

Residue Findings of QuPPE-Compounds in Samples of Plant Origin from the German Market in 2023

Reported by: EURL-SRM
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The aim of this compilation is to give an overview as to which highly polar (QuPPE-) compounds are currently encountered in food products of plant origin. This should help other institutions when it comes to taking decisions on how to expand the scope of targeted SRM analytes, on how to plan sampling and on which QuPPE compounds are worthwhile targeting in the various types of samples. With the knowledge communicated here we ultimately aim at contributing to a more targeted and efficient use of lab resources within the EU-OfLs.

At CVUA Stuttgart, 47 QuPPE-compounds were routinely monitored in 2023 (see Table 1). Some of these compounds are not legally relevant, as they are not part of the legal residue definition, yet still of interest for potential exposure and risk-assessment evaluations.

Despite not being a QuPPE compound, a very brief overview of the residue findings of 2-chloroethanol (2-CE; formed from the reaction of the fumigant ethylene oxide with chloride ions naturally contained in food products) in 2023 is given at the end of this document. 2-CE findings in various products have been causing severe turbulences in the EU-food market in 2020-2022, but the situations has improved from 2023 onwards.

Table 1: Scope of QuPPE-compounds that were routinely monitored by the CVUA Stuttgart in 2023

| Compound | Notes on legal aspects | General notes |
|------------------------------|---|--|
| 4-Methylimidazoline | Non regulated metabolite | Marker of propylene-bis-dithiocarbamates (propineb). Limited specificity at very low concentrations (<0.005 mg/kg). |
| Ammelide | Non-regulated metabolite and contaminant | Ammelide can originate from various sources, similar to ammeline. Ammelide (and ammeline) are formed as intermediates during the gradual transformation of melamine (a compound with multiple sources, see below) to cyanuric acid. Ammelide is reported as a metabolite of various triazine pesticides incl.: cyromazine (insecticide), anilazine (fungicide) and the herbicides terbuthylazine, prometryn, simazine, atrazine, ametryne and cyanazine. Among those, only terbuthylazine is currently approved within the EU. |
| Ammeline | Non-regulated metabolite and contaminant | Intermediate in the conversion of melamine to cyanuric acid. For more info, see ammeline |
| Amitrole | MRLs are set at the LOQ in all products | Non-approved herbicide, the most important of the very few triazole pesticides with herbicidal properties. Approval expired in mid-2016. |
| Bromide | MRLs refer to bromide ion. Background levels are generally considered in MRLs for food of plant origin, but rather not considered in MRLs for food of animal origin. Collection of data on background levels in the latter would be useful. | Reaction product of the fumigant methylbromide. Mostly originating from soil and irrigation water. Counter ion of certain quarternary ammonium cations such as benzalkonium, didcyldimethylammonium (DDA-), diquat and paraquat |
| Chlorate | New MRLs set in 2020 | Formerly used as herbicide, but nowadays mainly originating from chlorinated water, that is often used to irrigate fields or for washing harvested products or the equipment that is used for processing or storage of agricultural products. |
| Chloridazon-desphenyl | Regulated metabolite | Metabolite of chloridazon (approval expired on 31/12/2018). Chloridazon-desphenyl is quite persistent in the environment, thus residues in succeeding crops, and in water, have been reported. |
| Chlormequat | MRLs refer to chlormequat chloride | EU-Approved growth regulator with a wide range of applications. It has also been reported as a processing contaminant formed during the thermal degradation of choline (and its natural derivatives) in presence of chloride ions. Formation has been for example reported during thermal treatment of egg powder, wheat flour, barley and coffee. |

