with	1 complete card-box containing dressed sugar beet pills =100,000 pills per individual hopper **(after all pills per individual card-box were poured into each hopper, respectively, the empty card-box was shaken and tapped with its opening directed to the inside of the hopper to assure that all dust particles inside the card-box were quantitatively transferred into the hoppers)**
Total number of seeds drilled during dust drift measurement	116,959 pills/ha
Amount of clothianidin a.s. drilled on the total plot size	68.9 g a.s./ha
Amount of imidacloprid a.s. drilled on the total plot size	38.0 g a.s./ha
Soil codes of the study plot used for sugar pill drilling	OE 3521 [1] / OE 3522 [2]
Tested on field No.	6

[1] **This soil codes refer to a composite soil sample of the upper 10 cm of the study plot (soil texture, organic carbon, inorganic carbon, max. water holding capacity, CEC)**

[2] **This soil codes refer to a composite sample of the upper 5 cm of the study plot (water content of the soil immediately before drilling)**

Machine Z = Kverneland Accord Monopill SE

4.4 Test Organism

None, the study was focused on the determination of residue levels and not on direct effects on test organisms.

4.5 Schedule

Start and end of field activities:	August 20, 2008
Start and end of analytical activities:	August 21, 2008 - August 26, 2008

4.6 Test Location

The study was conducted in Leverkusen - Hitdorf, North Rhine-Westphalia, Germany on a study plot typically used for sugar beet growing (i.e Field No. 6 in the overview picture in Appendix 2, total field size: 5.6 ha, Field-ID: "Herbizidfeld VIII", Owner: Bayer Real Estate GmbH, Laacher Hof, Schleider Weg 52, D-40789 Monheim, Germany; further details are provided in Appendix 2). The previous crop on the study site was winter wheat. The entire area is characterized by sandy silt soils (for details of the soil characterisation see chapter 4.7).

In preparation of the sugar beet drilling, the experimental plot received a flat stubble processing, followed by ploughing. The final and sufficiently fine seed bed which allows sugar beet drilling according to typical European use conditions was prepared by additionally harrowing the field. On the downwind site adjacent to the drilling plot, the soil received a reduced soil processing (stubble processing and flat harrowing) and served as sampling area on which the Petri-dishes and the tripod-pylons for measuring atmospheric drift were placed. The Petri-dishes were placed in metal-placeholders (for details see chapter 4.8).

Drilling was performed at a time where the prevalent wind was coming from the perpendicular direction (i.e. 180° ± 30° relative to the sampling devices; see Appendix 3 for details). Each sampling and drilling plot was exactly calibrated by means of metering bands (100 & 50 m in length) and by marking relevant points by means of plastic sticks of approximately 1 m length, which were placed vertically upright into the soil.

4.7 Soil Characterization of the Study Plots and Clothianidin Soil Residue Analysis

Immediately before starting the sugar beet sowing operation, a composite soil sample from the upper 5 cm was taken (by means of a soil piercer) from at least 20 locations randomly selected on the drilling area and filled in a plastic bag. This composite sample was tightly sealed and analysed for its water content (Laboratory for Soil Organisms, Thorsten Leicher, BCS-D-ETX, non-GLP).

In addition, a further composite soil sample from the upper 10 cm was taken (by means of a soil piercer) from at least 20 locations randomly selected on the drilling area and filled in a plastic bag. This composite soil sample was sieved in the Bayer CropScience Laboratory for Soil Organisms (BCS-D-ETX) to < 2 mm. From this sieved fraction, approximately 250 g were isolated to be analyzed for the content of clothianidin; the soil residue analysis has been conducted in the laboratory of [redacted] (BCS-D-ROCS, according to the method 00540/M001, non-GLP). An appropriate fraction of the sieved soil had been further sent to the LUFA in Speyer (Landwirtschaftliche Untersuchungs- und Forschungs - Anstalt, Obere Langgasse 40, D-67346 Speyer, Germany) for the determination of the particle size distribution of the soil (soil texture), the content of organic and inorganic carbon, the cation exchange capacity (CEC) as well the max. water holding capacity (non-GLP analysis).

The analyses of the topsoil immediately before drilling revealed a water content in the upper 5 cm of ≈8%. This result verifies the dusty physical appearance of the soil surface during the sowing operation. Details of the water content analysis of the topsoil are provided in Table 8 as well as in Appendix 7.

The soil characterisation revealed a heavy loamy sand soil. Details of the soil characterization are provided in Table 8 as well as in Appendix 5.

The soil residue analysis, conducted according to the analytical method 00540/M001, revealed no clothianidin residues above the LOQ or LOD (LOD = limit of detection = 2 µg clothianidin/kg soil; LOQ = limit of quantification = 5 µg clothianidin/kg soil). Details of the soil residue analyses are provided in Table 8 as well as in Appendix 6.

4.8 Study Layout

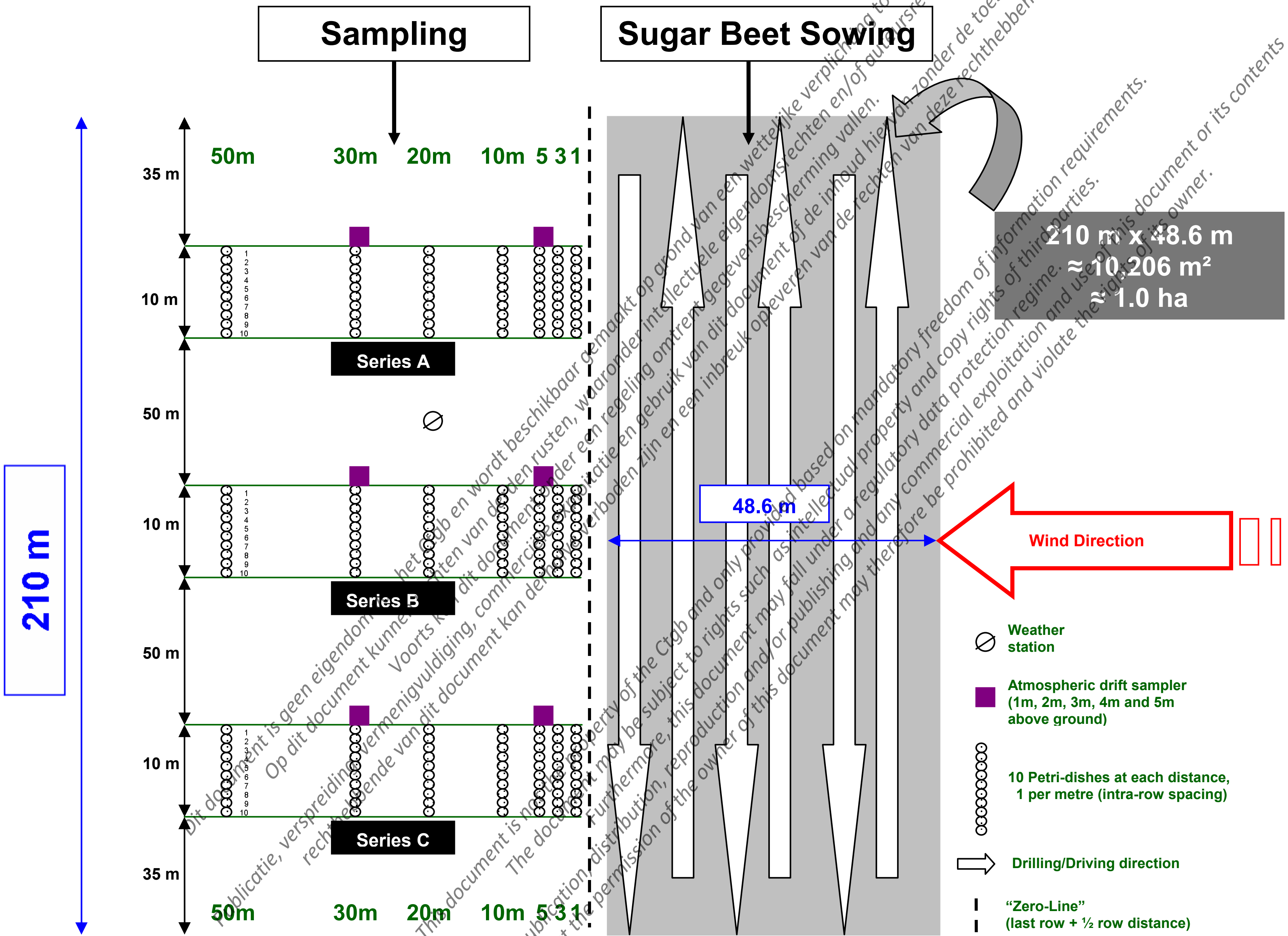
One typical mechanical single-kernel sugar beet pill sowing machine was tested, i.e. a Kverneland Accord Monopill SE, 12-row precision planter (12 hoppers), ID-code: “Machine Z”:

Commercially treated sugar beet pills (Cultivar ”Berenika”) were produced and packed by KWS Saat AG, Grimsehlstr. 31, P.O. Box 1463, D-37555 Einbeck, Germany. The sugar beet pills, treated with the insecticides clothianidin, imidacloprid and beta-cyfluthrin together with the fungicides thiram and hymexazol, were delivered in originally sealed card-boxes, each containing one Sugar Beet Unit (=100,000 seeds); the Thousand Pill Weight of the sugar beet pills was determined to be 29.7 g.

Before drilling, each hopper of the Kverneland Accord Monopill SE was filled on the yard of the machine-hall of Bayer CropScience’s Application Technology Unit, Building 5912, D-40789 Monheim, approximately 2 - 3 km away from the trial site (access of the trial site via paved roads [≈50%] and field paths [≈50%]). Each hopper was filled with 1 originally sealed card-box (= 1 Sugar Beet Unit = 1 × 100,000 pills). Particular care was taken to quantitatively transfer the entire content of each card-box into the hoppers, including any contained dust and transport-related seed treatment abrasion. Thus, in total 12 card-boxes were used (= 12 ×100,000 pills = 1,200,000 pills, sufficient for ≈ 9 - 12 ha).

The size of each drilling plot per machine was about 1.0 ha (48.6 m width, 210 m length = 10,200 m^2). At the time of drilling, the orientation of the drilling area was 180° (=perpendicular) ± 30° to the prevailing wind direction. Sowing was performed as typical for commercial agricultural practice (alternate directions).

All clothianidin-containing dust and abrasion particles which deposited at 1, 3, 5, 10, 20, 30 and 50 metres distance from the drilling area were sampled in polystyrene Petri-dishes (Ø 13.7 cm, 147.41 cm^2), filled with an acetonitrile-water mixture (2/8, v/v). For each sampling distance 3 arrays of 10 Petri-dishes each were installed with a distance of 1 metre between the dishes. Accordingly, a total of 30 samples were taken for each sampling distance. After the drilling was completed (i.e. after approximately 30 minutes for the 12-row planting machine), an additional waiting period of 15 minutes was employed before the start of sampling, in order to allow those dust particles which had not yet been deposited to settle on the sampling area.



4.9 Weather Data

Meteorological data such as relative humidity, temperature, wind direction and wind speed were measured on-site with a portable electronic weather station including wind gauge, which was placed inside the sampling area during drilling (at 20 m distance from the “zero line”). An average wind speed of 2 - 5 m/s and a derivation of wind direction of maximum ± 30° to the perpendicular wind direction (i.e. 180° to the sampling devices) were the target conditions during drilling. In addition to the on-site meteorological measurements, general climate recordings were obtained from Bayer CropScience AG’s meteorological station, positioned on the premises of the Bayer CropScience headquarter in D-40789 Monheim, Alfred-Nobel-Str. 50 (41 m above sea level), in close vicinity to the study site (≈ 2 - 3 km distance). The general climatic conditions during August 2008 are displayed in Table 7 (see below); the aggregated on-site climatic measurements per individual drilling path are displayed in Appendix 3.

4.10 Dust Sampling - Ground Deposition (“primary drift”)

Before drilling, ten polystyrene Petri-dishes (Ø 13.7 cm, height 1.7 cm, 147.41 cm^2 surface) per sampling row (1-10) and series (A, B, C) at seven different distances to the “zero line” were placed in metal placeholders on the field ground of the sampling area. In total, there was a sample surface of 147.41 cm^2 per individual Petri-dish and of $30 \times 0.014741\ m^2 = 0.442\ m^2$ per sampling distance. The Petri-dishes were filled with about 80 mL of an acetonitrile/water mixture (2/8, v/v) before drilling. This mixture reflects the extraction solution used in plant and animal metabolism studies. Sowing always started at the “zero line”. After the additional waiting period of 15 minutes elapsed, the content of each Petri-dish was quantitatively transferred into 250 mL polyethylene flasks by means of a polyethylene funnel. To take up quantitatively all possible clothianidin deposits inside the Petri-dish, each Petri-dish and its corresponding funnel were additionally rinsed with fresh acetonitrile/water (2/8, v/v) mixture (≈ 20 mL) and the rinse was combined with the content of the respective Petri-dish within the corresponding 250 mL polyethylene flask. After rinsing, each polyethylene flask was tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 metre distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with the ID-code of the sugar beet drilling machine (“Machine Z”), the solvent mixture actually used (acetonitrile/water), the No. of the respective series (A, B or C), the distance from the “zero line” (1, 3, 5, 10, 20, 30 or 50 m) and the No. of the respective Petri-dish per distance (1-10), giving in total 210 polyethylene flasks.

4.11 In-flight Dust Sampling ("atmospheric drift")

Passive dust-drift collectors have been installed at 1 m, 2 m, 3 m, 4 m and 5 m above the soil surface. The dust collectors were made of a polypropylene fabric mesh, build up of filaments with a 0.80 × 0.18 mm cross-section. This type of collector has a slightly oval shape with a length of ≈ 85mm and a diameter of ≈ 65mm; at its poles, the diameter is ≈ 50 mm (see Appendix 4 for pictures). The polypropylene fabric mesh collectors were pinned on each end of horizontal metal rods, which in turn were mounted at the respective height on a vertical tripod-pylon (height ≈ 6 m), giving in total 10 collectors per pylon (2 at each height). In all series (A, B and C), one pylon was installed at 5 and 30 m distance from the "zero line", respectively, resulting in 6 collectors per height per distance. Once the 30 m - line and later on, the 5 m - line had been reached during the sampling process of the Petri-dishes ("primary drift"; see above), the passive collectors were sampled and placed into polypropylene containers which were immediately sealed and labelled in the field. Each plastic container was unequivocally labelled with the ID-code of the sugar beet drilling machine ("Machine Z"), the number of the respective series (A, B or C), the distance from the "zero line" (5 or 30 m), the height above ground (1, 2, 3, 4 or 5 m) and the number per height (1 or 2), giving in total 60 polypropylene-mesh collectors. In the laboratory, each individual passive collector has been extracted with an appropriate volume of acetonitrile/water (2/8, v/v) within an ultrasonic bath to enhance the extraction process.

4.12 Aerial Dislocation of Ground Deposits ("secondary drift")

After the Petri-dishes filled with acetonitrile/water (2/8, v/v) and the passive dust-drift collectors have been sampled (see above), ten new polystyrene Petri-dishes (Ø 13.7 cm, height 1.7 cm, 147.41 cm² surface) were installed on the field ground of the sampling area in all series (A, B, C) at three distances from the "zero line" (1, 5 and 50 m), respectively. Again, the total sampling surface was 30 × 0.014741 m² = 0.442 m² per sampling distance (see also 4.10, above). The Petri-dishes were filled with about 100 mL of an glycerol/(Millipore-)water mixture (1/1, v/v) and were exposed for a period of 24 h after the last Petri-dish has been filled with glycerol/water, in order to quantify the amount of clothianidin which may be dislodged from the ground via secondary drift processes. After 24 h, the content of each Petri-dish was quantitatively transferred into 250 mL polyethylene flasks by means of a polyethylene funnel. To take up quantitatively all possible clothianidin deposits inside the Petri-dish, each Petri-dish and its corresponding funnel were additionally rinsed with Millipore Water (≈ 20 mL) and the rinse was combined with the content of the respective Petri-dish within the corresponding 250 mL polyethylene flask. After rinsing, each polyethylene flask was tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 metre distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with the ID-code of the sugar beet drilling machine ("Machine Z"), the solvent mixture actually used (glycerol/water), the number of the respective series (A, B or C), the distance from the "zero line" (1, 5, or 50 m) and the number of the respective Petri-dish per distance (1-10), giving in total 90 polyethylene flasks.

At the day of drilling (August 20, 2008), all 210 polypropylene flasks filled with acetonitrile/water (2/8, v/v) and all 60 passive polypropylene dust-drift collectors were transported by car to the laboratory of [redacted] (BCS-D-ROCS, Monheim). The following day (August 21, 2008), also the 90 polypropylene flasks filled with glycerol/water were transported by car to the laboratory of [redacted] Bayer CropScience AG Institute for Residues, Operator and Consumer Safety (BCS-D-ROCS). Sampling processing started at August 21, 2008. All samples were analysed for their clothianidin content according to method 00554/M001. A detailed analytical phase report of the residue analysis part (MR-08/163) is attached to this study report.

5 GLP ANALYSES PART

The GLP residue-analysis part is outlined and reported in the analytical phase report MR-08/163, which is attached to this final study report (see Appendix 9).

6 ENDPOINTS OF THE STUDY

Determination of the clothianidin deposition rate in g a.s./ha, which deposits at various distances from the treated area during drilling ("primary drift") of Poncho® Beta Plus treated sugar beet pills with a typical mechanical sugar beet drilling machine. In addition, also the in-flight clothianidin content ("atmospheric drift") is determined in µg a.s./passive dust collector as well as the clothianidin deposition rate in g a.s./ha, which deposits at various distances from the treated area via secondary drift processes within 24 hours after sowing (aerial dislocation of ground deposits, "secondary drift").

