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## Occurrence of residues of fipronil and other acaricides in chicken eggs and poultry muscle/fat

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### Abstract

Following the detection of fipronil residues in eggs resulting from a misuse of non-approved veterinary medicinal products in poultry farms against red mites, an ad-hoc monitoring programme was set up in the EU. Member States have provided results for 5,439 samples of eggs and chicken muscle/fat which were analysed for fipronil and a number of additional active substances which were proposed by the European Commission to be monitored in view of potential misuse in poultry farms against red mites. The report summarises the data sampled during the period 1 September 2017 to 30 November 2017 and reported to EFSA. It provides an overview on the results with regard to frequency of occurrence and compliance with the legal limits. The results are presented by country of origin, by type of food product, and by the active substances analysed.

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**Keywords:** fipronil, chicken eggs, poultry, acaricides, monitoring data, compliance with legal limit

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## Summary

As a consequence of the identified misuse of fipronil in chicken farms, Member States and the European Commission agreed in the PAFF Committee (European Commission Standing Committee on Plants, Animals, Food and Feed) meeting held on 30 August 2017 in Brussels to organise a specific monitoring programme.

The European Commission requested the European Food Safety Authority (EFSA) for technical assistance in the framework of Article 31 of Regulation (EC) No 178/2002 to set up the ad-hoc data collection and to summarise the results of the ad-hoc monitoring programme in a scientific report.

The major purpose of the ad-hoc monitoring programme was to get a comprehensive view on the contamination of eggs and poultry products related to illegal uses of acaricides. Member States were requested to take samples of chicken eggs, chicken fat and muscle between 1 September and 30 November 2017, to analyse them for fipronil and additional acaricides and to report the results to EFSA.

The information provided by Member States and Iceland was analysed as requested by the European Commission with regard to the number and percentage of samples with residues below/at or above the Limit of Quantification (LOQ) and the number and percentage of samples with residues below or at the European Union Maximum Residue Level (EU MRL)/above the EU MRL.

Overall, results for 5,439 samples were submitted to EFSA from the sampling period 1 September 2017 to 30 November 2017, covering in total 53,655 individual determinations. Among these samples, 742 samples contained residues exceeding the legal limit (i.e. above the MRL). MRL exceedances were almost exclusively related to fipronil and were associated with unprocessed chicken eggs (601 samples), fat of laying hens (134 samples), muscle of laying hens (5 samples) as well as dried egg powder (2 samples).

Samples that exceeded the legal limit originated from the Netherlands (664 samples), Italy (40 samples), Germany (13 samples), Poland (11 samples), Hungary (6 samples), France (5 samples), Slovenia (2 samples) and Greece (1 sample).

Among the 66 substances that were recommended to be analysed in the framework of the ad-hoc monitoring programme, the only substances found in quantifiable concentrations were fipronil (915 determinations) and amitraz (2 determinations).

The sampling strategy applied had a major influence on the MRL exceedances.

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## 1. Introduction

### 1.1. Background (provided by the requestor)

On 20 July 2017, Belgium informed the European Commission on the identification of eggs contaminated with fipronil. Since then, in many European Union (EU) countries and also in certain Third Countries a similar contamination of eggs was identified.

Fipronil is an acaricide, which is not classified as an 'allowed substance' for use as a veterinary medicinal product on food-producing animals. Fipronil is authorised as an active substance (insecticide) in plant protection products according to Regulation (EC) No 1107/2009<sup>1</sup>; however, residues of fipronil are not expected in eggs and poultry muscle/fat due to the fact that the authorisations exist only in a limited number of crops. Furthermore, there is no biocidal product containing fipronil, which is allowed for use against red mites in poultry stables in any EU Member State.

The fipronil contamination thus results from the illegal use of fipronil on laying hens and their farms.

In order to get a comprehensive view on the contamination of eggs and poultry muscle/fat due to possible illegal uses of acaricides, an ad-hoc data collection by the EU Member States was agreed in the Standing Committee on Plants, Animals, Food and Feed (SC PAFF) Section Novel Food and Toxicological Safety of the Food Chain of 30 August 2017. Residues of fipronil and other acaricides, for which misuse could be expected, shall be monitored in order to get a comprehensive view on the contamination of eggs and poultry muscle/fat due to illegal use of acaricides. The Member States agreed to focus the data collection on domestically produced fresh products, in order to facilitate the follow up in case of the identification of non-compliances. The Commission in collaboration with the European Food Safety Authority (EFSA) circulated the scope and reporting requirements of this data collection to the Member States in the document 'Ad hoc data collection as a follow up to the identified illegal use of fipronil in poultry farms'.

For fipronil and most substances within the scope of the monitoring exercise, maximum residue levels (MRLs) are established under Regulation (EC) No 396/2005<sup>2</sup> on maximum residue levels of pesticides in or on food and feed of plant and animal origin. Therefore the compliance or non-compliance of the residues of these substances in eggs and poultry muscle/fat needs to be evaluated according to the residue definitions and MRLs laid down in this legislation.

### 1.2. Terms of Reference (provided by the requestor)

EFSA is requested to perform the following tasks:

- 1) Identifying/liasing with the single national data providers in the national organisations;
- 2) Preparing technical documents/guidelines for the Standard Sample Description (SSD) coding of the fipronil and other substances residues results;
- 3) To set-up and run the data collection using the EFSA Data collection framework (DCF);
- 4) To provide support to the national data providers during the data collection;
- 5) To prepare a scientific report, which summarises for the samples taken between 1 September 2017 and 30 November 2017 the main findings in terms of
  - a) number and percentage of samples with residues below, at or above the Limit of Quantification (LOQ) and
  - b) residues below, at or above the EU Maximum Residue level (MRL) set in Regulation (EC) No 396/2005.

The above summary statistics (point 5.a and 5.b) should be performed by food item, by substance and by country of origin of the samples tested. The deadline for the publication of the scientific report was agreed to be 31 January 2018.

No Member State consultation is required on the scientific report as it will be a summary of occurrence data.

<sup>1</sup> Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC. OJ L 309, 24.11.2009, p. 1–50.

<sup>2</sup> Regulation (EC) No 396/2005 of the Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC. OJ L 70, 16.3.2005, p. 1–16.

The European Commission should be provided with an Excel table containing per commodity/substance combination the percentage of samples with residues below the LOQ, the percentage of samples with residues between the LOQ and the MRLs, the percentage of samples with residues above the MRL and the minimum and maximum reported LOQ. A table should be provided for the samples taken between 1 January 2017 and 31 August 2017 and another table for the samples taken between 1 September 2017 and 30 November 2017.

### 1.3. Interpretation of the Terms of Reference

In order to address bullet points 1–4 of the Terms of Reference, EFSA has set up the ad-hoc data collection inviting the competent national authorities to share their data via the EFSA DCF. A guidance document specifically focussing on the types of samples and active substances covered by the specific monitoring programme was prepared to ensure high data quality with regard to consistent coding of the data which is a prerequisite to perform meaningful data analysis. During the data submission phase, EFSA provided support to the data providers. Details on these activities however are not subject of this report.

The current report was prepared to address point 5.a and 5.b of the Terms of Reference, summarising the results of the specific monitoring programme established as a consequence of the recent findings of fipronil residues in chicken eggs resulting from illegal use of this acaricide.

Upon request of some Member States, the data collection was kept open until end of December 2017. In the current report, all results submitted until the 23 December 2017 were considered.

In the course of the detailed data analysis, it was noted that for a substantial number of samples the conventions on coding of the data were not fully respected.<sup>3</sup> Since these mistakes would have an influence on the validity of the data analysis, in agreement with the European Commission, EFSA contacted the reporting countries, asking for the correction of the data. On 26 March 2018 the last corrections have been submitted to EFSA.

When performing the data exploration, it became evident that Member States took different approaches for selecting samples; while some countries applied a risk based approach, focussing on producers that were under suspicion of misuse of fipronil, other countries selected random samples, without targeting products/producers that were expected to have violated legal provisions as regards the use of certain substances in animal production. Hence, without taking into account the sampling strategy, a comparison of the results provided by the different Member States would be misleading, since the sampling strategy is a parameter that has a major impact on the findings as regards MRL exceedance rate. EFSA therefore decided to include the sampling strategy as an additional parameter in the data analysis and to present the results separately for samples taken by random sampling and for suspect/targeted samples.

Taking into account the delays resulting from corrections of wrongly coded data and the additional data analysis, it was agreed with the European Commission to extend the deadline until 20 April 2018.

## 2. Data and methodologies

### 2.1. Data

Since the major purpose of the ad-hoc monitoring programme was to get a comprehensive view on the contamination of eggs and poultry products related to illegal uses of acaricides in chicken farms, the programme should focus on eggs, muscle and fat derived from laying hens. However, it was agreed with the European Commission that data on chicken broilers could be submitted as well. The complete list of food products covered by the specific monitoring programme is summarised in Table 1. The table also comprises the codes that should be used to describe the samples in the SSD format (EFSA, 2015, 2016, 2017).

<sup>3</sup> In a number of cases, Member States did not code the results with the appropriate parameter code for the legal residue definitions for fipronil or amitraz, but used a sub-code that was created to report individual components of the legal residue definitions. Without re-coding of these data by EFSA, the information would have been excluded from the data analysis, biasing the results. In addition, comparing the information provided via the Rapid Alert System for Food and Feed, it was noted that in certain cases the sample origin was not reported correctly. The data provider confirmed that the recoding of the information was appropriate. Moreover, the sample strategy was not reported correctly for a number of samples; hence these errors were also corrected.

**Table 1:** Food products to be analysed under the ad-hoc monitoring programme

MATRIX code	Description of the product	Details on the product analysed	Code describing the type of processing	Description of type of processing
P1030010A	Eggs (chicken)	Whole egg or whole egg liquid	T999A	Fresh/unprocessed
			T998A	Frozen
			T151A	Pasteurised
P1030010.1A	Eggs yolk (chicken)	Liquid egg yolk	T999A	Fresh/unprocessed
			T998A	Frozen
			T151A	Pasteurised
P1030010.2A	Eggs white (chicken)	Liquid egg white	T999A	Fresh/unprocessed
			T998A	Frozen
			T151A	Pasteurised
P1030010A	Eggs (chicken)	Whole egg powder	T131A	Dehydrated
P1030010.1A	Eggs yolk (chicken)	Egg yolk powder	T131A	Dehydrated
P1030010.2A	Eggs white (chicken)	Egg white powder	T131A	Dehydrated
P1016010_002.1B	Muscle (poultry)	Chicken (laying hens)	T999A	Fresh/unprocessed
			T998A	Frozen
P1016010_002.2B	Muscle (poultry)	Chicken (broilers) <sup>(a)</sup>	T999A	Fresh/unprocessed
			T998A	Frozen
P1016020_002.1A	Fat (poultry)	Chicken (laying hens)	T999A	Fresh/unprocessed
			T998A	Frozen
P1016020_002.2A	Fat (poultry)	Chicken (broilers) <sup>(a)</sup>	T999A	Fresh/unprocessed
			T998A	Frozen

(a): Supplementary results.

Under the specific monitoring programme, primarily fipronil and its related metabolites should be investigated, but eleven additional acaricides were included in the scope of the monitoring programme to ensure a comprehensive approach, i.e. amitraz, bifenthrin, cypermethrin, diazinon, etoxazole, flufenoxuron, ivermectin, pyridaben, pyriproxyfen, thiamethoxam and trichlorfon. The respective residue definitions and maximum residue levels (MRLs) for the target substances established under Regulation (EC) No 396/2005 and Regulation (EU) No 37/2010<sup>4</sup> are listed in Appendix A.1.

An extended list of an additional 54 substances was suggested by the European Commission to be taken into account for possible inclusion in the analytical methods used to analyse the samples (voluntary substances, see Appendix A.2). Member States were asked to submit results also for these active substances as far as analysed.

The sampling period was restricted to the time period from 1 September 2017 to 30 November 2017 (sampling window)<sup>5</sup>; all data submitted to EFSA until 23 December 2017 were taken into account in this report.

Member States were asked to take a sufficient number of samples to get a representative view on the situation as regards the chicken farms. A minimum number of samples per Member State and/or food product was not defined.

Competent authorities of 25 Member States and Iceland submitted monitoring data on samples analysed under the specific monitoring programme to EFSA. Malta, Lithuania<sup>6</sup> and Sweden<sup>7</sup> did not provide any data.

<sup>4</sup> Commission Regulation (EU) No 37/2010 of 22 December 2009 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin. OJ L 15, 20.1.2010, p. 1–72.