| Compound | Notes on legal aspects | General notes |
|-------------------------|--|--|
| Cyanuric acid | Non-regulated metabolite | Compound originating from multiple sources, e.g.: Triazine pesticides (incl. the herbicides terbuthylazine, atrazine, cyanazine, the fungicide; anilazine and the insecticide cyromazine). From the above only terbuthylazine is currently in use within the EU. Cyromazine has lost EU-approval in Dec. 2019. Cyanamide-based fertilizers. Cyanamide contained in fertilizers may convert to melamine through trimerization, which can further hydrolyze to cyanuric acid. Urea-based fertilizers or feed: especially at high temperatures urea loses ammonia converting to isocyanic acid (HNCO), which trimerises to cyanuric acid. Mono-, Di- and Trichloroisocyanurates: Used as disinfectants, algacides and bactericides, e.g. in sanitation liquids, bleaching agents as well as in swimming pools (pool-tabs) to retard the loss of chlorine in chlorinated water. In water, they gradually convert to cyanuric acid. Natural formation of cyanuric acid has also been reported (e.g. in humus). |
| Cyromazine | Since August 2023 all MRLs in food of plant origin are set at the LOQ. MRLs for food of animal origin are also set at the LOQ with exception of sheep tissues (The MRL for sheep milk is set at LOQ) | Non-approved fungicide (EU-approval expired in Dec. 2019). Also used as an ectoparasiticide (e.g. on sheep, but not on lactating sheep) and as a biocide on manure against fly larvae |
| Daminozide | MRLs are set at the LOQ in all products | Approved growth regulator |
| Difenzoquat | No specific MRLs established (0.01 mg/kg applies) | Non-approved herbicide |
| Dimethoate-O-desmethyl | Non-regulated metabolite | Also known as Metabolite X of dimethoate |
| Diquat | Specific MRLs set for products where desiccation takes place prior to harvest (e.g. potatoes, pulses, oilseeds) and for some other products, (e.g. citrus, stone fruit, tree nuts, oats, strawberries, eggs) | Non-approved herbicide, formerly used for desiccation in potatoes (EU-approval expired in Dec. 2019) |
| Diquat dipyrindone | Non-regulated metabolite | Metabolite of diquat |
| Diquat monopyridone | Non-regulated metabolite | Metabolite of diquat |
| Diquat Met. TOPPS | Non-regulated metabolite | Metabolite of diquat |
| Ethephon | | Approved growth regulator with multiple applications |
| HEPA | Non-regulated metabolite | Metabolite of ethephon. Likelihood of a natural formation by bacteria in the intestines of cattle under anaerobic conditions was reported by the EURL-SRM in 2019. Detected by the EURL-SRM in all analyzed samples of bovine liver (levels around 0.5 mg/kg). These levels are considered natural artefacts. Manure from cows, sheep and horse (collected from organic farms) and analysed by the EURL-SRM contained in all cases HEPA (highest level in cow manure at 1.3 mg/kg). Whether manure application in fields can explain the HEPA findings in some organic products is currently being investigated. |
| ETU (ethylene thiourea) | Non-regulated degradant | Degradant of ethylen-bis-dithiocarbamates. Reported as impurity in formulations based on EBTCs. Also formed during food processing. Marker compound for the screening of DTC-residues of products with EBTC-application history. Where EU and ETU are detected simultaneously the likelihood of relevant CS2 levels increases. Note: Most EBTCs lost approval within the EU (Mancozeb expired on Jan 2021 with the period of grace ending in Jan 2022; Maneb expired in Jan 2017, Zineb and Nabam didn't receive EU-approval). Metiram is still approved (current approval period ends Jan 24). |
| EU (2-Imidazolidinone) | Non regulated metabolite | Degradant of ethylen-bis-dithiocarbamates. Degradant of ETU (see above). Marker compound for the screening of DTC-residues of products with EBTC-application history. Where EU and ETU are detected simultaneously the likelihood of relevant CS2 levels increases. See also ETU. |
| Fosetyl | | Approved fungicide (converts to phosphonic acid, which is the active component) |
| Phosphonic acid | Regulated with parent fosetyl | Approved fungicide, used as such and also formed as a metabolite of fosetyl Phosphonate-based water-softening agents (e.g. ATMP, HEDP, DTPMP), that are used in cleansing agents contain some residual phosphonic acid, which may lead to small (rather insignificant) contamination of food, e.g. when in contact with surfaces that were not sufficiently rinsed after washing. Formation of phosphonic acid and derivatives thereof, such as HEPA, through the reduction of phosphates within the anaerobic environment in intestines of ruminants and other animals is likely. To which extend phosphonates formed here will lead to relevant residue levels in organic products needs to be investigated. |
| Glufosinate | Specific MRLs apply for many crops | Non-approved herbicide, also used in the cultivation of glufosinate-resistant transgenic crops. EU-approval expired in mid-2018 and not renewed |
| MPP (MPPA) | Included in residue definition of glufosinate | Metabolite of glufosinate |

