Fusarium toxins and ergot alkaloids in wheat and rye of the Federal State of Brandenburg harvested 2013

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INTRODUCTION

For many years, the IGV Institute of Cereal Processing, Ltd. has been concerned with the analysis of wheat and rye samples of integrated and organic cultivation as regards the components used, dough rheology, and milling and baking properties. During the years 2000 to 2013, the samples were analyzed additionally for Fusarium toxins in order to collect data on the contamination situation of the federal state Brandenburg over a period of several years. Since 2012 only wheat samples were analysed for Fusarium toxins whereas rye samples were analysed for ergot alkaloids. The analyses had the further aim to confirm differences between samples of organic and integrated cultivation with regard to the contamination by mycotoxins.

MATERIAL AND METHODS

During this period, appr. 60 wheat and 60 rye samples were analyzed in each year, these were collected from farmers of all the agricultural districts of the federal state, with special consideration of organic cultivation. The Fusarium toxins DON, zearalenone, nivalenol, T-2 and HT-2 toxins, 3-Acetyl-DON, 15-Acetyl-DON, DON-3-glucoside, DAS and fusarenon-X were analyzed by means of LC-MS/MS (2000-2008: HPLC). The twelve ergot alkaloids ergometrine, ergometrinine, ergosine, ergosinine, ergotamine, ergotaminine, ergocornine, ergocorninine, ergokryptine, ergokryptinine, ergocristine, ergocristinine were analysed using HPLC according to the BVL-method L 15.01/02-5:2012-01. The number of samples per agricultural district was determined by the total harvest yield. As with regard to the harvest yield, the number of samples of organic cultivation would have been too small for reliable conclusions, therefore the quantity of the samples of organic cultivation was increased. The proportion of samples of organic cultivation over the twelve years amounted to 10-16 % for wheat and 18-27 % for rye. Before analysis, impurities were removed from the samples. A certain part of each sample was ground for the determination of the components, quality parameters and mycotoxins. The larger part of the samples was used for the determination of the milling and baking properties.

Analysis by LC-MS/MS

The samples were extracted by shaking with acetonitrile/water (84:16, v/v) for 1 h. An aliquot of the filtered extract was mixed with internal standards (^{13}C-marked) and purified with the aid of Bond Elut Mycotoxin columns. The purified extract was concentrated to dryness by rotary evaporation at app. 50 °C, redissolved in HPLC eluent and injected to LC-MS/MS (API 4000 mass spectrometer, AB Sciex). Limits of quantification: DON/Nivalenol/DON-3-glucoside/ FUS-X: 15 µg/kg; zearalenone: 3 µg/kg; T-2/ HT-2 toxins, 3-/15-Ac- DON/DAS: 5 µg/kg.

For better comparability of the results analyzed by HPLC and LC-MS/MS, only concentrations of DON above 50 µg/kg and concentrations of zearalenone above 10 µg/kg were considered in the evaluation of the results.

RESULTS

Fusarium toxins:

DON was detected in wheat for almost all years of the investigation (except 2010). Over the years the measured contents of DON varied widely (see Fig.① and Tab.①). 2002, 2007 and 2012 were years with very high DON contaminations. In the other years the DON contamination was significantly lower.

2013 DON was detected in 26 % of the wheat samples. The maximum content was 2477 µg/kg, the mean value of all positive samples was 556 µg/kg (median: 290 µg/kg). ZEA was detected only in 3 % of the samples (max. 33 µg/kg). Nivalenol and DON-3-glucoside were detected both in 17 % of the samples. 3-Ac-DON, 15-Ac-DON were rarely detected. T-2-/HT-2 toxins, DAS and FUS-X were detected not at all (see Fig.②).

