



Research article

DEOXINIVALENOL AS A RISK FACTOR OF FOOD GRAIN CONTAMINATION: MONITORING RESULTS OF 1989–2018 YEARS HARVESTS IN RUSSIAN FEDERATION

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The paper dwells on the results obtained via long-term monitoring over food grain (wheat, barley, corn, oats, and rye) contamination with mycotoxin deoxynivalenol (DON). From 1989 to 2018 6,800 grain samples from Central, Southern, Volga, Ural, Siberian, North-Caucasian, Far Eastern, and Northwestern Federal Districts (FD) of the RF were analyzed. Depending on a year harvest, DON occurrence varied from 0 to 42 % and maximum toxin content reached 6.65 mg/kg. Over the whole examined period 10 % samples turned out to be contaminated and one forth of them contained the toxin in quantities exceeding maximum permissible levels (MPL). DON occurrence amounted to 24–42 % in years of mass epiphytotic (1989, 1992 and 1993) as well as in crops gathered in 2014 and 2017; DON was detected in quantities exceeding MPL in 9–27 % of examined samples in those years. 78 % contaminated samples came from Southern and North-Caucasian FD and another 10 % were from Far Eastern FD. A significant correlation between DON occurrence and a number of rainy and sunny days in May was established on the example of wheat samples from Krasnodar region. Analysis of contamination dynamics has revealed that over the last years there has been an ascending trend in frequency of DON detection in wheat that came not only from regions where Fusarium head blight was widely spread but also from regions in North-western, Siberian and Volga FD. Health risks related to DON intake with wheat grains processing products were assessed; the assessment revealed that DON intake higher than tolerable daily intake (TDI) for the residents of Southern and North-Caucasian FD in 1992, 1993, 2014 and 2017.

Average occurrence of DON was 4.2; 11.9; 3.0 and 0.6 % for barley, corn, rye, and oats samples and its maximum contents amounted to 8.95; 0.95; 0.96 and 0.44 mg/kg accordingly. Just as it was the case with wheat, the most of contaminated samples came from Southern, North-Caucasian and Far Eastern FD. Contamination tended to grow for all the examined grains and it calls for relevant measures aimed at controlling food grains safety

Key words: monitoring, mycotoxins, food grain, wheat, barley, oats, corn, rye, Fusarium head blight, occurrence, deoxynivalenol, health risk assessment, tolerable daily intake, weather, correlation analysis.

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Grain and the processed products are the traditional diet basis of most people in Russia; therefore, grain quality and safety is the most significant element of food safety in the Russian Federation. Phytopathogens, including toxigenic micromycetes are an integral part of any agricultural biocenosis. Improper crop rotations, technological breakdowns in grain cultivation and low quality of seed grains together with unfavorable weather make for “field” fungi development, micromycetes from *Fusarium* genus being among the most widely spread ones. Toxic secondary metabolites produced by microfungi, or mycotoxins, pose a health risk. The greatest health risk is associated with chronic intake of mycotoxins in small amounts with food [1–3].

Deoxynivalenol (DON) is the most widely spread fusariotoxin in the world. It was extracted in 1972 in Japan and the USA and later it was confirmed to be a constant contaminant in grain cultures in most regions all over the world. DON is the most frequently detected in wheat, a bit less frequently in corn, barley, rye, and oats, as well as in their processed grain products [4–12]. Its major producers, causing *Fusarium* head blight, are fungi from several *Fusarium* species including *F. graminearum*, *F. culmorum*, *F. nivale* [3, 6, 10, 11, 13]. Toxin accumulation depends on toxigenic properties of producing fungi strain, climatic and weather conditions, techniques applied to grow and protect plants, and storage conditions [14–16].

High humidity during and after flowering stage of the crop creates auspicious conditions for *Fusarium* head blight to appear. DON contents in contaminated grain grow starting from flowering and up to milky-wax ripeness and then they fall dramatically during wax and full ripeness [15, 17]. Moderate climate that is typical for North America, China, and Europe is optimal for *Fusarium* head blight (FHB) spreading in wheat [2, 6, 11, 16, 18]. In the Russian Federation grain grown in the North

Caucasian, the Southern, and the Far Eastern Federal Districts (FDs) is the most susceptible to the disease [2, 13, 15, 19].

Acute DON poisoning resulted in vomiting in animals studies (DON is also known as vomitoxin due to this fact). DON was established to cause alimentary toxicosis not only among farm animals [11, 16, 20] but among people as well [2, 20]. Studies revealed that it bonded to ribosome at molecular level and inhibited protein synthesis thus disrupting proper cell functioning [2]. DON in low doses is able to inhibit immunity. However, exposure to the toxin in its lethal dose can result in leukocytosis, bleeding, diarrhea, and endotoxemia [21]. The Joint FAO – WHO Expert Committee on Food Additives (JECFA) established provisional maximum tolerable daily intake (PMTDI) and acute reference dose of DON and acetylated metabolites (3-acetyl DON and 15-acetyl DON) for people at 1 and 8 µg/kg of body weight a day accordingly; the determined doses were results of toxicological studies [20]. The prevalence of the toxin and irrefutable evidence that it is truly hazardous for human health justified the introduction of hygienic standards for its contents in food in some countries. Codex Alimentarius Commission established international standards for DON contents in cereal grains (wheat, corn and barley) destined for further processing at the level of 2,000 µg/kg [22]. In the European Union countries maximum permissible levels (MPL) are regulated according to [23]: they should not exceed 1,750 µg/kg in unprocessed durum wheat, oats, and corn; 1,250 µg/kg in other unprocessed grains; 750 µg/kg in cereals and cereals products intended for direct human consumption. In the RF there are Technical Regulations of the Customs Union (TR CU) No. 021/2011 “On food safety”¹, and No. 015/2011 “Safety on grain”²; according to them DON MPLs in food wheat and barley as

¹ TR TS 021/2011. O bezopasnosti pishchevoi produktsii: tekhnicheskii reglament Tamozhennogo soyuza [TR CU 021/2011. On food safety: The Technical Regulations of the Customs Union. Approved by the Decision by the Customs Union Commission on December 9, 2011 No. 880]. *KODEKS: the electronic fund for legal and reference documentation*. Available at: [http://www.eurasiancommission.org/ru/db/techreglam/Documents/TR % 20TS % 20bezopProd.pdf](http://www.eurasiancommission.org/ru/db/techreglam/Documents/TR%20TS%20bezopProd.pdf) (March 12, 2021) (in Russian).