<sup>5</sup> The assessment of samples taken earlier in 2017 was not within the remit of the current report. (Results for 2,910 samples were submitted to EFSA related to the sampling period 1 January 2017 to 31 August 2017, covering in total 13,363 individual determinations. Among these samples, 123 samples contained residues exceeding the MRL; these exceedances were exclusively related to fipronil). This information was shared with the European Commission (see ToR) and will be reported in the framework of the annual report on pesticide residues prepared under Article 31 of Regulation (EC) No 396/2005.

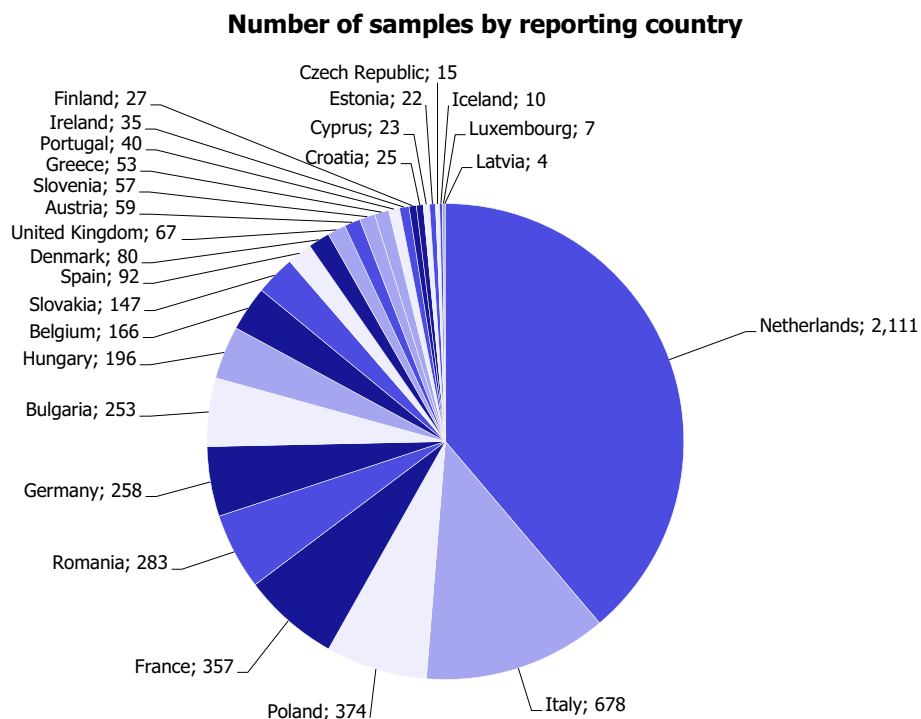
<sup>6</sup> Lithuania provided results for 19 samples, which were taken in August 2017, but no data were reported for samples taken between 1 September and 30 November 2017. The results of the samples taken in August 2017 are not presented in this report.

<sup>7</sup> Sweden informed EFSA that about 80 samples of Swedish produced fresh eggs were analysed within the national control programme of 2017, with no findings of fipronil. The results will be reported in the framework of the data submission under Article 31 of Regulation (EC) No 396/2005.

Overall, the reporting countries submitted valid results for 5,439 samples: 2,540 of these samples were classified by the reporting countries as suspect samples or samples that were targeted towards products/producers where a violation of the legal limits was presumed. The remaining 2,899 samples were taken randomly without specific targeting towards products that would be likely to be non-compliant.

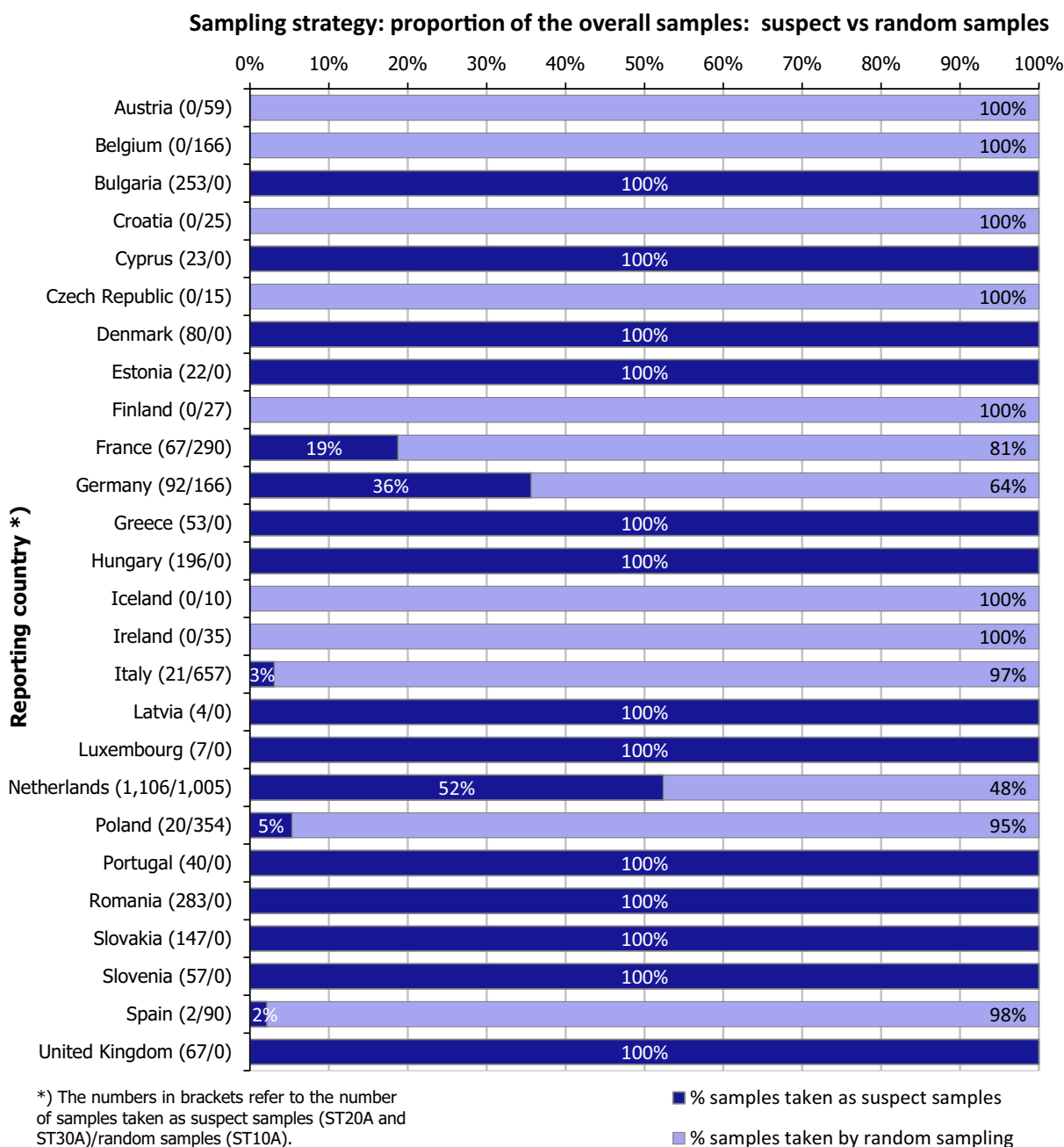
Only a small amount of the samples was related to production according to the principles of organic farming (374 of the 5,439 samples, 6.9%); the remaining samples were related to conventional production.

Figure 1 summarises the number of samples taken by Member States in the framework of the specific monitoring programme. Figure 2 provides the breakdown of random versus suspect/targeted samples by Member State. From this presentation, it is evident that Member States reported different sampling strategies. However, the results are not directly comparable between Member States and between sampling strategy because no specific criteria were defined concerning the sampling strategy.



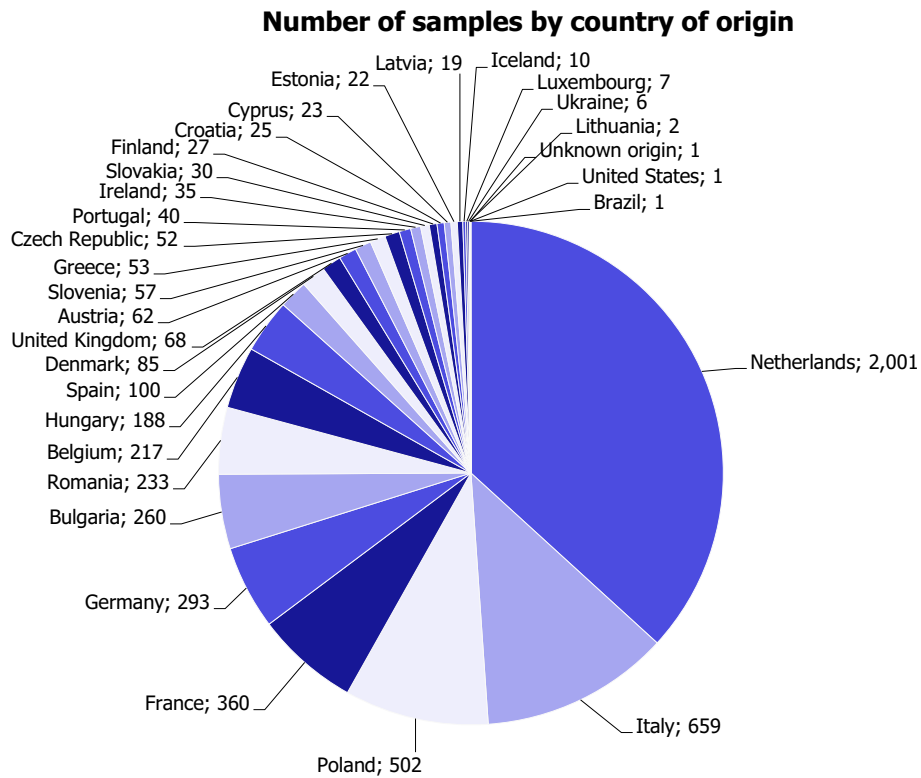
**Figure 1:** Number of samples analysed by Member States and Iceland





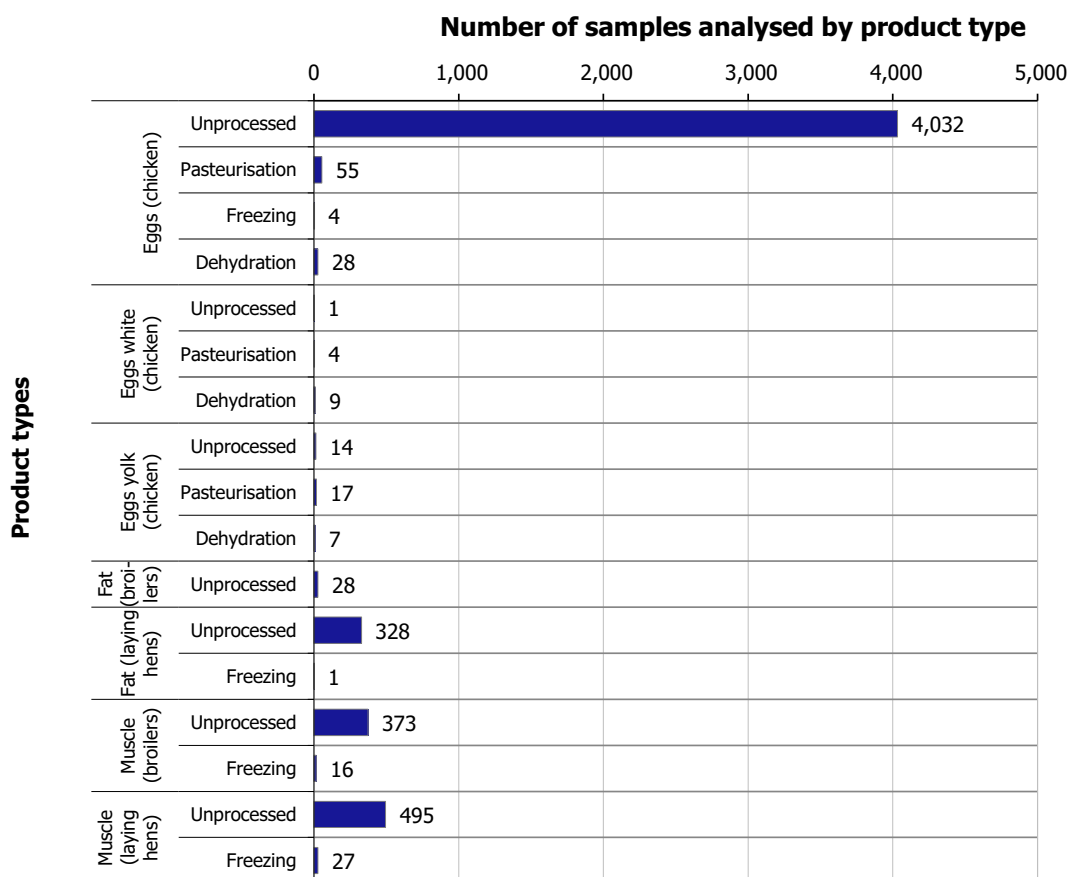
**Figure 2:** Proportion of the overall samples that were reported as suspect vs random samples per country

In most cases, the reporting countries analysed samples that were produced within their own territory. However, nine samples produced in third countries and 350 products produced in one EU Member State but analysed by another Member State were reported. Figure 3 provides the information on the number of samples analysed, broken down by country of origin.