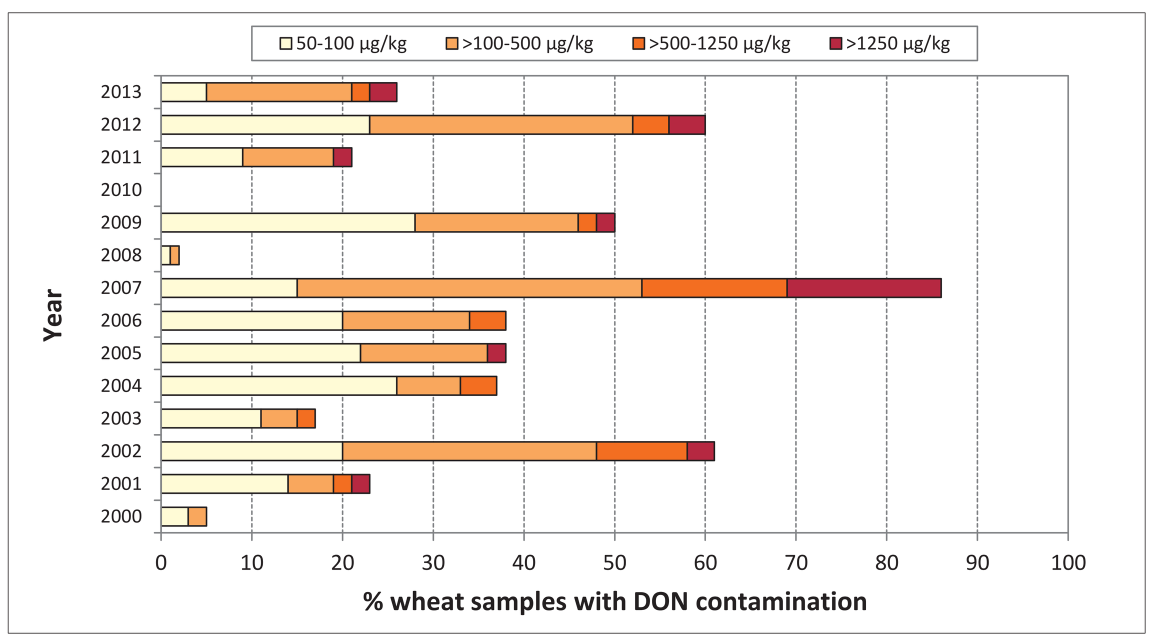

Fig.① DON in wheat samples (2000-2013)

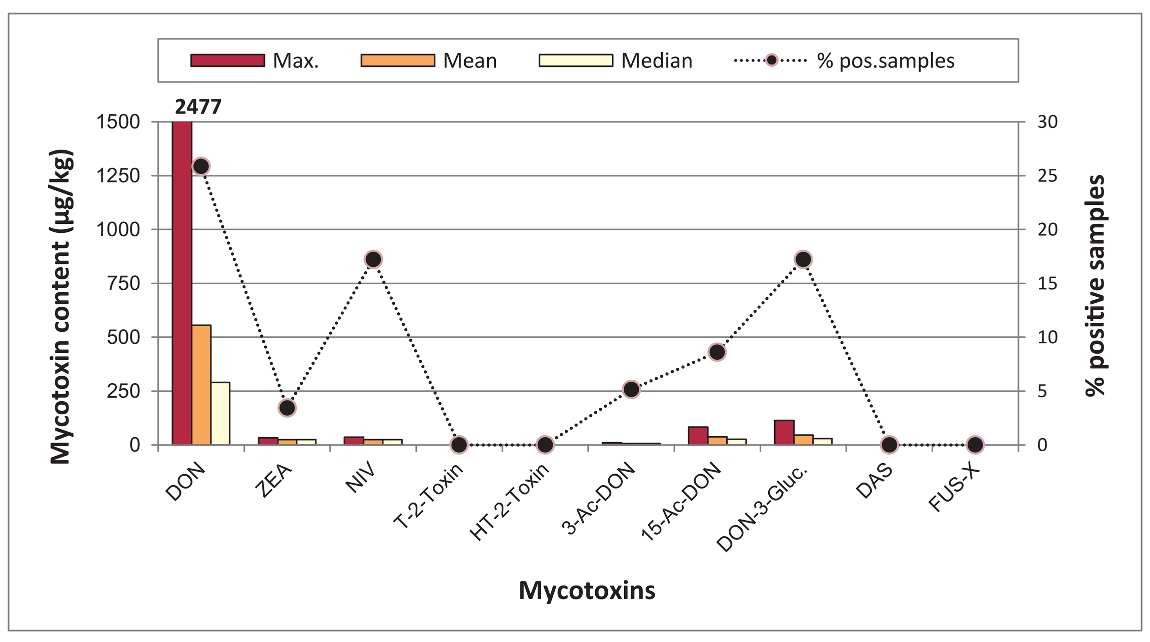

Fig.② Fusarium toxins in wheat samples (2013)

