Annual Report

The surveillance project for mycotoxins in food in Norway 2017 - Mycotoxins from *Fusarium* and ergot in wheat and rye

Veterinærinstituttet
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The surveillance programme for mycotoxins in food in Norway 2017 – Mycotoxins from *Fusarium* and ergot in wheat and rye

Content

Authors
Aksel Bernhoft, Ellen Christensen, Lada Ivanova, Chiek Er, Mona Torp

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Summary

Several mycotoxins, with emphasis on trichothecenes and ergot alkaloids as well as the ergot fungus *Claviceps purpurea* were analysed in samples of wheat and rye for human consumption collected from mills and food stores during 2017. The aim of the programme was to survey the mycotoxin status of Norwegian whole and milled wheat and rye and *C. purpurea* in whole wheat and rye in 2017.

In wheat, deoxynivalenol (DON) was present at insignificant concentrations and other trichothecenes and zearalenone were hardly detectable. In rye, these mycotoxins were generally not detected. Ergot alkaloids were, however, detected in some samples and at a similar level in wheat and rye. The highest measured concentrations of ergot alkaloids are worth attention, but there is no regulation for them and the knowledge base for risk assessment is scarce. The producer of ergot alkaloids, *C. purpurea,* was detected in about half of the wheat samples and in most samples of rye of which one sample above the maximum limit in EU and Norway (500 mg/kg). However, no ergot alkaloids were found in the sample with *C. purpurea* above maximum level, and no significant correlations were found between ergot alkaloids and *C. purpurea* in wheat nor in rye. Regulation of ergot alkaloid levels instead of on *C. purpurea* sclerotia would improve the risk management.

Sammendrag på norsk

Forskjellige mykotoksiner med hovedvekt på trikotecener og meldrøye-toksiner (alkaloider), samt meldrøye-soppen *Claviceps purpurea* ble analysert i prøver av mathvete og -rug samlet inn fra møller og matbutikker i 2017. Målet med programmet var å få en oversikt over mykotoksininnholdet i norsk hel og malt hvete og rug, og *C. purpurea* i hel hvete og rug i 2017.

I hvete var det ubetydelige konsentrasjoner av deoksynivalenol (DON), og andre trikotecener og zearalenon ble knapt påvist. I rug var disse mykotoksinene stort sett ikke påvisbare. Meldrøye-alkaloider ble imidlertid påvist i noen prøver og i lignende nivå i hvete og rug. De høyeste påviste konsentrasjonene av meldrøye-toksiner gir grunn til oppmerksomhet, men det er ingen lovgivning for dem, og kunnskapsgrunnlaget for risikovurdering er mangelfullt. Soppen som produserer meldrøye-alkaloider, *C. purpurea*, ble påvist i omtrent halvparten av hveteprøvene og i de fleste rugprøvene, hvorav *en* prøve var over maksimalgrensen i EU og Norge (500 mg/kg). Imidlertid ble ingen meldrøye-alkaloider funnet i prøven med *C. purpurea* over maksimalt nivå, og det var ingen signifikante korrelasjoner mellom meldrøye-alkaloider og *C. purpurea* i hvete eller i rug. Regelverk på meldrøye-alkaloider istedenfor på *C. purpurea* ville forbedre risikohåndteringen.

Introduction

The surveillance programme for mycotoxins in foods is a collaboration between the Norwegian Food Safety Authority (NFSA) and the Norwegian Veterinary Institute (NVI) where NFSA decides the extent of the surveillance programme based on scientific advices from NVI. NFSA is responsible for sampling and NVI for analysing and reporting of the results.

Genus *Fusarium* is the most important mycotoxin-producing fungi that primarily infects cereals in the field during the growing season. It produces important mycotoxins like the trichothecenes deoxynivalenol (DON), T-2 toxin (T-2) and HT-2 toxin (HT-2), as well as zearalenone (ZEN). Surveillance of DON in Norwegian cereals over the last two decades has found in some years that DON can occur at high concentrations, particularly in oats and wheat. DON constitutes a health risk in cereals if ingested above certain levels by animals and humans [1]. Prominent effects of DON exposure include gastrointestinal disorders such as reductions of feed intake and growth rate, which are well documented in pigs [1]. T-2 and HT-2 are usually present at significant concentrations only in oats and products containing oats. The toxic effects of T-2 and HT-2 are similar to DON but more potent. They can cause gastrointestinal lesions as well as immune suppression [1]. The oestrogenic mycotoxin ZEN is produced by the same *Fusarium* species as DON. The few data available so far have shown this toxin to be at insignificant concentrations in Norwegian cereals [1].