**Figure 3:** Number of samples by country of origin of the samples analysed

To get a better understanding of the type of products analysed, Figure 4 gives a breakdown of the number of samples by food product. The majority of the samples analysed were chicken eggs and egg products (76.7% of the samples), followed by muscle of laying hens (9.6%) and fat of laying hens (6.0%). The specific monitoring programme was primarily targeted towards products from laying hens and not from broiler chickens; thus, the results for muscle and fat of broilers, which accounted for 7.2% and 0.5% of the total number of samples analysed, are considered as supplementary information only.



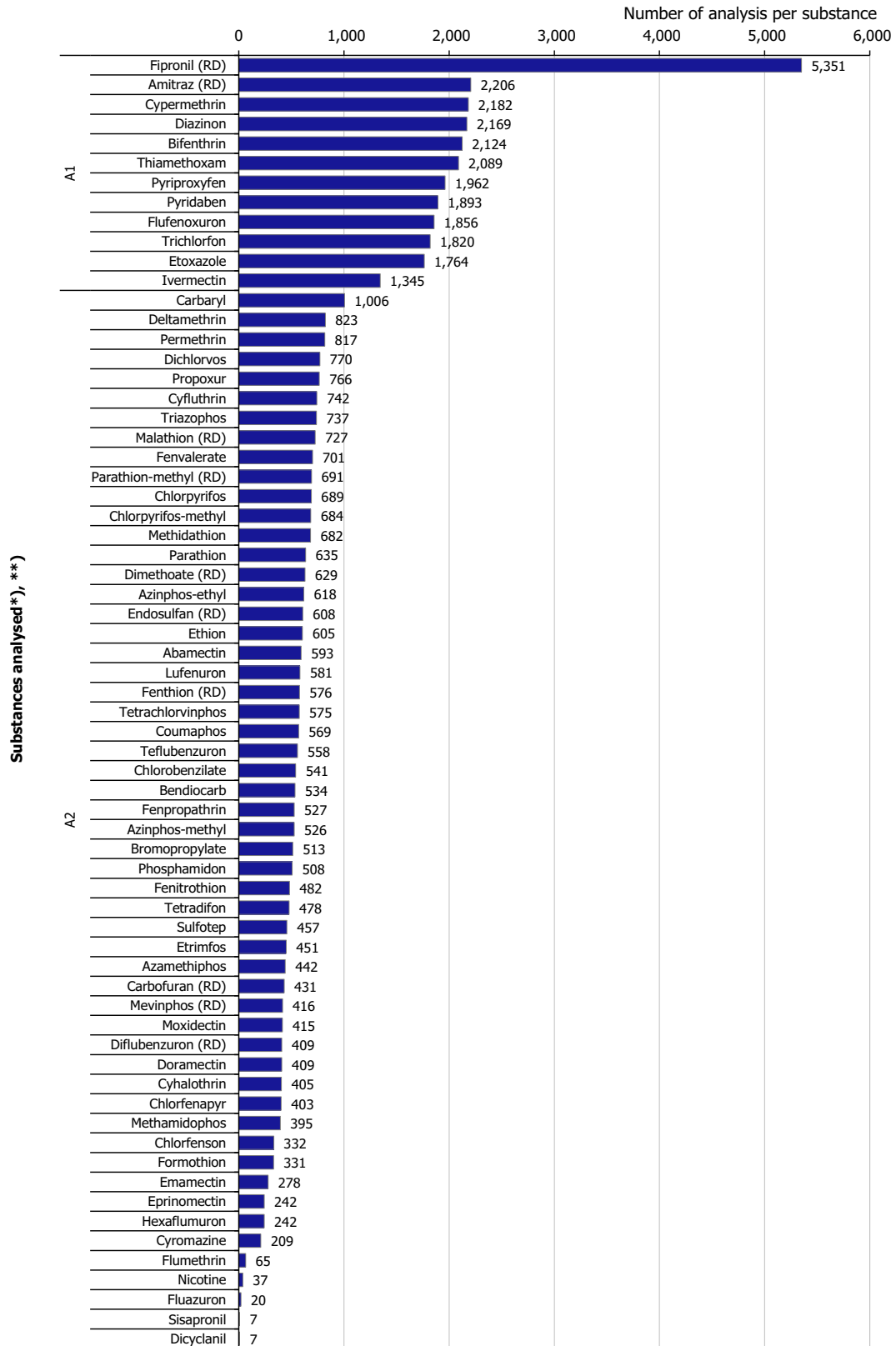
**Figure 4:** Number of samples by product category

Overall, 53,655 individual results (analytical measurements) for the 66 different substances listed in Appendix A.1 and A.2 that were subject of this specific monitoring programme were provided.

In addition, 15,964 additional results on individual metabolites were reported as far as they could be quantified separately, e.g. fipronil sulfone. However, the current mandate did not require a specific analysis of the pattern of metabolites in the samples taken under this ad-hoc monitoring programme and therefore these results are not included in this report.

Almost all samples were analysed for fipronil residues (5,351 out of 5,439 samples). A high number of samples was analysed for amitraz residues (2,206 samples), cypermethrin (2,182 samples), diazinon (2,169 samples), bifenthrin (2,124 samples), thiamethoxam (2,089 samples), pyriproxyfen (1,962 samples), pyridaben (1,893 samples), flufenoxuron (1,856 samples), trichlorfon (1,820 samples), etoxazole (1,764 samples) and ivermectin (1,345 samples). For the additional substances recommended for analysis (Appendix A.2), the available database is less robust: with the exception of carbaryl, for all of these substances less than 1,000 results are available (Figure 5).

The number of substances analysed by the different reporting countries differed considerably (analytical scope): while some reporting countries focussed exclusively on fipronil (e.g. Bulgaria, Latvia and the United Kingdom), other Member States covered a wide range of substances (e.g. 58 substances covered by Germany and Hungary); a wide range of substances (equal to or more than 50 substances) was also analysed by Denmark, Austria, Poland, the Czech Republic, Ireland and Portugal.



\*) Results in accordance with the legal residue definition established for the substance concerned. (RD) after the name of the substance refers to cases where the legal residue definition covers not only the parent compound, but also metabolites.

\*\*) A1: Substances of Appendix A.1.  
A2: Substances of Appendix A.2.

Figure 5: Number of analytical determinations by substance

## 2.2. Methodologies

The information provided by Member States and Iceland was analysed as requested in the Terms of Reference by food product type, by country of origin and by pesticide. The following categories were used to classify results as 'residues below, at or above the Limit of Quantification' and as 'residues below, at or above the EU Maximum Residue Level':

- Residues below the LOQ: samples without any detectable residues and samples with detectable residues, but all below the LOQ were classified as samples without quantifiable residues.
- Residues at or above the LOQ: samples with residue concentrations for at least one substance at or above the LOQ were classified as samples with quantifiable residues.
- Residues below or at the MRL: samples without any detectable/quantifiable residues and samples with detectable/quantifiable residues all below or at the legal limit were described as samples compliant with the MRL.
- Residues above the MRL: samples with residues for at least one substance above the legal limit were considered as samples exceeding the MRL. The evaluation of whether a substance exceeded the MRL was performed by the reporting countries. The measurement uncertainty is not taken into account when deciding whether a result is exceeding the MRL.<sup>8</sup>

The MRL exceedance was assessed only for the 58 substances for which legal limits are established in Regulation (EC) No 396/2005 (see Appendix A); it was restricted to unprocessed products and to fipronil in dehydrated chicken eggs, egg yolk powder and egg white powder.

When data were presented separately for processed and unprocessed products, frozen and pasteurised products were considered as unprocessed since these processing techniques are not expected to have an influence on the magnitude or nature of residues compared to unprocessed products. Egg powder of whole eggs, powder of egg yolk and egg white were classified as processed products.

Since the sampling strategy was considered to be a major factor having an influence on the quantification rate of fipronil or the MRL exceedance rate, the results are presented separately for samples taken randomly and suspect samples, i.e. samples that were targeted towards products/producers where a violation of the legal limits was presumed.

### 2.2.1. Colour codes in the figures

The data are mainly presented in figures which provide a better overview of the results. The following colour codes were used to visualise the type of information:

- Dark blue/dark orange refers to suspect samples;
- Light blue/light orange is used for presenting data on samples taken randomly without targeting towards suspicious producers/products;
- Dark orange/light orange is used to highlight results which exceeded the MRL or where residues occurred at or above the LOQ (quantifiable residues);
- Dark blue/light blue is used to describe results within the legal limit or below the LOQ.

## 3. Results

### 3.1. Results by food product

#### 3.1.1. Residues below and residues at or above the LOQ by food product

As requested in the Terms of Reference, EFSA assessed the frequency of samples with residues below and residues at or above the LOQ. It is noted that the presence of residues in eggs and poultry products as such is not giving any indications of illegal practices. In general, residues in animal

<sup>8</sup> In order to decide whether a sample exceeds the legal limit the measured residue concentration is compared with the MRL applicable for a certain substance in or on a food product. In regulatory enforcement practice, the measurement uncertainty is taken into account to decide whether a sample containing measurable residues is compliant with the legal limit. Thus, even if the numerical residue concentration measured exceeds the MRL, a sample can be still considered compliant with the legal limit. Usually, a measurement uncertainty of 50% is used, unless the analysing laboratory provides specific information on the analytical measurement uncertainty related to the analyte measured. The decision on the MRL compliance is at the discretion of the national competent authority. Thus, not all results which exceed the MRL numerically triggered follow-up enforcement actions at national level.

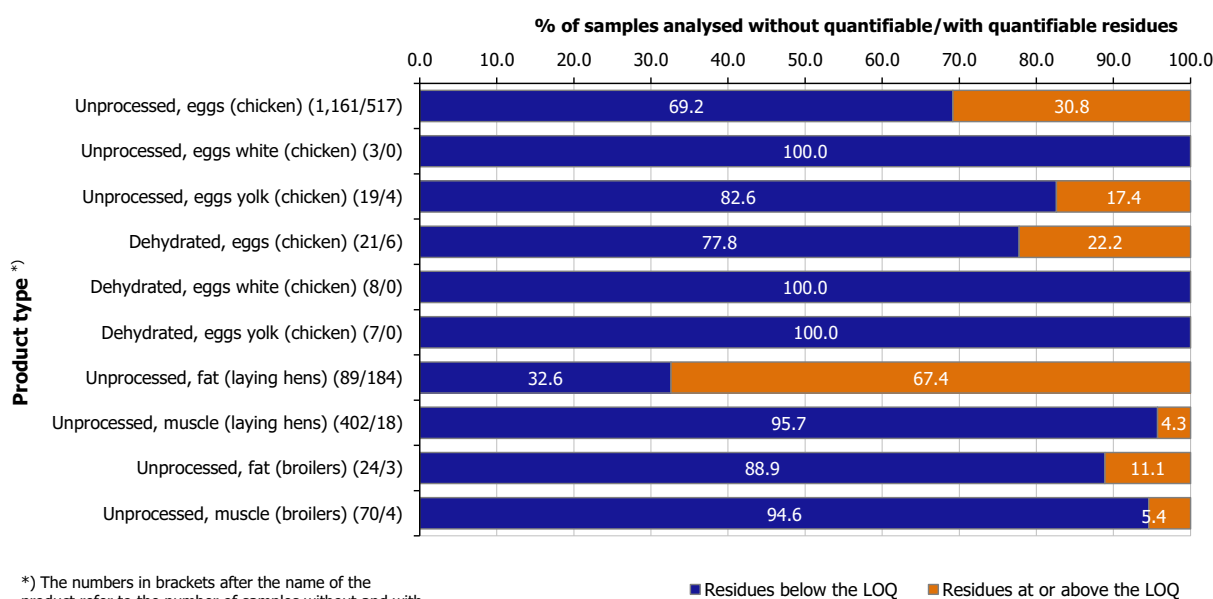
products may occur due to the use of veterinary medicinal products or via carry-over of residues of pesticides in feed. As long as the residues are within the legal limits, the products are considered as compliant with the EU food safety standards. However, the presence of certain substances in food, even if within the legally permitted limits, may give an indication of a possible misuse and should be taken as an indication to further investigate the source of the substance.