| Compound | Notes on legal aspects | General notes |
|--|---|--|
| N-Acetyl Glufosinate | Included in residue definition of glufosinate | Metabolite of glufosinate |
| Glyphosate | Specific MRLs apply for many crops | Approved herbicide, also used in the cultivation of glyphosate-resistant transgenic crops. Current approval periods end in Dec 2022. |
| AMPA | Non-regulated metabolite. | Metabolite of glyphosate. Planned inclusion in RD of glyphosate. |
| N-Acetyl-Glyphosate | Non-regulated metabolite. | Metabolite of glyphosate. Planned inclusion in RD of glyphosate. |
| Maleic hydrazide | Plant product MRLs set at 0.2* / 0.5* except for Potatoes, Carrots, Parsnips, Onions, Garlic, Shallots, Chicory | Approved sprouting inhibitor. |
| Matrine | MRL of 0.01 mg/kg applies (listed in EU-pesticide database) | Natural quinolizidine alkaloid, that is considered (together with oxymatrine) as the active ingredient of biopesticides based on extracts of certain plants of the <i>Sophora</i> family. Not approved within the EU as PPP, neither in conventional nor in organic production. Registered in various countries in Asia, Africa and South America. There were cases of illegal addition of <i>Sophora</i> root extracts in fertilizers in Italy. Together with oxymatrine, often found in so-called "acacia honey" from China, which mostly originated from flowers of <i>Sophora</i> plants. <i>Sophora</i> extracts are also used in traditional Asian medicine and cosmetics. Co-harvesting of licorice and <i>Sophora</i> roots results in a considerable contamination of licorice and licorice products with matrine (and oxymatrine). |
| Oxymatrine | MRL of 0.01 mg/kg applies (listed in EU-pesticide database) | Quinolizidine alkaloid present in <i>Sophora</i> extracts, remarks on matrine apply |
| Melamine | Regulated by Reg. 1881/2006/EC as a contaminant | Metabolite of cyromazine (pesticide and vet. drug). May also originate from cyanamide fertilizers (trimerization of cyanamide) and from urea fertilizers, where it is formed through trimerisation of urea to triuret and subsequent elimination of ammonia and carbon dioxide (Note: biuret and triuret are related non-cyclic products formed from the di- and trimerisation of urea respectively). Melamine hydrolyses to cyanuric acid via ammeline and ammelide. Melamine is widely used for the synthesis of melamine-formaldehyde resins that are employed in synthetic surfaces of furniture, textiles, kitchenware as well as in moulding and packaging materials. Also used as a fire-retardant. |
| Mepiquat | MRLs refer to mepiquat chloride | Approved growth regulator. Similar to chlormequat, mepiquat has been reported to be formed as a natural processing contaminant through maillard-like reactions, e.g. during roasting of coffee and barley grain with the latter being used for the production of brewery malt, which is a main ingredient of beer. |
| Mepiquat, 4-Hydroxy | Non-regulated metabolite | Metabolite of mepiquat, mainly relevant for food of animal origin |
| Morpholine | Not regulated as a pesticide | Additive of waxes. Typically used together with oleic acid to assist emulsification of wax and facilitate wax handling. In the EU, the use of morpholine in fruit coating is not permitted, but it is widely used in other parts of the world. |
| Nereistoxin | Non-regulated metabolite | Transformation product of various members of the nereistoxin pesticides (e.g. bensultap, sultap, cartap, thiocyclam). There are rumors that it is illegally used as such in agriculture. |
| Nicotine | Specific MRLs set for rose hips, herbs, edible flowers, wild fungi, teas, herbal infusions and spices. MRLs will be revised when information on origin of background levels become available. | Non-approved insecticide. Nicotine originating from tobacco may contaminate food at all stages of food production, through air, soil and human contact. Crops experiencing intensive human contact during harvest or processing are particularly affected |
| Paraquat | MRL at LOQ 0.02 to 0.05 mg/kg | Non-approved herbicide, EU-approval expired in Dec. 2007 |
| Perchlorate | Regulated as a contaminant, see Reg. (EC) 1881/2006/EC | Persistent and ubiquitous environmental contaminant. Mainly originating from fertilizers, may be also formed as a byproduct of disinfection of drinking water. Temporarily inhibits the intake of iodine in the thyroid gland. |
| Propamocarb | Current approval end on 15/06/2025 | Approved fungicide, mainly relevant for vegetables, e.g. root-, bulb-, fruiting-, and leafy vegetables |
| Propamocarb N-desmethyl | Non regulated metabolite | Metabolite of propamocarb |
| Propamocarb-N-oxide | Non regulated metabolite | Metabolite of propamocarb |
| PTU (propylene thiourea = 4-Methyl-2-imidazolidinethione) | Formerly regulated in infant- and baby food Reg. EC 125/2006 and 141/2006. Nowadays not regulated | Degradant of propylen-bis-DTCs (i.e. propineb). Also impurity in propineb-based PPPs. Also formed from propineb during food processing (heating). Useful marker for propineb-application history. Note: Propineb is not any more approved within the EU (approval expired in March 2018) |
| Thiocyanate | No specific MRLs set. Formally, the default MRL of 0.01 mg/kg applies. Collection of data on background levels would help introduce reasonable MRLs that consider background levels. | Non-approved fungicide. Also naturally formed in various cultivated plants of the brassica and allium family. Temporarily inhibits the intake of iodine in the thyroid gland. Eggs and milk may contain higher levels if the animals are fed with brassica crops. Also naturally formed in animals (e.g. in saliva). |
| Thiram metabolite M1 | Non regulated metabolite | Marker of dimethyldithiocarbamates. Specificity questionable at low levels. |