² TR TS 015/2011. O bezopasnosti zerna: tekhnicheskii reglament Tamozhennogo soyuza [TR CU 015/2011. Safety on grain (last edited on September 15, 2017): The Technical Regulations of the Customs Union. Approved by the Decision by the Customs Union Commission on December 9, 2011 No. 874]. *KODEKS: the electronic fund for legal and reference documentation*. Available at: <https://docs.cntd.ru/document/902320395> (March 12, 2021) (in Russian).

well as their products are equal to 0.7 mg/kg and 1.0 mg/kg respectively.

Long-term mycotoxicological monitoring has been accomplished to examine food grain contamination with mycotoxins, both in areas where *Fusarium* head blight is widely spread and in other regions where grain is grown. This research work focuses on long-term 30-year monitoring results on DON contamination in food grain (wheat, barley, corn, rye, and oats) harvested in 1989–2018 in order to reveal basic health risks and identify activities for those risks to be reduced.

Materials and methods. Food grain samples (6,800 overall) were provided by Rospotrebnadzor regional offices from Central, Southern, Volga, Ural, Siberian, North Caucasian, Far Eastern, and Northwestern Federal Districts of the RF³. Experts examined in total 4,009 wheat samples, 1,293 barley samples, 1,020 rye samples, 278 corn samples and 200 oats samples harvested from 1989 to 2018.

Grain samples were taken from homogeneous lots stored at grain reception and grain processing facilities in conformity with GOST (State Standard) R ISO 24333-2011⁴; DON contents in samples were determined with enzyme-linked immunosorbent assay (2009–2012), high performance liquid chromatography with diode-array and ultraviolet detection (HPLC-UV)⁵ (1989–2018) and liquid chromatography with tandem mass spectrometry (HPLC-MS/MS)⁶ (2018).

Data were statistically analyzed in IBM SPSS Statistics 23 (Statistical package for social sciences, USA) and Microsoft Office Excel 2007 (Microsoft Corp., USA). DON contents data in samples were presented in simple

mean (M) and median (Me) values and 90-th percentile (90 %) (contamination levels below minimum detectable concentration for the selected procedures (0.05 mg/kg) were taken as 0). We accomplished two-factor dispersion and correlation analysis to study correlations between frequency of DON contamination in wheat harvested in 2006–2018 in Krasnodar region and temperature and humidity, duration of sunny weather days and precipitations from May to August. Data were analyzed within the Latin square 3x3 where lines were numbers of samples and columns were years of harvest, values of dependent variables for a selected wheat class were given in cells with Latin alphabet letters, and frequency of DON contamination was a dependent variable (the significance level was 0.1). Additionally, to establish influence of a parameter on the dependent variable in contamination analysis, we applied Mann – Whitney non-parametric test after performing class-interval grouping into two class-intervals for each factor, upper and lower levels, 1 and 2 accordingly. If after dispersion analysis there were more than two deviations in null hypothesis and levels for a fixed factor, we applied Tukey's range test to perform multiple comparisons of simple means with the significance level being 0.05.

Health risks caused by wheat grain being contaminated with DON were assessed on the basis of comparing total daily DON intake with wheat-based foods and PMTDI DON. Estimated daily DON intake with wheat-based foods was calculated as per the following equation (1):

$$N_{calc} = \frac{M \cdot P \cdot 1000}{w}, \quad (1)$$

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⁴ GOST R ISO 24333-2011. Zerno i produkty ego pererabotki. Otbor prob [GOST R ISO 24333-2011. Cereals and cereal products – Sampling]. *KODEKS: the electronic fund for legal and reference documentation*. Available at: <https://docs.cntd.ru/document/1200092274> (March 11, 2021) (in Russian).

⁵ MU 5177-90. Metodicheskie ukazaniya po obnaruzheniyu, identifikatsii i opredeleniyu soderzhaniya dezoksinivalenola (vomitoksina) i zearalenona v zerne i zernoproduktakh [Methodical Guidelines 5177-90. Methodical guidelines on detecting, identifying and determining contents of deoxynivalenol (vomitoxin) and zearalenone in grain and grain products]. Available at: <http://www.gostrf.com/normadata/1/4293828/4293828870.pdf> (March 13, 2021) (in Russian).

⁶ MVI 410/4-2020. Metod mul'tidetektsii miktotoksinov v zerne i pervichnykh produktakh ego pererabotki [Measuring procedures 410/4-2020. A procedure for multidetection of mycotoxins in grains and primary produces of its processing]. Moscow, 2020 (in Russian).

where N_{calc} is estimated daily DON intake ($\mu\text{g}/\text{kg}$ of body weight); M is the average DON contents in wheat grains (mg/kg); P is consumption of processed wheat products (kg/day); w is body weight (kg); average body weight of a person is taken as 60 kg ; $1,000$ is conversion factor to converse into μg .

Results and discussion. Wheat grain contamination harvested in 1989–2018 all over the RF. Frequency of detection and average DON contamination in wheat varied from 0 to 42% and from 0 to $0.43 \text{ mg}/\text{kg}$ accordingly depending on a crop year (Table 1). In total, 10.0% out of $4,009$ grain lots harvested from 1989 to 2018 were contaminated; con-

tamination levels varied from 0.05 to $6.65 \text{ mg}/\text{kg}$ (Table 1). DON contents were higher than MPL in 101 (2.5%) grain samples.

26% samples from 1989 harvest contained DON and its contents exceeded MPL in 9% . Frequency of DON contamination in 1992 was maximum, 42% with the exceeding of MPL in 27% cases. The frequency of DON detection in wheat harvested in 1993 was also high, it reached up to 24% of the examined samples, and a share of samples with DON contents being higher than MPL reached 15% . DON content in contaminated samples varied from 0.05 to $6.65 \text{ mg}/\text{kg}$ in 1998 ; from 0.05 to $5.63 \text{ mg}/\text{kg}$ in 1992 ; and from 0.1 to $3.95 \text{ mg}/\text{kg}$ in 1993 .