In total, 917 samples of the 5,439 samples taken between 1 September and 30 November 2017 (16.9%) contained residues in concentrations at or above the LOQ. Overall, the quantification was higher in the subset of suspect samples (736 samples) compared with the samples taken randomly (181 samples).

Figure 6 visualises the frequency of quantifiable residues in the 2,540 suspect samples for the different food product types (residues below the LOQ vs residues at or above the LOQ). The highest frequency of quantifiable residues were identified for unprocessed fat of laying hens (67.4% of the 273 samples analysed), followed by unprocessed chicken eggs (including frozen and pasteurised eggs) (30.8% of the 1,678 samples), egg powder (dehydrated chicken eggs) (22.2%), unprocessed egg yolks (17.4%), fat of broilers (11.1%), muscle of broilers (5.4%) and unprocessed muscle of laying hens (4.3%). No quantifiable residues were identified in samples of unprocessed egg white, egg white powder and egg yolk powder.

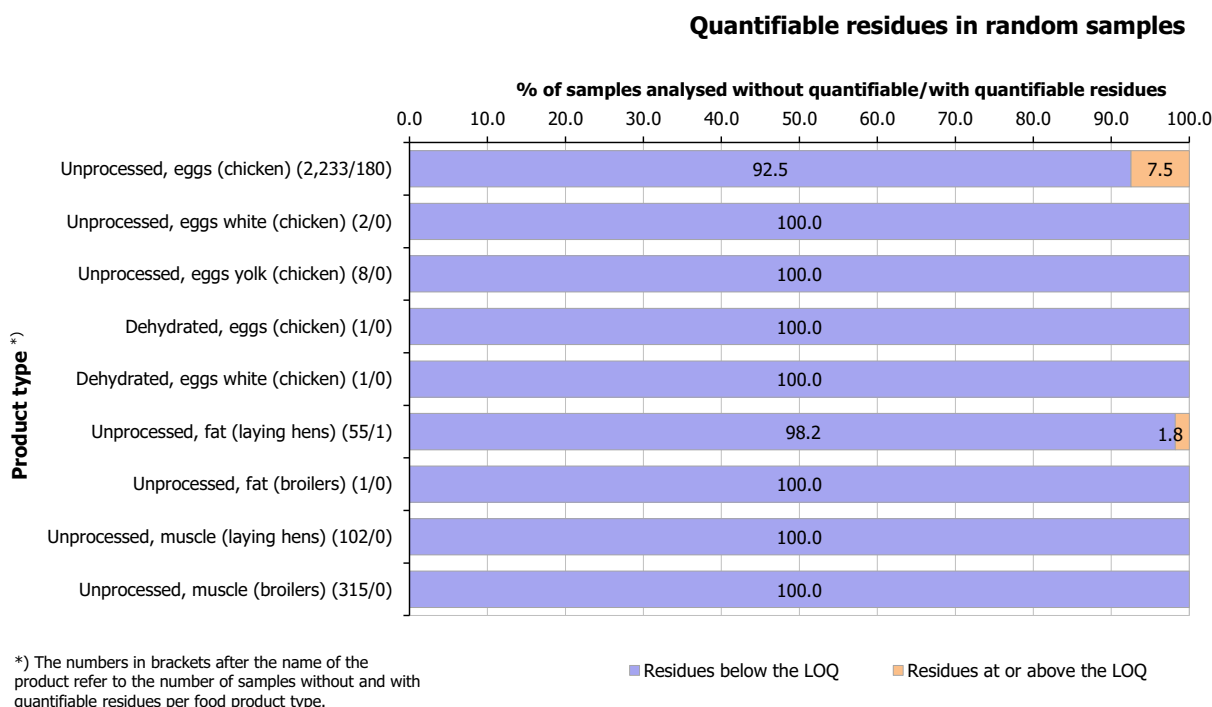
In Figure 6, the number of samples without and with quantifiable residues is reported in brackets next to the name of the product type. The products with the highest number of samples with residues at or above the LOQ were unprocessed chicken eggs (517 samples) followed by unprocessed fat of laying hens (184 samples) and muscle of laying hens (18 samples). For the remaining food products the number of samples with quantifiable residues was below 10. Among the suspect samples, the results above the LOQ were related to fipronil residues only.

### Quantifiable residues in suspect samples



**Figure 6:** Number and percentage of samples with residues below the LOQ and residues at or above the LOQ by product type – suspect samples only

In Figure 7, the same type of information as in Figure 6 is reported for the 2,899 samples taken as random samples. Quantifiable residues were only found in unprocessed chicken eggs (7.5%, 180 samples of the total of 2,413 chicken egg samples taken randomly) and fat of laying hens (1.5%, one sample out of 56 samples analysed). While in two samples of chicken eggs, amitraz was found, all other cases of quantifiable residues were related to fipronil.



**Figure 7:** Number and percentage of samples with residues below the LOQ and residues at or above the LOQ by product type – samples taken by random sampling

### 3.1.2. Residues below or at and residues above the MRL by food product

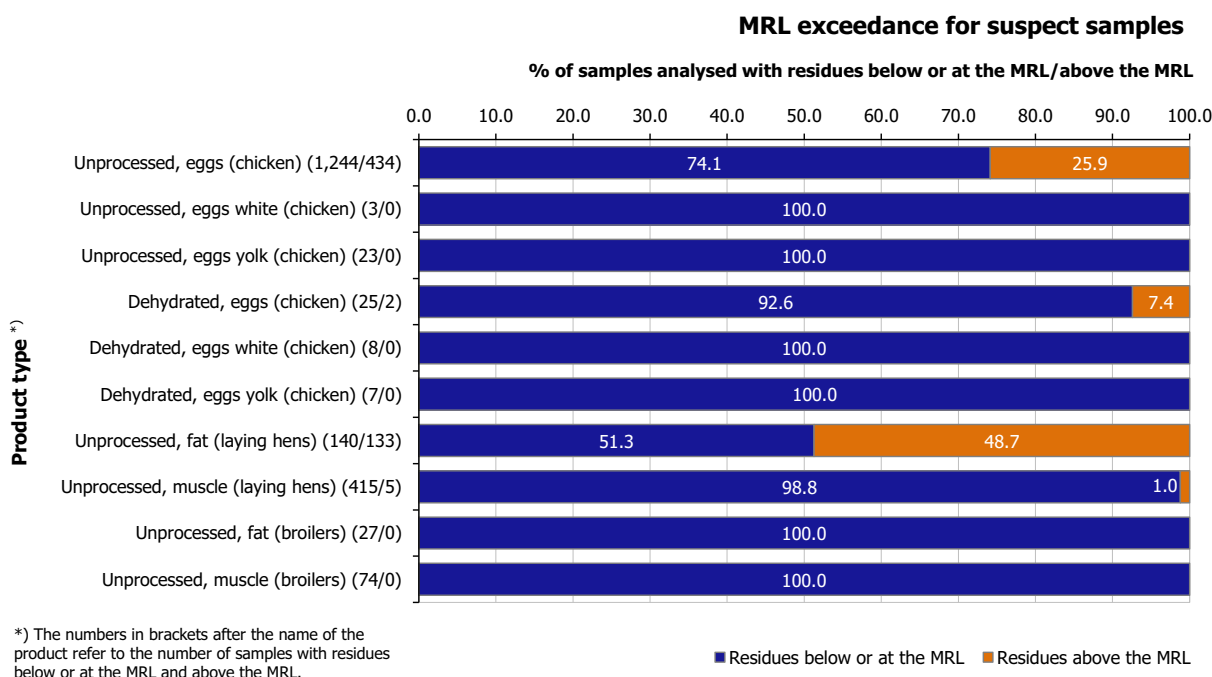
In this section, the results are reported as regards the MRL exceedance based on the evaluation of the reporting country. Overall, 742 samples of the total 5,439 samples taken (13.6%) contained residues in concentrations exceeding a legal limit (i.e. 603 samples of chicken eggs, 134 samples of fat of laying hens and 5 samples of muscle of laying hens). Among the 2,540 suspect samples, 574 contained residues above the legal limit. Considering the 2,899 random samples, 168 contained residues above the legal limit.

Taking the subset of suspect samples, MRL exceedances were related to fat of laying hens (133 samples, 48.7%), unprocessed chicken eggs (434 samples, 25.9%), whole egg powder (2 samples, 7.4%) as well as muscle of laying hens (5 samples, 1%). No MRL exceedances were noted for unprocessed egg white, egg yolk, egg white powder, egg yolk powder, fat and muscle of broiler. In Figure 8, the frequency of MRL exceedances is presented for the different food products analysed. MRL exceedances are visualised in orange while samples without quantifiable residues or residues below or at the legal limit are presented in blue.

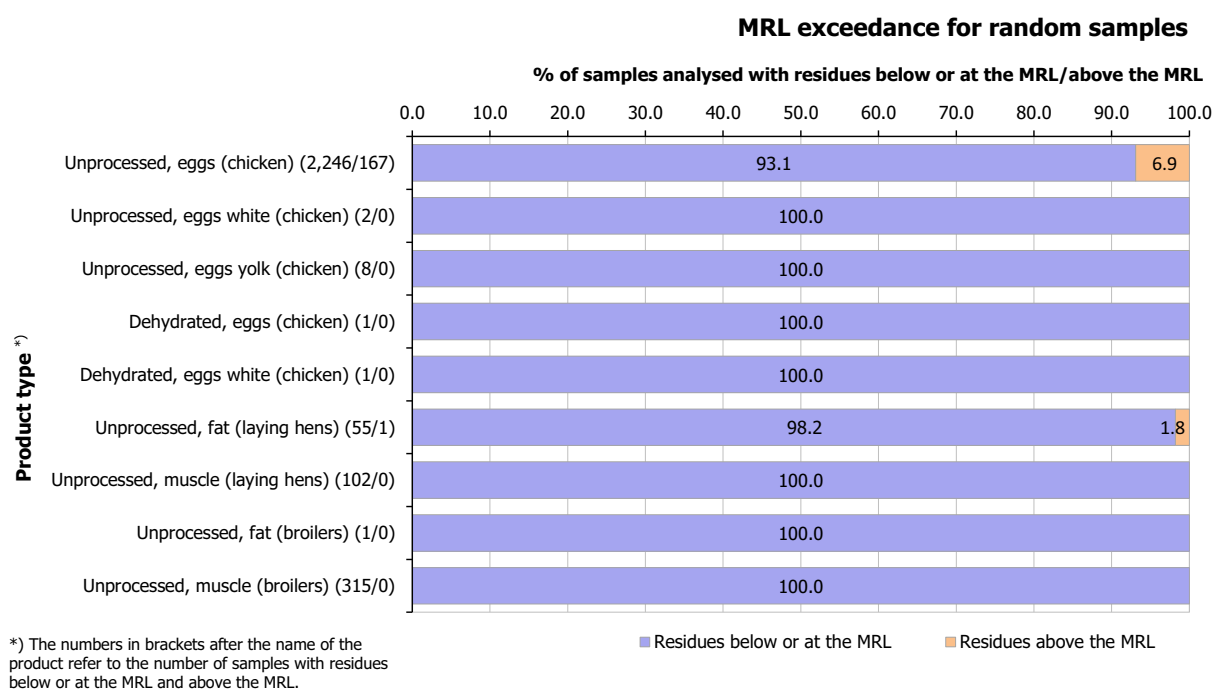
For samples taken by random sampling, the highest frequency of MRL exceedances was identified for unprocessed chicken eggs with 6.9% MRL exceedances (167 samples) followed by fat of laying hens (1.8% of the 56 samples analysed). The results for random samples are presented in Figure 9.

Comparing the results presented in Figures 8 and 9 shows that the frequency of MRL exceedances was considerably lower in the samples taken randomly, compared with the suspect samples.

All MRL exceedances except one were related to fipronil residues. In one sample of chicken eggs (random sampling) an MRL exceedance was noted for amitraz residues.



**Figure 8:** Number and percentage of samples with residues below or at the EU MRL and residues above the EU MRL by product type – suspect samples only



**Figure 9:** Number and percentage of samples with residues below or at the EU MRL and residues above the EU MRL by product type – samples taken by random sampling

### 3.2. Results by sample origin

This section is dedicated to the analysis of the results by country of origin of the sample. As reported before, in most cases, the reporting countries analysed samples that were produced within their own territory. Only nine samples produced in third countries (the United States, Ukraine and Brazil) and 350 products produced in one EU Member State but analysed by another Member State were reported.