7 FILING

All raw data pertaining to this study and the original final report are stored in the central GLP archive of Bayer CropScience AG, Alfred-Nobel-Str. 50, D-40789 Monheim for as long as required by GLP principles.
Reserve samples of the reference items are stored in the archives of Bayer CropScience GmbH, Product Technology-Analytics Frankfurt, Industriepark Hoechst (D-65926 Frankfurt). The test and reference items are stored as long as their quality still guarantees an evaluation.

8 RESULTS

8.1 Ground Deposition

A detailed compilation of all ground deposition results ("primary drift") is presented in Table 9 below.

Table 1 90th%ile ground deposition ("primary drift")

	Conservative[1] 90th%ile Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
3 m	<LOD
5 m	<LOD
10 m	<LOD
20 m	<LOD
30 m	<LOD
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Table 2 Average ground deposition ("primary drift")

	Conservative[1] Average Ground Deposition ("Primary Drift") [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOQ
3 m	<LOQ
5 m	<LOQ
10 m	<LOD
20 m	<LOQ
30 m	<LOQ
50 m	<LOD
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, "<LOD" was replaced by 0.004 g a.s./ha and "<LOQ" was replaced by 0.014 g a.s./ha

Overall, in one out of 210 measurements concerning "primary drift" (7 distances × 30 Petri-dishes per distance), the LOQ of 0.014 g clothianidin a.s./ha has been exceeded and in five further cases, the residues were between the LOQ (i.e. 0.014 g clothianidin a.s./ha) and the LOD (i.e. 0.004 g clothianidin a.s./ha).

8.2 Aerial Dislocation of Ground Deposits

A detailed compilation of all ground deposition results (“secondary drift”) is presented in Table 10 below.

Table 3 90[th]%ile aerial dislocation of ground deposits (“secondary drift”)

	Conservative[1] 90[th]%ile Aerial Dislocation of Ground Deposits (“Secondary Drift”) [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, “<LOD” was replaced by 0.004 g a.s./ha and “<LOQ” was replaced by 0.014 g a.s./ha

Table 4 Average aerial dislocation of ground deposits (“secondary drift”)

	Conservative[1] Average Aerial Dislocation of Ground Deposits (“Secondary Drift”) [g a.s./ha]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Distance from 0-line	
1 m	<LOD
5 m	<LOQ
50 m	<LOQ
Limit of detection (LOD) = 0.004 g a.s./ha; limit of quantification (LOQ) = 0.014 g a.s./ha	

[1] For the calculation, “<LOD” was replaced by 0.004 g a.s./ha and “<LOQ” was replaced by 0.014 g a.s./ha

Overall, twenty-four hours post-sowing, clothianidin residues were quantifiable in three out of 90 samples (3 distances × 30 Petri-dishes per distance); seven further samples revealed residues between the LOQ (i.e. 0.014 g clothianidin a.s./ha) and the LOD (i.e. 0.004 g clothianidin a.s./ha).

8.3 Atmospheric Drift

A detailed compilation of all in-flight dust measurements ("atmospheric drift") is presented in Table 11 below.

Table 5 90th%ile measurements of in-flight dust ("atmospheric drift")

	Conservative[1] 90th%ile Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.072
2 m	0.188
3 m	0.131
4 m	0.074
5 m	0.103
	30 m distance from the "zero-line"
1 m	0.128
2 m	0.181
3 m	0.064
4 m	0.224
5 m	0.290
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector

Table 6 Average measurements of in-flight dust ("atmospheric drift")

	Conservative[1] Average Atmospheric Drift (Polypropylene-Mesh-Collectors) [µg a.s./collector]
Machine ID-No.	**Z**
Producer/Type	Kverneland Accord Monopill SE
Seed separation principle	Mechanical, no air assistance
Height above ground	***5 m distance from the "zero-line"***
1 m	0.056
2 m	0.093
3 m	0.069
4 m	0.041
5 m	0.052
	30 m distance from the "zero-line"
1 m	0.056
2 m	0.082
3 m	0.040
4 m	0.090
5 m	0.135
Limit of detection (LOD) = 0.006 µg a.s./collector; Limit of quantification (LOQ) = 0.020 µg a.s./collector	

[1] For the calculation, "<LOD" was replaced by 0.006 µg a.s./collector and "<LOQ" was replaced by 0.020 µg a.s./collector

Table 7 Climate Data

Climate data for the study area were obtained from Bayer CropScience AG's meteorological station located on the premises of the Bayer CropScience-headquarter (Alfred-Nobel-Str. 50, D-40789 Monheim am Rhein, Germany), in close vicinity to the study sites (≈ 2 - 3 km distance).

Day	Air Temperature (2 m above ground) [°C]			Rain [mm]	Rel. Humidity [%]	Air Pressure [hPa]	Wind Speed [m/s]	
[dd/mm/yyyy]	mean	min.	max.	total	min.	mean	mean	max.
01/08/2008	23.5	16.4	28.6	0.0	60	1009.7	9.6	2.6
02/08/2008	19.8	15.5	25.2	1.3	65	1011.4	8.4	2.2
03/08/2008	20.4	15.3	25.2	4.6	70	1008.2	9.8	3.1
04/08/2008	18.7	15.5	23.5	13.6	70	1004.6	13.3	3.7
05/08/2008	18.4	13.9	25.4	0.0	67	1011.2	10.0	2.1
06/08/2008	24.2	15.7	34.7	0.0	56	1008.9	5.7	1.4
07/08/2008	21.9	16.0	30.0	1.5	69	1001.8	16.1	2.4
08/08/2008	17.6	14.7	21.3	1.5	80	1002.7	11.4	2.8
09/08/2008	18.4	12.1	25.7	0.0	63	1009.7	9.3	2.2
10/08/2008	18.6	16.4	22.0	4.0	74	1003.4	9.4	2.4
11/08/2008	19.2	14.1	24.9	0.0	63	1000.9	6.4	2.0
12/08/2008	19.2	15.1	25.7	2.1	71	992.6	12.0	2.9
13/08/2008	17.6	13.8	22.6	1.4	63	1001.9	19.3	3.9
14/08/2008	17.2	11.9	23.5	0.0	61	1009.6	7.7	2.1
15/08/2008	17.2	9.2	24.3	0.1	63	1009.4	6.5	1.5
16/08/2008	18.5	9.2	27.5	0.0	54	1007.8	4.8	1.1
17/08/2008	19.3	12.7	26.2	0.0	54	1004.8	7.5	2.0
18/08/2008	19.6	14.5	24.8	0.1	66	1004.9	10.5	2.3
19/08/2008	19.7	16.2	24.1	0.0	65	1004.0	10.4	3.1
20/08/2008	17.2	13.5	20.4	7.8	71	1008.2	10.9	3.5
21/08/2008	18.6	12.7	24.2	0.5	66	1011.7	10.7	2.6
22/08/2008	16.3	13.1	21.8	11.2	84	1008.1	7.9	1.1
23/08/2008	14.5	10.6	18.2	1.5	80	1007.4	10.9	2.8
24/08/2008	16.6	11.4	22.2	0.6	70	1008.9	6.1	1.8
25/08/2008	16.7	13.3	22.5	1.1	78	1011.4	6.9	2.1
26/08/2008	18.2	15.9	21.8	0.0	73	1017.1	6.6	2.1
27/08/2008	17.9	16.6	21.3	0.0	74	1017.2	7.7	2.2
28/08/2008	17.8	15.7	20.6	0.0	78	1015.4	6.5	1.8
29/08/2008	18.7	14.0	24.6	0.0	75	1015.1	6.3	1.6
30/08/2008	20.6	11.6	30.4	0.0	65	1013.7	5.8	1.2
31/08/2008	22.2	13.8	31.6	3.9	59	1008.1	15.1	2.0

Table 8 Soil Characteristics and Results of the Soil Clothianidin Analysis (Before Sugar Drilling)

	Machine Z
	Kverneland Accord Monopill SE
Soil codes of the study plot used for sugar pill drilling [1]	**OE 3521 / OE 3522**
Sand [%]	54.0
Silt [%]	31.9
Clay [%]	14.1
Soil type	Heavy loamy sand
Total carbon [%]	1.24
Organic carbon [%]	1.24
Inorganic carbon [%]	n.d. [2]
Maximum water holding capacity [%]	31.8
Cation Exchange Capacity (CEC) [meq/100 g]	10.0
Water content of the soil directly before drilling [%]	8.1
Clothianidin soil concentration before sugar beet drilling	< LOD (i.e. < 2 µg a.s./kg soil)

[1] **Both soil codes refer to identical study plot, however, OE 3521 refers to a composite sample of the upper 10 cm, whereas OE 3522 refers to a composite sample of the upper 5 cm (water content only)**

[2] **n.d. = not detected**

Table 9 Clothianidin residue values – ground deposition

	Single values, Series A		Single values, Series B		Single values, Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]
1 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-7	*< LOD*	< LOD	*< LOQ*	< LOQ	*< LOD*	< LOD
1 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-5	*0.042*	0.029	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
3 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-4	*< LOD*	< LOD	*< LOQ*	< LOQ	*< LOD*	< LOD
5 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Table 9 Clothianidin residue values – ground deposition (continued)

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]
10 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
10 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-1	*< LOD*	< LOD	*< LOQ*	< LOQ	*< LOD*	< LOD
20 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-3	*< LOQ*	< LOQ	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
20 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
30 meter-10	*< LOD*	< LOQ	*< LOD*	< LOD	*< LOD*	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish

LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Table 9 Clothianidin residue values – ground deposition (continued)

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]
50 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Table 10 Clothianidin residue values - 24 hours measurements after drilling

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]	*[µg a.s. / Petri-dish]*	[g a.s. / ha]
1 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
1 meter-10	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*0.052*	0.035
5 meter-2	*< LOD*	< LOD	*< LOQ*	< LOQ	*< LOD*	< LOD
5 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-4	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOQ*	< LOQ
5 meter-6	*< LOQ*	< LOQ	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-9	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
5 meter-10	*< LOD*	< LOD	*< LOQ*	< LOQ	*< LOD*	< LOD
50 meter-1	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-2	*< LOD*	< LOD	*< LOD*	< LOD	*< LOQ*	< LOQ
50 meter-3	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-4	*< LOD*	< LOD	*0.023*	0.015	*< LOD*	< LOD
50 meter-5	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-6	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-7	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOQ
50 meter-8	*< LOD*	< LOD	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-9	*< LOQ*	< LOQ	*< LOD*	< LOD	*< LOD*	< LOD
50 meter-10	*< LOD*	< LOD	*0.034*	0.023	*< LOD*	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish

LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Table 11 Clothianidin residue values - passive dust drift collectors

5 m distance to the "zero-line"						
	[µg a.s./collector]					
Height above ground [m]	**Single values, Series A**		**Single values, Series B**		**Single values, Series C**	
	I	**II**	**I**	**II**	**I**	**II**
1	*0.040*	*0.046*	*0.054*	*0.050*	*0.083*	*0.060*
2	*0.045*	*0.098*	*0.184*	*0.192*	*< LOQ*	*< LOQ*
3	*0.028*	*0.020*	*0.169*	*0.092*	*0.072*	*0.035*
4	*< LOQ*	*0.024*	*0.086*	*0.061*	*< LOQ*	*0.034*
5	*0.037*	*0.169*	*0.031*	*0.032*	*< LOQ*	*< LOQ*
30 m distance to the "zero-line"						
	[µg a.s./collector]					
Height above ground [m]	**Single values, Series A**		**Single values, Series B**		**Single values, Series C**	
	I	**II**	**I**	**II**	**I**	**II**
1	*< LOQ*	*< LOQ*	*0.077*	*0.178*	*< LOQ*	*0.022*
2	*0.164*	*0.044*	*0.032*	*0.197*	*< LOQ*	*0.035*
3	*0.072*	*0.029*	*< LOQ*	*0.055*	*< LOQ*	*0.043*
4	*0.028*	*< LOQ*	*0.158*	*0.290*	*< LOQ*	*0.021*
5	*0.027*	*0.230*	*0.160*	*0.350*	*< LOQ*	*< LOQ*

LOQ = 0.020 µg a.s./passive PP-mesh dust collector; LOD = 0.006 µg a.s./passive PP-mesh dust collector

Appendix 1 Analysis of Treated Seeds

Bayer CropScience

STUDY TITLE

Analysis of the content of clothianidin and imidacloprid in dressed sugar beet pills used in study E 308 3545-8

TEST ITEM

Dressed sugar beet pills from KWS Saat AG

AUTHOR

[redacted]

STUDY COMPLETION DATE

2008-09-08

PERFORMING LABORATORY

Bayer CropScience AG
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Appendix 1 (continued)

Signature

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Laboratory Responsible Scientist:

[redacted]

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Page: 2

Appendix 1 (continued)

Analysis of the content of clothianidin and imidacloprid in dressed sugar beet pills used in study E 308 3545-8

1. Test material
A sample of dressed sugar beet pills (cultivar Berenika, Reg. No. 16-8023A) from KWS Saat AG was analyzed. The pills had an expected target content of 60 mg of clothianidin and 30 mg of imidacloprid per pill.

2. Description of the analytical method
Clothianidin and imidacloprid were extracted from dressed pills using the conditions described here and quantified by gradient HPLC using UV detection.

2.1. Reagents
All reagents were of A.R. quality or HPLC-grade quality.

2.2. Extraction of the treated seeds
3 aliquots consisting of 100 dressed pills each were extracted with 100 ml each of an acetonitrile/water/phosphoric acid mixture (80/19/1) using an ultrasonic bath for 30 minutes. The extracts were centrifuged and the clear supernatants were analyzed by gradient HPLC for their content of clothianidin and imidacloprid, respectively.

2.3. Reference compound
Bayer CropScience-certified lots of the reference compounds clothianidin and imidacloprid were used in this study.

2.4. Instrumentation
An Agilent type 1100 HPLC instrument was used. The detector was an Agilent 1100 diode array detector. Integration of the chromatograms was performed using the Agilent Cerity chromatography software.

2.5. Chromatography

2.5.1 Column
Stationary phase : Zorbax Extend C18 (Agilent)
Diameter of stationary phase particles: 3.5 µm
Column dimensions: 75 mm x 4.6 mm

2.5.2 Eluant
Component A: 1 L H_2O (Millipore quality) + 5 mL 1 N sulphuric acid
Component B: Acetonitrile

Page: 3

Appendix 1 (continued)

2.5.3 Chromatographic conditions

Gradient:

Time (min)	% A	% B	Flow rate (mL/min)
0.0	80	20	2
2.0	0	100	2
2.6	0	100	3

Temperature: 50°C
Injected volume: 3 µl
Run time: ca. 2.6 min
UV detection: 230 nm

3. Results

3.1. Content clothianidin and imidacloprid

	Clothianidin (mg/100 pills)	Clothianidin (mg/pill)	Loading clothianidin (% of target)	Imidacloprid (mg/100 pills)	Imidacloprid (mg/pill)	Loading imidacloprid (% of target)
Aliquot 1	58.2743			32.3033		
Aliquot 2	59.9832			33.1568		
Aliquot 3	58.5538			32.0381		
Average	**58.94**	**0.5894**	**98.2**	**32.50**	**0.3250**	**108.3**

3.2 Loading of the seeds:
The loading of the analyzed sugar beet pills was 98.2 % for Clothianidin and 108.3 % for Imidacloprid.

4. Summary of results
The dressed sugar beet pills (cultivar Berenika, Reg. No. 16-8023A) from KWS Saat AG had a loading of 98.2% for clothianidin and 108.3% for imidacloprid, respectively.