| Compound | Notes on legal aspects | General notes |
|-----------|---|---|
| Trimesium | MRLs of dry commodities do not always take into account the amounts formed during the drying process. | Counter-ion of glyphosate, also naturally formed as an artefact during the drying process of food. Also known as trimethylsulfonium cation. |

Residue Findings:

In 2023, a total of 2385 samples, mainly fruit and vegetables, but also cereals, pulses, processed goods, tea and others, were analyzed for QuPPE-amenable compounds at the CVUA Stuttgart. 1631 samples (68 %) contained quantifiable residues of one or more of the tested QuPPE compounds.

Table 2 sorts the compounds based on the frequency of finding above the reporting limit. Table 3 shows a compilation of all the results concerning the above-listed highly polar compounds.

Table 2: Residue findings of QuPPE-compounds (CVUA Stuttgart 2023) by detection frequency

| Frequency of findings > respective RL | Compounds (pesticides and legally relevant metabolites shown in bold) |
|---------------------------------------|---|
| > 10 % of samples. | <i>Cyanuric acid, Phosphonic acid, Chlorate and Perchlorate</i> |
| > 1 - 10 % of samples. | <i>Melamine, Thiocyanate²⁾, Propamocarb, Propamocarb-N-oxide, 2-Chloroethanol¹⁾, Paraquat⁴⁾, Propamocarb-N-desmethyl, Nicotine, Ethephon, Ammelide, Trimesium, Chlormequat-chloride, Bromide³⁾, and Chloridazon-desphenyl</i> |
| 0.1 -1% of samples | <i>Ethephon metabolite HEPA, Maleic hydrazide, Thiram metabolite M1, Diquat⁴⁾, Glyphosate, Mepiquat-chloride, Dimethoate-O-desmethyl, Glufosinate met. MPPA, EU, 4-Methylimidazole, Fosetyl, Morpholine, ETU, Mepiquat- 4-hydroxy and Matrine</i> |
| <0.1 - LOQ | <i>Cyromazine, Oxymatrine, Nereistoxin, N-Acetyl-glufosinate, Daminozide</i> |
| Not detected above LOQ | <i>Amitrole, Ammeline, Difenzoquat, Diquat-dipyridone, Diquat met. TOPPS, Diquat-monopyridone, Ethylene oxide ¹⁾, Glufosinate, and Glyphosate met. N-Acetylgllyphosate, Glyphosate met. AMPA and PTU</i> |

¹⁾ EO/2-CE are technically not QuPPE compounds but still shown here (see towards the end of this document), note EO/2-CE analysis was semi-targeted with dry commodities being the main focus.
²⁾ No MRLs were deemed necessary for potassium thiocyanate.
³⁾ Bromide is ubiquitous and virtually every sample is positive. The RL of 5 mg/kg represents the lowest MRL in food of plant origin.
⁴⁾ Paraquat and Diquat analysis was semi-targeted. Main focus were dry commodities such as pulses, oily seeds and cereals as well as potatoes