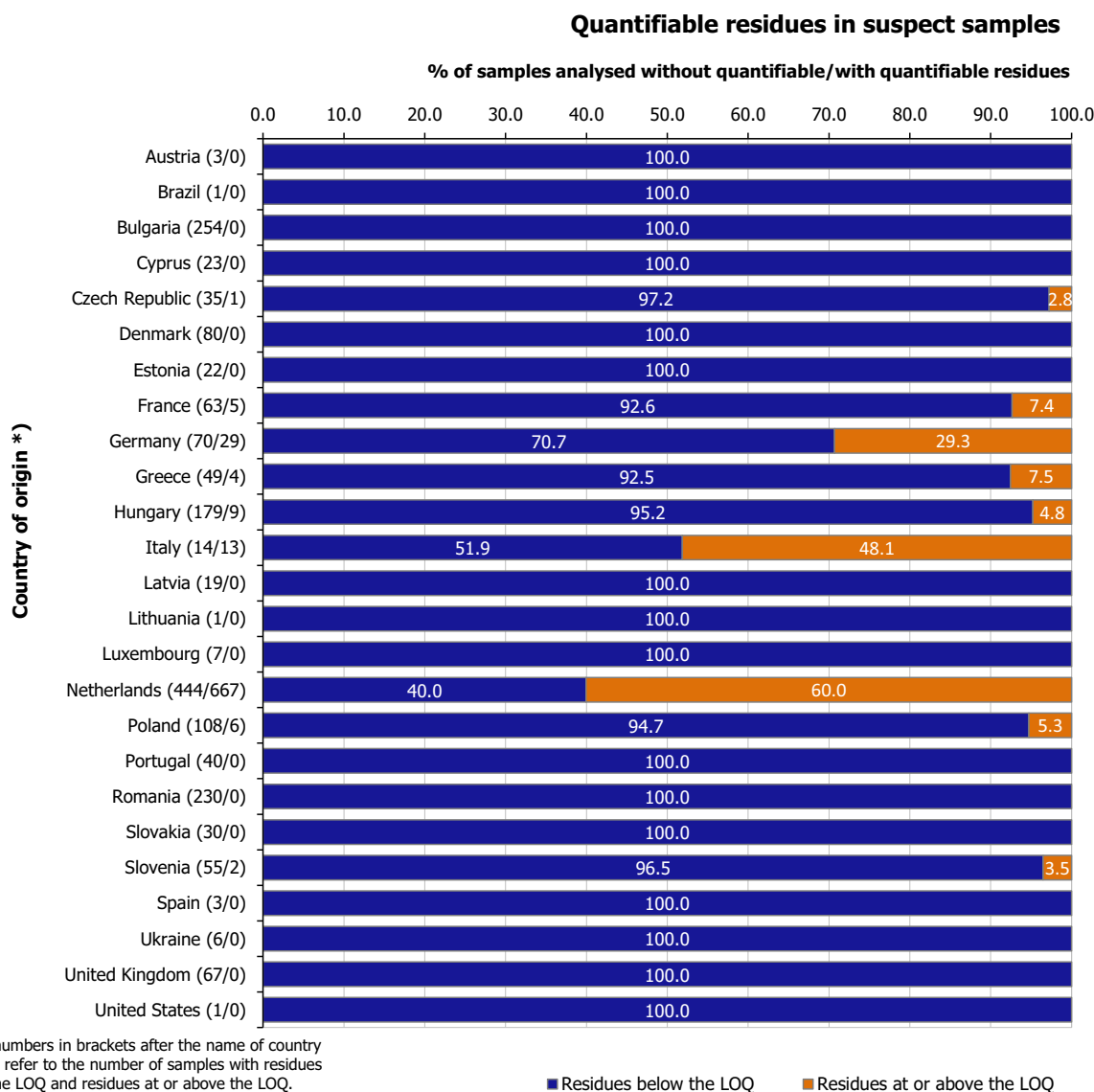
### 3.2.1. Residues below and residues at or above the LOQ by country of origin

Taking into account all samples (suspect and random samples), quantifiable residues were found in products produced in 10 countries, i.e. the Netherlands (803 samples), Italy (47 samples), Germany (30 samples), Poland (14 samples), Hungary (9 samples), France (5 samples), Greece (4 samples), Slovenia and Belgium (2 samples, respectively) and the Czech Republic (1 sample).

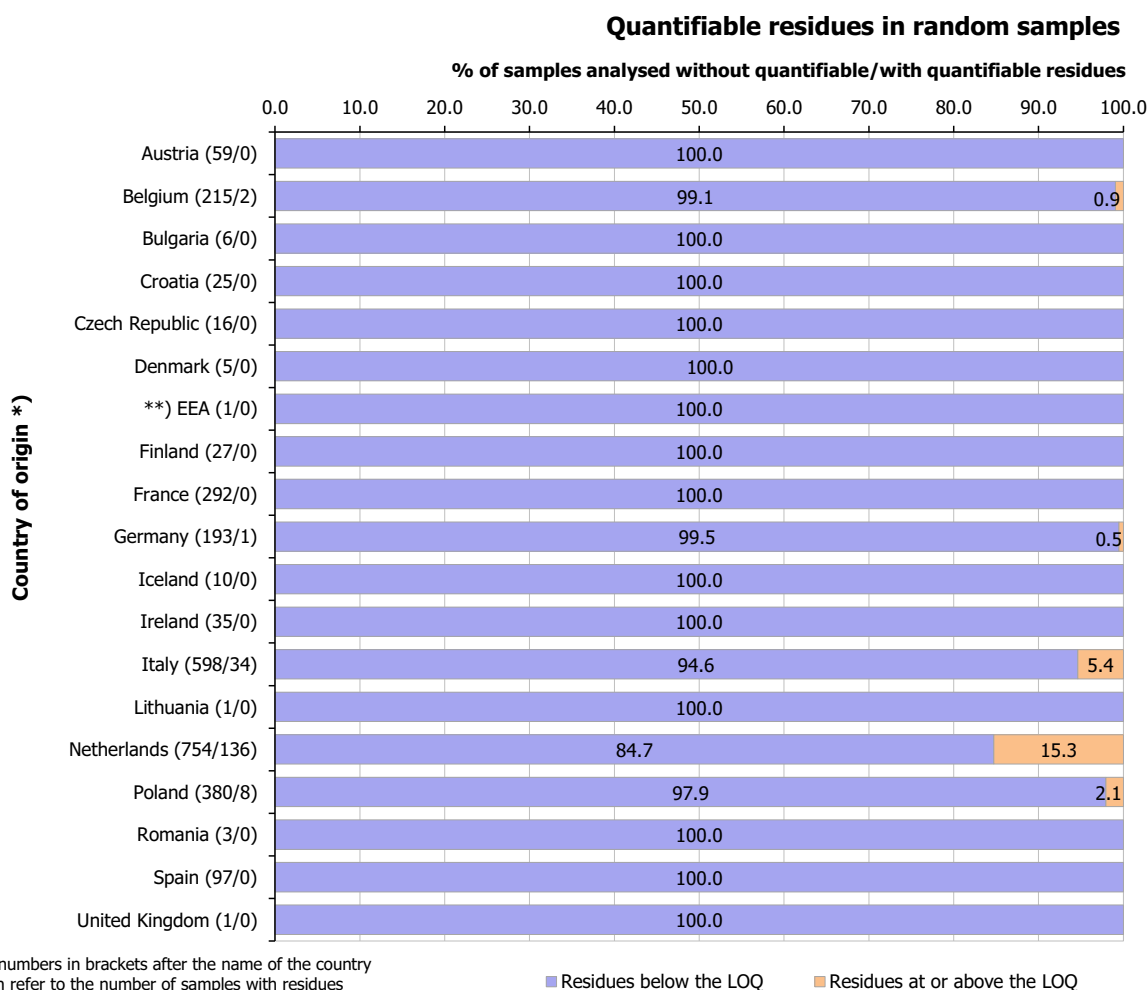
Focussing on suspect samples, the highest number of quantifiable residues was reported in samples produced in the Netherlands (667 of the 1,111 samples analysed), followed by Italy (13 of the 27 samples analysed), Germany (29 of the 99 samples analysed), Greece (4 of the 53 samples), France (5 of the 68 samples) and Poland (6 of the 114 samples).

Figure 11 shows the results for the random samples, where samples from five countries showed quantifiable residues (i.e. the Netherlands (136 samples), Italy (34 samples), Poland (8 samples), Belgium (2 samples) and Germany (1 sample).

Comparing the results of Figures 10 and 11 confirms the previous observation that the sampling strategy was a factor that had a major impact on the quantification rate.



**Figure 10:** Number and percentage of samples with residues below the LOQ and residues at or above the LOQ by country of origin – suspect samples

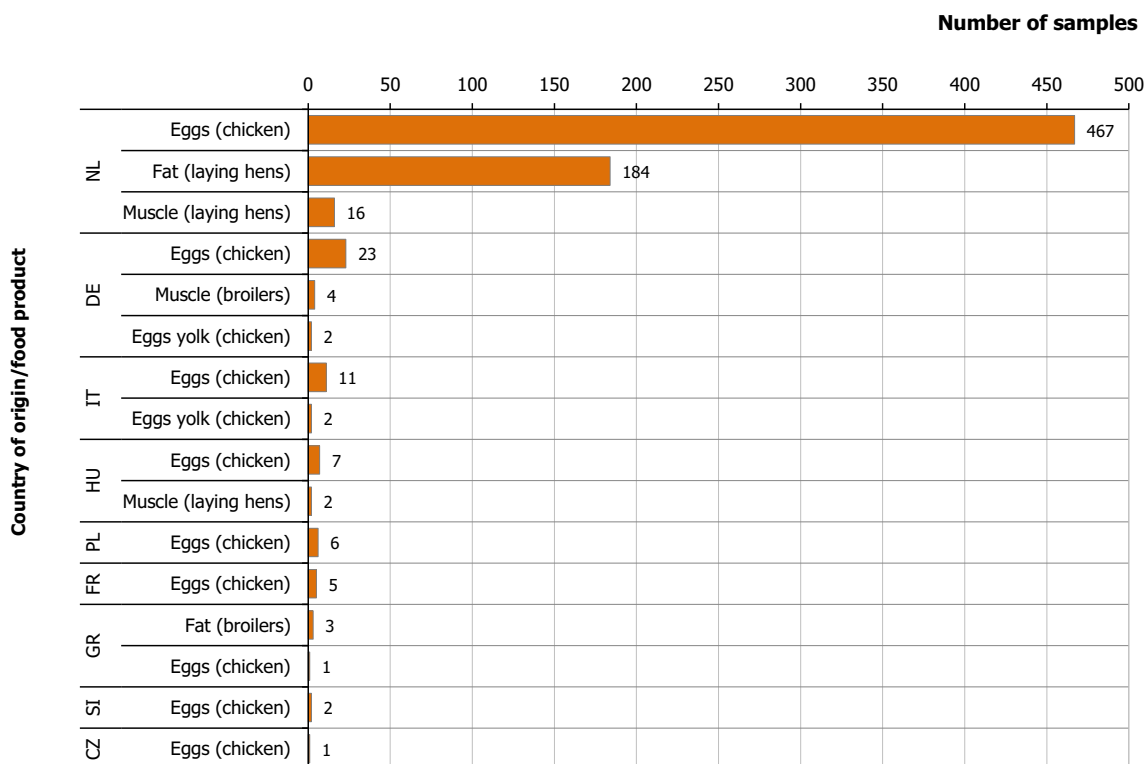


**Figure 11:** Number and percentage of samples with residues below the LOQ and residues at or above the LOQ by country of origin – samples taken by random sampling

In Figures 12 and 13, the number of samples with quantifiable residues is split by country of origin and product type. Considering the suspect samples only (Figure 12), quantifiable residues were found most frequently in samples of chicken eggs (467 samples) produced in the Netherlands, followed by fat of laying hens (184 samples) also originating from the Netherlands. In addition, samples of eggs and egg yolk from Germany (25 samples), Italy (13 samples), Hungary (7 samples), Poland (6 samples), France (5 samples), Slovenia (2 samples), Greece and the Czech Republic (1 sample, respectively) were found to contain quantifiable residues. Samples of muscle of laying hens produced in the Netherlands (16 samples) and Hungary (2 samples), as well as muscle of broilers from Germany (4 samples) and fat of broilers produced in Greece (3 samples) also contained residues in quantifiable amounts.