Ergot alkaloids are emerging mycotoxins of considerable interest in EU and occurrence data are desired [2]. They show moderately acute toxicity, but have neurotoxic properties, and may inhibit blood circulation and interfere with hormonal levels. Amongst cereal species produced in Norway, the producer of ergot alkaloids, the ergot fungus *Claviceps purpurea,* is usually mainly found in rye.

Aims

The surveillance programme aims to provide reliable documentation on the occurrence of important mycotoxins in cereals for human consumption, with special emphasis on trichothecenes and ergot alkaloids and to examine the correlation between the content of ergot alkaloids and *C. purpurea* in the grain.

Materials and methods

In 2017, the surveillance programme for mycotoxins in foods consisted of: a) 26 samples of whole wheat, 25 samples of milled wheat, 26 samples of whole rye and 22 samples of milled rye. The numbers deviated only slightly from the original sampling plan of 25 samples per product.

The samples collected by NFSA were according to a specific plan for sampling involving the various NFSA regions. They were collected at mills in grain production areas, or at food stores in Hamar and Oslo, and sent to NVI throughout the year. The sampling procedure followed the EU Regulation 401/2006 to achieve representative samples for each product. The sampling procedures took into account the size/volume of selected lot, like distribution pattern of the substances in the grain, number of incremental samples necessary, sampling tools, size of final samples etc.

The samples were analysed at NVI in Oslo for selected mycotoxins and metabolites. Whole wheat and whole rye were also analysed for *C. purpurea*.

Quantitative determination of *Claviceps purpurea*

The method calculated *C. purpurea* sclerotia in milligram per kg cereal according to the recomandation of EFSA. The samples were weighed and then spread over a large light surface for visual inspection. Detected sclerotia of *C. purpurea* were picked out and weighed separately.

Margin of error for analysis was considered: There is always uncertainty associated with visual inspection, primarily in relation to the risk of underestimation. However, careful examination can spot without difficulty the black sclerotia from the grains, and they appear larger than the normal grains. All sclerotia were assessed by experts or by DNA sequencing for verification of morphological results. The results are therefore considered highly specific, without the risk of false positives and with minimal risk of underestimation.

Chemical analysis

The multi-mycotoxin liquid chromatography–high-resolution mass spectrometry (LC-HRMS/MS) method was applied for the simultaneous determination of selected mycotoxins in the various cereal samples. In 2016, the developed LC-HRMS/MS multi-mycotoxin method was validated in order to ensure the quality and reliability of collected data [3]. The performance parameters linearity, selectivity, limit of detection (LODs) and limit of quantifications (LOQs) were validated. The evaluation of matrix effects was performed by utilizing the signal suppression or enhancement (SSE) approach based on a relative difference of the slope of calibration curves constructed with and without matrix extract. The matrix effects were observed for all selected mycotoxins, varying from 64 to 148 %. Reasonable levels of signal suppression or signal enhancement was achieved for only 30 % of targeted mycotoxins. Therefore, in order to control improve the accuracy of the method, we introduced stable-isotope labelled internal standards (IS) for eight of the analysed mycotoxins including DON, 3-acetyl-DON (3-Ac-DON), 15-acetyl DON (15-Ac-DON),DON-3-glucoside (DON-3-G), nivalenol (NIV), T-2, HT-2 and ZEN. For ergot alkaloids, selection of appropriate IS (bromocriptine mesylate or methysergide maleate) was confirmed by proficiency test (2017) for the national reference laboratories (NRLs) and appointed official control laboratories (OCLs) regarding the determination of ergot alkaloids in rye. The accuracy of an analytical method was assessed by analysing a blank sample matrix of interest spiked at a known concentration level (200 ng/kg).