Table 1
Frequency and levels of DON contamination in wheat harvested in 1989–2018

Year	Number of samples			DON contents in contaminated samples, mg/kg	DON content in total samples, mg/kg		
	total, items	Contaminated DON items (% of the total number)	containing DON -exceeding MPL		M	Me	90 %
1989	57	15 (26)	5 (9)	0.05 – 6.65	0.23	0	0.44
1990/91	67	4 (6)	1 (1)	0.05 – 0.74	0.02	0	0
1992	139	59 (42)	37 (27)	0.05 – 5.63	0.43	0	1.06
1993	156	38 (24)	24 (15)	0.10 – 3.95	0.22	0	0.63
1994	254	16 (6)	6 (2)	0.17 – 0.96	0.03	0	0
1995	169	11 (6)	0 (0)	0.07 – 0.70	0.03	0	0
1996	120	15 (13)	0 (0)	0.06 – 0.70	0.02	0	0.07
1997	137	15 (11)	1 (0.7)	0.05 – 1.14	0.02	0	0.05
1998	126	12 (10)	1 (0.8)	0.05 – 1.09	0.03	0	0.03
1999	132	0 (0)	0 (0)	< 0.05	0	0	0
2000	222	6 (3)	1 (0.5)	0.09 – 0.77	0.01	0	0
2001	252	12 (5)	0 (0)	0.05 – 0.62	0.01	0	0
2002	158	6 (4)	1 (0.6)	0.05 – 0.78	0.01	0	0
2003	375	5 (1)	0 (0)	0.05 – 0.07	0	0	0
2004	213	2 (1)	0 (0)	0.07; 0.08	0	0	0
2005	147	12 (8)	0 (0)	0.07 – 0.69	0.02	0	0
2006	85	11 (13)	0 (0)	0.05 – 0.34	0.02	0	0.06
2007	98	9 (9)	1 (1)	0.06 – 0.91	0.05	0	0
2008	73	4 (5)	1 (1.4)	0.06 – 1.03	0.02	0	0
2009	109	8 (7)	0 (0)	0.06 – 0.12	0.01	0	0
2010	122	10 (8)	2 (2)	0.06 – 1.26	0.03	0	0
2011	158	13 (8)	0	0.05 – 0.44	0.01	0	0
2012	34	0 (0)	0	< 0.05	0	0	0
2013	111	9 (8)	0	0.05 – 0.52	0.01	0	0
2014	57	21 (37)	6 (10)	0.07 – 5.85	0.29	0	0.66
2015	64	6 (9)	0	0.05 – 0.33	0.01	0	0
2016	154	29 (19)	2 (1.3)	0.05 – 1.43	0.05	0	0.54
2017	105	32 (31)	9 (9)	0.05 – 2.46	0.18	0	0.51
2018	115	18 (16)	3 (2.6)	0.10 – 1.27	0.06	0	0.20
TOTAL	4,009	399 (10.0)	101 (2.5)	0.05 – 6.65	0.05	0	0.05

Note: DON MPL, mg/kg , is not higher than 0.7 for wheat (TR CU 015/2011 “Safety of grain”).

Over the next 20 years from 1993 to 2013 frequency of DON contamination in wheat was relatively rare and varied from 0 to 9 %. DON contents higher than MPL were detected in very few cases, and maximum DON content in contaminated samples amounted to 1.26 mg/kg. A diagram that showed harvests distributed as per average DON contents revealed a peak at 0.01–0.03 mg/kg.

Starting from 2014 frequency of wheat grain contamination grew to 9–37 % together with increasing numbers of samples with DON contents being higher than MPL. The only exception was the crop of 2015 due to lack of samples contaminated with DON at levels higher than MPL.

Therefore, wheat harvested in 1999, 2003, 2004 and 2012 turned out to be the least contaminated with DON since a share of contaminated samples didn't exceed 1 % and contamination levels were quite low. Frequency of DON contamination varied from 3 to 13 % in wheat harvested in 1990–1991, 1994–1998, 2000–2002, 2005–2011, 2013 and 2015; average DON contents were from 0 to 0.05 mg/kg; and 90-th percentile, from 0 to 0.07 mg/kg. Contamination with the toxin that was higher than MPL was detected in isolated samples harvested in 1990–1991, 1997, 1998, 2000, 2002, 2007, 2008 and 2010. A peak in DON contamination was detected not only in mass epiphytotic years (1989, 1992 and 1993) but also in recent years of 2014, 2016, 2017 and 2018. In these years frequency of DON contamination varied from 16 to 37 %; average DON contents, from 0.06 to 0.29 mg/kg; 90-th percentile, from 0.20 to 0.66 mg/kg. Grain samples with DON contents being higher than MPL were the most frequent in wheat harvested in 1989, 1992, 1993, 2014 and 2017 (9–27 % of all the examined samples).

Wheat contamination harvested in 1999–2018 throughout regions. DON contents in wheat were different depending on the region of grain production (Figure 1). Analysis of distribution of contaminated wheat harvested in 1999–2018 showed that 78 % samples (159 out of 205 contaminated samples) were grown in the Southern FD (Republics of Adygei and Kalmykia, Crimea, Astrakhan region,

Volgograd region, and Rostov region) and the North Caucasian FD (the Republics of Ingushetia, North Ossetia – Alania, Kabardino-Balkaria, Karachay-Cherkessia, and Stavropol region); all these regions are primary areas in Russia where *Fusarium* head blight is widely spread in grains. DON contents varied from 0.05 to 5.85 mg/kg in these samples. 10 % of contaminated samples were received from the Far Eastern FD and 5 % from the Central FD. Frequency of DON contamination was significantly lower in harvests gathered in the Siberian, the Volga, the Northwestern, and the Ural FDs and amounted to 1–2 %.