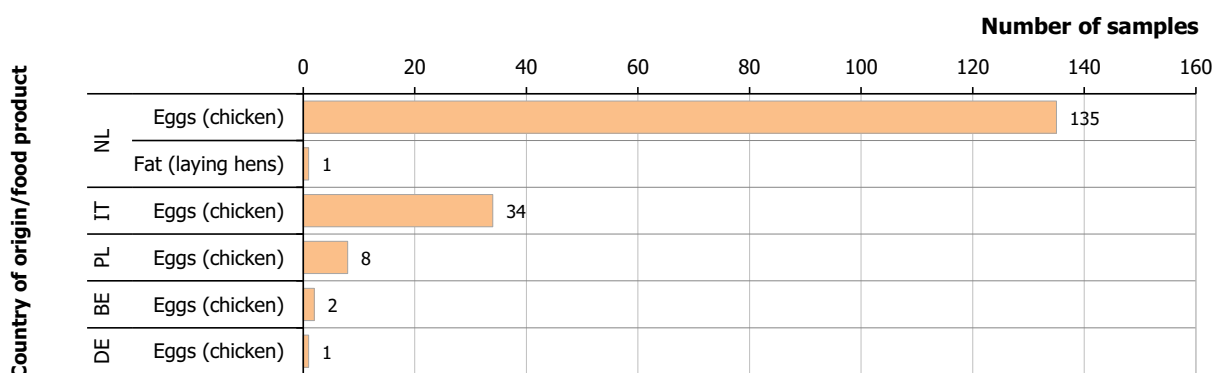
Among the random samples (Figure 13), quantifiable residues were found in products originating from the Netherlands (135 samples of chicken eggs and 1 sample of fat of laying hens), Italy (34 samples of chicken eggs), Poland (8 samples of chicken eggs), Belgium (2 samples of chicken eggs) and Germany (1 sample of chicken eggs).

### Samples of chicken eggs, muscle and fat with quantifiable residues, suspect samples



**Figure 12:** Number of samples with residues at or above the LOQ by country of origin and food product – suspect samples

### Samples of chicken eggs, muscle and fat with quantifiable residues, random samples

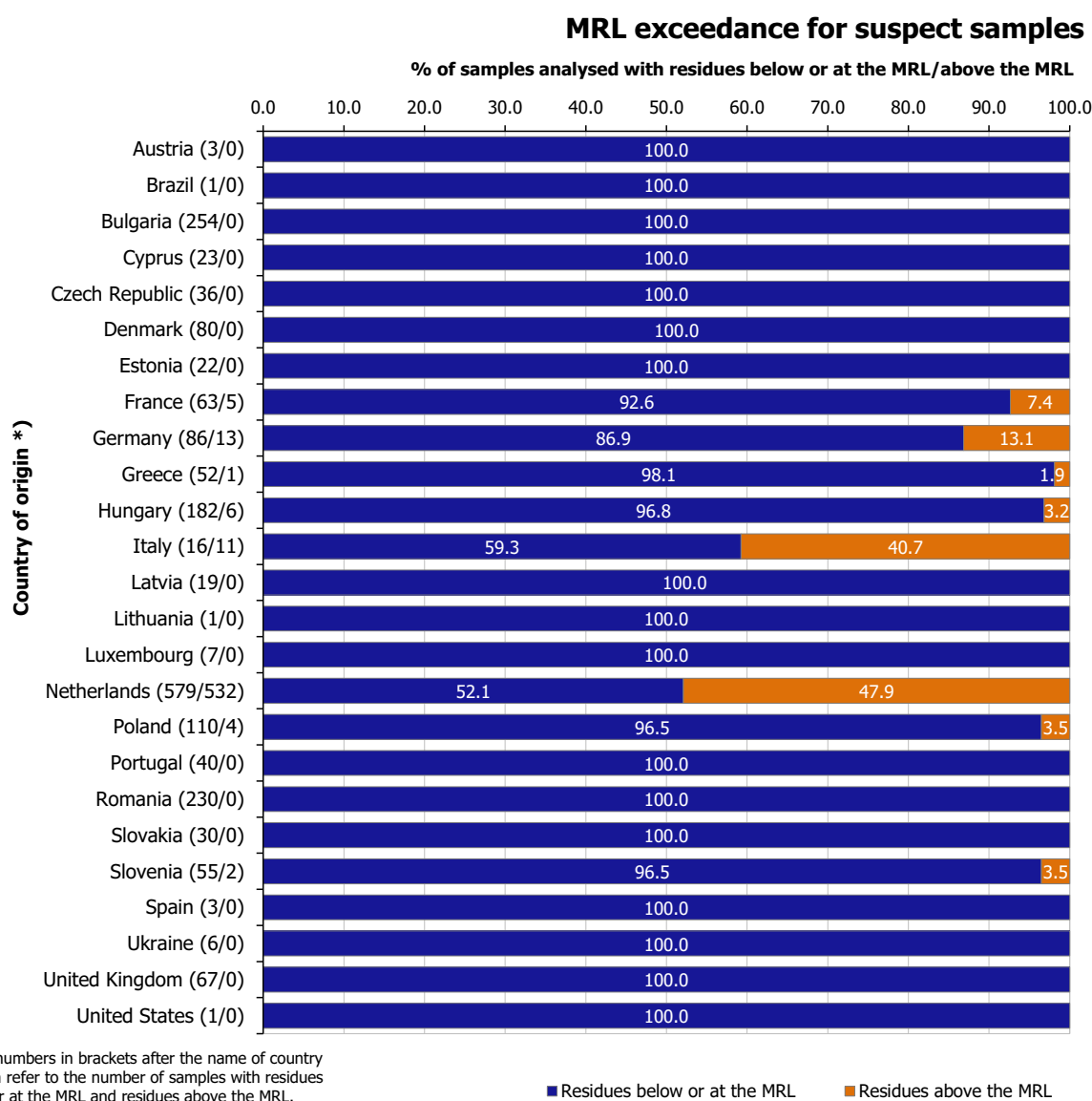


**Figure 13:** Number of samples with residues at or above the LOQ by country of origin and food product – samples taken by random sampling

#### 3.2.2. Residues below or at the MRL and residues above the MRL by country of origin

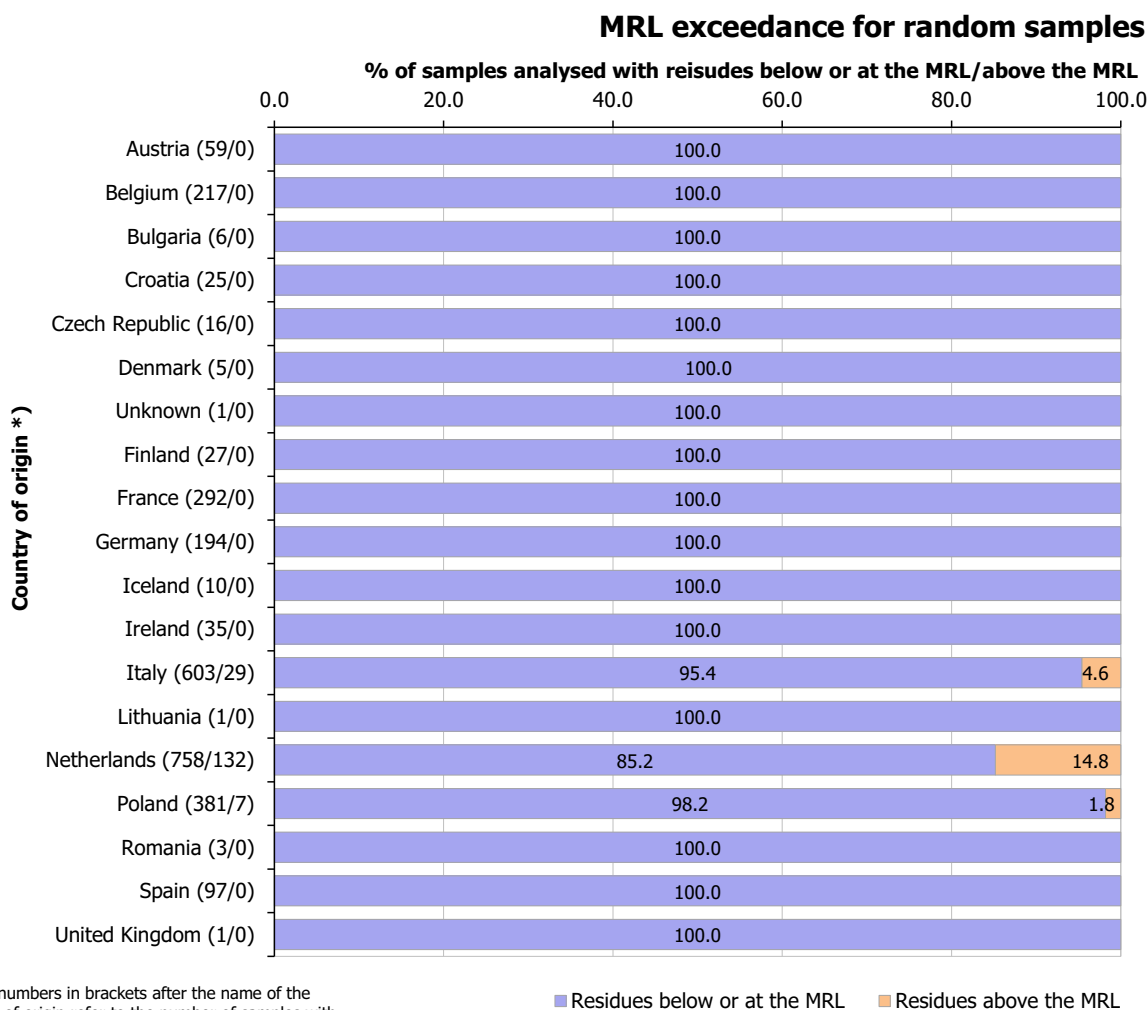
Considering all samples (reported as suspect and random samples), MRL exceedances were reported for products originating from eight Member States, i.e. the Netherlands (664 samples), Italy (40 samples), Germany (13 samples), Poland (11 samples), Hungary (6 samples), France (5 samples), Slovenia (2 samples) and Greece (1 sample).

Among the samples reported as suspect/targeted samples, the highest number of MRL exceedances was reported in samples originating from the Netherlands (532 samples), followed by Germany (13 samples), Italy (11 samples), Hungary (6 samples), Poland (4 samples), Slovenia (2 samples) and Greece (1 sample) (Figure 14).



**Figure 14:** Number and percentage of samples with residues below or at the EU MRL and above the EU MRL by country of origin – suspect samples only

In Figure 15, the MRL exceedances for random samples are reported. While in random samples produced in three countries MRL exceedances were noted (i.e. 132 of randomly sampled products produced in the Netherlands, 29 of the random samples produced in Italy and 7 of the random samples produced in Poland), none of the samples produced in other countries was found to exceed the legal limits.