In order to improve the extraction methodology with respect to polar and nonpolar compounds, a two-step extraction was carried out (MeCN:H_2O:HCOOH, 80:19.9:0.1, v/v/v and MeCN:H_2O:HCOOH, 20:79.9:0.1, v/v/v). LC-HRMS analyses were performed on a Q-Exactive™ Hybrid Quadrupole-Orbitrap mass spectrometer equipped with a heated electrospray ion source (HESI-II) and coupled to an UHPLC Dionex Ultimate 3000 system (Thermo FisherScientific).

Analytes and limit of detections

DON: 66 µg/kg, 3-ADON: 16 µg/kg, 15-ADON: 52 µg/kg, DON-3-G: 80 µg/kg, T-2: 14 µg/kg, HT-2: 22 µg/kg, NIV: 30 µg/kg, ZEN: 10 µg/kg, ergonovine: 56 µg/kg, ergosine: 12 µg/kg, ergotamine: 40 µg/kg, ergocornine: 12 µg/kg, alpha-ergocryptin: 190 µg/kg, ergocristine: 24 µg/kg.

Statistical analysis

Half detection limits were used for calculation purposes. Pearson correlation method was used for determination of correlation coefficients between *C. purpurea* and ergot alkaloids.

Results and discussion

Fusarium mycotoxins

Analysis of trichothecenes analysed, consisting of DON, 3- and 15-Ac-DON, DON-3-G, NIV, T-2 and HT-2, found only DON in several samples of whole (50 %) and milled wheat (12 %), but at insignificant levels (Table 1). Trichothecenes were hardly detected in rye. ZEA was hardly detected in any samples.

In comparison with data for DON in Norwegian wheat analysed at NVI during 1990-2011 and in 2016 the present mean DON concentrations in wheat appeared at the lower end of the yearly means during these years [1, 4]. The low level of DON in wheat collected in 2017 (harvested in 2016-17) is consistent with the finding of low levels of DON also in oats and barley in Norway these years [5, 6]. The finding of non-detectable concentrations of T-2, HT-2 and NIV in wheat is consistent with previous results in wheat analysed at NVI during these years. Few data are available for metabolites of DON in Norwegian cereals. In a study by Uhlig et al [7] of several mycotoxins in Norwegian cereal from the 2011 season, a year with considerable DON concentrations, 15 % DON-3-G and 4 % 3-Ac-DON compared with the wheat concentration of DON were found. Those results and our results from 2016 [4] and the present results indicate that the levels of DON-metabolites in wheat are considerably lower than the level of DON itself.

Few data are available for trichothecenes in rye in Norway. However, VKM [1] presented some data analysed at NVI, which indicated that rye has so far generally not been a cereal species considerably contaminated with DON, T-2 or HT-2. Our surveillance programmes from 2016 [4] and 2017 (present results) verify that lower trichothecene contamination was found in rye compared with wheat.

Except for trace concentration of ZEN in a single sample of whole wheat, this toxin was not found in the present samples. ZEN has not been systematically surveyed in Norwegian cereal but the restricted available data indicate detection at mostly low levels [1, 4, 7].

Table 1. Concentrations (µg/kg) of deoxynivalenol (DON), 3-acetyl-DON (3-Ac-DON), 15-acetyl-DON (15-Ac-DON), DON-3-glucoside (DON-3-G), T-2 toxin (T-2), HT-2 toxin (HT-2), sum T-2 + HT-2 toxin (T-2+HT-2), nivalenol (NIV) and zearalenone (ZEN) in whole wheat, milled wheat, whole rye and milled rye sampled in Norway during 2017.