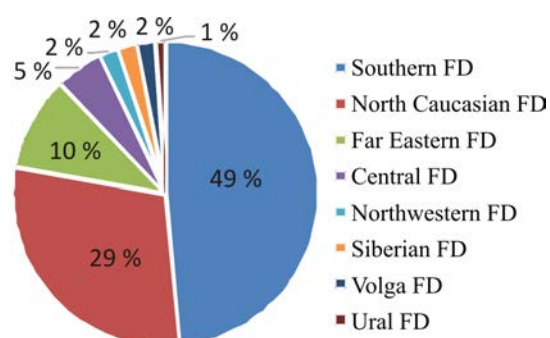


Figure 1. Region distribution of DON contaminated wheat harvested in 1999–2018 (% of the total contaminated samples)

According to Rosstat data, in 2018 the greatest contributions in total grain harvest belonged to Rostov region, 12.9 %; Krasnodar region, 12.4 %; and Stavropol region, 9.9 %. They were followed by Altai region and Volgograd region, 4.2 % each; Voronezh, Kursk, and Omsk region, from 3.1 to 3.8 %; Tatarstan, Lipetsk, Novosibirsk, Tambov, and Orel regions – from 2 to 2.7 %; Orenburg, Penza, Kurgan, Tula, Chelyabinsk, Samara, and Krasnoyarsk regions – from 1.5 to 1.8 %⁶. As said above, grain produced in leading regions in wheat harvesting, namely Rostov, Krasnodar, and Stavropol regions, tended to be more heavily contaminated with DON than grain produced in other regions. Harvested wheat is further processed and consumed not only by people living in the Southern and the North Caucasian FDs but also by people living in other FDs; hence, DON intake due to consumption of contaminated wheat-based products can contribute significantly to DON in-

take with food products estimated for the country population as a whole.

Dynamics of grain contamination with DON was analyzed on the example of several wheat-producing regions in the RF; Table 2 contains the results.

From 1999 to 2018 wheat samples grown in *the Southern FD* mostly received from Krasnodar, Rostov, and Volgograd regions, and Adygei and Kalmyk Republics. DON grain contamination in these regions was significantly different depending on the area where grain was grown. For example, grain received from Kalmyk Republic and Volgograd region practically was not contaminated with DON or its content was very low. On the contrary, frequency of the toxin detection was the highest in grain received from Krasnodar

region and Adygei Republic. Rostov region was somewhere in between as per this parameter within the Southern FD (Table 2).

Totally, 262 grain samples from Krasnodar region were analyzed; samples contaminated with DON were detected in 10 out of 17 harvests gathered in different years and frequency of detection varied from 0 to 79 % depending on a crop year. In some years a share of samples contained the toxin in concentration higher than MPL varied from 5.9 % (2016) to 42.9 % (2017) and maximum contamination levels reached 3.21 and 5.85 mg/kg.

65 grain samples from Adygei Republic were analyzed and DON was detected in grains from 8 out of 13 harvests gathered in different years in quantities varying from 0.08 to 0.78 mg/kg.

Table 2

Dynamics of wheat DON contamination harvested in 1999–2018 from different FDs in the RF

Region	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018
The Southern Federal District (694 samples)																				
Krasnodar region (n** = 262)	3/0/0* (0)	4/0/0 (0)	10/2/0 (20)	1/0/0 (0)	5/0/0 (0)	3/0/0 (0)	1/0/0 (0)	15/7/0 (47)	15/2/0 (13)	–	–	77/7/0 (9)	8/0/0 (0)	–	51/2/0 (4)	14/8/2 (57)	9/2/0 (22)	17/11/1 (65)	14/11/6 (79)	15/8/2 (53)
Adygei Republic (n = 65)	–	12/2/0 (17)	8/1/0 (13)	5/2/1 (40)	14/1/0 (7)	–	–	–	–	1/0/0 (0)	–	–	5/2/0 (40)	–	1/0/0 (0)	1/1/0 (100)	1/1/0 (100)	1/0/0 (0)	1/0/0 (0)	1/1/0 (100)
Rostov region (n = 271)	25/0/0 (0)	8/2/1 (25)	21/8/0 (38)	8/2/0 (25)	11/0/0 (0)	14/1/0 (7)	25/5/0 (20)	12/1/0 (8)	7/0/0 (0)	5/0/0 (0)	34/1/0 (3)	4/0/0 (0)	–	–	25/0/0 (0)	8/0/0 (0)	28/2/0 (7)	21/6/0 (29)	7/0/0 (0)	8/0/0 (0)
The North Caucasian Federal District (333 samples)																				
Stavropol region (n = 215)	3/0/0 (0)	4/1/0 (0)	5/0/0 (0)	2/1/0 (0)	6/0/0 (0)	6/0/0 (0)	4/2/0 (50)	2/1/0 (50)	8/0/0 (0)	1/0/0 (0)	16/1/0 (6)	9/1/0 (11)	17/2/0 (12)	24/0/0 (0)	19/0/0 (0)	17/7/3 (41)	20/0/0 (0)	19/3/0 (0)	15/9/1 (60)	18/2/1 (11)
Kabardino-Balkaria, North Ossetia – Alania (n = 56)	–	–	–	–	–	–	–	–	1/1/1 (100)	–	10/4/0 (40)	–	16/6/0 (38)	–	5/2/0 (40)	5/4/1 (80)	4/1/0 (25)	1/0/0 (0)	2/1/0 (50)	3/0/0 (0)
Dagestan, Chechnya (n = 44)	–	2/0/0 (0)	–	–	–	–	11/2/0 (18)	–	24/2/0 (8)	2/0/0 (0)	–	–	2/0/0 (0)	–	–	5/0/0 (0)	–	–	–	–
The Far Eastern Federal District (56 samples)																				
Amur region (n = 12)	–	1/0/0 (0)	–	–	–	–	–	–	–	1/0/0 (0)	–	–	–	–	–	1/1/0 (100)	1/0/0 (0)	2/2/1 (100)	3/3/1 (100)	3/2/0 (67)
Primorye (n = 27)	–	1/0/0 (0)	–	1/0/0 (0)	–	–	4/3/0 (75)	1/1/0 (100)	1/1/0 (100)	3/3/1 (100)	6/2/0 (33)	1/1/1 (100)	–	–	–	–	–	–	2/1/1 (50)	–
The Central Federal District (693 samples)																				
Orel region (n = 150)	1/0/0 (0)	10/0/0 (0)	–	53/0/0 (0)	22/0/0 (0)	–	3/0/0 (0)	–	–	–	–	1/0/0 (0)	20/1/0 (5)	–	–	–	–	6/1/0 (17)	5/2/0 (40)	3/0/0 (0)
Voronezh region (n = 109)	7/0/0 (0)	10/0/0 (0)	–	7/0/0 (0)	11/0/0 (0)	14/0/0 (0)	15/0/0 (0)	5/0/0 (0)	19/3/0 (16)	–	–	1/0/0 (0)	–	–	–	–	–	–	3/0/0 (0)	3/0/0 (0)
Other regions (n = 434)	42/0/0 (0)	55/0/0 (0)	1/0/0 (0)	15/0/0 (0)	76/0/0 (0)	47/0/0 (0)	27/0/0 (0)	18/0/0 (0)	31/0/0 (0)	28/1/0 (4)	13/0/0 (0)	4/0/0 (0)	17/1/0 (6)	–	–	1/0/0 (0)	–	17/1/0 (5)	19/0/0 (0)	17/0/0 (0)
The Volga FD (331 samples)	1/0/0 (0)	29/0/0 (0)	4/0/0 (0)	69/0/0 (0)	23/0/0 (0)	65/0/0 (0)	5/0/0 (0)	5/0/0 (0)	10/0/0 (0)	13/0/0 (0)	5/0/0 (0)	3/0/0 (0)	42/0/0 (0)	10/0/0 (0)	–	–	–	18/0/0 (0)	10/2/0 (20)	19/2/0 (11)
The Siberian FD (266 samples)	2/0/0 (0)	48/0/0 (0)	55/0/0 (0)	19/0/0 (0)	33/0/0 (0)	25/0/0 (0)	9/0/0 (0)	–	4/0/0 (0)	3/0/0 (0)	8/0/0 (0)	10/0/0 (0)	19/1/0 (5)	–	–	3/0/0 (0)	–	5/0/0 (0)	12/1/0 (8)	11/2/0 (18)
The Ural FD (69 samples)	–	5/0/0 (0)	–	12/0/0 (0)	5/0/0 (0)	10/0/0 (0)	–	–	–	–	–	–	5/0/0 (0)	–	–	–	–	21/2/0 (10)	3/0/0 (0)	8/0/0 (0)
The North-western FD (35 samples)	–	16/0/0 (0)	–	1/0/0 (0)	1/0/0 (0)	7/0/0 (0)	–	–	–	–	1/0/0 (0)	–	1/0/0 (0)	–	–	–	–	5/3/0 (60)	3/2/0 (67)	–