Of the cereals tested, 2 wheat samples exceeded the legal limits for DON in food cereals, both of these samples originated from integrated cultivation. Furthermore, Fig.③ and Tab.① show that, of all other cereals tested, those from integrated cultivation had higher levels of toxins than those from organic cultivation. The samples of organic cultivation were lower contaminated compared to the samples from integrated cultivation. DON was detected only in 13 % of the samples from organic cultivation (mean value/median of the positive samples: 78 µg/kg) but in 30 % of the samples from integrated cultivation (mean value of the positive samples: 629 µg/kg, median value: 409 µg/kg).

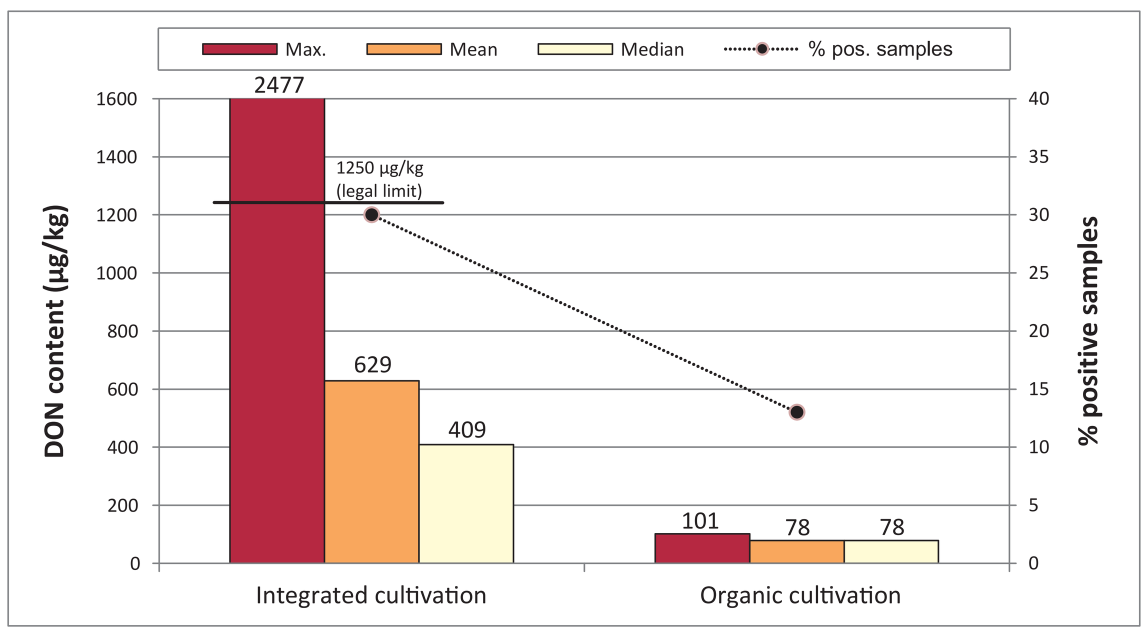

Fig.③ Maximum, mean and median of the DON content of wheat samples according to the type of cultivation (2013)