	DON	3-Ac-DON	15-Ac-DON	DON-3-G	T-2	HT-2	T-2+ HT-2	NIV	ZEN
Whole wheat (n = 26)									
Mean	89	<16	<52	<80	<14	<22	<36	<30	<10
Median	<66	<16	<52	<80	<14	<22	<36	<30	<10
Min-max	<66-256	<16	<52	<80	<14	<22	<36	<30	<10-12
St. deviation	67	0	0	0	0	0	0	0	1
% samples with detectable conc.	50	0	0	0	0	0	0	0	4
Milled wheat (n = 25)									
Mean	<66	<16	<52	<80	<14	<22	<36	<30	<10
Median	<66	<16	<52	<80	<14	<22	<36	<30	<10
Min-max	<66-142	<16	<52	<80	<14	<22-22	<36	<30	<10
St. deviation	29	0	0	0	0	2	2	0	0
% samples with detectable conc.	12	0	0	0	0	4	0	0	0
Whole rye (n = 26)									
Mean	<66	<16	<52	<80	<14	<22	<36	<30	<10
Median	<66	<16	<52	<80	<14	<22	<36	<30	<10
Min-max	<66-74	<16	<52-61	<80	<14	<22	<36	<30	<10
St. deviation	8	0	7	0	0	0	0	0	0
% samples with detectable conc.	4	0	4	0	0	0	0	0	0
Milled rye (n = 22)									
Mean	<66	<16	<52	<80	<14	<22	<36	<30	<10
Median	<66	<16	<52	<80	<14	<22	<36	<30	<10
Min-max	<66	<16	<52	<80	<14	<22-27	<36	<30	<10
St. deviation	0	0	0	0	0	3	3	0	0
% samples with detectable conc.	0	0	0	0	0	5	0	0	0

Ergot and alkaloids

Claviceps purpurea sclerotia were present in most samples of whole rye, where one sample was above the maximum limit at 500 mg/kg in EU and Norway [8] (Table 2). Sclerotia were found in fewer samples and at lower levels in whole wheat, which is similar to our previous findings [4, 9]. Ergot alkaloids were found in some samples of wheat (whole) and rye (whole and milled) at similar levels. However, ergot alkaloids were not detected in milled wheat (Table 2). A similar occurrence of the sum of these toxins in wheat and rye was observed in 2016 [4] but the maximum concentrations of sum ergot alkaloids were higher in 2017. Maximum sum of ergot alkaloids around 1000-1500 mg/kg may possibly be of human health concern, but no legislation is available and the data for risk assessment is scarce. No significant correlations were found between *C. purpurea* sclerotia and any of the individual ergot alkaloids or the sum of them in wheat or rye ($r<0.388$, $p>0.05$) (Table 3). Furthermore, no ergot alkaloids were found in the sample that had *C. purpurea* sclerotia above maximum legislated level. However, significant correlations were shown between some individual ergot alkaloids; between ergosine and ergocristine in wheat and rye and also between ergocristine and α-ergocryptine in rye. Thus, the significant correlations were found between the most detected toxins. Correlation between ergot fungus and toxins was also not found in the corresponding surveillance programme in 2016 [4].

Table 2. Concentrations of *Claviceps purpurea* sclerotia (mg/kg) in whole wheat and whole rye, and ergot alkaloids (µg/kg) in whole wheat, milled wheat, whole rye and milled rye sampled in Norway during 2017. n.d.= not determined.

	C. purpurea sclerotia	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine	Σ Ergot alkaloids
Whole wheat (n = 26)								
Mean	14	<56	34	<40	18	<190	45	<334
Median	0	<56	<12	<40	<12	<190	<24	<334
Min-max	0-136	<56	<12-444	<40	<12-315	<190	<24-434	<334-1026
St. deviation	29	0	92	0	61	0	96	193
% samples with detectable conc.	46	0	19	0	4	0	15	15
Milled wheat (n = 25)								
Mean	n.d.	<56	<12	<40	<12	<190	<24	<334
Median	n.d.	<56	<12	<40	<12	<190	<24	<334
Min-max	n.d.	<56	<12	<40	<12	<190	<24	<334
St. deviation	n.d.	0	0	0	0	0	0	0
% samples with detectable conc.	n.d.	0	0	0	0	0	0	0
Whole rye (n = 26)								
Mean	174	<56	25	<40	<12	<190	50	<334
Median	136	<56	<12	<40	<12	<190	<24	<334
Min-max	0-603	<56	<12-430	<40	<12-73	<190-211	<24-409	<334-987
St. deviation	172	0	84	0	13	23	102	180
% samples with detectable conc.	81	0	8	0	4	4	15	12
Milled rye (n = 22)								
Mean	n.d.	<56	33	<40	28	<190	<24	<334
Median	n.d.	<56	<12	<40	<12	<190	<24	<334
Min-max	n.d.	<56	<12-579	<40	<12-459	<190-375	<24-146	<334-1472
St. deviation	n.d.	0	122	0	96	60	29	278
% samples with detectable conc.	n.d.	0	9	0	9	5	9	5

Table 3. Correlation coefficients between *Claviceps purpurea* and the determined individual and sum ergot alkaloids in whole wheat and whole rye. Significant correlations are shown in **bold** (p<0.05, r>0.388).