Appendix 1 (continued)

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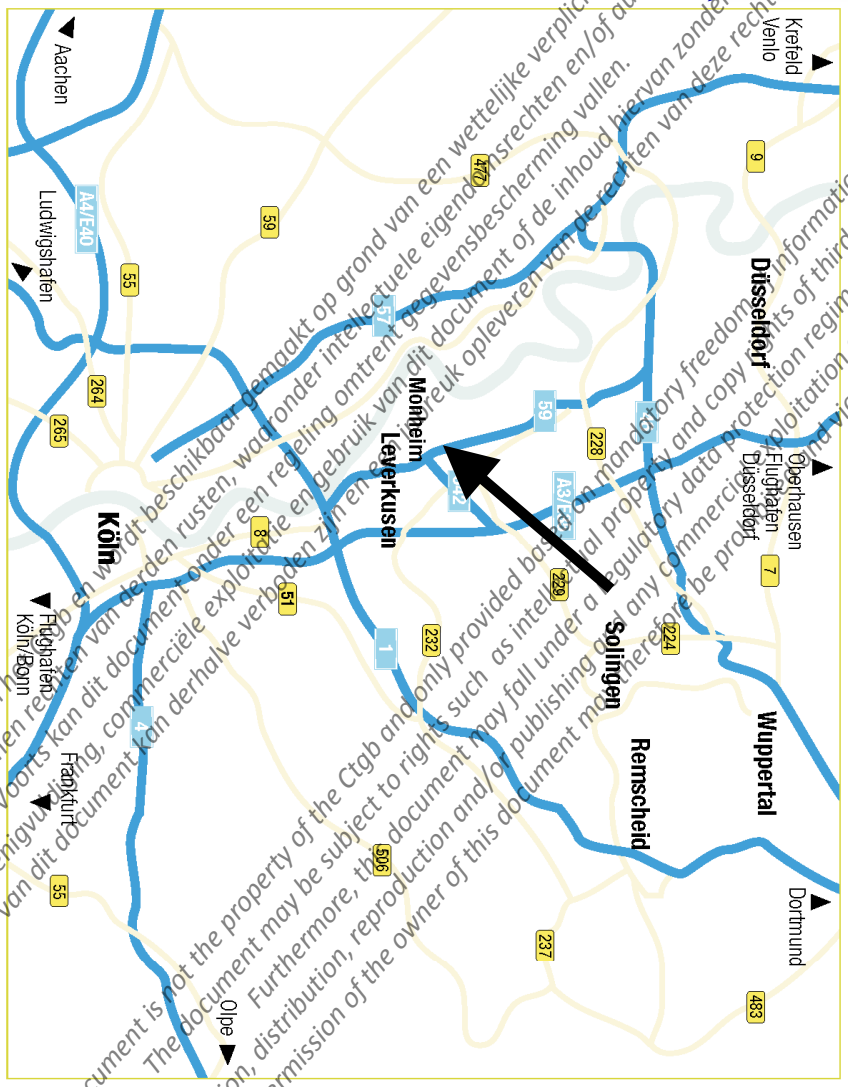
[redacted]

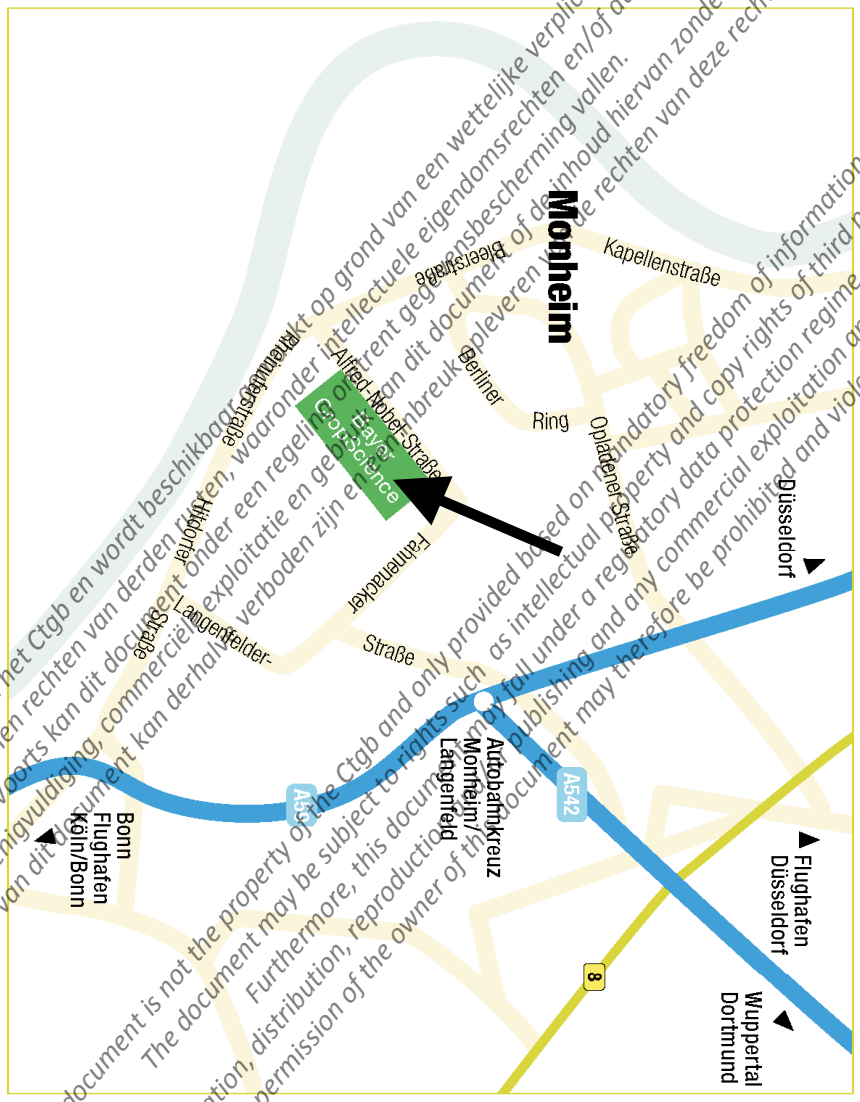
Page: 5

Appendix 2 Study Region and Study Plot Area

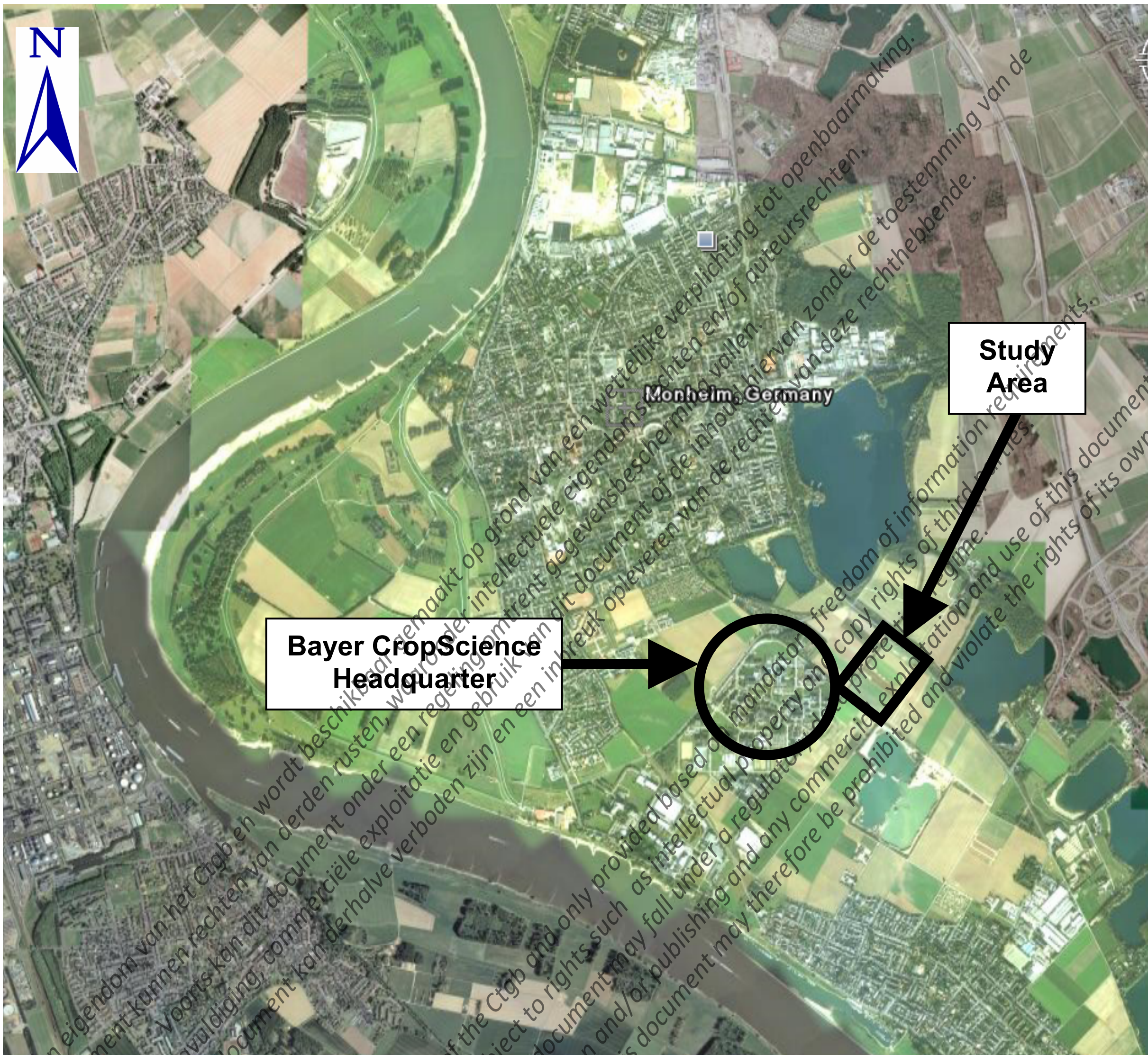
Appendix 2 (continued)



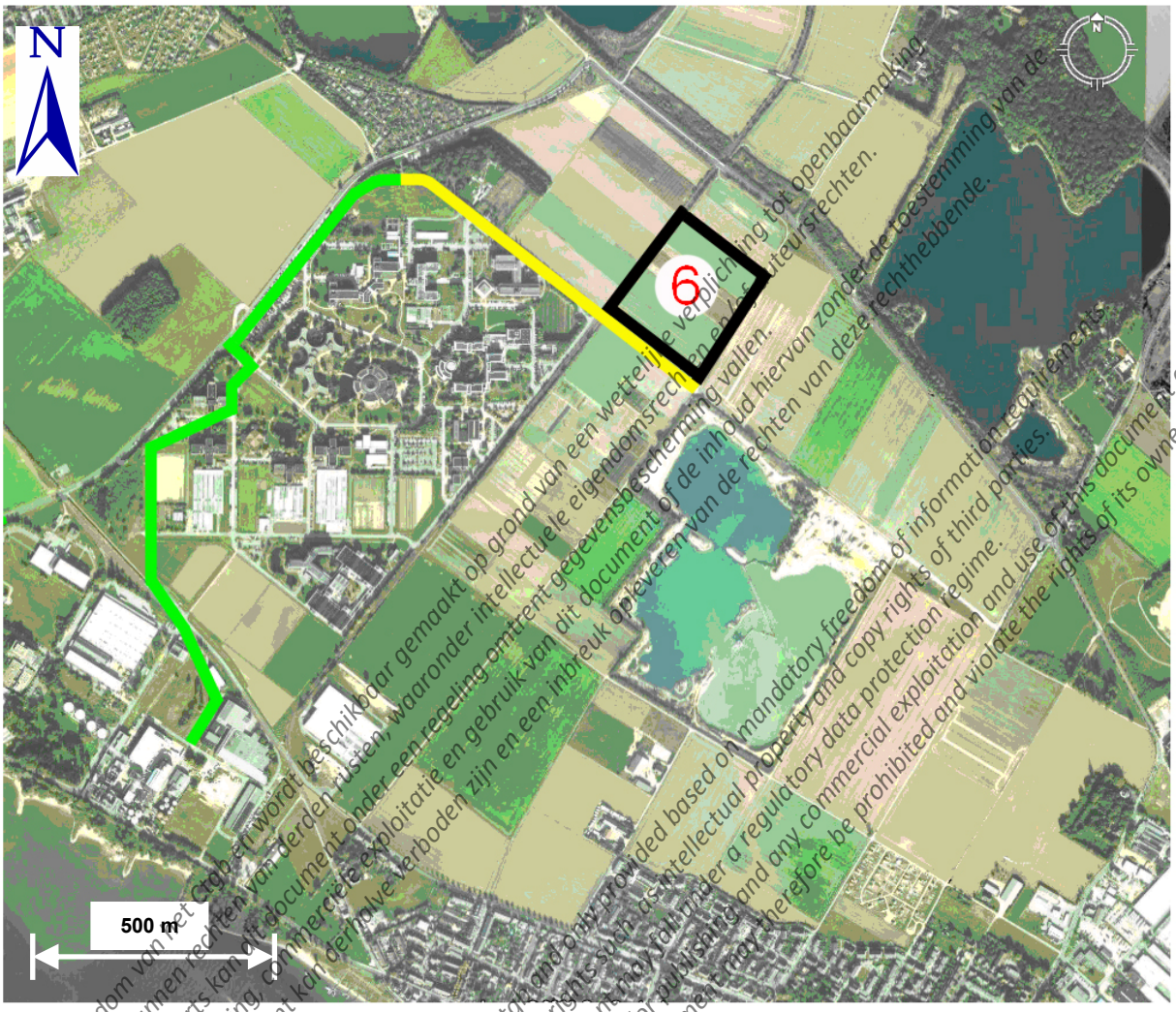
Appendix 2 (continued)



Appendix 2 (continued)



Appendix 2 (continued)



	Public road, good quality
	Farm track, moderate quality

Field No. 6	5.6 ha	Study plot location for testing Machine Z

Appendix 3 On-site Wind Speed And Wind Direction Measurements

Statistical parameters	Temperature	Relative humidity	Wind speed 2 m above ground	Wind [1] Direction	Δ^2 Wind Direction
[-]	[°C]	[%]	[m/s]	[Degree]	[Degree]
Drilling path No. 1					
Arithmetic mean	19.2	70.9	5.5	174.0	-6.0
Geometric mean	19.2	70.9	5.5	173.9	n.a.
10th%ile	19.1	70.9	4.5	167.7	-12.3
90th%ile	19.4	70.9	6.3	179.5	-0.5
Drilling path No. 2					
Arithmetic mean	19.2	71.3	7.3	165.1	-14.9
Geometric mean	19.2	71.3	7.2	164.9	n.a.
10th%ile	19.1	71.1	6.4	152.1	-27.9
90th%ile	19.4	71.8	8.1	174.7	-5.3
Drilling path No. 3					
Arithmetic mean	19.1	71.3	6.1	171.2	-8.8
Geometric mean	19.1	71.3	6.0	171.1	n.a.
10th%ile	19.0	71.3	5.3	164.0	-16.0
90th%ile	19.1	71.3	7.2	177.2	-2.8
Drilling path No. 4					
Arithmetic mean	19.4	71.4	5.3	161.9	-18.1
Geometric mean	19.4	71.4	5.2	161.7	n.a.
10th%ile	19.2	71.3	4.2	150.3	-29.7
90th%ile	19.5	71.6	7.0	173.0	-7.0
Drilling path No. 5					
Arithmetic mean	19.2	71.3	5.4	170.8	-9.2
Geometric mean	19.2	71.3	5.4	170.1	n.a.
10th%ile	19.1	71.2	4.7	152.5	-27.5
90th%ile	19.4	71.8	6.2	190.7	10.7
Drilling path No. 6					
Arithmetic mean	19.4	71.0	5.4	170.3	-9.7
Geometric mean	19.4	71.0	5.3	170.1	n.a.
10th%ile	19.4	70.9	4.4	158.3	-21.7
90th%ile	19.5	71.3	6.2	179.5	-0.5
Drilling path No. 7					
Arithmetic mean	19.3	70.9	7.0	161.6	-18.4
Geometric mean	19.3	70.9	7.0	158.9	n.a.
10th%ile	19.0	70.7	5.9	150.5	-29.5
90th%ile	19.5	70.9	8.1	179.3	-0.7

[1] A wind direction of 180° means that the wind is blowing exactly perpendicularly to the sampling devices
[2] The delta (Δ) indicates the difference to the exactly perpendicular wind direction (i.e. 180°)
n.a. = not applicable

Appendix 3 (continued)

Statistical parameters	Temperature	Relative humidity	Wind speed 2 m above ground	Wind [1] Direction	Δ^2 Wind Direction
[-]	[°C]	[%]	[m/s]	[Degree]	[Degree]
Drilling path No. 8					
Arithmetic mean	19.2	70.8	6.1	169.4	-10.6
Geometric mean	19.2	70.8	6.0	169.2	n.a.
10^{th}%ile	19.1	70.5	4.7	161.0	-19.0
90^{th}%ile	19.4	70.9	7.6	179.4	-0.6
Drilling path No. 9					
Arithmetic mean	19.1	71.1	5.9	168.2	-11.8
Geometric mean	19.1	71.1	5.9	168.0	n.a.
10^{th}%ile	19.1	70.9	5.3	157.3	-22.7
90^{th}%ile	19.2	71.3	7.0	177.9	-2.1

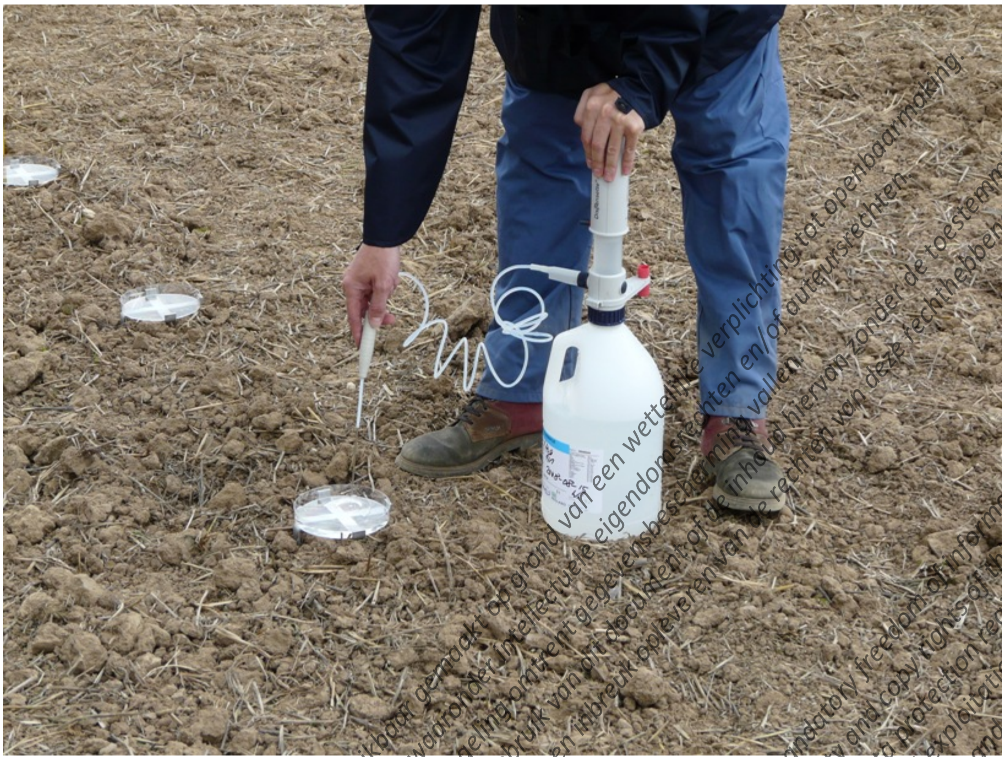
[1] A wind direction of 180° means that the wind is blowing exactly perpendicularly to the sampling devices
[2] The delta (Δ) indicates the difference to the exactly perpendicular wind direction (i.e. 180°)
n.a. = not applicable