Tab.① DON and ZEA in wheat samples of integrated / organic cultivation

Year	Culti-va-tion	Ana-lyzed samp-les	DON					ZEA				
			Number of pos. samples	Content, µg/kg	Mean	Me-dian	Samples > 1250 µg/kg	Number of pos. samples	Content, µg/kg	Mean	Me-dian	Samples > 100 µg/kg
2000	int.	47	3	50 - 280	140	80	-					
	org.	16	0	(<50)	-	-	-					
2001	int.	47	12	50 - 1350	310	90	2	4	10 - 68	14	8	-
	org.	12	1	70			-	0	(<10)	-	-	-
2002	int.	46	31	50 - 4870	470	240	2	18	11 - 447	61	32	2
	org.	14	6	60 - 200	100	90	-	1	12			-
2003	int.	46	9	60 - 540	140	90	-	1	84			-
	org.	10	0	(<50)	-	-	-	0	(<10)	-	-	-
2004	int.	41	15	50 - 1120	195	70	1	0	(<10)	-	-	-
	org.	16	6	50 - 220	100	80	-	1	18			-
2005	int.	42	20	50 - 1730	212	98	1	4	11 - 105	54	51	1
	org.	13	1	87			-	0	(<10)	-	-	-
2006	int.	43	20	50 - 1018	200	88	-	0	(<10)	-	-	-
	org.	14	1	147			-	0	(<10)	-	-	-
2007	int.	43	40	50 - 10439	1211	428	10	21	10 - 451	76	37	5
	org.	15	10	69 - 782	262	133	-	3	29 - 75	45	30	-
2008	int.	44	2	50 - 287	169		-	0	(<10)	-	-	-
	org.	16	0	(<50)	-	-	-	0	(<10)	-	-	-
2009	int.	45	24	51 - 2040	251	98	1	0	(<10)	-	-	-
	org.	12	4	51 - 79	62	58	-	0	(<10)	-	-	-
2010	int.	44	0	(<50)	-	-	-	0	(<10)	-	-	-
	org.	13	0	(<50)	-	-	-	0	(<10)	-	-	-
2011	int.	45	11	50 - 3300	402	121	1	9	10 - 304	48	15	1
	org.	13	1	131			-	1	16			-
2012	int.	41	27	51 - 8250	594	210	2	5	10 - 51	34	23	-
	org.	15	6	50-385	141	90	-	0	(<10)	-	-	-
2013	int.	43	13	65 - 2477	629	409	2	2	18 - 33	26	26	0
	org.	15	2	54 - 101	78	78	-	0	(<10)	-	-	-

In rye samples DON was found over the years more rarely than in wheat samples. Transgressions of the legal limit value did not occur in rye samples. But also in rye the maximum content, mean value and median were higher in the samples of integrated cultivation than in the samples of organic cultivation. Zearalenone was detected on even fewer occasions than in wheat samples.

Ergot alkaloids:

Ergot alkaloids were detected in rye samples in 2012 very rarely. 2013 was a year with frequent ergot occurrence. Ergot alkaloids were detected in 67 % of the samples. The correlation between the content of ergot sclerotia and the content of ergot alkaloids was low (r = 0,51). The measured contents of ergot alkaloids varied widely (see Fig.④). High ergot alkaloid contents were measured. The total ergot alkaloid contents were between 10 and 4850 µg/kg. The mean value of all positive samples was 536 µg/kg (median: 237 µg/kg).