	C. purpurea sclerotia	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine	∑ Ergot alkaloids
Whole wheat (n=26)								
C. pur. sclerotia	1.000							
Ergonovine	-	-						
Ergosine	0.249	-	1.000					
Ergotamine	-	-	-	-				
Ergocornine	-0.097	-	-0.063	-	1.000			
α-Ergocryptine	-	-	-	-	-	-		
Ergocristine	0.340	-	**0.981**	-	-0.071	-	1.000	
∑ Ergot alkaloids	0.258	-	**0.946**	-	0.249	-	**0.944**	1.000
Whole rye (n=26)								
C.pur. sclerotia	1.000							
Ergonovine	-	-						
Ergosine	0.184	-	1.000					
Ergotamine	-	-	-	-				
Ergocornine	0.163	-	0.117	-	1.000			
α-Ergocryptine	0.230	-	-0.046	-	-0.040	1.000		
Ergocristine	0.313	-	**0.699**	-	-0.075	**0.512**	1.000	
∑ Ergot alkaloids	0.305	-	**0.865**	-	0.080	**0.394**	**0.953**	1.000

Conclusions

The results of analyses of several trichothecenes and ZEN in wheat and rye were insignificant in concentrations, if present. However, ergot alkaloids were present in some samples and at a similar level in wheat and rye. The maximum concentrations may be worth attention for health reasons but no legislation is available and the data for risk assessment is scarce. *C. purpurea* sclerotia were present in most samples of whole rye but in fewer samples and at lower levels in whole wheat. There were no significant correlations between ergot alkaloids and *C. purpurea* sclerotia in the cereal samples, and thus, the legislative control of ergot alkaloid levels instead of on *C. purpurea* sclerotia would improve the risk management.

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Appendix

Appendix Table 1. Concentrations of various mycotoxins (µg/kg) and *Claviceps purpurea* (mg/kg) in individual samples of **whole wheat** collected in Norway during 2017. DON=deoxynivalenol, 3-Ac-DON=3-acetyl-DON, 15-Ac-DON=15-acetyl-DON, DON-3-G=DON-3-glucoside, T-2=T-2 toxin, HT-2=HT-2 toxin, NIV=nivalenol, ZEN=zearalenone, *C. purpurea*=*Claviceps purpurea*.

Jnr.	DON	3- Ac-DON	15- Ac-DON	DON-3-G	T-2	HT-2	NIV	ZEN	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine	*C. purpurea*
21-90-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
21-162-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-34-1	179	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	6
23-34-2	256	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	25
23-35-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	315	<190	<24	0
23-35-2	220	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-35-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	4
23-35-4	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	5
23-36-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-36-2	122	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-57-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-70-2	120	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-81-2	100	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-97-2	72	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	14
23-112-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	444	<40	<12	<190	434	41
23-122-2	156	<16	<52	<80	<14	<22	<30	<10	<56	161	<40	<12	<190	232	24
23-140-1	144	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	57
23-170-2	130	<16	<52	<80	<14	<22	<30	<10	<56	12	<40	<12	<190	66	136
23-186-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	3
23-187-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-200-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-201-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-223-1	121	<16	<52	<80	<14	<22	<30	<10	<56	130	<40	<12	<190	185	34
23-251-1	152	<16	<52	<80	<14	<22	<30	12	<56	20	<40	<12	<190	<24	10
23-285-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-307-1	123	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0

Appendix Table 2. Concentrations of various mycotoxins (µg/kg) in individual samples of **milled wheat** collected in Norway during 2017. DON=deoxynivalenol, 3-Ac-DON=3-acetyl-DON, 15-Ac-DON=15-acetyl-DON, DON-3-G=DON-3-glucoside, T-2=T-2 toxin, HT-2=HT-2 toxin, NIV=nivalenol, ZEN=zearalenone.