Appendix 4 Additional Photos

Passive polypropylene-mesh dust collector

Tripod-pylons with mounted passive polypropylene-mesh dust collectors

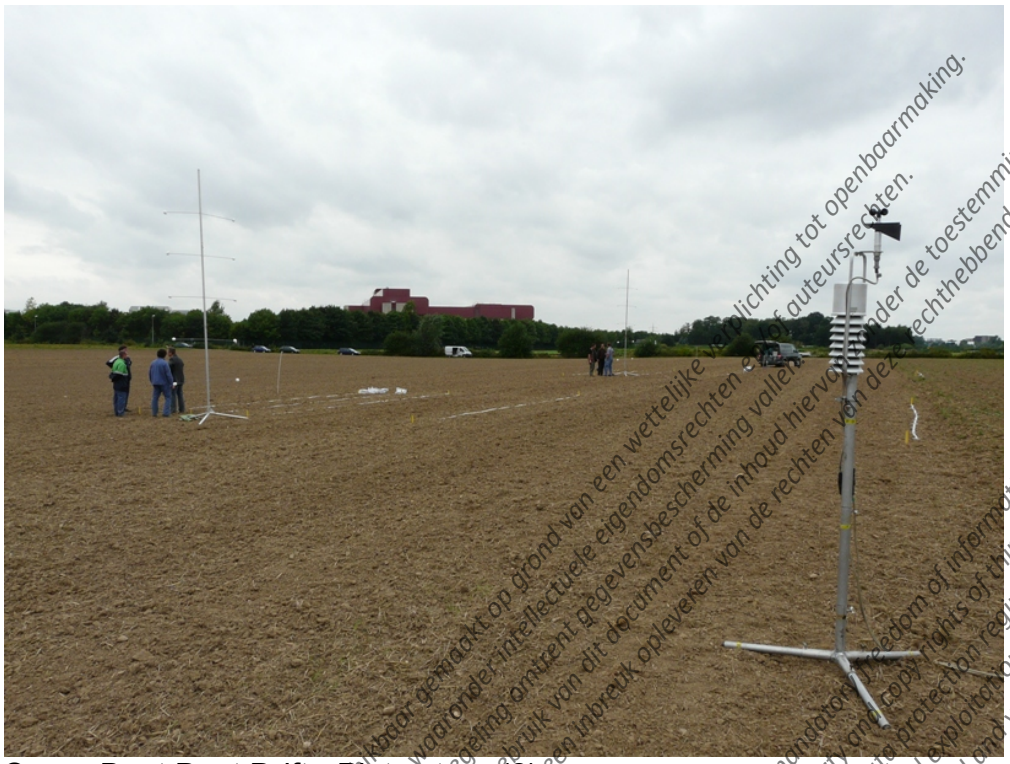
Appendix 4 (continued)



Filling of the Petri-dishes

Sugar-Beet Dust Drift - Plot set-up (1)

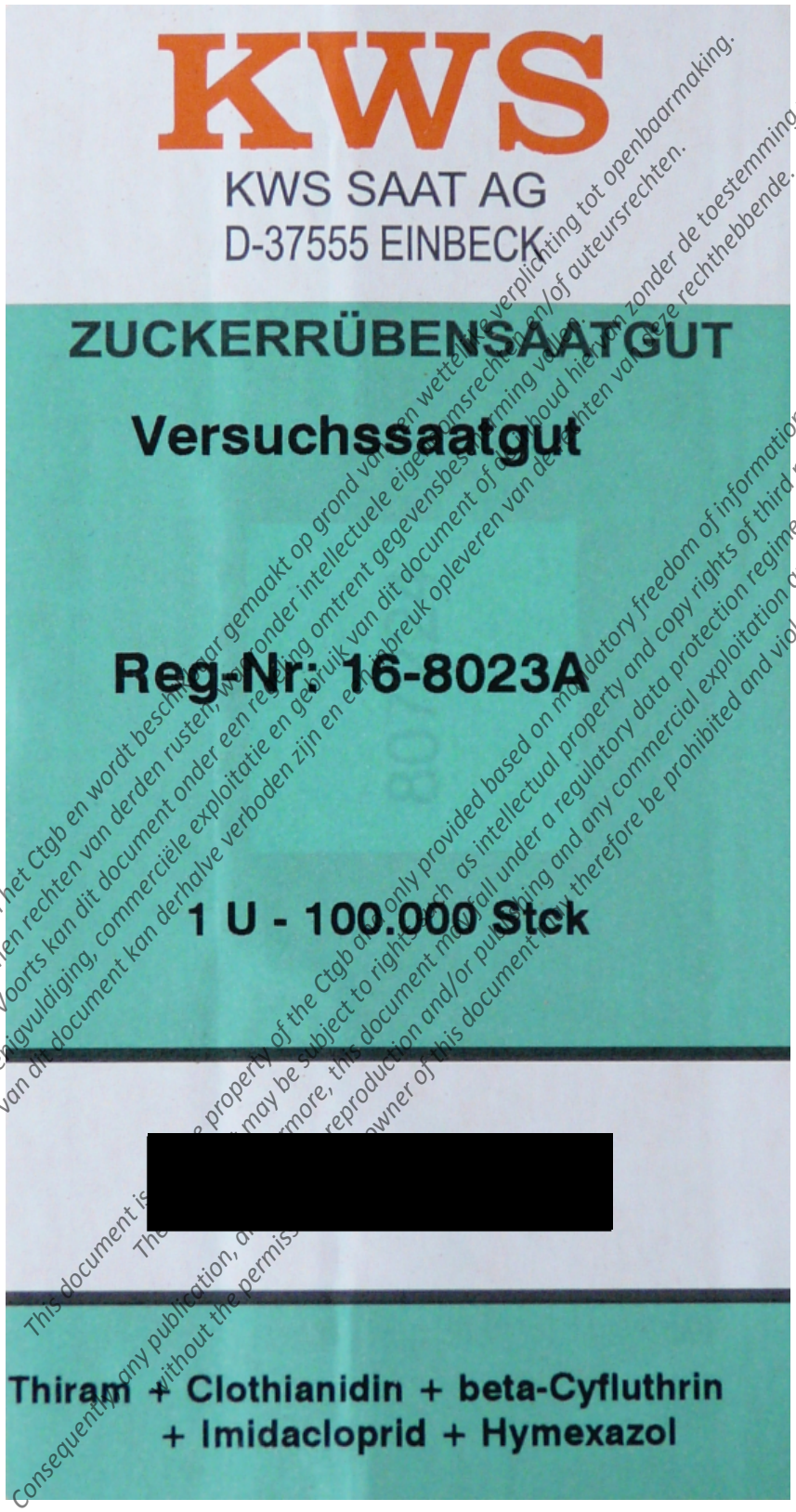
Appendix 4 (continued)



Sugar-Beet Dust Drift - Plot set-up (2)

Sugar-Beet Dust Drift - View from the moving tractor towards the sampling area

Appendix 4 (continued)



Seed label of the employed sugar beet pills

Appendix 5 Soil Characterization of The Study Plot

LUFA Speyer

BEZIRKSVERBAND PFALZ

LANDWIRTSCHAFTLICHE UNTERSUCHUNGS- UND FORSCHUNGSANSTALT SPEYER

Obere Langgasse 40
67346 Speyer

Landwirtschaftliche Untersuchungs- • und Forschungsanstalt • Postfach 1629 • 67326 Speyer

[redacted]

Bayer CropScience AG
Development-Ecotoxicology, BCS AG-D-ETX
Alfred-Nobel-Str. 50
Building 6600, Room 025
40789 Monheim / Rhein

[redacted]

Untersuchungsbericht
zu Studie E 308 3545-8

	OE 3521 B42711/08
Korngrößenverteilung:	
Bodenart	stark lehmiger Sand
Ton < 0,002 mm (%)	14,1
Feinschluff 0,002-0,006 mm (%)	5,5
Mittelschluff 0,006-0,020 mm (%)	9,0
Grobschluff 0,020-0,063 mm (%)	17,4
Feinsand 0,063-0,200 mm (%)	18,8
Mittelsand 0,200-0,630 mm (%)	26,1
Grobsand 0,630-2,000 mm (%)	9,1
Cges (%)	1,24
Canorg (%)	n.n.
Corg (%)	1,24
Wkmax (%)	31,8
Kationenaustauschkapazität (meq/100g)	10,0

n.n. = nicht nachweisbar

LUFA Speyer

[redacted]

USt-ID-Nr. DE149392971

Seite 1 von 1

Kreis- und Stadtsparkasse Speyer
BLZ 547 500 10
Kto. 62 471

Sprechzeiten:
Montag bis Freitag
8.30 - 12.00 Uhr
14.00 - 16.00 Uhr

Deutscher Akkreditierungs Rat
DAP-PL-2910.00

Appendix 6 Clothianidin Soil Residue Analysis

Bayer CropScience AG
BCS-D-ROCS

Result Report
E 308 3545-8

Determination of clothianidin in one soil samples according analytical method 00540/M001

Author

[redacted]

Completion Date

2008-09-05
(yyyy-mm-dd)

Sponsor

Bayer CropScience AG
Ecotoxicology
Alfred-Nobel-Str. 50
D-40789 Monheim am Rhein
Germany

Test Facility

Bayer CropScience AG
Development - Residues, Operator and Consumer Safety
Agricultural Centre Monheim
Alfred-Nobel-Str. 50
D-40789 Monheim am Rhein
Germany

Appendix 6 (continued)

Bayer CropScience AG **Result Report**
BCS-D-ROCS **E 308 3545-8**

Analytical method

This method describes the determination of the active ingredient TI-435. Soil samples of 20 g are extracted in a microwave extractor with 50 mL of a mixture of water / acetonitrile. After extraction, parts of the samples are centrifuged to remove fine particles of the soil or sediment. Identification and quantitation of the active substance is done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode. Isotopically labelled internal standard (*d_3-TI-435*) is used to compensate for possible matrix effects in the MS/MS-detector.
The limit of quantification (LOQ) is 5 µg/kg for TI-435 and the limit of detection (LOD) is 2 µg/kg for TI-435.

Results

Sample ID	Sample Weight [g]	Moisture [%]	Result [µg/kg]
OE 3521/1	20	1.84	< LOD

Residues above the LOD of 2 µg/kg for clothianidin could not be detected.

Head of Laboratory [redacted]
Bayer CropScience AG

[redacted]

Appendix 7 Water Content Analysis of the Top Soil of Each Drilling Plot

Bayer CropScience AG Report LRT-SV-BKD **04/08**
Internal Test No.: 3566– Result Report to E 308 3545-8

Determination of the water content of one soil sample according to method SOP 3009, version 6,
Bayer CropScience AG, BCS-D-ETX

Author

[redacted]

Completion Date

2008-10-17
(yyyy-mm-dd)

TESTING FACILITY

Bayer CropScience AG
Ecotoxicology
Alfred-Nobel-Str. 50
D-40789 Monheim

Page 1 of 2 Pages

Appendix 7 (continued)

Materials & Method

The soil was sampled from the 0-5 cm toplayer of one study plot directly before sugar-beet seed drilling. Approximately 2.5 kg soil was received in a sealed plastic bag, labeled and stored at room temperature in the dark. The sample was processed within one week after arrival.
To determine the water content of the soil, the soil sample was sieved down to a size of 2 mm. The water content was measured in three replicates, each consisting of 10 g sieved soil (+/- 0.02 g), which was weighed into a ceramic cup, whose tare weight was determined before the addition of soil. After a drying period of 20 minutes at the highest power level of a micro wave, the ceramic cup, including the soil, was weighed. Subsequently the sample was dried a second time for 5 minutes at the highest power level and weighed again. In case the difference between the first and the second weighing was higher than 0.05 g, this procedure was repeated until the mass loss between the drying periods was below 0.05 g (Weight $_t$ - Weight $_{t+5\ minutes}$ < 0.05g). If the mass loss of the sample was below 0.05 g the initial water content was calculated for each sample as follows:

Mass loss [g]/Initial soil weight * 100

The results for the water content of the soil sample is reported as single values of the replicates as well as calculated mean in %.

Results

ID No. (OE No.)	Cup No.	Moist wt. of 10 g Soil + Cup [g] (*)	1st weight after drying [g]	2nd weight after drying [g]	Water Content [%]	Water Content: Mean Value [%]
OE 3522	a	59.38	58.55	58.57	8.3	8.10
	b	55.70	54.90	54.90	8.0	
	c	56.71	55.91	55.90	8.0	

(*) The originally weighted-in quantity of the soil samples took place with an accuracy of ± 0.02 g

[redacted] Date: 2008-10-17

Appendix 8 Dust Abrasion of Treated Seeds and Loose Dust in Seed Bags

Bayer CropScience

STUDY TITLE

Laboratory study to investigate the dust abrasion, volatile dust and loose dust in packages of commercially Poncho Beta Plus treated sugar beet pills used for a field dust drift study

TEST ITEMS

Poncho Beta FS 453
Spec. No.: 102000008400
Mat. No.: 06264247

Gaucho WS 70
Spec. No.: 102000006811
Mat. No.: 04175778

STUDY DIRECTOR

[redacted]

STUDY COMPLETION DATE

2008-11-05

TESTING FACILITY

Bayer CropScience AG
Portfolio Management
Seed Treatment
D-40789 Monheim
Germany

STUDY NUMBER

Bayer CropScience Study No. HSF2008PonchoBetaPlus001

Page 1 of 8

Appendix 8 (continued)

Bayer CropScience AG Study No. HSF2008PonchoBetaPlus001

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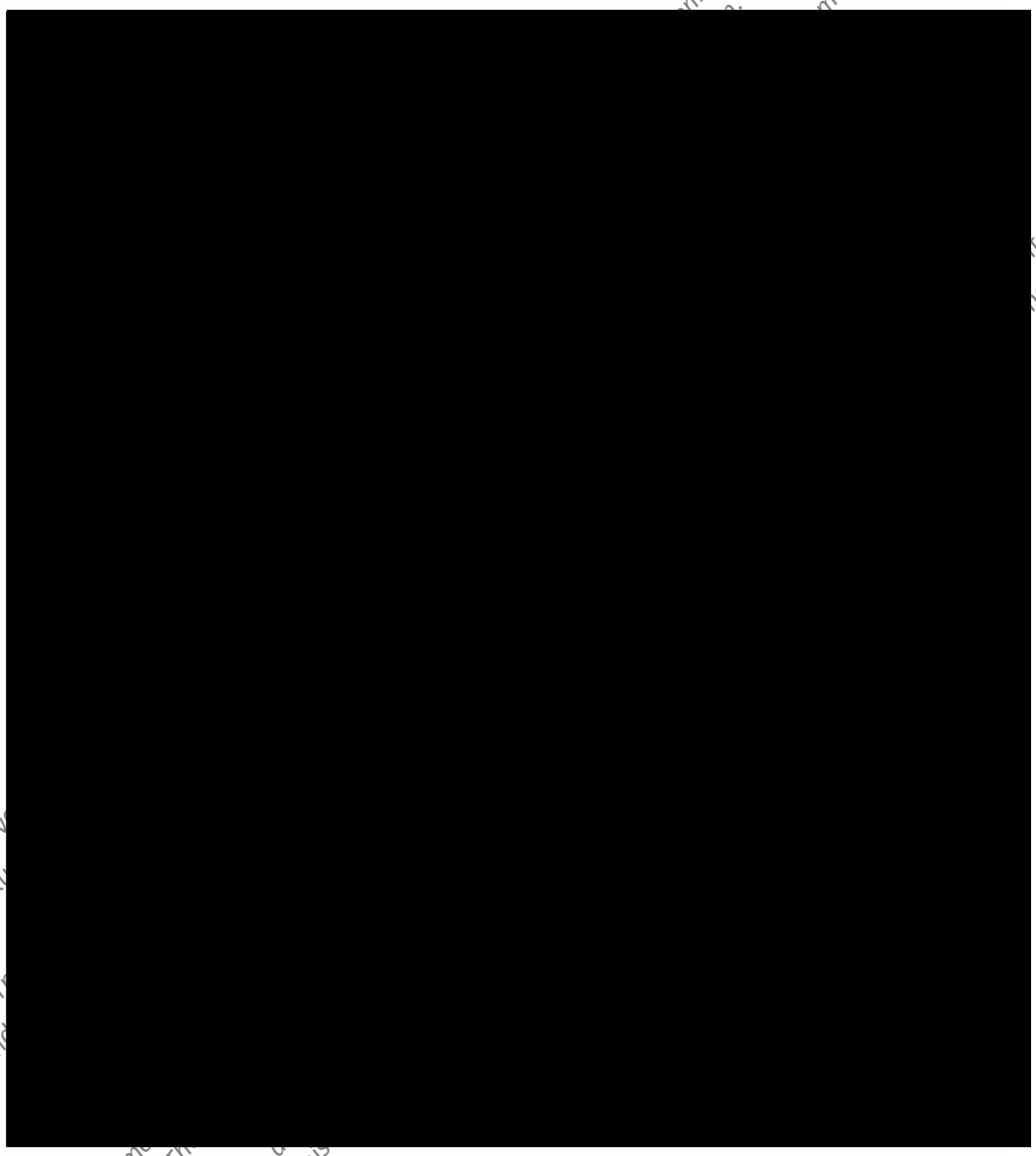
Appendix 8 (continued)



Appendix 8 (continued)

Table of Contents

Appendix 8 (continued)

Laboratory study to investigate the dust abrasion, volatile dust and loose dust in packages of commercially Poncho Beta Plus treated sugar beet pills used for a field dust drift study

1. Introduction

Various laboratory experiments were conducted in August 2008 to investigate dust abrasion and volatile dust of Poncho Beta Plus treated sugar beet pills used in a field dust drift study (E 308 3845-8). In addition, the quantity of loose dust present in seed packages was determined.