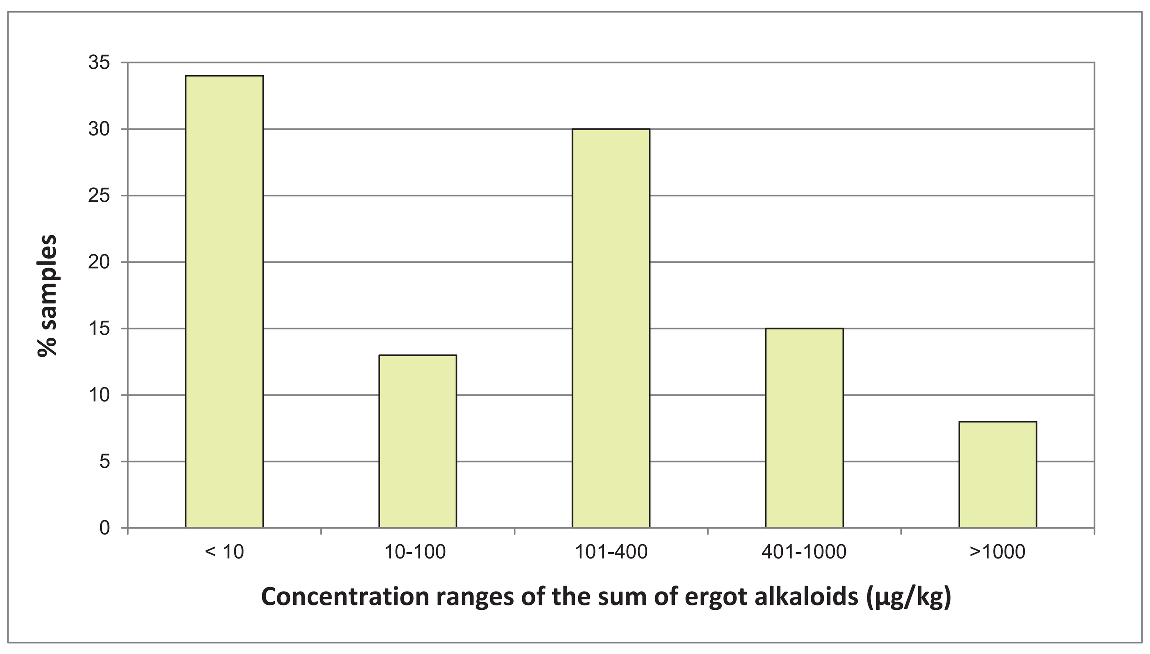

Fig.④ Ergot alkaloids in rye samples (2013)

The individual ergot alkaloids were relatively evenly distributed.
A **»leading alkaloid«** could not be found (see Fig.⑤).

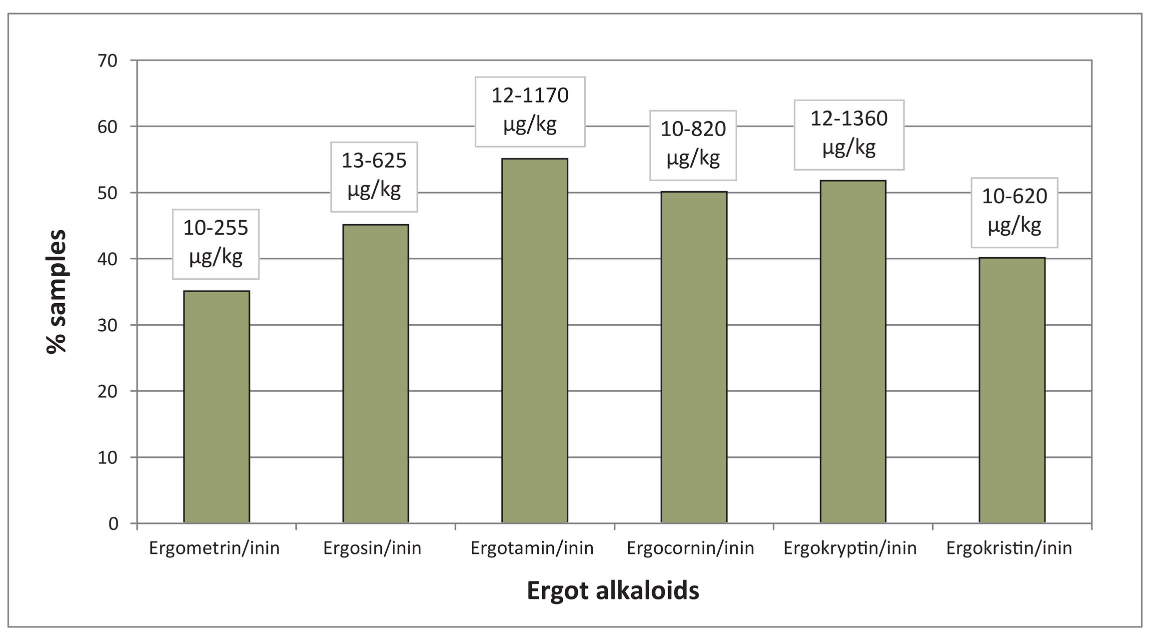

Fig.⑤ Ergot alkaloids in rye samples (2013)

In the contaminated samples from 2 to 12 ergot alkaloids were detectable (see Fig.⑥). Highly contaminated samples contained from 10 to 12 ergot alkaloids (17 respectively 18 % of all analyzed samples). In the samples with medium content from 4 to 12 ergot alkaloids were detected. Samples with very low content contained from 2 to 8 ergot alkaloids.