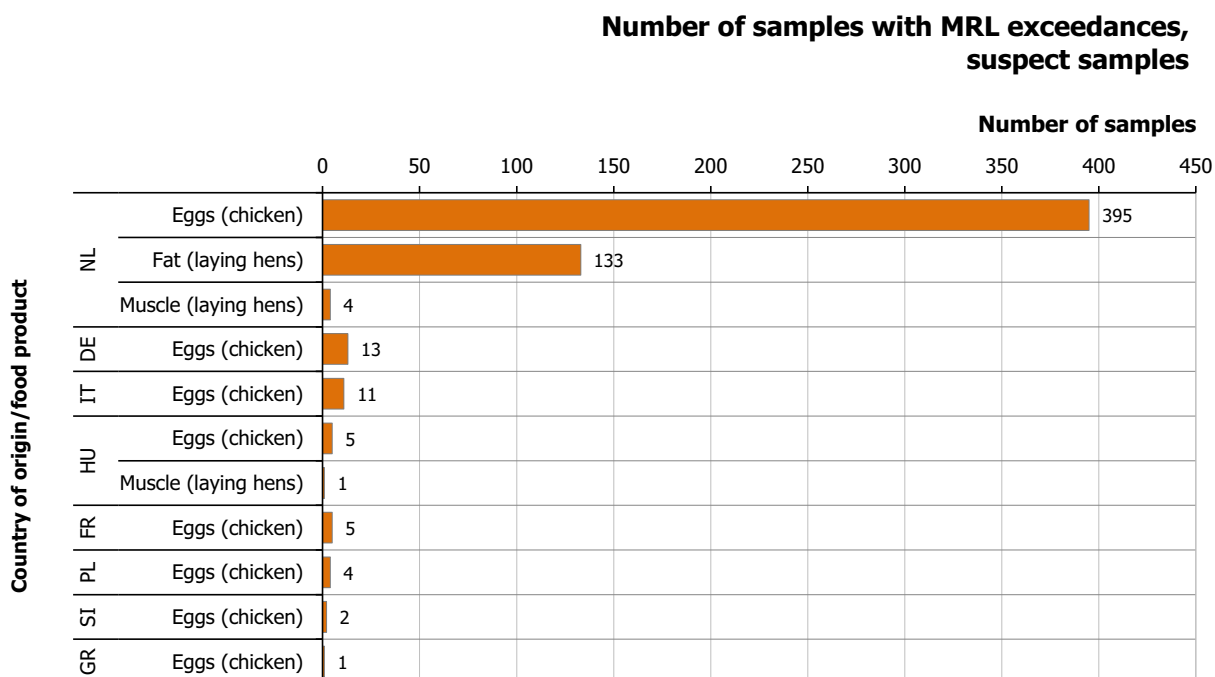


**Figure 15:** Number and percentage of samples with residues below or at the EU MRL and above the EU MRL by country of origin – samples taken by random sampling

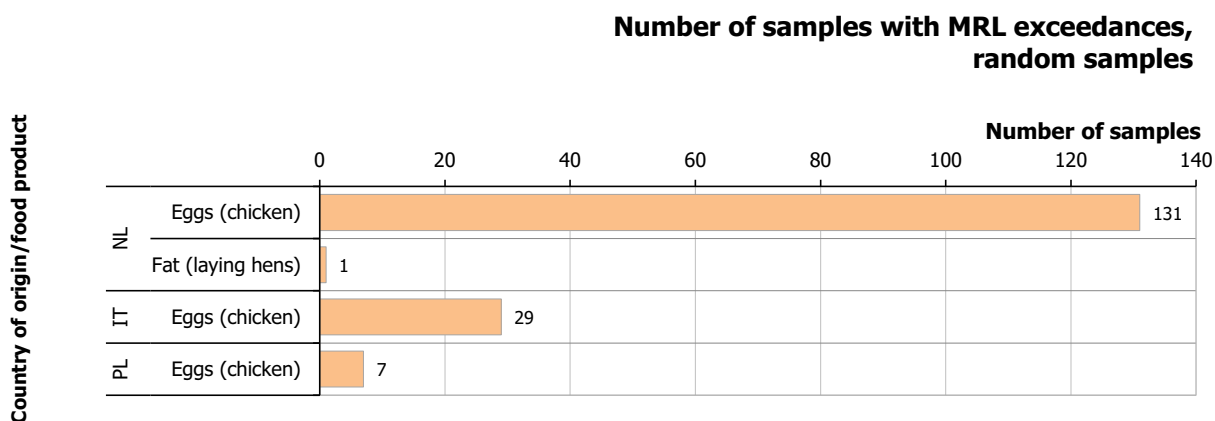
In Figures 16 and 17, the number of samples with residues above the MRL split by country of origin and product type is shown.

Among the 574 suspect samples with MRL exceedances, chicken eggs produced in the Netherlands were the product with the highest number of MRL exceedances (395 samples), followed by Dutch chicken fat (laying hens) with 133 samples, German eggs (13 samples), Italian eggs (11 samples), Hungarian and French eggs (5 samples each), Polish eggs (4 samples), Slovenian eggs (2 samples), and one Greek egg sample. In addition, four Dutch and one Hungarian sample of chicken muscle of laying hens exceeded the applicable MRL for this product.

The 168 MRL exceedances noted for samples taken as random samples were mainly attributed to Dutch chicken eggs (131 samples) and Italian eggs (29 samples); in addition, 7 samples of Polish eggs and 1 sample of chicken fat from the Netherlands exceeded the legal limit.



**Figure 16:** Number of samples with residues above the EU MRL by country of origin and food product – suspect samples

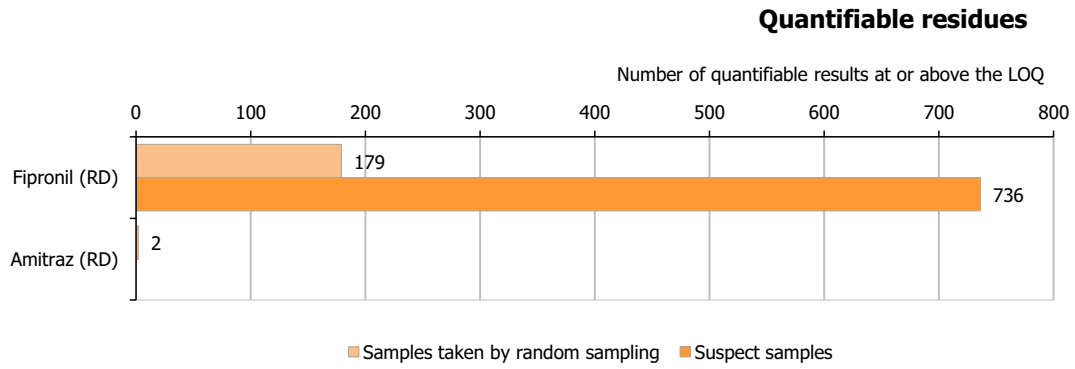


**Figure 17:** Number of samples with residues above the EU MRL by country of origin and food product – samples taken by random sampling

### 3.3. Results by pesticide

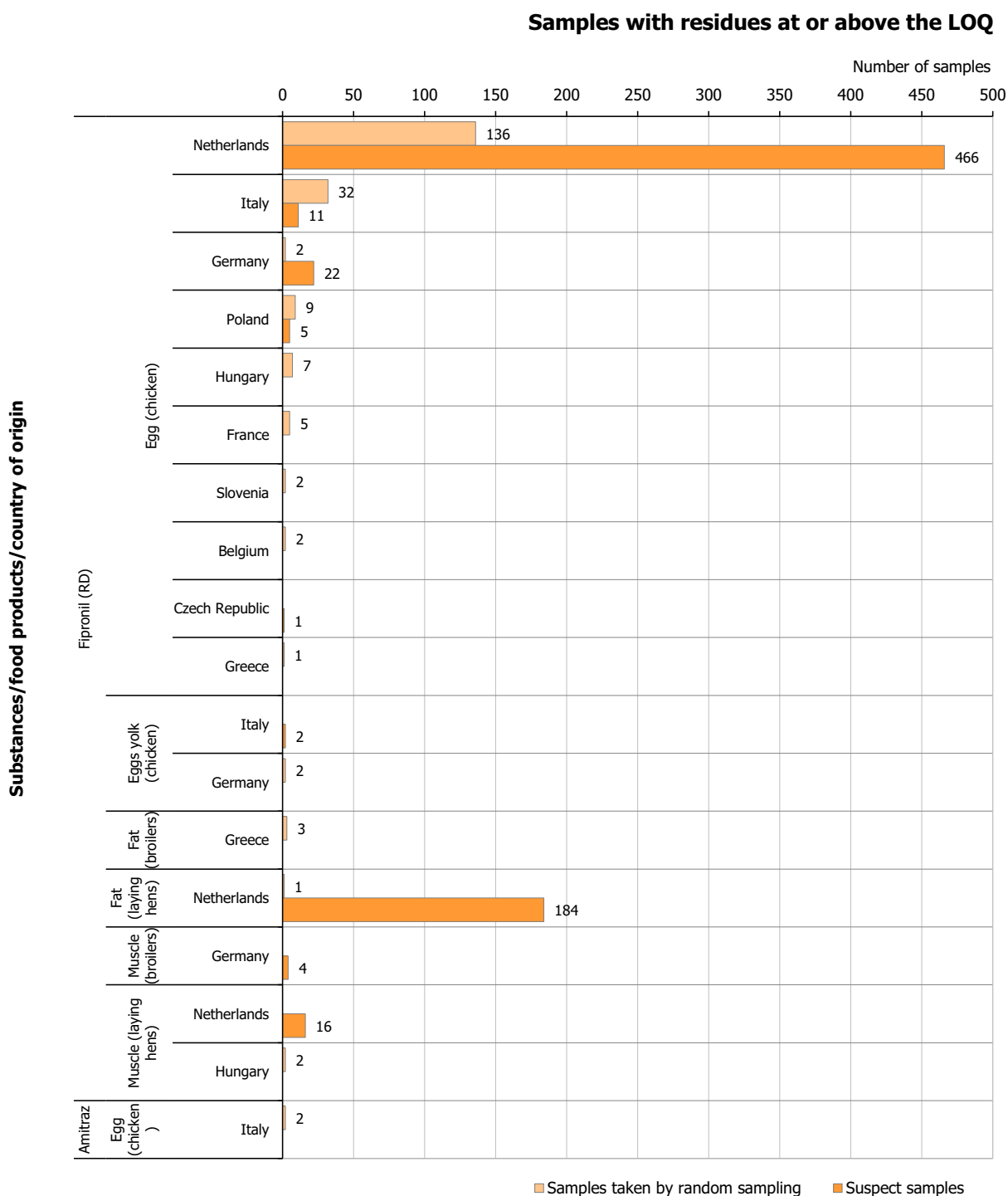
#### 3.3.1. Residues below and residues at or above the LOQ by pesticide

Among all 53,655 results reported for the 5,439 samples taken (random and suspect samples together), in 917 cases, a substance was measured in concentrations greater than or equal to the limit of quantification. The majority of the positive results (results  $\geq$  LOQ) were related to fipronil (915 results). In addition, amitraz was quantified in two samples. None of the other substances listed in Appendix A.1 or A.2 was found in any of the products analysed. It was also noted that none of the samples analysed contained multiple residues. The results are presented in Figure 18 split by sampling strategy.



**Figure 18:** Number of results with residues at or above the LOQ by substance and sampling strategy

For the substance-food product combinations with quantifiable residues, more details are given in Figure 19, showing, for each country of origin, the number of random samples and suspect samples analysed for the respective substance with quantifiable residues.



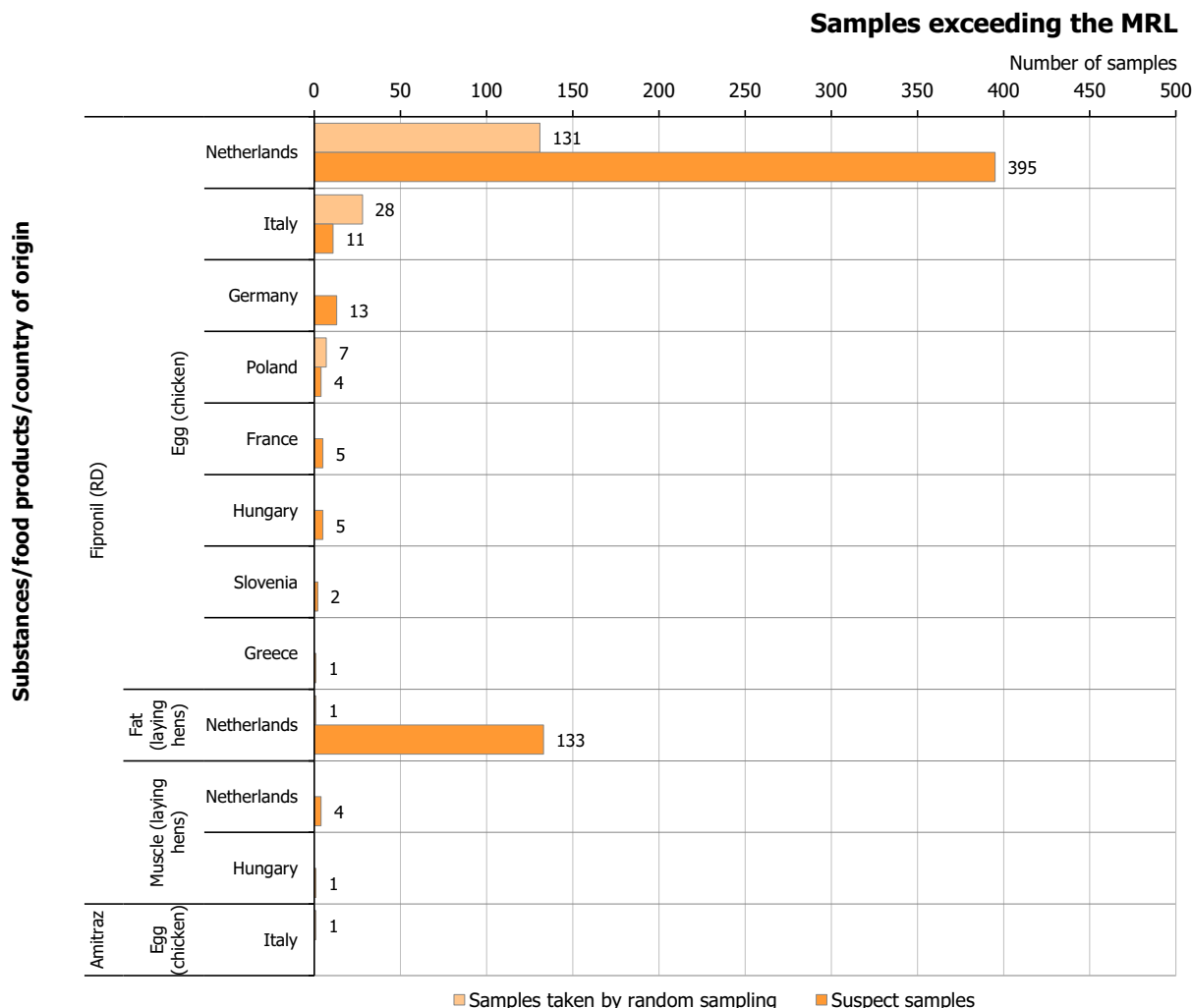
**Figure 19:** Number of samples with residues at or above the LOQ by substance, food product, sample origin and sampling strategy

### 3.3.2. Residues below or at and residues above the MRL by pesticide

Residue concentrations exceeding the legal limit for the respective products were reported in 742 of the overall samples (574 from suspect sampling and 168 from random sampling). The MRL exceedances were almost exclusively related to fipronil residues (741 samples); in addition, one case of an exceedance of the legal limit for amitraz was identified in a sample of chicken eggs produced in Italy. Focussing on the food products with MRL exceedances, the number of samples by country of origin of the product was further investigated (Figure 20).