Note: * means a number of examined samples, items / a number of samples contaminated with DON, items / a number of contaminated samples with DON contents being higher than MPL, items, (frequency of detection, %). ** is a number of samples received from a given region over the monitoring period from 1999 to 2018.

Grain samples (271 overall) from Rostov region were provided for monitoring purposes practically every year. DON was detected in grain from 9 out of 17 harvests gathered in different years in concentration varying from 0.05 to 0.77 mg/kg and frequency of its detection was lower than in Krasnodar region and Adygei Republic. DON contents were higher than MPL only in one out of 271 analyzed samples; it was a grain sample from the harvest gathered in 2000.

Grain samples from harvests gathered in 1999–2018 in *the North Caucasian FD* were mostly received from Stavropol region; samples from Kabardino-Balkaria, Dagestan, and North Ossetia–Alania were rarer (harvests gathered in 9 different years). In total 333 grain samples were received to be analyzed over the examined period; 215 out of them were from Stavropol region; 56 samples, from Kabardino-Balkaria and North Ossetia–Alania (Table 2). Frequency of DON detection in contaminated grain harvested in 1999–2018 in Stavropol region varied from 6 % (2009) and 60 % (2017) and maximum contents reached 1.56, 0.97 and 0.73 mg/kg in some grain samples in 2014, 2017 and 2018 crops accordingly. 9-year monitoring over wheat grain contamination in Kabardino-Balkaria and North Ossetia–Alania revealed the toxin contents in 7 out of 9 examined grain harvests (Table 2). DON contents higher than MPL were detected in grains from harvests gathered in 2007, 2010 and 2014 and contamination levels reached 0.91, 0.71 and 0.83 mg/kg accordingly.

DON content was analyzed in 56 samples of wheat from harvests of 14 different years in *the Far Eastern FD*. The toxin was detected in wheat from 9 out of 14 grain harvests gathered in Primorye and its contents exceeded MPL in some grain samples in 2008, 2010, 2016 and 2017 crops (Table 2). DON was detected in grains from 4 out of 7 harvests gathered in different years and its contamination levels were higher than MPL in grains in 2016 and 2017 crops.

DON was rarely detected in wheat from *the Central FD*. The toxin was detected in small quantities, from 0.05 to 0.16 mg/kg, in

1.4 % out of 693 examined samples of grains from harvests gathered in 20 different years.

Low (1.2 %) frequency of DON detection was revealed for grain samples received from *the Volga FD*. The toxin was detected in quantities from 0.09 to 0.37 mg/kg in few samples out of 331 examined ones. It should be noted that samples contaminated with DON were from harvests gathered in 2017 and 2018.

Low (from 0.18 to 0.25 mg/kg) DON contents were detected in very few samples of grain from harvests gathered in 2011, 2017 and 2018 in *the Siberian FD*.

DON contamination in grain received from *the Ural* and *the Northwestern FDs* was analyzed in few grain samples, 69 and 35 accordingly, from harvests gathered in 8 different years. Frequency of the toxin detection was 3 and 14 % accordingly. Contaminated samples were detected only in 2016 and 2017 crops. It should be noted that approximately 2/3 of samples received from the Northwestern FD, namely Kaliningrad region, were contaminated with DON in quantities varying from 0.14 to 0.46 mg/kg. High frequency of DON detection in Kaliningrad region is well in line with data collected in neighboring regions. Thus, DON was detected in 83 % out of 92 examined wheat samples received from Poland and harvested in 2016 in the amount from 0.01 to 1.27 mg/kg².

Therefore, analysis of dynamics of wheat grain contamination harvested in 1999–2018 in various RF regions revealed a growing trend for DON occurrence not only in the North Caucasian, the Southern, and the Far Eastern FDs where contamination was the highest during the survey but also in the North Western, the Siberian, and the Volga FDs recent years.