2. Test Materials

Poncho® Beta FS 453 (a.i. clothianidin, beta-cyfluthrin),
Gaucho® WS 70 (a.i. imidacloprid)
Spec. No.: see above
Mat. No.: see above

Seed material: "KWS sugar beet pills", variety "Berenika", Thousand-Pill-Weight: 29.7 g, commercial pills purchased from KWS Saat AG, Grimsehlstr. 31, D-37555 Einbeck, Germany, Reg.-No.: 16-8023A

3. Experimental

The laboratory tests were conducted in August 2008 to investigate the dust abrasion (Heubach dust abrasion test), volatile dust (Ceres dust test) and loose dust fractions in packages of sugar beet pills used for a field dust drift study (E 308 3845-8). The sugar beet variety "Berenika" was commercially treated by KWS and delivered to the Bayer CropScience Seed Treatment Application Centre.

3.1. Seed treatment of sugar beet pills

Commercial sugar beet pills (original packages), variety "Berenika" has been received from KWS. The pills had been treated with Poncho Beta Plus, containing clothianidin and imidacloprid (60 + 30 g/Unit*).

* 1 Sugar Beet Unit = 100,000 pills (for variety "Berenika" equivalent to 2.97 kg)

Appendix 8 (continued)

3.2. Seed conditioning

Prior to laboratory analyses (Heubach dust abrasion test and Ceres dust test) a sub-sample of the treated pills was conditioned for a minimum of 48 hours in a constant climate chamber at 20 ºC and 50% RH.

3.3. Laboratory test (Heubach dust abrasion test)

Samples of 100 grams of pre-conditioned seed (20 ºC, 50% RH) were weighed (±1/100 g) and filled into the drum of the Heubach dustmeter equipment. In total, 3 replications were performed. After a glass fiber filter disc (Whatman GF92) was placed into the filter holding unit, the same was weighed (± 0.1 mg). After fitting the filter unit the rotation cycle was started. The following settings were used:

Rotation speed: 30 rpm;

Air flow: 20 L/min.

Rotation time: 2 min.

After finishing the cycle the filter unit was disconnected and weighed (± 0.1 mg). The difference in weight constitutes the amount of fine dust collected on the filter. The dust values were calculated in g/100 kg and g/100,000 pills. After each measurement (replication) all parts of the instrument were cleaned.
Note: The above described method can be only used for relative comparison between treatments as the absolute quantities of dust will depend on method parameters, i.e. rotation time, rotation speed and air-low.

3.4. Laboratory test (CERES volatile dust test)

Samples of 2 x 250 grams of pre-conditioned pills (20 ºC, 50% RH) per treatment were weighed (±1/100 g) and filled into a beaker. A filter disc (AAWP 03700) is placed into the filter holding unit (Millipore, Filt-Air Q50). The unit with the filter disc was precisely weighed (± 0.1 mg). After fitting the filter unit to the dust-meter equipment (see appendix) the vacuum pump (15 L/min. airflow) was started and the pre-weighed pills poured into the tube of the equipment. After 5 min. of air suction the filter holding unit was disconnected and weighed again (± 0.1 mg). The difference in weight constitutes the amount of fine dust collected on the filter. The dust values were calculated in g/100 kg and g/100,000 pills. After each measurement (replication) all parts of the instrument were cleaned. In total, 2 replications were performed.

3.5. Laboratory test (determination of loose dust in seed packages)

Each package of sugar beet pills was opened at the top and the pills were carefully poured in small quantities at a time onto a round 2.5 mm metal sieve placed on top of metal pan. The screen with the pills was shaken in order for the loose dust particles to fall through the screen. This procedure was repeated until the whole seed package had been emptied and all loose dust particles removed from the package and collected in the metal pan. The collected loose dust was quantitatively transferred from the metal pan into a PE-bottle and the total amount of dust was weighed. In total, 3 replications were performed.

Appendix 8 (continued)

4. Results

Table 1 shows the amount of abraded dust (Heubach dust abrasion test), volatile dust (Ceres volatile dust test) and loose dust in sugar beet seed packages.

Table 1: Average quantities of abraded dust (Heubach test), volatile dust (Ceres test) and total loose dust in seed package, August 2008.

KWS- sugar beet pills, variety "Berenika"	**[g/100 kg]**	**[g/100,000 pills] ***
Heubach abraded dust	0.174	0.00517
Ceres volatile dust	0.380	0.01130
Loose dust in seed package (total)	1.000	0.02970

*** 100,000 pills = 1 Sugar Beet Unit (for variety "Berenika" equivalent to 2.97 kg)**

5. Conclusions

The average amount of abraded dust (Heubach dust abrasion test) was 0.174 g/100 kg or 0.00517 g/100,000 pills. The average amount of volatile dust (Ceres volatile dust test) was 0.38 g/100 kg or 0.0113 g/100,000 pills. The average total amount of loose dust in seed package was 1.0 g/100 kg or 0.0297 g/100,000 pills.

Appendix 8 (continued)

6. Attachment

Bayer CropScience

Declaration of Quality Standards

[redacted]

Appendix 9 Full Analytical Phase Report

Bayer CropScience AG
BCS-D-ROCS
D-40789 Monheim am Rhein

Study No.: E 308 3545-8
Report No.: MR-08/163

Study Title

Drift deposition pattern of seed treatment particles abraded from Poncho® Beta Plus treated sugar beet pills and emitted by a typical mechanical sowing machine

Purpose

The aim of the study was to measure the deposition rate of clothianidin-containing seed treatment particles (g a.s./ha) at various distances from a field drilled with Poncho® Beta Plus treated sugar beet pills during sowing with a typical mechanical sugar beet in acetonitrile/water and glycerol/water out of Petri dishes and from passive dust collectors.

Data Requirement

EU-Ref: Council Directive 91/414/EEC of July 15, 1991,
Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed
Pre-Registration: SANCO/3029/99 Rev. 4, 2000-07-11

Author

[redacted]

Report Completion Date

2008-09-12
Date: yyyy-mm-dd

Analytical Test Facility

Bayer CropScience AG
Development - Residues, Operator and Consumer Safety
BCS-D-ROCS
Alfred-Nobel-Str. 50
D-40789 Monheim am Rhein

Laboratory Project ID

P672084721

Lynx ID: EBTIL043

Page 1 (38)

Appendix 9 (continued)

Data Confidentiality Statement

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Appendix 9 (continued)

[redacted]

Appendix 9 (continued)

Certification of Authenticity

[redacted]

Appendix 9 (continued)

Print Date: 12 SEP 2008

Report No **E-308-3545-8**
Page

Quality Assurance (GLP)

Quality Assurance Statement

[redacted]

Appendix 9 (continued)

Table of Contents **Page**

Appendix 9 (continued)

1 Summary

The aim of the study was to measure the deposition rate of clothianidin-containing seed treatment particles (g a.s./ha) at various distances from a field drilled with Poncho® Beta Plus treated sugar beet pills during sowing with a typical mechanical sugar beet in acetonitrile/water and glycerol/water out of Petri dishes and from passive dust collectors.

The field part of this study (collecting of the solvent of the Petri dishes, and the passive dust collectors) as well as the transport of the samples to the laboratory of [redacted] was not conducted under GLP. Therefore nothing will be documented in the RAW data of this GLP part. This will be part of the final report.

All clothianidin-containing dust and abrasion particles which deposited at 1, 3, 5, 10, 20, 30 and 50 meters distance from the drilling area were sampled in polystyrene Petri-dishes (Ø 13.7 cm. 147.41 cm²), filled with an acetonitrile-water mixture (2/8. v/v). For each sampling distance 3 arrays of 10 Petri-dishes each were installed with a distance of 1 meter between the dishes. Accordingly, a total of 30 samples were yielded for each sampling distance.

After the drilling was completed (i.e. after approximately 30 minutes for the 12-row mechanical planting machine), an additional waiting period of 15 minutes was employed to allow those dust particles which had not yet been deposited to settle on the sampling area. Thereafter, the acetonitrile-water mixture of each Petri-dish was quantitatively transferred into a 250 mL polyethylene flask by means of a polyethylene funnel. Both, the Petri-dish and the funnel were rinsed with acetonitrile/water (2/8. v/v) and the rinse was combined with the content of the respective Petri-dish inside the 250 mL polyethylene flask before being tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 meter distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with machine ID-code ("Machine Z"). the number of the respective series (A. B or C), the distance from the drilling area (1, 3, 5, 10, 20, 30 or 50 m) and the number of the respective Petri-dish per distance (1-10), giving in total 210 flasks.

In all series (A. B and C) at 5 and 30 m distance from the 'zero-line', respectively, passive dust-drift collectors made of polypropylene have been installed at 1 m, 2 m, 3 m, 4 m and 5 m above the soil surface. At each end of a horizontal pole in the respective height, one passive collector has been installed, giving in total 6 passive collectors per height per distance. Once the 30 m - line and later on, the 5 m - line has been reached during the sampling process of the Petri-dishes (filled with acetonitrile/water. 2/8. v/v; see above), the passive dust collectors were sampled and placed into plastic containers which were immediately sealed and labelled in the field. In the laboratory, each individual passive dust collector has been extracted with an appropriate volume of acetonitrile/water (2/8. v/v) within an ultrasonic bath to enhance the extraction process.

Appendix 9 (continued)

1 Summary (contd)

After sampling of all 210 Petri-dishes (filled with acetonitrile/water. 2/8. v/v) and all 60 passive dust collectors per machine was completed, new Petri-dishes were disposed in all three series (A. B. C) at the 1 m, 5 m and 50 m distance from the 'zero-line', giving per machine another 90 Petri-dishes. This second installation of Petri-dishes was filled with about 100 mL of a glycerol/water mixture (1/1. v/v) and was exposed (lids open) for a period of 24 h in order to quantify the amount of clothianidin which may enter the Petri-dishes via secondary drift processes.

After 24 h. the content of each Petri-dish was quantitatively transferred into 250 mL polyethylene flask by means of a polyethylene funnel. Both, the Petri-dish and the funnel were rinsed with water and the rinse was combined with the content of the respective Petri-dish inside the 250 mL polyethylene flask before being tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 meter distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with machine ID-code ("Machine Z"), the number of the respective series (A. B or C), the distance from the drilling area (1, 5 or 50 m) and the number of the respective Petri-dish per distance (1-10), giving in total 90 flasks.

At the day of drilling / sampling (August 20, 2008), all 210 polypropylene flasks (acetonitrile/water. 2/8. v/v) together with all 60 passive dust collectors were transported by car to the laboratory of Ralf Schoening at Bayer CropScience AG's Institute for Residues, Operator and Consumer Safety (BCS-D-ROCS). The following day (August 21, 2008), the 90 polypropylene flasks (glycerol/water. 1/1. v/v) were also transported by car to the laboratory of Ralf Schoening (BCS-D-ROCS). All samples were analysed for their clothianidin content according to methods 00554/M001.

To the content of the 250 mL polyethylene bottles 20 µL of a 5.0 mg/L Ti 435-d3 internal standard solution was added to each of the samples and shaked well. 1 mL of the content of the well mixed 250 mL polyethylene bottles were transferred into a HPLC vial and subjected to the MS/MS procedure. For the analysis of the passive dust collectors 100 mL of acetonitrile/water (1/4. v/v) and 20 µL of a 5.0 mg/L Ti 435-d3 internal standard solution was added to each plastic container. The samples were placed in an ultrasonic bath and an aliquot of 1 mL was transferred into a HPLC vial and subjected to the MS/MS procedure.

The Limit of Quantitation (LOQ) for clothianidin, defined as the lowest validated fortification level, was 0.02 µg a.s./Petri dish or 0.014 g a.s./ha and 0.02 µg a.s./passive dust collector. The LOD was estimated from the linearity data and from the control samples and was 0.006 µg a.s./Petri dish or 0.004 g a.s./ha and 0.006 µg a.s./passive dust collector.

The individual recovery values for clothianidin ranged from 96 to 109% with mean recoveries of 98% (Petri dishes) and 103% (passive dust collectors) and relative standard deviations (RSD's) of 2.2 (Petri dishes) and 4.8% (passive dust collectors) (n = 4, each). All results of the method validation were in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Appendix 9 (continued)

1 Summary (contd)

For the sowing machine tested, residues of clothianidin were between < LOD (0.006 g a.s./ha) and 0.029 g a.s./ha in the acetonitrile/water solutions from the Petri dishes, between < LOD and 0.035 g a.s./ha in the glycerol/water solutions from the Petri dishes collected after 24 h and from < LOQ (0.02 µg a.s./passive dust collectors) to 0.350 µg absolute a.s. on the passive drift collectors.

For detail information see Table 4 to Table 6.

Appendix 9 (continued)

2 Organisation and Staff

2.1 Organisation of Project

Study Director	[redacted]
PI Analysis and Head of Laboratory	
Analytical Test Facility	Bayer CropScience AG BCS-D-ROCS Alfred-Nobel-Str. 50 D-40789 Monheim am Rhein
Head of Analytical Test Facility	[redacted]

2.2 Responsible Personnel for Residue Analysis

PI Analysis and Head of Laboratory	[redacted]
Technicians	

Appendix 9 (continued)

2.3 Archiving

All raw data pertaining to this study and the original final report are stored in the central GLP archive of Bayer CropScience AG, Alfred-Nobel-Str. 50, D-40789 Monheim am Rhein for as long as required by GLP principles.

Retain samples of the reference items are stored in the archives of Bayer CropScience AG, Product Technology-Analytics Frankfurt, Industriepark Höchst, D-65926 Frankfurt. The reference items are stored as long as their quality still guarantees an evaluation.

2.4 Quality Assurance Unit

Bayer CropScience AG
BCS-D-GLP/QA
Agricultural Centre Monheim
Alfred-Nobel-Str. 50
D-40789 Monheim am Rhein

2.5 Schedule

Start of Experimental Phase (First sample preparation)	2008-08-21
End of Experimental Phase (Last printout of a chromatogram)	2008-08-26 Date: yyyy-mm-dd

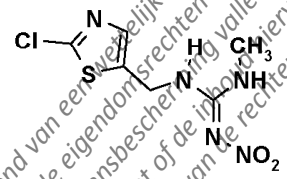
3 Introduction and Purpose of the Study

The aim of the study was to measure the deposition rate of clothianidin-containing seed treatment particles (g a.s./ha) at various distances from a field drilled with Poncho® Beta Plus treated sugar beet pills during sowing with a typical mechanical sugar beet in acetonitrile/water and glycerol/water out of Petri dishes and from passive dust collectors.

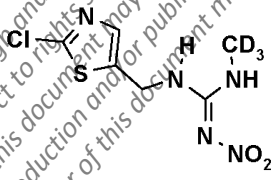
Appendix 9 (continued)

4 Compounds

4.1 Reference Item

- Name of the Compound	**Clothianidin (TI 435)**
- Certificate of Analysis	AZ13320, dated 2006-04-18
- Mol-ID	114
- Structure	
- Chemical Name of the Compound	(E)-1-(2-chloro-1,3-thiazol-5-ylmethyl)-3-methyl-2-nitroguanidine
- Product Code	AE 1283742 00 1B99 0001
- Empirical Formula	$C_6 H_8 Cl N_5 O_2 S$
- Molar Mass	249.68 g/mol
- Purity	99.4%
- Expiry Date	April 2009
- Batch No.	KTS10061-1-1

4.2 Internal standard

- Name of the Compound	**d_3-Clothianidin (d_3-TI 435)**
- Certificate of Analysis	M28418, 2002-12-02
- Structure	
- Empirical Formula	$C_6 H_5 Cl D_3 N_5 O_2 S$
- Molar Mass	252.7 g/mol
- Purity	99.9%

Appendix 9 (continued)

5 Sample Material and Sample Preparation

At the day of drilling / sampling (August 20, 2008), all 210 polypropylene flasks (acetonitrile/water, 2/8, v/v) together with all 60 passive dust collectors were transported by car to the laboratory of [redacted] Bayer CropScience AG's Institute for Residues, Operator and Consumer Safety (BCS-D-ROCS). The following day (August 21, 2008), the 90 polypropylene flasks (glycerol/water, 1/1, v/v) were also transported by car to the laboratory [redacted] (BCS-D-ROCS). All samples were analysed for their clothianidin content as described below in accordance to method 00554/M001.

6 Residue Analyses

6.1 Analytical Method

Note: The field part of this study (collecting of the solvent of the Petri dishes, and the passive dust collectors) as well as the transport of the samples to the laboratory of [redacted] was not conducted under GLP. Therefore nothing will be documented in the RAW data of this GLP part. This will be part of the final report.