From this presentation, it can be concluded that the contamination of chicken products leading to exceedances of the MRLs was limited to fipronil and amitraz. The food products affected were mainly chicken eggs and to a lower extent fat of laying hens. Sporadically, MRL exceedances were reported for muscle of laying hens. MRL exceedances were reported by nine Member States and found in samples originating from eight Member States (the Netherlands, Italy, Germany, Poland, France, Hungary, Slovenia and Greece).



**Figure 20:** Number of samples with residues above the MRL by substance, food product and country of origin

#### 4. Conclusions

The data sampled by Member States and Iceland in the framework of this ad-hoc monitoring programme give a comprehensive overview on the occurrence of fipronil and other substances with acaricidal activity in chicken eggs, egg products and chicken fat/muscle from the sampling period 1 September 2017 to 30 November 2017.

Based on the detailed analysis of the available data, it is concluded that the contamination of chicken products leading to exceedance of the legal limits was almost exclusively related to fipronil. Only one case of amitraz residues in chicken eggs exceeding the MRL was reported. The food products affected were mainly chicken eggs (including products of egg yolk and egg white) and fat of laying hens. Sporadically, MRL exceedances were reported for muscle of laying hens. Samples with MRL exceedance were reported by nine Member States and originated from eight Member States (the Netherlands, Italy, Germany, Poland, Hungary, France Slovenia and Greece).

It is recommended that fipronil and other acaricides be included in the future monitoring activities of the Member States.

## References

- EFSA (European Food Safety Authority), 2015. Guidance for reporting data on pesticide residues in food and feed according to Regulation (EC) No 396/2005 (2014 data collection). EFSA Journal 2015;13(7):4196, 61 pp. <https://doi.org/10.2903/j.efsa.2015.4195>
- EFSA (European Food Safety Authority), 2016. Guidance for reporting data on pesticide residues in food and feed according to Regulation (EC) No 396/2005 (2015 data collection). EFSA Journal 2016;14(5):4496, 33 pp. <https://doi.org/10.2903/j.efsa.2016.4496>
- EFSA (European Food Safety Authority), 2017. Guidance for reporting data on pesticide residues in food and feed according to Regulation (EC) No 396/2005 (2016 data collection). EFSA Journal 2017;15(5):4792, 48 pp. <https://doi.org/10.2903/j.efsa.2017.4792>

## Abbreviations

BE	Belgium
CZ	Czech Republic
DCF	Data collection framework
DE	Germany (Deutschland)
FR	France
GR	Greece
HU	Hungary
IT	Italy
LOQ	Limit of Quantification
MRL	Maximum Residue Level
MSDVet	Merck Sharp & Dohm Corp Veterinary Manual
NL	The Netherlands
PL	Poland
RD	Residue definition (active substances for which the legal residue definition comprises not only the active substance, but also metabolites are usually labelled with the suffix (RD))
SC PAFF	Standing Committee on Plants, Animals, Food and Feed
SI	Slovenia
SSD	Standard Sample Description

## Appendix A – Scope of the ad-hoc monitoring programme

### A.1. Substances to be analysed under the ad-hoc monitoring programme, including legal limits (MRLs) for the relevant substance/commodity combinations

Legal framework	Regulation (EC) No 396/2005		Regulation (EU) No 37/2010	
	Substances	MRLs	Residue definition	MRLs
<b>Amitraz</b>	0.05* mg/kg (poultry all tissues) 0.01* mg/kg (eggs)	Amitraz (amitraz including the metabolites containing the 2,4-dimethylaniline moiety expressed as amitraz)	No MRLs for poultry or eggs	Sum of amitraz and all metabolites containing the 2,4-DMA moiety, expressed as amitraz
<b>Bifenthrin</b>	0.05* mg/kg (poultry muscle and fat) 0.01* mg/kg (other poultry tissues and eggs)	Bifenthrin (sum of isomers) (F)	N/A <sup>(b)</sup>	N/A
<b>Cypermethrin</b>	0.1 mg/kg (poultry muscle/fat) other poultry tissues and eggs 0.05* mg/kg	Cypermethrin (cypermethrin including other mixtures of constituent isomers (sum of isomers)) (F)	No MRLs for poultry or eggs	Cypermethrin (sum of isomers)
<b>Diazinon</b>	0.02 mg/kg (poultry muscle/edible offals) 0.01* mg/kg (poultry other tissues) 0.02* mg/kg (eggs)	Diazinon (F)	No MRLs for poultry or eggs	Diazinon
<b>Etoxazole</b>	0.01* mg/kg (all poultry tissues and eggs)	Etoxazole	N/A <sup>(b)</sup>	N/A
<b>Fipronil (RD)</b>	0.006 mg/kg (poultry fat) 0.005* mg/kg (other poultry tissues and eggs) For processed products, see Appendix B, Table B.1)	Fipronil (sum fipronil + sulfone metabolite (MB46136) expressed as fipronil) (F)	No MRLs. Authorised for use in cats and dogs	N/A
<b>Flufenoxuron</b>	0.05* mg/kg (all poultry tissues and eggs)	Flufenoxuron (F)	N/A <sup>(b)</sup>	N/A
<b>Ivermectin</b>	N/A <sup>(a)</sup>	N/A	No MRLs for poultry or eggs	22, 23-dihydro-ivermectin B1a
<b>Pyridaben</b>	0.02* mg/kg (all poultry tissues and eggs)	Pyridaben (F)	N/A <sup>(b)</sup>	N/A
<b>Pyriproxyfen</b>	0.05* mg/kg (all poultry tissues and eggs)	Pyriproxyfen (F)	No MRLs for poultry or eggs	N/A

Legal framework	Regulation (EC) No 396/2005		Regulation (EU) No 37/2010	
	Substances	MRLs	Residue definition	MRLs
<b>Thiamethoxam</b>	0.01* mg/kg (all poultry tissues and eggs)	Thiamethoxam	N/A <sup>(b)</sup>	N/A
<b>Trichlorfon (metrifonate)</b>	0.01* mg/kg (all poultry tissues and eggs)	Trichlorfon	N/A <sup>(b)</sup>	N/A

N/A: not applicable; MRL: maximum residue level.

(a): Since the active substance is not covered by Regulation (EC) No 396/2005; no MRLs are established for any food product.

(b): Active substance not covered by Regulation (EU) No 37/2010.

(F): Fat-soluble residue.

## A.2. Additional substances to be analysed

As agreed in the PAFF meeting held on 30 August 2017, the substances listed in the table below should be taken into account when analysing the samples covered by the ad-hoc monitoring programme. The substances were selected according to the following selection criteria:

- Acaricides;
- Substances that can be analysed by multiresidue methods;
- Substances described in the MSDVet Manual for treatment of poultry against mites;
- Substances not specifically approved but in literature reported to be widely used against red mites;
- Substances banned for use on red mites in the EU;
- Substances against ectoparasites for which MRLs have been established under the legislation for veterinary medicinal products for different food species.

Substance	Substance	Substance
Abamectin (synonym avermectin)	Deltamethrin	Lufenuron
Azamethiphos <sup>(a)</sup>	Dichlorvos	Malathion
Azinphos ethyl	Dicyclanil <sup>(a)</sup>	Methamidophos
Azinphos-methyl	Diflubenzuron	Methidathion
Bendiocarb	Dimethoate	Mevinphos
Bromopropylate	Doramectin <sup>(a)</sup>	Moxidectin <sup>(a)</sup>
Carbaryl	Emamectin	Nicotine
Carbofuran	Endosulfan	Parathion
Chlorfenapyr	Eprinomectin <sup>(a)</sup>	Parathion-methyl
Chlorfenson (synonym chlorfenizon)	Ethion (synonym diethion)	Permethrin
Chlorobenzilate	Etrimfos	Phosphamidon
Chlorpyrifos	Fenitrothion	Propoxur
Chlorpyrifos-methyl	Fenpropathrin	Sisapronil <sup>(a)</sup>
Coumaphos	Fenthion	Sulfotep
Cyfluthrin	Fenvalerate	Teflubenzuron
Cyhalothrin	Fluazuron <sup>(a)</sup>	Tetrachlorvinphos (stirofos)
Cyromazine	Flumethrin	Tetradifon
	Formothion	Triazophos
	Hexaflumuron	

(a): No MRLs are established for any food product covered by the specific monitoring programme, neither under Regulation (EC) No 396/2005 nor under Regulation (EU) No 37/2010.

## Appendix B – MRLs for fipronil residues in unprocessed and processed products

For this specific monitoring programme, the MRLs for fipronil listed in Table B.1 below had to be taken into account. The MRLs for processed products were agreed at the Meeting of the SC Committee PAFF, Section Novel Food and Toxicological Safety of the Food Chain held on 30 August 2017.

In addition, the table below contains the fipronil MRLs for chicken fat and chicken muscle.

**Table B.1:** Legal limits for fipronil residues

MATRIX code	MATRIX code description	prodText	prodTreat code	prodTreat description	MRL (mg/kg)
P1030010A	Eggs (chicken)	Whole egg or whole egg liquid	T999A or T998A or T151A	Fresh/unprocessed or Freezing or Pasteurisation	0.005* (LOQ MRL)
P1030010.1A	Eggs yolk (chicken)	Liquid egg yolk	T999A or T998A or T151A	Fresh/unprocessed or Freezing or Pasteurisation	0.016
P1030010.2A	Eggs white (chicken)	Liquid egg white	T999A or T998A or T151A	Fresh/unprocessed or Freezing or Pasteurisation	0.002*–0.005* (LOQ MRL) <sup>(a)</sup>
P1030010A	Eggs (chicken)	Whole egg powder	T131A	Dehydration	0.021
P1030010.1A	Eggs yolk (chicken)	Egg yolk powder	T131A	Dehydration	0.037
P1030010.2A	Eggs white (chicken)	Egg white powder	T131A	Dehydration	7.17 × LOQ achievable for liquid egg white or 0.005 <sup>(b),(c),(d)</sup>
P1016010_002.1B P1016010_002.2B	Muscle (poultry)	Chicken (laying hens and broilers)	T999A or T998A	Fresh/unprocessed or Freezing	(LOQ MRL)
P1016020_002.1A P1016020_002.2A	Fat (poultry)	Chicken (laying hens and broilers)	T999A or T998A	Fresh/unprocessed or Freezing	0.006

LOQ: limit of quantification; MRL: maximum residue level.

- (a): Taking into account that maximum 10% of fipronil residues in the whole egg are present in liquid egg white, the calculated residue concentration is 0.00074 mg/kg. Thus, residues above the LOQ are not expected to occur in liquid egg white produced from whole eggs compliant with the legal limit established for whole eggs.
- (b): Residues above the LOQ are not expected to occur in liquid egg white produced from whole eggs compliant with the legal limit established for whole eggs.
- (c): An MRL of 0.005 mg/kg should be applied in case there is no information on/evidence of the compliance of the liquid egg white used for the production of the egg white powder.
- (d): Food business operators producing egg white powder must ensure that the liquid egg used for the production of egg white powder is compliant with EU legislation, i.e. the level of fipronil in egg white liquid is below LOQ.