Table 3: Residue findings of QuPPE-compounds (CVUA Stuttgart 2023) by compound

| Compound | # samples | # pos. ¹⁾ | % pos. | Max (mg/kg) | Mean ²⁾ (mg/kg) | Median ²⁾ (mg/kg) | # >MRL ³⁾ | % >MRL | RL ⁴⁾ |
|--------------------------------|-----------|----------------------|--------|-------------|----------------------------|------------------------------|----------------------|--------|------------------|
| Cyanuric acid | 2381 | 706 | 30 | 4,9 | 0,0558 | 0,0135 | Not part of RD | | 0.005 |
| Fosetyl, sum (Phosphonic acid) | 2381 | 454 | 19 | 106 | 4,9 | 1,4 | 11 | 0.5 | 0.05 |
| Chlorate | 2381 | 370 | 16 | 3 | 0,0458 | 0,012 | 7 | 0,3 | 0.005 |
| Perchlorate | 2381 | 349 | 15 | 15,7 | 0,108 | 0,012 | 6 ⁵⁾ | 0,3 | 0.005 |
| Melamine | 2382 | 229 | 9,6 | 6,5 | 0,104 | 0,031 | 0 ⁴⁾ | 0 | 0.01 |
| Thiocyanate | 2381 | 173 | 7,3 | 51,1 | 10,8 | 6,2 | 0 | 0 | 0.1 |
| Propamocarb | 2382 | 102 | 4,3 | 3,5 | 0,182 | 0,028 | 2 | 0,08 | 0.005 |
| Propamocarb-N-oxide | 2382 | 82 | 3,4 | 0,34 | 0,0479 | 0,017 | 0 | 0 | 0.005 |
| Paraquat | 245 | 7 | 2,9 | 0,2 | 0,0626 | 0,032 | 2 | 0,8 | 0.01 |
| Propamocarb-N-desmethyl | 2382 | 55 | 2,3 | 0,11 | 0,0207 | 0,009 | Not part of RD | | 0.005 |
| Ammelide | 2381 | 39 | 1,6 | 0,59 | 0,0845 | 0,029 | Not part of RD | | 0.005 |
| Ethephon | 2381 | 37 | 1,6 | 1 | 0,245 | 0,16 | 0 | 0 | 0.01 |
| Nicotine | 2382 | 38 | 1,6 | 0,58 | 0,0693 | 0,0215 | 13 | 0,5 | 0.01 |
| Bromide | 2381 | 31 | 1,3 | 159 | 34 | 23 | 0 | 0 | 5 ⁷⁾ |
| Chlormequat-chloride | 2382 | 30 | 1,3 | 0,81 | 0,108 | 0,044 | 3 | 0,1 | 0.005 |
| Trimesium | 2382 | 32 | 1,3 | 0,52 | 0,0632 | 0,023 | 9 ⁶⁾ | 0,4 | 0.005 |
| Chloridazon-desphenyl | 2382 | 27 | 1,1 | 0,027 | 0,0117 | 0,009 | 0 | 0 | 0.005 |
| Ethephon metabolite HEPA | 2381 | 25 | 1,0 | 0,54 | 0,095 | 0,042 | Not part of RD | 0 | 0.01 |
| Maleic hydrazide | 2381 | 22 | 0,9 | 27,7 | 5,6 | 4,1 | 0 | 0 | 0.01 |
| Diquat | 245 | 2 | 0,8 | 0,041 | 0,0295 | 0,030 | 0 | 0 | 0.01 |
| Thiram M1 ⁸⁾ | 2382 | 20 | 0,8 | 0,049 | 0,0123 | 0,006 | Not part of RD | 0 | 0.002 |
| Glyphosate | 2381 | 16 | 0,7 | 0,99 | 0,225 | 0,093 | 2 | 0,08 | 0.02 |

| Compound | # samples | # pos. ¹⁾ | % pos. | Max (mg/kg) | Mean ²⁾ (mg/kg) | Median ²⁾ (mg/kg) | # >MRL ³⁾ | % >MRL | RL ⁴⁾ |
|-----------------------------------|-----------|----------------------|--------|-------------|----------------------------|------------------------------|----------------------|--------|------------------|
| Dimethoate-O-desmethyl | 2381 | 13 | 0,5 | 0,2 | 0,0646 | 0,033 | 0 | 0 | 0.005 |
| Mepiquat chloride | 2382 | 11 | 0,5 | 0,17 | 0,0377 | 0,021 | 1 | 0,04 | 0.005 |
| 4-Methylimidazoline ⁸⁾ | 2382 | 9 | 0,4 | 0,022 | 0,0117 | 0,011 | Not part of RD | 0 | 0.004 |
| EU ⁷⁾ | 2382 | 10 | 0,4 | 0,52 | 0,135 | 0,098 | Not part of RD | 0 | 0.02 |
| Glufosinate met. MPPA | 2381 | 9 | 0,4 | 0,13 | 0,0329 | 0,02 | 0 | 0 | 0.01 |
| Fosetyl | 2381 | 6 | 0,3 | 0,65 | 0,261 | 0,19 | 0 | 0 | 0.01 |
| Morpholine | 2382 | 6 | 0,3 | 3,6 | 0,75 | 0,092 | additive | 0 | 0.02 |
| ETU ⁸⁾ | 2382 | 5 | 0,2 | 0,35 | 0,201 | 0,21 | Not part of RD | 0 | 0.02 |
| Matrine | 2385 | 3 | 0,1 | 0,21 | 0,159 | 0,185 | 0 | 0 | 0.01 |
| Mepiquat, 4-hydroxy | 2382 | 3 | 0,1 | 0,095 | 0,0658 | 0,095 | Not part of RD | 0 | 0.005 |
| Cyromazine | 2382 | 2 | 0,08 | 0,015 | 0,0135 | 0,0135 | 1 | 0,04 | 0.005 |
| Daminozide | 2382 | 1 | 0,04 | 0,006 | 0,00637 | | 0 | 0 | 0.005 |
| N-Acetyl-glufosinate | 2381 | 1 | 0,04 | 0,13 | 0,13 | | 0 | 0 | 0.02 |
| Nereistoxin | 2382 | 1 | 0,04 | 0,035 | 0,035 | | Not part of RD | 0 | 0.005 |