6.1.1 Petri Dishes or Contend of the Polypropylene Flasks

1. Add 20 µL of a 5.0 mg/L Ti 435-d_3 internal standard solution to each of the samples and shake the samples well.
2. Transfer 1 mL of the content of the well mixed 250 mL polypropylene flasks into a HPLC vial and subjected to the MS/MS procedure.

6.1.2 Passive Dust Collectors

1. Add 100 mL of acetonitrile/water (1/4, v/v) and 20 µL of a 5.0 mg/L Ti 435-d_3 internal standard to each of the passive dust collectors placed in a plastic container and put the containers for approx 5 min. into an ultra sonic bath.
2. Transfer 1 mL of the content of the well mixed plastic container into a HPLC vial and subjected to the MS/MS procedure.

Appendix 9 (continued)

6.1.3 Liquid chromatography and MS/MS-Determination

An aliquot of the solution from step 3 is injected into the high performance liquid chromatograph, chromatographed under gradient reversed phase conditions and detected by Tandem Mass Spectrometry with electrospray ionization.

6.1.4 HPLC Conditions

Example for HPLC conditions for the determination of clothianidin.

Instrument: HP 1200
Injector: HTS PAL, CTC Analytics
Column: Phenomenex, Luna C18 (2) - HTS, 2.5 µm, 50 x 2.0 mm i.d.
Injection Volume: 25 µL
Oven temperature: 60° C
Mobile Phase: A: Water/Methanol (90/10, v/v) + 10 mmol Ammonium Formate/L + 120 µL Formic Acid/L
B: Water/Methanol (10/90, v/v) + 10 mmol Ammonium Formate/L + 120 µL Formic Acid/L

Time Table:

Time [min]	A [%, v/v]	B [%, v/v]
0.00	80	20
0.10	80	20
1.30	10	90
1.50	10	90
1.51	80	20
2.00	80	20

Stoptime: 2.00 min
Flow (Column): 0.80 mL/min
Flow (into MS): 0.80 mL/min
Retention times: Clothianidin, d_3-TI 435: approx. 0.7 min

Note: Different HPLC conditions were used for the determination of clothianidin. This is only one example for the determination. All other conditions are similar and were documented in the raw data.

Appendix 9 (continued)

6.1.5 Detection

The detection by MS/MS was performed on a triple-quadrupole tandem mass spectrometer, equipped with a Turbo Ion Spray (ESI) interface operated in the positive ion mode under MRM conditions. Unit mass resolution was established and maintained in each mass resolving quad by maintaining a full width at half-maximum of approx. 0.7 DA.

Table 1: General Mass Spectrometer Operating Key Parameters*

Triple Quadrupol LC-MS/MS, e.g. API 4000 (AB MDS Sciex)				
Ionization:	Electrospray Potential: Temperature:	(Turbo Ion Spray) 4800 V 600°C (Source)		
Polarity:	Positive			
Scan Type:	MRM-Mode (Multiple Reaction Monitoring Mode)			
Resolution:	Q1: Unit, Q3: Unit (Unit denotes 0.6 – 0.8 amu full width at half maximum FWHM)			
Software:	Analyst 1.4.1 application software run under Windows XP			
Gas Settings*:	Nebulization Gas (Gas 1): Turbo Gas (Gas 2): Curtain Gas (CUR): Collision Gas (CAD):	60 80 25 6	Nitrogen 5.0 Nitrogen 5.0 Nitrogen 5.0 Nitrogen 5.0	

Table 2: Test Item Depending Mass Spectrometer Operating Key Parameters*

Test item	Precursor Ion	Precursor Ion Q1 Mass (amu)	Product Ion Q3 Mass (amu)	Dwell Time (msec)	Collision Energy (eV)
Clothianidin 1st MRM	$[M+H]^+$	250	169	75	19
Clothianidin 2nd MRM		250	132	75	21
d_3-TI 435		253	172	100	19

The Limit of Quantitation (LOQ) for clothianidin, defined as the lowest validated fortification level, was 0.02 µg a.s./Petri dish or 0.014 g a.s./ha and 0.02 µg a.s./passive dust collector. The LOD was estimated from the linearity data and from the control samples and was 0.006 µg a.s./Petri dish or 0.004 g a.s./ha and 0.006 µg a.s./passive dust collector.

Data on method validation are given in Chapter 6.2.

Note: Different MS/MS conditions were used for the determination of clothianidin. This is only one example for the determination. All other conditions are similar and were documented in the raw data.

Appendix 9 (continued)

6.2 Method Validation

The analytical method was validated by running concurrent recoveries at the LOQ and 10-fold LOQ. Recovery experiments were done by spiking control samples with a defined amount of clothianidin. Fortification levels and recovery data are given in Table 3. In deviation to SOP 1550 only one recovery set (control, 2 x LOQ and 2 x 10 x LOQ) was prepared for the samples from the Petri Dishes and one recovery set for the passive dust collectors. This has no impact of the quality of the study.

Table 3: Recovery Data for **Clothianidin** in Acetonitrile/Water Solutions from Petri Dishes and Passive Dust Collectors; FL: Fortification Level

Sample Material	FL [µg-absolute]	Recoveries [%] (Single Values)		Mean per FL [%]	RSD per FL [%]	Mean overall [%]	RSD overall [%]
Petri dishes	0.02	96	97	97	0.7	98	2.2
	0.20	98	101	100	2.1		
Passive Dust Collectors	0.02	102	109	106	4.7	103	4.8
	0.20	97	103	100	4.2		

RSD: Relative Standard Deviation, LOQ: Practical Limit of Quantitation.

The individual recovery values for clothianidin ranged from 96 to 109% with mean recoveries of 98% (Petri dishes) and 103% (passive dust collectors) and relative standard deviations (RSD's) of 2.2 (Petri dishes) and 4.8% (passive dust collectors) (n = 4, each). All results of the method validation were in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Representative chromatograms are included in Appendix 1.

Appendix 9 (continued)

6.3 Analytical Results

Table 4 gives an overview about the residue concentration of clothianidin in the analysed acetonitrile/water solutions from the Petri dishes.

Table 4: Clothianidin residue values Kverneland Monopill S SE (Machine Z).

	Single values, Series A		Single values, Series B		Single values, Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]	[g a.s./ha]	[µg a.s. / Petri-dish]	[g a.s./ha]
1 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-7	< LOD	< LOD	< LOQ	< LOQ	< LOD	< LOD
1 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-5	0.042	0.029	< LOD	< LOD	< LOD	< LOD
3 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
3 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-4	< LOD	< LOD	< LOQ	< LOQ	< LOD	< LOD
5 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Appendix 9 (continued)

6.3 Analytical Results (contd)

Table 4: Clothianidin residue values Kverneland Monopill S SE (Machine Z) (contd).

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]
10 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
10 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-1	< LOD	< LOD	< LOQ	< LOQ	< LOD	< LOD
20 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-3	< LOQ	< LOQ	< LOD	< LOD	< LOD	< LOD
20 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30 meter-10	< LOD	< LOQ	< LOD	< LOD	< LOD	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Appendix 9 (continued)

6.3 Analytical Results (contd)

Table 4: Clothianidin residue values Kverneland Monopill S SE (Machine Z) (contd)

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]
50 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Appendix 9 (continued)

6.3 Analytical Results (contd)

Table 5 gives an overview about the residue concentration of clothianidin in the analysed glycerol/water solutions from the Petri dishes, 24 h secondary drift.

Table 5: Clothianidin residue values Kverneland Monopill S SE (Machine Z), 24 h Secondary Drift.

	Single values. Series A		Single values. Series B		Single values. Series C	
	[µg a.s. / Petri-dish]	[g a.s. / ha]	[µg a.s. / Petri-dish]	[g a.s./ha]	[µg a.s. / Petri-dish]	[g a.s./ha]
1 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-2	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
1 meter-10	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-1	< LOD	< LOD	< LOD	< LOD	0.052	0.035
5 meter-2	< LOD	< LOD	< LOQ	< LOQ	< LOD	< LOD
5 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-4	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-5	< LOD	< LOD	< LOD	< LOD	< LOQ	< LOQ
5 meter-6	< LOQ	< LOQ	< LOD	< LOD	< LOD	< LOD
5 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-9	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5 meter-10	< LOD	< LOD	< LOQ	< LOQ	< LOD	< LOD
50 meter-1	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-2	< LOD	< LOD	< LOD	< LOD	< LOQ	< LOQ
50 meter-3	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-4	< LOD	< LOD	0.023	0.015	< LOD	< LOD
50 meter-5	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-6	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-7	< LOD	< LOD	< LOD	< LOD	< LOD	< LOQ
50 meter-8	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
50 meter-9	< LOQ	< LOQ	< LOD	< LOD	< LOD	< LOD
50 meter-10	< LOD	< LOD	0.034	0.023	< LOD	< LOD

LOQ = 0.02 µg a.s./Petri Dish; LOD = 0.006 µg a.s./ /Petri Dish
LOQ = 0.014 g a.s./ha; LOD = 0.004 g a.s./ha; surface area of Petri-dish: 147.41 cm²

Note: for the calculation of the values in [g/ha] non-rounded values in [µg a.s./Petri-dish] were used.

Appendix 9 (continued)

6.3 Analytical Results (contd)

Table 6 gives an overview about the residue concentration of clothianidin in the analysed acetonitrile/water solutions from the passive dust collectors.

Table 6: Clothianidin residue values Kverneland Monopill S SE (Machine Z). Passive Dust Collectors

	Single values. Series A	Single values. Series B	Single values. Series C
	[µg a.s. / Pas. Sampler]	[µg a.s. / Pas. Sampler]	[µg a.s. / Pas. Sampler]
5 m distance 5 m above ground-1	0.037	0.031	< LOQ
5 m distance 4 m above ground-1	< LOQ	0.086	< LOQ
5 m distance 3 m above ground-1	0.028	0.169	0.072
5 m distance 2 m above ground-1	0.045	0.184	< LOQ
5 m distance 1 m above ground-1	0.040	0.054	0.083
5 m distance 5 m above ground-2	0.169	0.032	< LOQ
5 m distance 4 m above ground-2	0.024	0.061	0.034
5 m distance 3 m above ground-2	0.020	0.092	0.035
5 m distance 2 m above ground-2	0.098	0.102	< LOQ
5 m distance 1 m above ground-2	0.046	0.050	0.060
30 m distance 5 m above ground-1	0.027	0.160	< LOQ
30 m distance 4 m above ground-1	0.028	0.158	< LOQ
30 m distance 3 m above ground-1	0.072	< LOQ	< LOQ
30 m distance 2 m above ground-1	0.164	0.032	< LOQ
30 m distance 1 m above ground-1	< LOQ	0.077	< LOQ
30 m distance 5 m above ground-2	0.230	0.350	< LOQ
30 m distance 4 m above ground-2	< LOQ	0.290	0.021
30 m distance 3 m above ground-2	0.029	0.055	0.043
30 m distance 2 m above ground-2	0.044	0.197	0.035
30 m distance 1 m above ground-2	< LOQ	0.178	0.022

LOQ = 0.02 µg a.s./Passive Dust Collector; LOD = 0.006 µg a.s./Passive Dust Collector

Appendix 9 (continued)

7 Evaluation and Discussion

The aim of the study was to measure the deposition rate of clothianidin-containing seed treatment particles (g a.s./ha) at various distances from a field drilled with Poncho® Beta Plus treated sugar beet pills during sowing with a typical mechanical sugar beet in acetonitrile/water and glycerol/water out of Petri dishes and from passive dust collectors.

The field part of this study (collecting of the solvent of the Petri dishes, and the passive dust collectors) as well as the transport of the samples to the laboratory of [redacted] was not conducted under GLP. Therefore nothing will be documented in the RAW data of this GLP part. This will be part of the final report.

All clothianidin-containing dust and abrasion particles which deposited at 1, 3, 5, 10, 20, 30 and 50 meters distance from the drilling area were sampled in polystyrene Petri-dishes (Ø 13.7 cm. 147.41 cm²), filled with an acetonitrile-water mixture (2/8. v/v). For each sampling distance 3 arrays of 10 Petri-dishes each were installed with a distance of 1 meter between the dishes. Accordingly, a total of 30 samples were yielded for each sampling distance.

After the drilling was completed (i.e. after approximately 30 minutes for the 12-row mechanical planting machine), an additional waiting period of 15 minutes was employed to allow those dust particles which had not yet been deposited to settle on the sampling area. Thereafter, the acetonitrile-water mixture of each Petri-dish was quantitatively transferred into a 250 mL polyethylene flask by means of a polyethylene funnel. Both, the Petri-dish and the funnel were rinsed with acetonitrile/water (2/8. v/v) and the rinse was combined with the content of the respective Petri-dish inside the 250 mL polyethylene flask before being tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 meter distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with machine ID-code ("Machine Z"). the number of the respective series (A. B or C), the distance from the drilling area (1, 3, 5, 10, 20, 30 or 50 m) and the number of the respective Petri-dish per distance (1-10), giving in total 210 flasks.

In all series (A. B and C) at 5 and 30 m distance from the 'zero-line', respectively, passive dust-drift collectors made of polypropylene have been installed at 1 m, 2 m, 3 m, 4 m and 5 m above the soil surface. At each end of a horizontal pole in the respective height, one passive collector has been installed, giving in total 6 passive collectors per height per distance. Once the 30 m - line and later on, the 5 m - line has been reached during the sampling process of the Petri-dishes (filled with acetonitrile/water. 2/8. v/v; see above), the passive dust collectors were sampled and placed into plastic containers which were immediately sealed and labelled in the field. In the laboratory, each individual passive dust collector has been extracted with an appropriate volume of acetonitrile/water (2/8. v/v) within an ultrasonic bath to enhance the extraction process.

Appendix 9 (continued)

7 Evaluation and Discussion (contd)

After sampling of all 210 Petri-dishes (filled with acetonitrile/water. 2/8. v/v) and all 60 passive dust collectors per machine was completed, new Petri-dishes were disposed in all three series (A. B. C) at the 1 m, 5 m and 50 m distance from the 'zero-line', giving per machine another 90 Petri-dishes. This second installation of Petri-dishes was filled with about 100 mL of a glycerol/water mixture (1/1. v/v) and was exposed (lids open) for a period of 24 h in order to quantify the amount of clothianidin which may enter the Petri-dishes via secondary drift processes.

After 24 h. the content of each Petri-dish was quantitatively transferred into 250 mL polyethylene flask by means of a polyethylene funnel. Both, the Petri-dish and the funnel were rinsed with water and the rinse was combined with the content of the respective Petri-dish inside the 250 mL polyethylene flask before being tightly closed with its corresponding polypropylene screw cap. Each Petri-dish and each funnel was only used once before being ultimately discarded. Sampling always started at the 50 meter distance and proceeded towards the drilling area in order to avoid cross-contamination. Each polyethylene flask was unequivocally labelled with machine ID-code ("Machine Z"), the number of the respective series (A. B or C), the distance from the drilling area (1, 5 or 50 m) and the number of the respective Petri-dish per distance (1-10), giving in total 90 flasks.

At the day of drilling / sampling (August 20, 2008), all 210 polypropylene flasks (acetonitrile/water. 2/8. v/v) together with all 60 passive dust collectors were transported by car to the laboratory [redacted] at Bayer CropScience AG's Institute for Residues, Operator and Consumer Safety (BCS-D-ROCS). The following day (August 21, 2008), the 90 polypropylene flasks (glycerol/water. 1/1. v/v) were also transported by car to the laboratory [redacted] (BCS-D-ROCS). All samples were analysed for their clothianidin content according to methods 00554/M001.

To the content of the 250 mL polyethylene bottles 20 µL of a 5.0 mg/L Ti 435-d_3 internal standard solution was added to each of the samples and shaked well. 1 mL of the content of the well mixed 250 mL polyethylene bottles were transferred into a HPLC vial and subjected to the MS/MS procedure. For the analysis of the passive dust collectors 100 mL of acetonitrile/water (1/4. v/v) and 20 µL of a 5.0 mg/L Ti 435-d_3 internal standard solution was added to each plastic container. The samples were placed in an ultrasonic bath and an aliquot of 1 mL was transferred into a HPLC vial and subjected to the MS/MS procedure.

The Limit of Quantitation (LOQ) for clothianidin, defined as the lowest validated fortification level, was 0.02 µg a.s./Petri dish or 0.014 g a.s./ha and 0.02 µg a.s./passive dust collector. The LOD was estimated from the linearity data and from the control samples and was 0.006 µg a.s./Petri dish or 0.004 g a.s./ha and 0.006 µg a.s./passive dust collector.

The individual recovery values for clothianidin ranged from 96 to 109% with mean recoveries of 98% (Petri dishes) and 103% (passive dust collectors) and relative standard deviations (RSD's) of 2.2 (Petri dishes) and 4.8% (passive dust collectors) (n = 4, each). All results of the method validation were in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Appendix 9 (continued)

7 Evaluation and Discussion (contd)

For the sowing machine tested, residues of clothianidin were between < LOD (0.006 g a.s./ha) and 0.029 g a.s./ha in the acetonitrile/water solutions from the Petri dishes, between < LOD and 0.035 g a.s./ha in the glycerol/water solutions from the Petri dishes collected after 24 h and from < LOQ (0.02 µg a.s./passive dust collectors) to 0.350 µg absolute a.s. on the passive drift collectors.