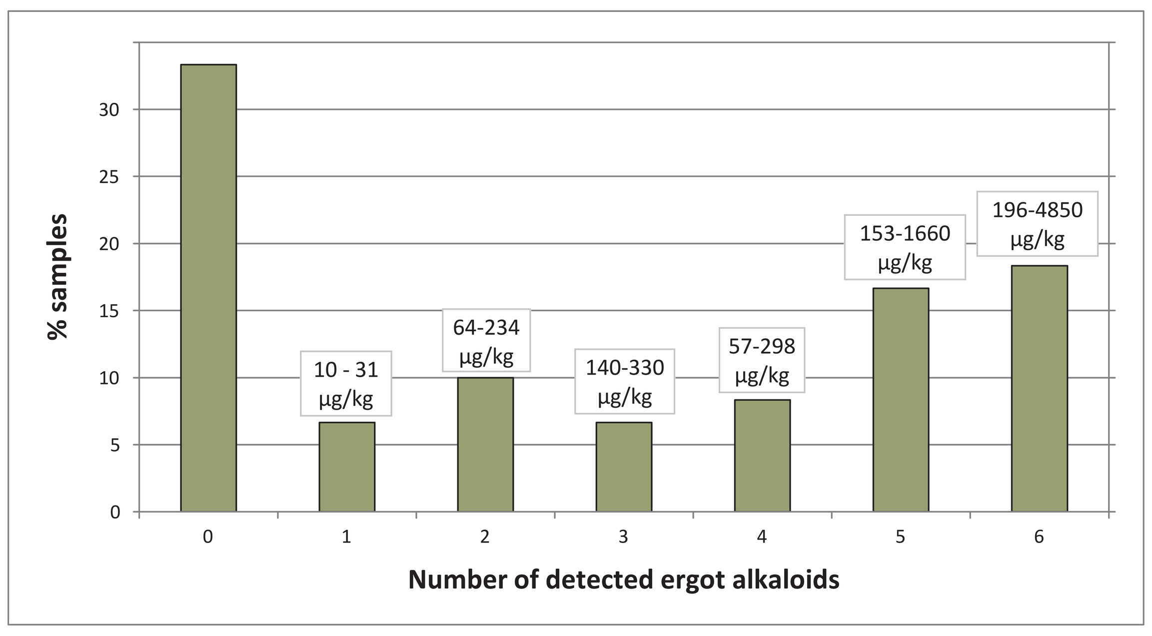

Fig.⑥ Number of detected ergot alkaloids (in+inin) in the samples

Occurrence of DON and ergot alkaloids was higher in the integrated rye samples as opposed to those collected from organic cultivation. The highest contents of ergot alkaloids were only found in samples from integrated cultivation (up to 4855 µg/kg). Ergot alkaloids were detected in 74 % of the 39 rye samples from integrated cultivation (mean value of the positive samples: 620 µg/kg, median value: 260 µg/kg). The maximum content of the samples from organic cultivation was 776 µg/kg. Only in 48 % of the 21 rye samples from organic cultivation ergot alkaloids were detected (mean value of the positive samples: 281 µg/kg, median: 179 µg/kg).