¹⁾ The number of positive samples refers to samples exceeding the RL.

²⁾ Mean and median of positives

³⁾ Numerical MRL-exceedances

⁴⁾ RL= Reporting Limit (exemplary for fruits and vegetables)

⁵⁾ Perchlorate and melamine are legally contaminants. In six cases the ML of perchlorate, as defined in Reg.(EC) No. 1881/2006, was exceeded

⁶⁾ MRL-exceedances of trimesium are in most cases most likely due to the formation of trimesium during processing

⁷⁾ Bromide is ubiquitous and virtually every sample is positive. The RL of 5 mg/kg represents the lowest MRL in food of plant origin. Analytically much lower levels can be quantified.

⁸⁾ Additional findings below LOQ were used for dithiocarbamate screening purposes. The reporting limits given are semi-quantitative

MRL exceedances

In 58 samples (thereof 9 organic) MRLs of different compounds were numerically exceeded. In 28 of these samples (thereof 4 organic) the MRLs were exceeded even after deducting 50% measurement uncertainty. Table 4 gives an overview of these exceedances. In 9 samples (thereof 3 organic) the MRL-exceedance concerned trimesium, which is known to be generated naturally during the drying process of plants.

Table 4: Samples with residues of QuPpe-compounds exceeding existing MRLs* (CVUA Stuttgart 2023)

| Compound | Commodity | Country of Origin | Conc. (mg/kg) | >2x MRL** | Note |
|--|----------------------------|-------------------|---------------|-----------|--|
| Chlorate <i>(herbicide. but chlorinated water used in irrigation or sanitation is mostly responsible for levels found in food)</i> | Baby and infant foods | Unknown | 0.017 | | organic |
| | Sesame | Nigeria | 3 | x | |
| | Sesame | Unknown | 0.074 | | |
| | Grapes | Chile | 0.605 | x | |
| | Lime | Mexico | 0.077 | | |
| | Leafy vegetables, dried | Germany | 0.21 | x | |
| | Moringa oleifera | Spain | 0.21 | x | organic |
| Chlormequat chloride | Peanut, roasted with shell | Turkey | 0.812 | x | Might have been formed as a processing contaminant during roasting |
| | Potato | Germany | 0.012 | | |
| | Garlic | China | 0.025 | x | |
| Cyromazine | Bell peppers | Turkey | 0.015 | | |
| Fosetyl, sum <i>(phosphonic acid was the only detected compound in most cases)</i> | Chick pea | Unknown | 12.1 | X | |
| | Peanut, roasted with shell | Egypt | 6.2 | x | |
| | Peanut, roasted with shell | Turkey | 11.3 | x | |
| | Peanut, roasted with shell | Unknown | 12.2 | x | |
| | White bean | Argentina | 7.9 | X | |
| | White bean | Brazil | 6.7 | X | |
| | Cherry | Germany | 3.9 | | |
| | Sour Cherry, frozen | Unknown | 11.3 | x | |
| | Green beans | Morocco | 3.1 | | |
| | Onion | New Zealand | 56.3 | | |
| | Spring onion | Germany | 39.9 | | |
| | Glyphosate | Kaki | Spain | 0.17 | |
| Kaki | | Spain | 0.23 | x | |
| Mepiquat chloride | Sweet potato | Egypt | 0.04 | | organic |