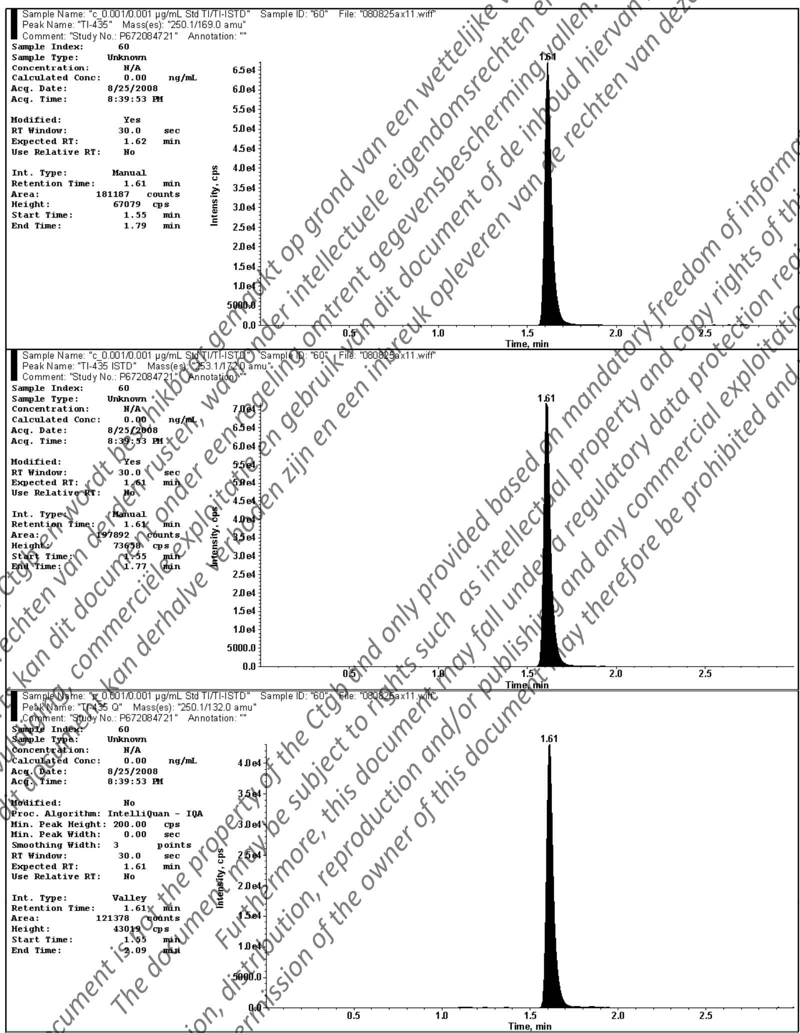
For detail information see Table 4 to Table 6.

Appendix 9 (continued)

Appendix 1: Representative Chromatograms

Clothianidin

Figure 1 : Top: Standard Solution 1.0 µg/L Clothianidin 1st MRM; Middle: 1 µg/L d_3-Clothianidin; Bottom: Standard Solution 1.0 µg/L Clothianidin 2nd MRM

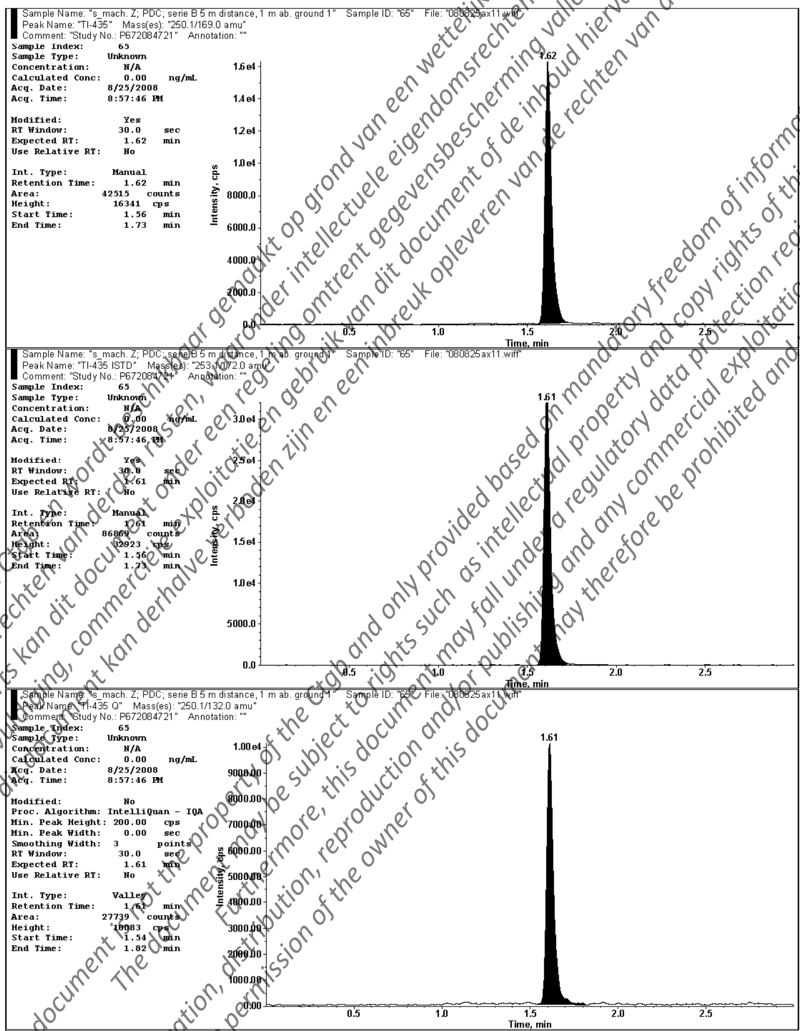


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 2 : Top: Machine Z. Passive Dust Collector, 5 m Distance, 1 m Above the Ground, Clothianidin 1st MRM; Middle: 1 µg/L d_3-Clothianidin; Bottom: Machine Z, Passive Dust Collector, 5 m Distance, 1 m Above the Ground, Clothianidin 2nd MRM

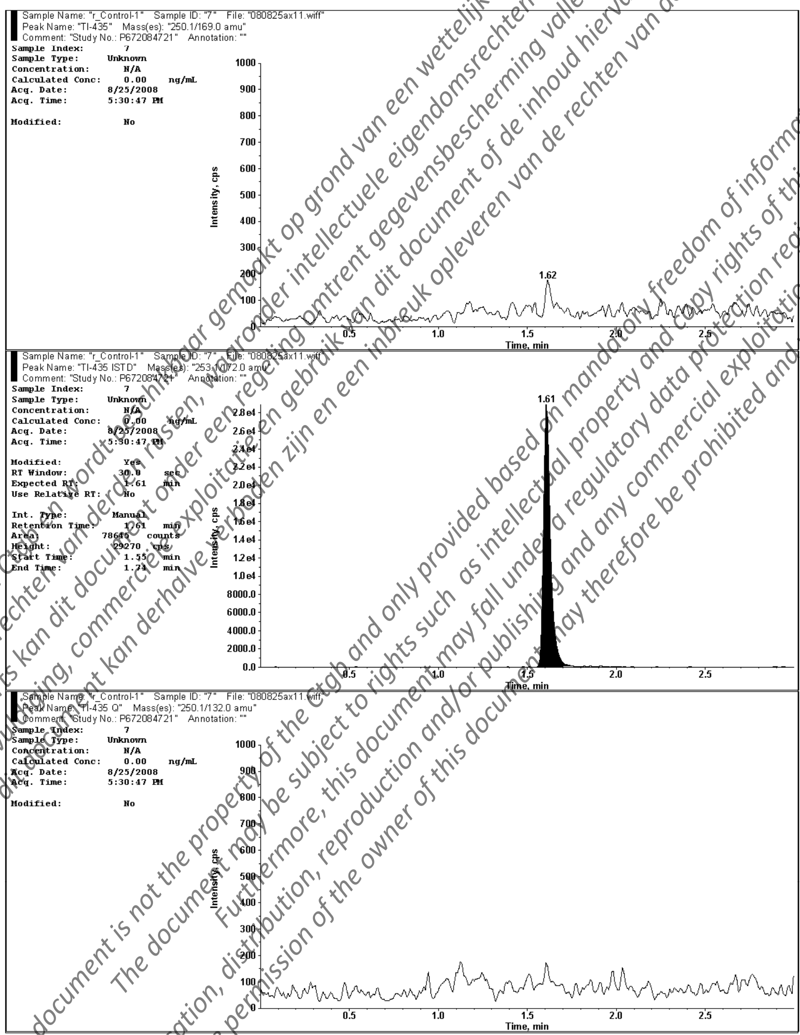


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 3 : Top: Control Passive Dust Collector for Recovery Sample, Clothianidin 1st MRM; Middle: 1 µg/L d_3-Clothianidin; Bottom: Control Passive Dust Collector for Recovery Sample, Clothianidin 2nd MRM

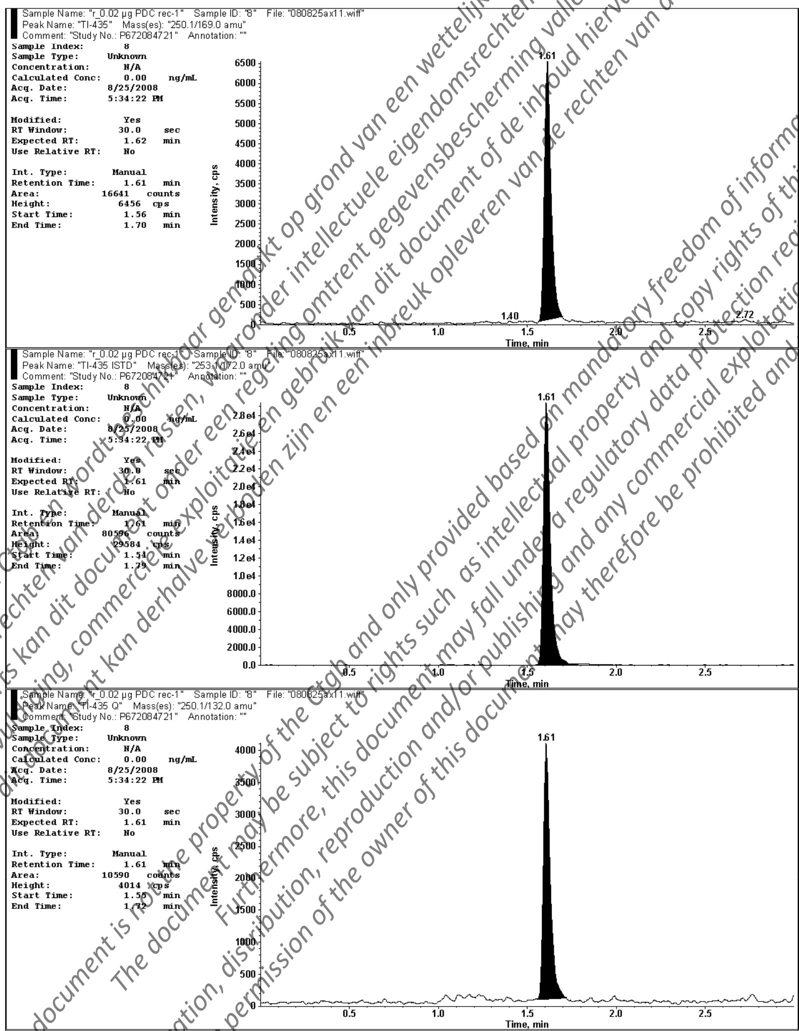


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 4 : Top: Recovery Sample 0.02 µg/Passive Dust Collector, Clothianidin 1st MRM; Middle: 1 µg/L d_3-Clothianidin; Bottom: Recovery Sample 0.02 µg/ Passive Dust Collector Clothianidin 2nd MRM

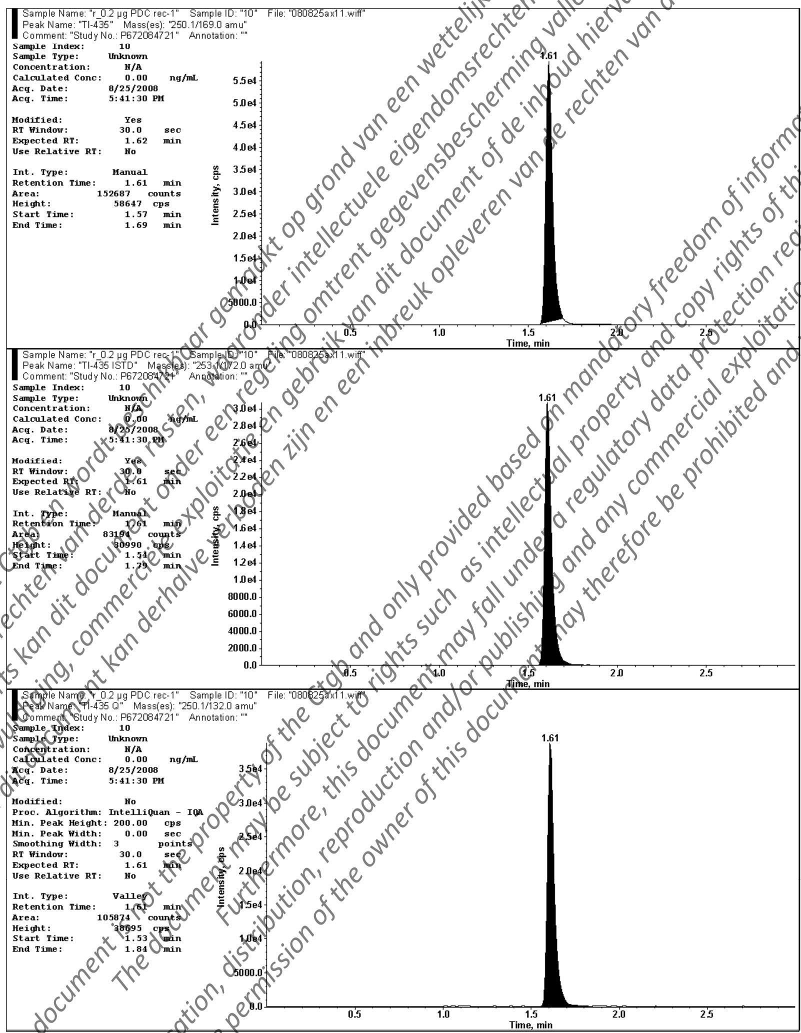


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 5 : Top: Recovery Sample 0.2 µg/Passive Dust Collector, Clothianidin 1st MRM; Middle: 1 µg/L d_3-Clothianidin; Bottom: Recovery Sample 0.2 µg/ Passive Dust Collector Clothianidin 2nd MRM

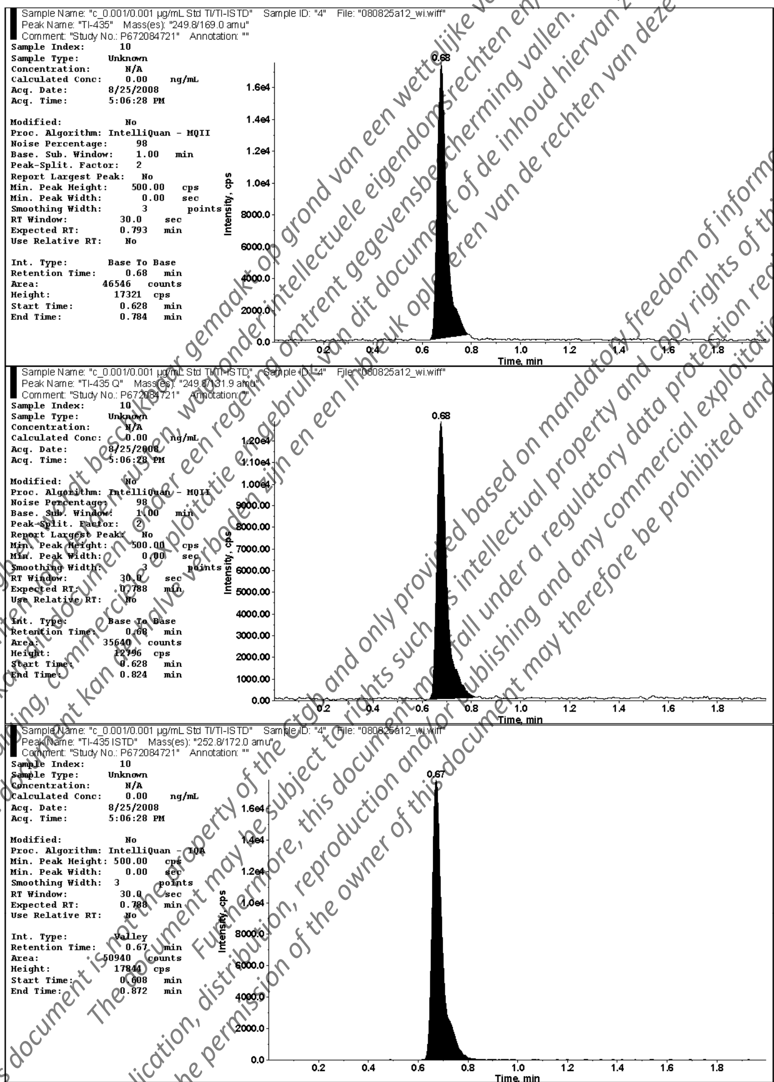


Appendix 9 (continued)

Appendix 1: Representative Chromatograms (contd)

Clothianidin

Figure 6 : Top: Standard Solution 1.0 µg/L Clothianidin, 1st MRM; Middle: Standard Solution 1.0 µg/L Clothianidin, 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