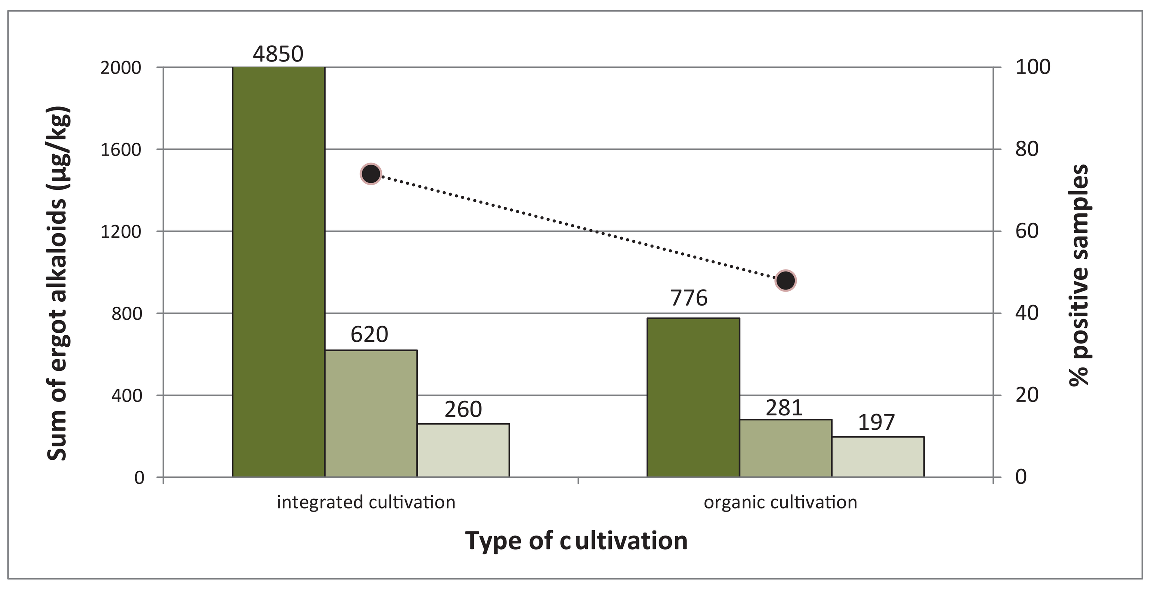

Fig.⑦ Maximum, mean and median of the ergot alkaloid content according to the type of cultivation

The kind of tillage (plough or ploughless) was found to have a greater influence on the content of ergot alkaloids, as opposed to the type of rye cultivars (population or hybrid variety) (see Fig.⑧ and ⑨).