| Compound | Commodity | Country of Origin | Conc. (mg/kg) | >2x MRL** | Note |
|---|----------------------------|-------------------|---------------|-----------|---------|
| Nicotine (insecticide, but tobacco-related contamination mostly responsible for levels found in food) | Chick peas | India | 0.021 | | |
| | Pumpkin seeds | China | 0.039 | | |
| | Triphala powder | Germany | 0.33 | x | organic |
| | Clementine | Croatia | 0.018 | | |
| | Medlar | Italy | 0.013 | | |
| | Cumin seeds | Turkey | 0.0785 | | |
| | Ginger | Peru | 0.0405 | x | |
| | Lamb's lettuce | Germany | 0.015 | | organic |
| | Lamb's lettuce | Germany | 0.022 | x | |
| | Leek | The Netherlands | 0.015 | | |
| | Rucola | Germany | 0.03 | x | |
| | Spinach | Germany | 0.013 | | |
| Spinach | Italy | 0.012 | | | |
| Oxymatrine | Fennel tea | Unknown | 0.022 | x | |
| Paraquat | Brown lentil | Unknown | 0.11 | x | |
| | Mung bean | Argentina | 0.2 | x | |
| Perchlorate (contaminant in fertilizers) | Prickly pear | Turkey | 0.061 | | |
| | Basil dried | Unknown | 15.7 | x | |
| | Moringa oleifera | Germany | 5.2 | x | |
| | Bell peppers | Germany | 0.25 | x | |
| | Moringa oleifera | Spain | 4.2 | x | organic |
| | Pak choi | Belgium | 0.074 | | |
| Propamocarb | Peanut, roasted with shell | Egypt | 0.029 | x | |
| | Pepper | Iraq | 0.054 | | |
| Trimesium (Counter ion of glyphosate but also natural formation during drying process of crops) | Moringa oleifera | Germany | 0.061 | | organic |
| | Moringa oleifera | Germany | 0.051 | | |
| | Parsley, dried | Unknown | 0.52 | | |
| | Black tea | Sri Lanka | 0.088 | | |
| | Black tea | Unknown | 0.39 | x | organic |
| | Black tea | Unknown | 0.067 | | |
| | Black tea | Unknown | 0.09 | | |
| | Moringa oleifera | Germany | 0.12 | x | |
| Moringa oleifera | Spain | 0.094 | | organic | |

* >1xMLR

** >2xMRL means that the sample exceeded MRL even after deducting measurement uncertainty of 50%

Table 5: Top 15 residue levels of the most-frequently found QuPPE-compounds (with > 50 findings in total)

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|--|----------------------------|-------------------|-----------------------|
| Cyanuric acid (non-regulated pesticide metabolite and contaminant in fertilizers) | Algae, dried | Germany | 4.9 |
| | Shiitake, dried | China | 1.5 |
| | Potato | Germany | 0.95 |
| | Cultivated mushroom, dried | China | 0.78 |
| | Mandarine | Israel | 0.76 |
| | Pineapple | Costa Rica | 0.74 |
| | Cordyceps capsules | Germany | 0.71 |
| | Orange juice | Unknown | 0.68 |
| | Apple juice | Germany | 0.66 |
| | Chili powder | Unknown | 0.63 |
| | Paprika spice | Unknown | 0.62 |
| | Paprika spice | Germany | 0.61 |
| | Pineapple | Costa Rica | 0.60 |
| | Chili powder | Unknown | 0.46 |
| | Pineapple | Costa Rica | 0.44 |
| Phosphonic acid (Metabolite of Fosetyl but also used as such. Shows high persistence in plants. and residues in crops of perennial plants may originate from previous seasons) | Almond | USA | 79.2 |
| | Hops | Germany | 56.1 |
| | Onion | New Zealand | 41.9 |
| | Raisin and Sultana | South Africa | 41.8 |
| | Hops pellets | Germany | 38.4 |
| | Avocado | Brazil | 38.2 |
| | Spring onion | Germany | 29.7 |
| | Strawberry | Germany | 29.3 |
| | Hops pellets | Germany | 25.9 |
| Strawberry | Italy | 22.6 | |