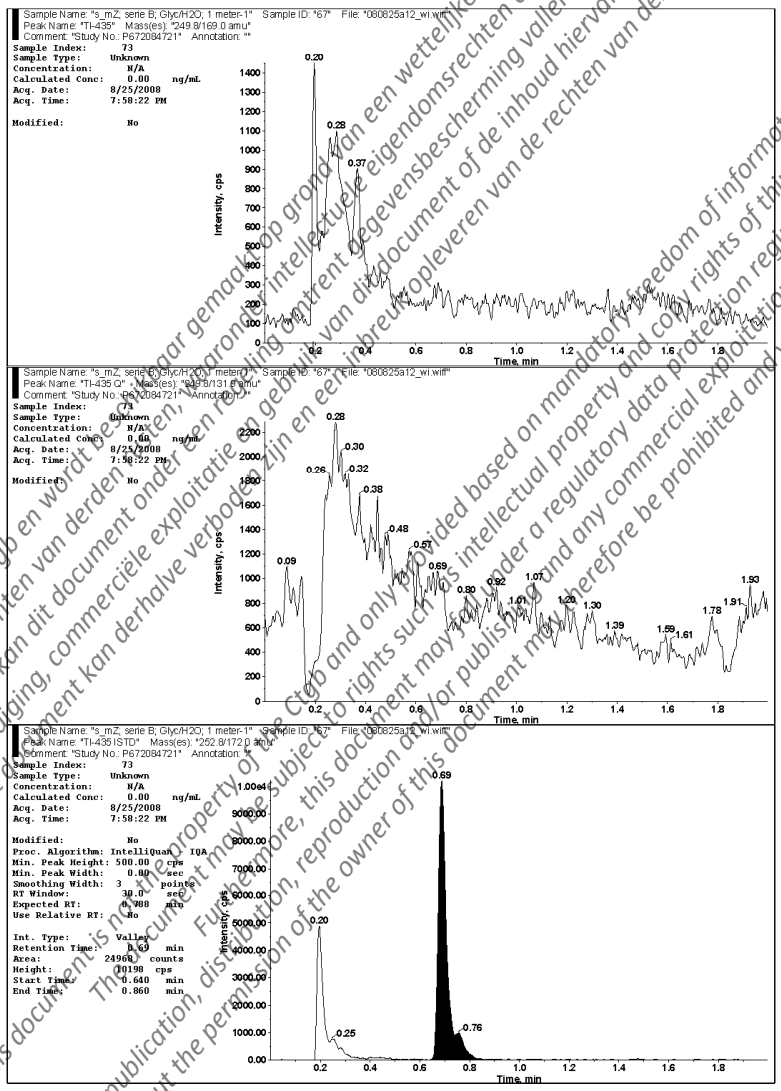


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 7 : Top: Machine Z, Petri Dish 24 h, Series B, 1 m Distance, Petri Dish 1, Clothianidin 1st MRM; Middle: Machine Z, Petri Dish 24 h, Series B, 1 m Distance, Petri Dish 1, Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

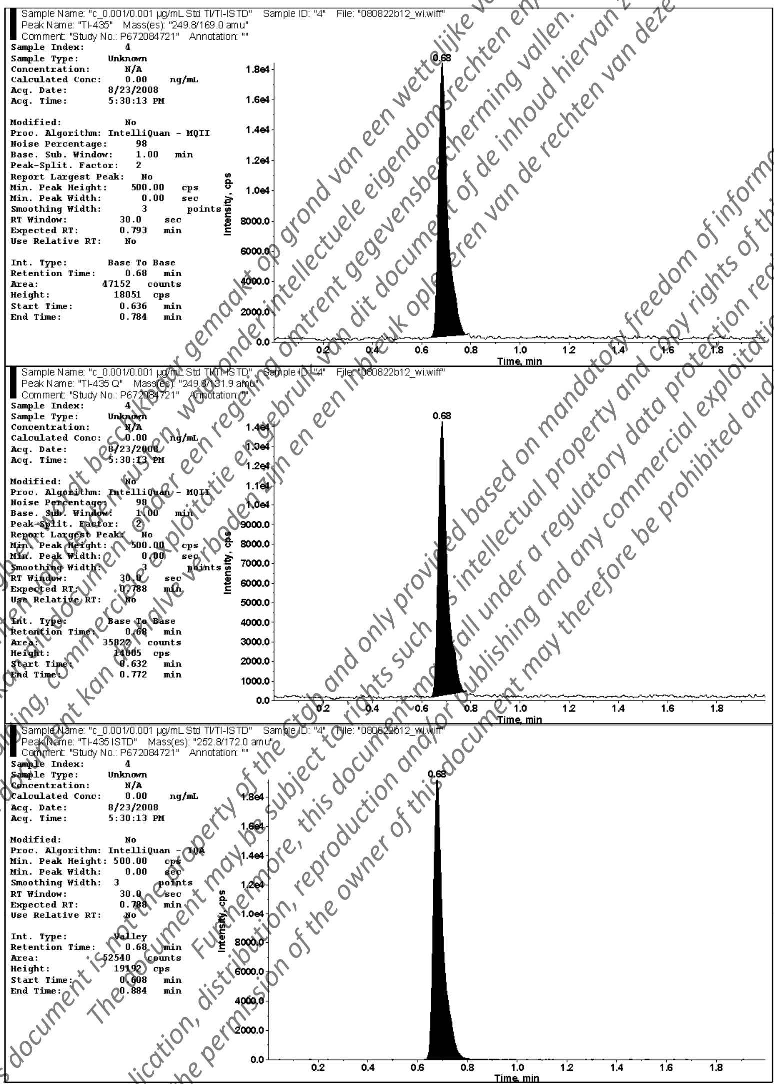


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 8 : Top: Standard Solution 1.0 µg/L Clothianidin 1st MRM; Middle: Standard Solution 1.0 µg/L Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

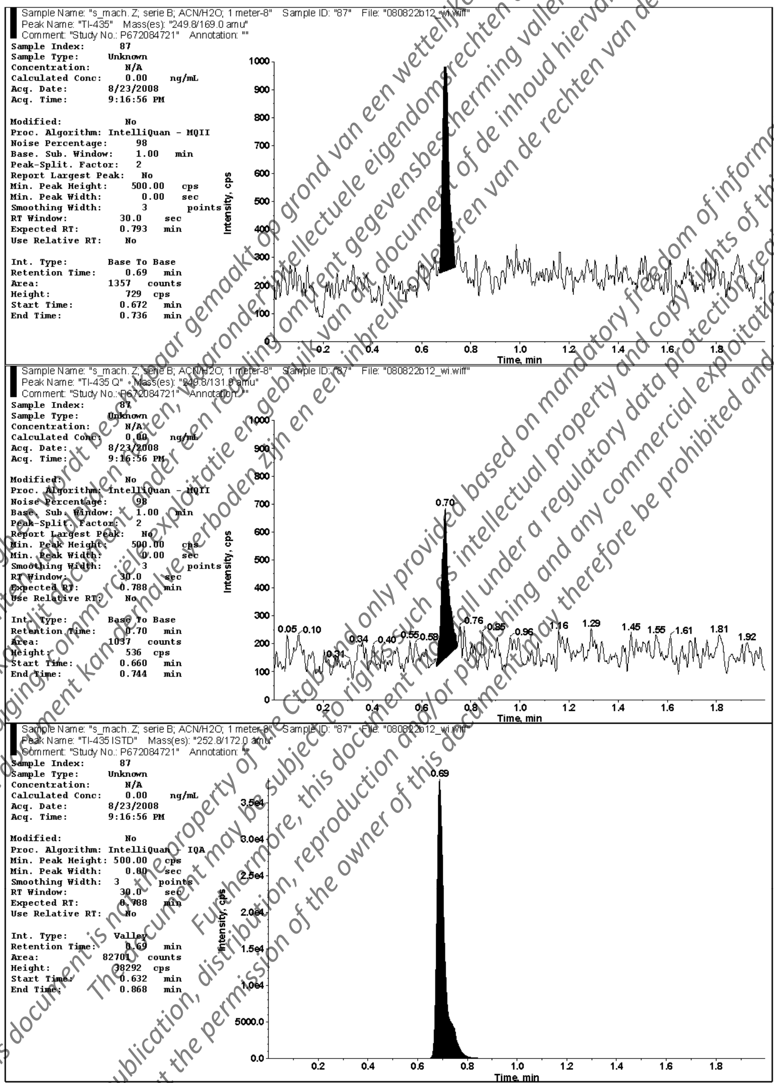


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 9 : Top: Machine Z, Petri Dish, Series B, 1 m Distance, Petri Dish 8, Clothianidin 1st MRM; Middle: Machine Z, Petri Dish, Series B, 1 m Distance, Petri Dish 8, Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

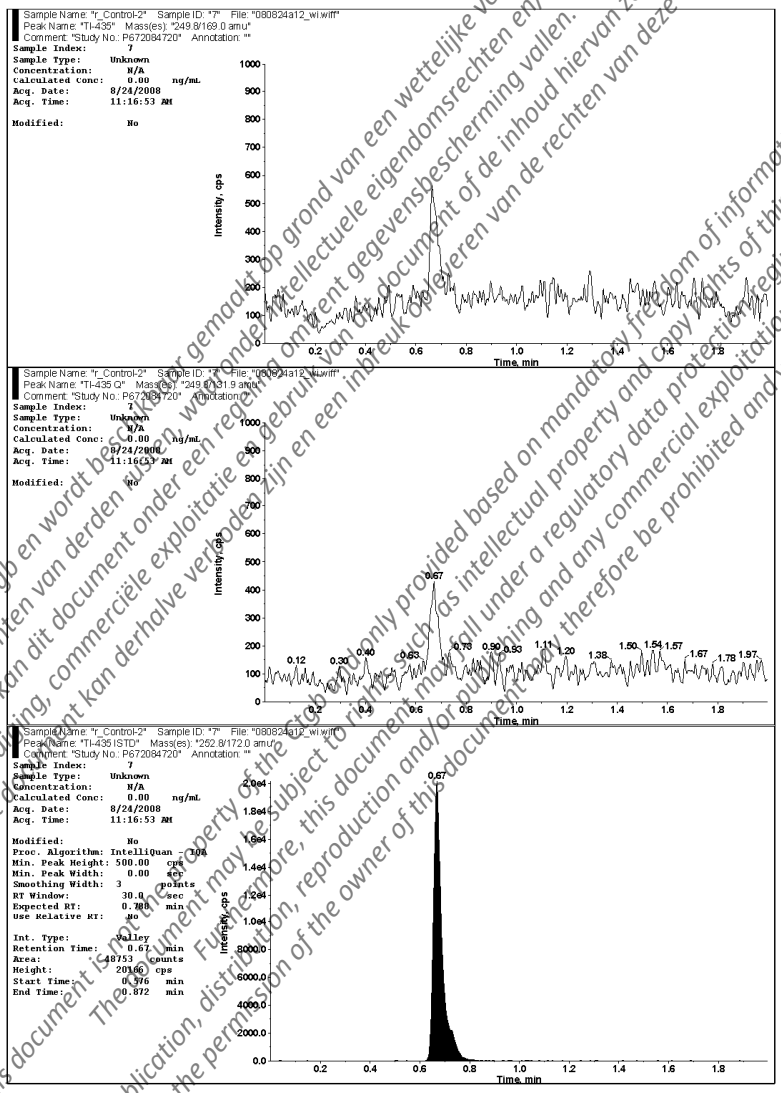


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 10 : Top: Control Petri Dish for Recovery Sample, Clothianidin 1st MRM; Middle: Control Petri Dish for Recovery Sample, Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

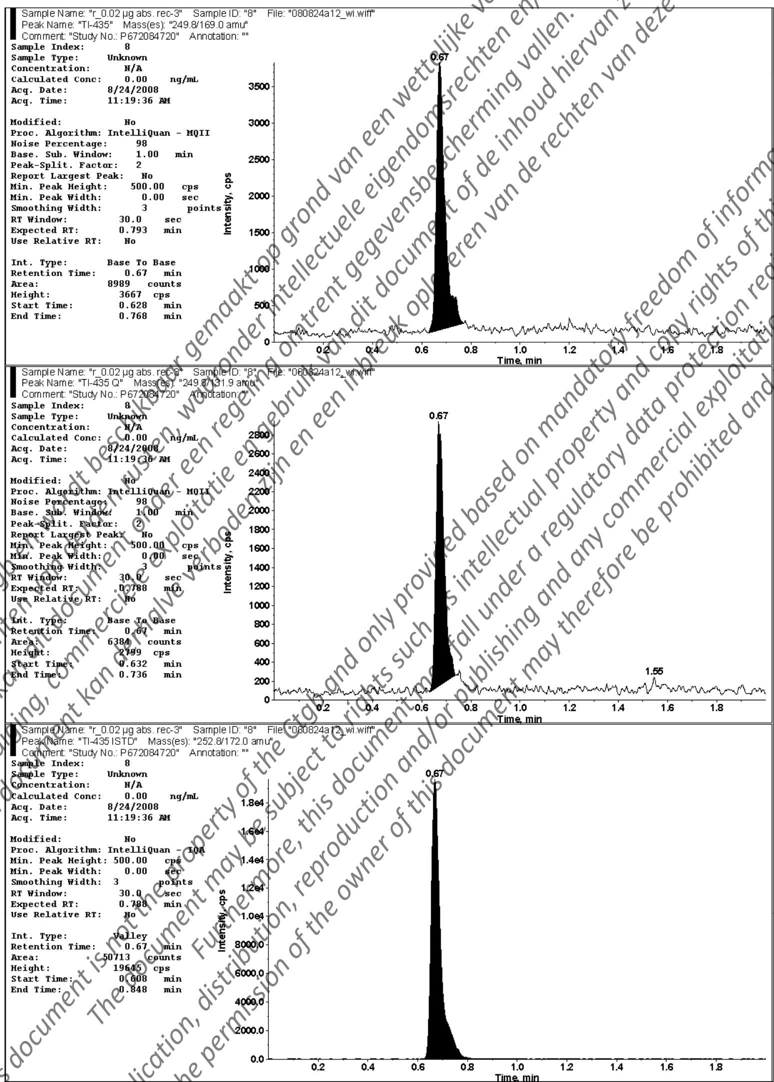


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 11 : Top: Recovery Sample 0.02 µg/Petri Dish, Clothianidin 1st MRM; Middle: Recovery Sample 0.02 µg/ Petri Dish, Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin

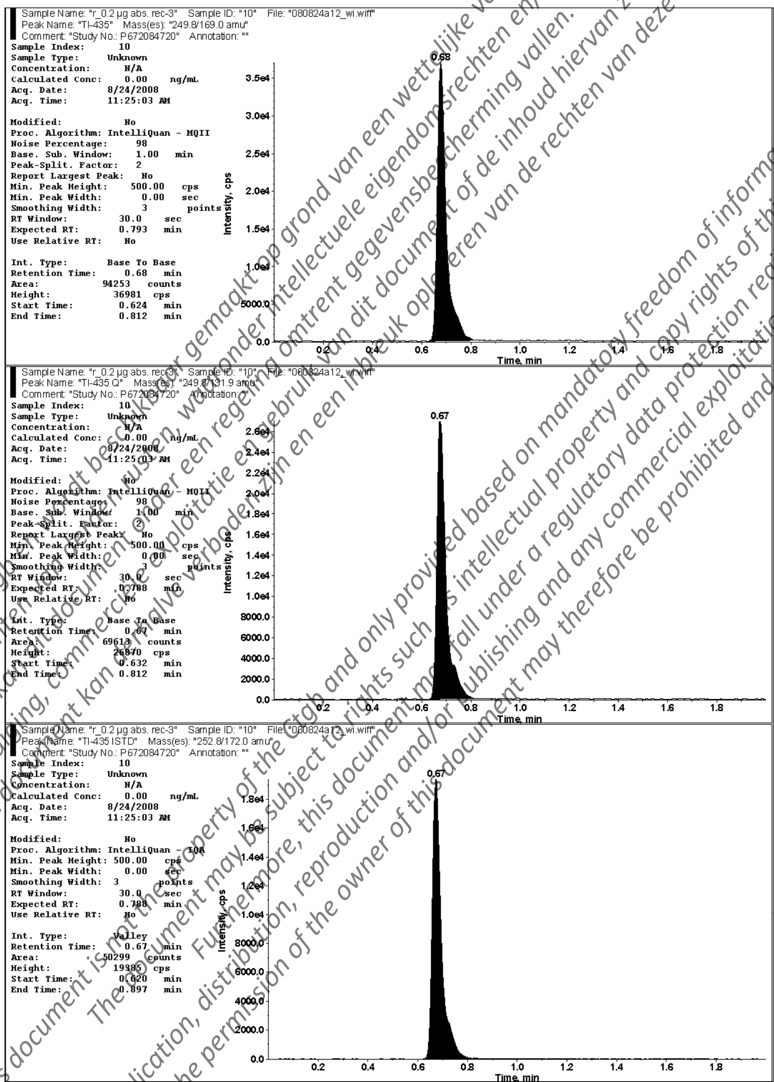


Appendix 9 (continued)

Appendix 1:
Representative Chromatograms (contd)

Clothianidin

Figure 12 : Top: Recovery Sample 0.2 µg/Petri Dish, Clothianidin 1st MRM; Middle: Recovery Sample 0.2 µg/Petri Dish, Clothianidin 2nd MRM; Bottom: 1 µg/L d_3-Clothianidin



Appendix 9 (continued)

Appendix 2:
GLP Certificate

[redacted]

Appendix 9 (continued)

Appendix 2
GLP Certificate (contd)