Jnr.	DON	3-Ac-DON	15-Ac-DON	DON-3-G	T-2	HT-2	NIV	ZEN	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine
21-20-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-21-2	142	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-21-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-21-4	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-23-1	130	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-34-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-51-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-56-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-78-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-99-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-103-1	67	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-104-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-122-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-123-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-129-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-130-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-134-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-167-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-176-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-181-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-182-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-183-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
23-81-5	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
23-170-4	<66	<16	<52	<80	<14	22	<30	<10	<56	<12	<40	<12	<190	<24
23-187-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24

Appendix Table 3. Concentrations of various mycotoxins (µg/kg) and *Claviceps purpurea* (mg/kg) in individual samples of **whole rye** collected in Norway during 2017. DON=deoxynivalenol, 3-Ac-DON=3-acetyl-DON, 15-Ac-DON=15-acetyl-DON, DON-3-G=DON-3-glucoside, T-2=T-2 toxin, HT-2=HT-2 toxin, NIV=nivalenol, ZEN=zearalenone, *C. purpurea*=*Claviceps purpurea*.

Jnr.	DON	3-Ac-DON	15-Ac-DON	DON-3-G	T-2	HT-2	NIV	ZEN	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine	*C. purpurea*
21-132-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	16
23-45-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	332
23-57-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	80	425
23-60-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	44
23-63-1	74	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	343
23-70-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	300
23-81-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	603
23-96-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	236	219
23-97-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	211	307	367
23-112-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	73	<40	73	<190	<24	311
23-122-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	173
23-140-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	273
23-146-1	<66	<16	61	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	182
23-170-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	357
23-187-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-201-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	99
23-216-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	37
23-223-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	56
23-264-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	430	<40	<12	<190	409	308
23-265-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	40
23-268-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-286-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-293-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	5
23-293-2	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-301-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	0
23-302-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24	27

Appendix Table 4. Concentrations of various mycotoxins (µg/kg) in individual samples of **milled rye** collected in Norway during 2017. DON=deoxynivalenol, 3-Ac-DON=3-acetyl-DON, 15-Ac-DON=15-acetyl-DON, DON-3-G=DON-3-glucoside, T-2=T-2 toxin, HT-2=HT-2 toxin, NIV=nivalenol, ZEN=zearalenone.

Jnr.	DON	3-Ac-DON	15-Ac-DON	DON-3-G	T-2	HT-2	NIV	ZEN	Ergo-novine	Ergo-sine	Ergot-amine	Ergo-cornine	α-Ergo-cryptine	Ergo-cristine
21-20-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	579	<40	459	375	<24
21-21-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-26-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-32-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-33-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-35-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-48-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-56-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-100-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-118-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	35
21-119-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-131-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-133-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-163-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-167-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	17	<40	<12	<190	<24
21-168-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
21-176-1	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
23-81-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	26	<190	<24
23-81-4	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
23-140-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	146
23-170-3	<66	<16	<52	<80	<14	<22	<30	<10	<56	<12	<40	<12	<190	<24
23-187-4	<66	<16	<52	<80	<14	27	<30	<10	<56	<12	<40	<12	<190	<24

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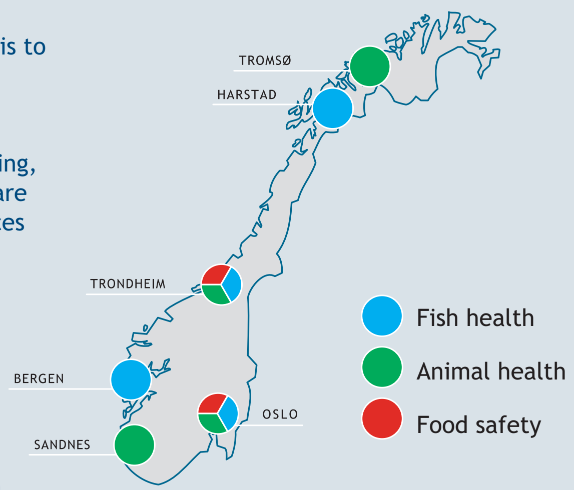

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Oslo postmottak@vetinst.no
Trondheim vit@vetinst.no
Sandnes vis@vetinst.no
Bergen post.vib@vetinst.no
Harstad vih@vetinst.no
Tromsø vitr@vetinst.no

www.vetinst.no

Veterinærinstituttet
Norwegian Veterinary Institute