Analysis of environmental factors affecting the grain contamination with DON. Climatic and weather conditions are known to have their influence on DON accumulation by toxicogenic fungi of *Fusarium* genus [14, 15]. We estimated a correlation between grain contamination with DON (Figure 2) and several environmental factors on the example of grain samples from harvests gathered in 2016–2018 in Krasnodar region. The examined factors in-

cluded the following: average monthly humidity and temperature, a number of sunny hours (a period of time in each month when the sun was above the horizon in a given area and not hidden behind the clouds) and a number of days with precipitations exceeding 1 mm from May to August. Meteorological data were taken from “Aisori – remote access to DDL-archives”, an online achieve that contained data on climatic research⁷.

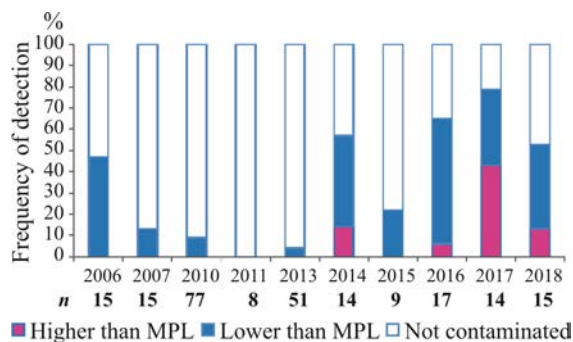


Figure 2. Dynamics of DON contamination in wheat harvested in 2006–2018 in Krasnodar region (*n* is a number of samples)

Correlation analysis confirmed there was an authentically significant correlation between frequency of the toxin detection and its average contents in contaminated grain samples harvested in 2006–2018: correlation coefficient was $r = 0.68$ and authenticity was $p = 0.02$ at the significance level $\alpha = 0.05$. Keeping in mind that variation coefficient for frequency of DON detection was significantly lower we took the former coefficient to use in

further statistical analysis as a leading one. We didn't detect any correlation between frequency of DON detection and the examined weather factors from June to August. At the same time weather conditions in May, namely duration of sunny weather and a number of days with precipitations exceeding 1 mm authentically had the most significant influence on DON contamination, the significance level being 0.05 (Table 3). These data are well in line with the fact that wheat grain is the most susceptible to fungi infections during its milky ripeness and in Krasnodar region this period is from mid May to mid June. Clear and sunny weather and a decrease in a number of rainy days in May make for declining wheat contamination with DON and vice versa.

The detected correlation was confirmed with using Mann – Whitney non-parametric test. To apply it, we divided a parameter within a month into two class-intervals, upper and lower ones, and frequencies of DON detection were written down in a relevant class-interval. The analysis revealed that authenticity was higher than significance ($p > 0.05$) for average monthly air humidity and temperature in May; hence, they had only insignificant influence on contamination levels. But the significance was $p < 0.05$ for duration of sunny days and a number of rainy days with precipitation exceeding 1 mm and this indicates there is an authentic correlation between these two weather factors and grain contamination.

Table 3

Correlations between frequency of DON detection in wheat grain and examined weather conditions for harvests gathered in 2006–2018

Parameter	Correlation coefficient, r (authenticity, p)			
	May	June	July	August
Average monthly air humidity	0.49 (0.120)	–0.05 (0.868)	–0.22 (0.504)	–0.08 (0.811)
Average monthly air temperature	–0.46 (0.150)	–0.03 (0.916)	0.02 (0.946)	0.12 (0.718)
Duration of sunny hours per month	–0.65 (0.029)	–0.08 (0.805)	–0.07 (0.837)	–0.09 (0.782)
A number of days with precipitations exceeding 1 mm	0.74 (0.008)	–0.21 (0.525)	–0.11 (0.747)	–0.11 (0.756)

⁷ Veselov V.M., Pribyl'skaya I.R., Mirzebasov O.A. Spetsializirovannye massivy dlya klimaticheskikh issledovaniy. «Aisori – Udalennyi dostup k YaOD-arkhivam [Specialized data arrays for climatic research. “Aisori – remote access to DDL-archives”]. VNIIGMI-MC Aisori D. Available at: <http://aisori-m.meteo.ru/waisori/index0.xhtml> (March 03, 2021) (in Russian).

Table 4

Two-factor dispersion analysis of a grain quality category, year of a harvest, and contamination

Dependent variable (DON content in a sample)					
Source	Sum of type III squares	Degree of freedom	Mean square	F-criterion	Significance
Adjusted model	3.422 ^a	3	1.141	1.300	0.284
Year	0.503	1	0.503	0.573	0.452
Grain quality category	0.811	1	0.811	0.924	0.341
Year × category	2.034	1	2.034	2.319	0.134
Error	47.380	54	0.877		
Total	68.325	58			
Adjusted result	50.802	57			

^a – R-square = 0.067 (Adjusted R-square = 0.016)

Table 5

Frequency and levels of DON contamination in food barley, oats, rye, and corn harvested in 1989–2018

Grain	Number of samples			DON contents in contaminated samples, mg/kg	DON contents in total samples, mg/kg		
	Total, item	Containing DON items (% of total number)	DON contents higher than MPL		<i>M</i>	<i>Me</i>	90 %
				Barley			
Corn	278	33 (11.9)	MPL not fixed	0.05 – 0.95	0.04	0	0.07
Oats	200	6 (3.0)	MPL not fixed	0.06 – 0.96	0.01	0	0
Rye	1,020	6 (0.6)	MPL not fixed	0.06 – 0.44	0.00	0	0

Note: DON MPL, mg/kg, not exceeding 1.0 for barley (TR CU 015/2011 “Safety of grain”).

There was a growth in grain contamination observed from 2006 to 2018. Correlation analysis revealed a directly proportional correlation between a year of a harvest and frequency of DON detection: correlation coefficient was $r = 0.60$ and authenticity was $p = 0.049$ at the significance level $\alpha = 0.05$. Although it seems hardly possible to use this correlation to predict future situation due to $\alpha \approx p$, we still can't ignore it.