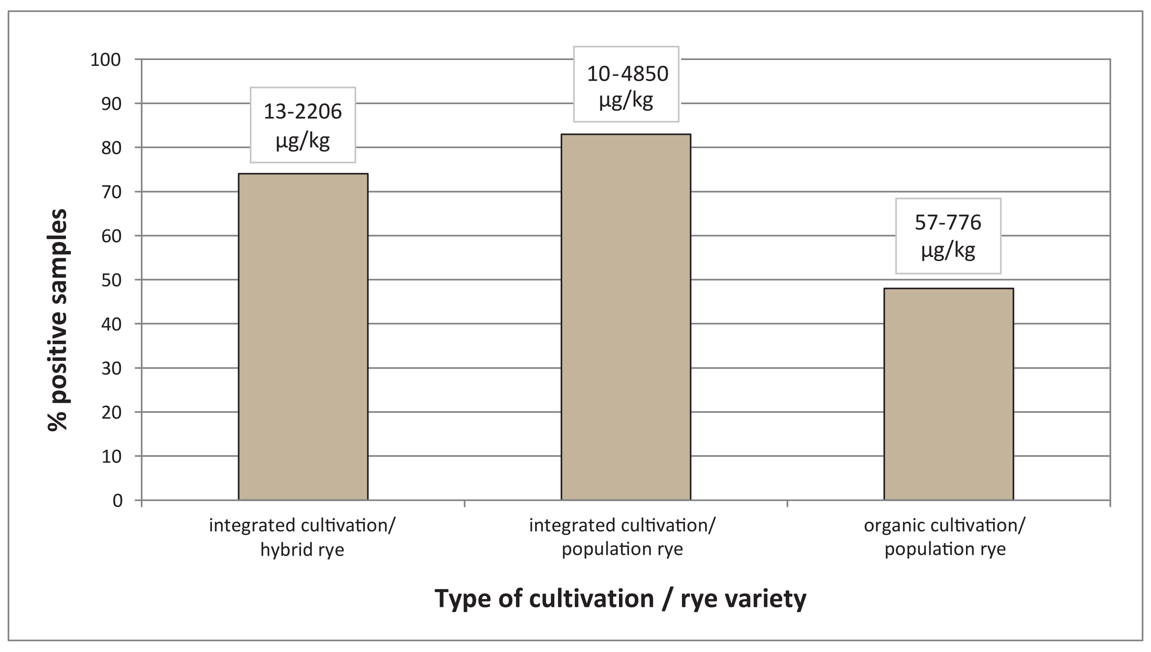

Fig.⑧ Ergot alkaloid contents depending on the type of rye cultivar in integrated and organic cultivation

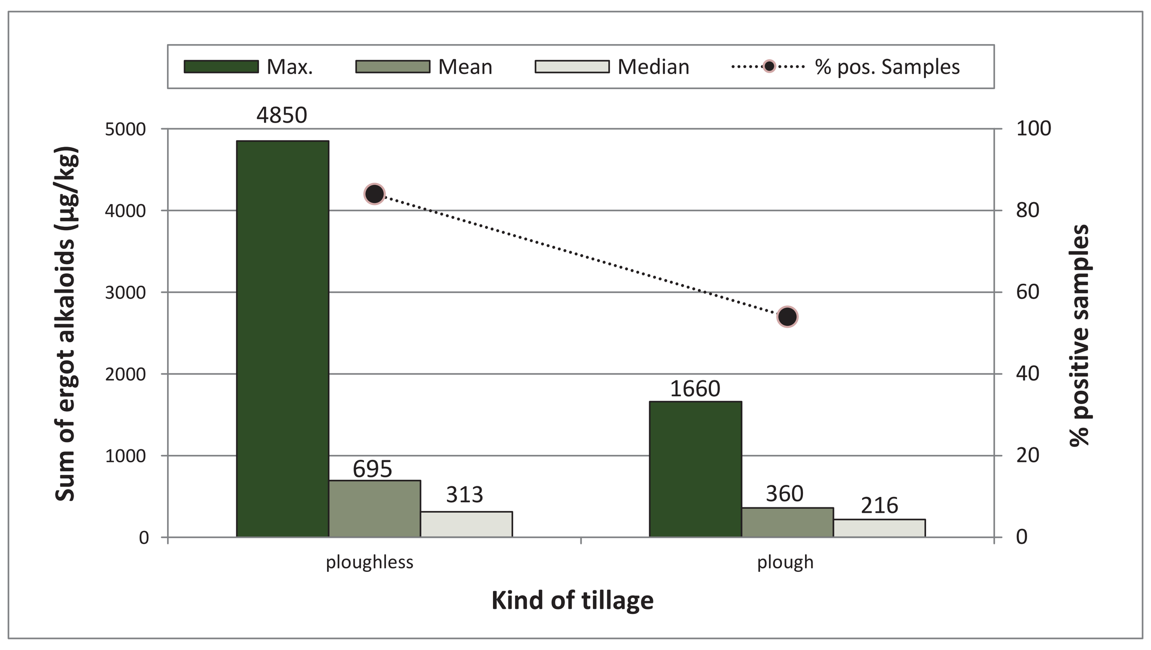

Fig.⑨ Maximum, mean and median of the ergot alkaloid content depending on the kind of tillage

After maize as pre-crop not only the DON contamination risk is higher but also the risk of an ergot contamination seems to rise.

CONCLUSION

2013 was a crop year of the Federal State of Brandenburg with moderate to high DON contamination in wheat samples and with a high contamination of ergot alkaloids in rye samples, respectively.

ACKNOWLEDGEMENTS

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REFERENCES

[1] Meister U (2005), *Fusarium toxins* in cereals of integrated and organic cultivation from the Federal State of Brandenburg (Germany) harvested in the years 2000-2007. Mycotoxin Research (2009) 25:133-139

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