We performed two-factor dispersion analysis of a grain quality category, a year of a harvest, and DON contents in a sample (Table 4). A year of a harvest was used as fixed factor A, and grain quality category, fixed factor B.

Analysis of barley, oats, rye and corn contamination harvested in 1989–2018. Aggregated data on DON contamination in barley, oats, rye, and corn harvested in 1989–2018 are given in Table 5. DON contamination *in food barley* was examined on the example 1,293 grain samples harvested in 1989–2018. Frequency of DON contamination in barley was lower than in wheat (10 %) and corn. DON was

detected in 4 % samples (54 overall) in quantities varying from 0.05 to 8.95 mg/kg, and 5 samples contained DON in quantities higher than MPL. DON was detected most frequently in grains in 2017 and 2014 crops, 44 and 30 % accordingly, and this frequency was substantially lower in grains harvested in 2015 (22 %), 2016 (19 %), 1989 (16 %), 1992 and 2009 (14 % in both these years). In other years, frequency varied from 0 to 7 %. Notably, over the recent years, starting from 2014, DON contamination in barley has been growing.

Approximately 80 % of all barley production is located in the Central, the Volga, the Southern and the Siberian FDs [24]. The greatest number of samples was received from the Central and the Southern FDs. The toxin was detected in 11 % samples from the Southern FD in quantities varying from 0.05 to 1.7 mg/kg and its contents were higher than MPL in one sample of grain harvested in 2017. Barley received from Krasnodar region was the most contaminated with DON was, since 23 % out of 39 samples were contaminated

with DON in the amount from 0.05 to 1.7 mg/kg; the least contaminated barley was received from Rostov region: 7.5 % out of 40 samples were contaminated with DON in the amount from 0.05 to 0.11 mg/kg. According to our data the toxin was most frequently detected in barley received from the North Caucasian FD; DON was detected in 17 % of 64 examined samples, in particular, in low quantities in samples from Stavropol region (0.10 and 0.11 mg/kg). The range of DON contamination in grain from Kabardino-Balkaria was wider, from 0.07 to 8.95 mg/kg with exceeding MPL DON in grain harvested in 2017. DON in low quantities was detected in rare cases in grain received from Ingushetia and North Ossetia.

Although samples from the Far Eastern FD were sent for analysis irregularly and in relatively few numbers, approximately half of examined samples (10 out of 21) were contaminated with DON in quantities varying from 0.05 to 2.83 mg/kg with exceeding of MPL in one sample harvested in Amur region in 2018. Barley lots harvested in 2005–2009 in Primorye contained the toxin in low quantities, from 0.06 to 0.21 mg/kg.

Our research results revealed that, just as it was the case with wheat, samples received from the Far Eastern, the North Caucasian, and the Southern FDs were the most contaminated.

Corn grain was similar to wheat grain (11.98 % against 10 %) as per DON contaminations. DON contents in contaminated corn samples varied from 0.05 to 0.95 mg/kg (average contents amounted to 0.04 mg/kg). For reference, DON contents in contaminated wheat samples amounted to 0.05–6.65 mg/kg. Having analyzed dynamics of domestic corn contamination with DON, we revealed that DON occurred only in grains harvested in 2000, 2002, and 2012–2018, and the highest frequency of DON contamination was detected in recent years (2014, 2016–2018).

The Southern, the North Caucasian and the Central FDs are basic corn producers in Russia⁸. The greatest number of samples was

sent for analysis from the Southern (217) and the North Caucasian FDs (47). The toxin was the most frequently detected in corn from the North Caucasian FD, namely, in 32 % out of 47 examined lots, in quantities varying from 0.05 to 0.68 mg/kg, 0.09 mg/kg on average. The toxin was detected less frequently, namely, in 6 % out of samples received from the Southern FD; its amount varied from 0.05 to 0.95 mg/kg, 0.02 mg/kg on average. DON was detected in rare corn lots received from the Central FD in quantities equal to 0.29 and 0.32 mg/kg. It should be noted that 4 out of 6 examined corn samples received from the Far Eastern FD was contaminated with DON in quantities varying from 0.13 to 0.55 mg/kg.

Oats contamination with DON was examined in 200 samples of grains harvested in 1999–2018. The toxin was detected in 3 % cases in quantities from 0.05 to 0.96 mg/kg, 0.008 mg/kg on average (Table 5). DON was detected in grains harvested in 2009, 2016 and 2017.

It should be noted that approximately 39 % of overall oats produced in the country is grown in Altai, Krasnoyarsk, Novosibirsk, Tyumen, Kemerovo, and Omsk regions and Bashkortostan [25]. The toxin was not detected in samples received from these regions. Contamination was detected only in samples from the Far Eastern (4 cases) and the North Caucasian FD (2 cases). Oats samples from Amur region and Primorye were the most contaminated with DON, its amounts varying from 0.09 to 0.96 mg/kg. The toxin was less frequently detected and in lower quantities in oats received from Ingushetia and Stavropol region (0.06; 0.09 mg/kg accordingly).

Generally the toxin was detected in **rye** samples were received from the North Caucasian FD during epiphytotic (harvests gathered in 1989 and 1992). The toxin was detected in rare cases in low quantities in grains harvested in 1996 and 2007, at the level of 0.22 and 0.06 mg/kg in the North Caucasian and the Central FDs accordingly.

⁸ Sel'skoe khozyaistvo v Rossii. 2019: statisticheskii sbornik [Agriculture in Russia. 2019: statistical data collection]. Rosstat. Moscow, 2019, 91 p. (in Russian).

Approximately 74.4 % of the total grain harvest of rye in the country is grown in the Volga FD, Bashkortostan, Tatarstan, Orenburg and Saratov regions [24]. Rye samples received from these regions were not contaminated with DON.

According to monitoring results, it can be concluded that wheat and corn are the most frequently contaminated with DON among grain, and, barley to a lesser extent. The growth in DON occurrence in grains in recent years (2014, 2017 and 2018) could be explained not only by weather conditions auspicious for toxin production by *Fusarium* genus but also by changes in *F. graminearum* areas. According to Gagkaeva and others (2014), over the last few years these fungi spread into new territories located to the north from their usual habitat [13]. Starting from 2003, resistant populations of *F. graminearum* species occurred in Primorye and North Caucasus, spread into new areas, for example, into the north-western part of Russia, during local weather changes characterized with climatic conditions auspicious for microfungi [24]. The authors believe that the existing climate warming, especially in winter, makes for *F. graminearum* survival in new areas and the fungi get adapted to colder environment [13]. Agriculture intensification might be another reason for growing DON contamination.

Monitoring results confirmed that the Southern, the North Caucasian, and Far Eastern FDs remained the primary areas where *Fusarium* head blight was widely spread. At the same time in 2016–2018 frequency of DON detection grew in areas located to the north from the aforementioned regions.

Assessment of health risks caused by DON contaminations in food wheat grains.

Wheat is a primary source of DON intake with food. When calculating probable DON intake into the human body, we used average mycotoxin contents in samples of grains harvested in the same year (*M*, Table 1). Toxin intake per person was calculated within a sce-

nario when DON contents in food products made from processed wheat corresponded to its contents in grain; that is, processing didn't produce any significant effects on DON contents in a finished product. This approach corresponds to results obtained in studies on stability of the toxin during food processing and cooking: when grain is refined, the toxin contents usually remain the same or go down to 22–23 % [15, 27]; any thermal treatment doesn't influence DON contents [15, 27, 28].

Data on consumption of wheat products (wheat bread, grocery, flour confections, wheat flour, semolina, and macaroni) were taken from budget statistical reports issued by the Federal State Statistic Service and based on a sample survey over budgets spent by households⁹. Average consumption of food products made of processed wheat decreased over the last 25 years, from 267 g in 1993 to 209 g in 2018; it was taken into account when DON intake was calculated. Average person's weight was taken as equal to 60 kg.

Calculated total DON intake per person on average in Russia was significantly different from year to year. DON intake varied from 0.2 % (in 1999) to 140 % (in 1992) of DON PMTDI but remained lower than fixed DON PMTDI, 1 µg/kg of body weight, in the most cases except in 1992 (140 %) and 2014 (102 %) (Table 6) [20].

We should note that high average calculated DON intake with food products made of wheat harvested in 2014 for the country population didn't differ significantly from the intake calculated for the most risky regions; it was partly due to the fact that 88 % of all grain samples for analysis were received from the Southern and the North Caucasian FDs. More profound analysis of estimated daily DON intakes revealed that toxin intake was higher in the Southern and the North Caucasian FDs than on average in the country during the survey and varied from 3 % (in 2004 and 2009) to 410 % of PMTDI (in 1992). Calculated DON intake was also higher than PMTDI in the

⁹Potreblenie produktov pitaniya v domashnikh khozyaistvakh (byulleten') [Food products consumption in households (bulletin)]. *Rosstat*. Available at: <https://rosstat.gov.ru/compendium/document/13292> (March 03, 2021) (in Russian).

Table 6

Calculated daily DON intake with food products made of wheat grain harvested in 1989–2018

A harvest year	Estimated daily intake ($N_{calc.}$), $\mu\text{g}/\text{kg}$ of b.w. a day (% of PMTDI)	
	On average in Russia	The Southern and the North Caucasian FD
1989	0.96 (96.0)	–
1990–1991	0.07 (7.0)	–
1992	1.40 (140.0)	4.10 (410.0)
1993	0.89 (89.0)	2.18 (218.0)
1994	0.12 (12.0)	0.29 (29.0)
1995	0.12 (12.0)	0.68 (68.0)
1996	0.07 (7.0)	0.18 (18.0)
1997	0.08 (8.0)	0.20 (20.0)
1998	0.12 (12.0)	0.48 (48.0)
1999	0.002 (0.2)	–
2000	0.04 (0.4)	0.30 (30.0)
2001	0.036 (3.6)	0.22 (22.0)
2002	0.028 (2.8)	0.28 (28.0)
2003	0 (0)	0 (0)
2004	0.004 (0.4)	0.03 (3.0)
2005	0.084 (8.4)	0.36 (36.0)
2006	0.096 (9.6)	0.18 (18.0)
2007	0.066 (6.6)	0.24 (24.0)
2008	0.07 (7.0)	0.17 (17.0)
2009	0.025 (2.5)	0.03 (3.0)
2010	0.18 (18.0)	0.14 (14.0)
2011	0.04 (4.0)	0.12 (12.0)
2012	0 (0)	0 (0)
2013	0.04 (4.0)	0.04 (4.0)
2014	1.02 (102.0)	1.12 (112.0)
2015	0.05 (5.0)	0.05 (5.0)
2016	0.16 (16.0)	0.26 (26.0)
2017	0.63 (63.0)	1.09 (109)
2018	0.12 (12.0)	0.56 (56.0)

Southern and the North Caucasian FDs in 1993, 2014, and 2017 and accounted for 218, 112 and 109 % of PMTDI accordingly. The aforementioned ascending trend in wheat contamination calls for implementing necessary activities aimed at control over food grain safety.

Conclusions. We have analyzed the results obtained through long-term monitoring over food grain contamination with mycotoxins accomplished by the Federal Service for Surveillance over Consumer Rights Protection and Human Well-being with active participation by the Federal Research Center of Nutrition, Biotechnology, and Food Safety. The analysis confirmed that grains grown in the Southern, the North Caucasian, and the Far Eastern FDs were the most susceptible to DON contamination. At the same time, there is a growth in frequency of DON detection in

grains grown in more northern regions and this indicates that *Fusarium* head blight is spreading over new areas and grain contamination with mycotoxins producers is becoming more probable. This area of contamination can increase when grain is transported from one region to another and this means it is necessary to control grain quality, primarily, when it is used as seed grain.

The highest frequency of DON detection and highest DON contents are typical for wheat and corn among all examined grains. It was shown that calculated daily DON intake with food wheat products was higher than PMTDI DON set by JECFA in some years over the examined period. DON intake was more likely to exceed PMTDI for people living in the Southern and the North Caucasian FDs.

To reduce health risks caused by food grain contamination with DON, it is vital to implement activities aimed at stricter control over spread of *Fusarium* head blight in the RF. Special attention should be paid to monitoring over safety of wheat and corn food grains, in particular, those grown in the Southern and the North Caucasian FDs.

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