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|---|----------------------------|-------------------|-----------------------|
| | Strawberry | Germany | 22.1 |
| | Bell peppers | Turkey | 20.4 |
| | Grapes | Italy | 16.3 |
| | Potato | France | 16.1 |
| | Strawberry | Germany | 15.9 |
| Chlorate <i>(herbicide, but chlorinated water used in irrigation or sanitation mostly responsible for levels found in food)</i> | Sesame | Nigeria | 3.0 |
| | Paprika spice | Unknown | 1.1 |
| | Parsley, dried | Unknown | 0.68 |
| | Chili powder | Unknown | 0.68 |
| | Grapes | Chile | 0.61 |
| | Parsley | Spain | 0.54 |
| | Algae, dried | Germany | 0.41 |
| | Paprika spice | Germany | 0.34 |
| | Chili powder | Unknown | 0.29 |
| | Bell peppers | Belgium | 0.22 |
| | Moringa oleifera | Germany | 0.21 |
| | Moringa oleifera | Spain | 0.21 |
| | Dill leaves | Unknown | 0.21 |
| | Chives, frozen | Unknown | 0.19 |
| | Chives, frozen | The Netherlands | 0.17 |
| Perchlorate <i>(Regulated as contaminant)</i> | Basil dried | Unknown | 15.7 |
| | Moringa oleifera | Germany | 5.2 |
| | Moringa oleifera | Spain | 4.2 |
| | Parsley, dried | Unknown | 0.62 |
| | Moringa oleifera | Germany | 0.42 |
| | Cumin seeds | Turkey | 0.42 |
| | Moringa oleifera | Tanzania | 0.41 |
| | Moringa oleifera | Germany | 0.37 |
| | Oregano dried | Unknown | 0.33 |
| | Cumin seeds | Unknown | 0.31 |
| | Cumin seeds | Unknown | 0.27 |
| | Oregano dried | Unknown | 0.27 |
| | Bell peppers | Germany | 0.25 |
| | Cumin seeds | Unknown | 0.25 |
| | Cumin seeds | Germany | 0.24 |
| Melamine <i>(Metabolite of cyromazine, but also contaminant originating from multiple sources; regulated as contaminant)</i> | Cultivated mushroom, dried | China | 6.5 |
| | Potato | Germany | 1.30 |
| | Parsley | Italy | 1.18 |
| | Chives | Germany | 0.68 |
| | Leek | Germany | 0.52 |
| | Parsley | Germany | 0.52 |
| | Hops pellets | Germany | 0.45 |
| | Celeriac | Germany | 0.42 |
| | Carrot | Germany | 0.41 |
| | Hops pellets | Germany | 0.39 |
| | Potato | Germany | 0.38 |
| | Hops | Germany | 0.36 |
| | Cucumber | Spain | 0.33 |
| | Coriander leaves | Germany | 0.27 |
| | Lambs lettuce | Italy | 0.25 |
| Thiocyanate <i>(mostly of natural origin, especially in brassica crops)</i> | Red cabbage | Germany | 51.1 |
| | Brussels sprout | The Netherlands | 47.2 |
| | Brussels sprout | The Netherlands | 44.4 |
| | Red cabbage | Germany | 43.9 |
| | Broccoli | Spain | 42.1 |
| | White cabbage | Germany | 41.8 |
| | Brussels sprout | The Netherlands | 37.0 |
| | Brussels sprout | The Netherlands | 34.3 |
| | Brussels sprout | The Netherlands | 34.3 |
| | Brussels sprout | The Netherlands | 34.0 |
| | Brussels sprout | The Netherlands | 33.0 |
| | Red cabbage | Germany | 31.7 |
| | Red cabbage | Germany | 31.0 |

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|-------------------------|------------------------------|-------------------|-----------------------|
| Propamocarb | Red cabbage | Germany | 30.0 |
| | White cabbage | Germany | 29.7 |
| | Head lettuce | Belgium | 3.5 |
| | Cucumber | Germany | 1.3 |
| | Cucumber | Spain | 1.2 |
| | Cucumber | Spain | 1.2 |
| | Cucumber | Spain | 1.1 |
| | Cucumber | Spain | 1.0 |
| | Cucumber | Unknown | 0.98 |
| | Cucumber | Spain | 0.81 |
| | Cucumber | Spain | 0.73 |
| | Cucumber | Greece | 0.49 |
| | Tomato | Spain | 0.48 |
| | Melon | Costa Rica | 0.41 |
| | Cucumber | Spain | 0.39 |
| Iceberg lettuce | Germany | 0.39 | |
| Cucumber | Spain | 0.37 | |
| Propamocarb-N-oxide | Cucumber | Spain | 0.34 |
| | Cucumber | Spain | 0.32 |
| | Cucumber | Germany | 0.31 |
| | Cucumber | Spain | 0.29 |
| | Cucumber | Spain | 0.26 |
| | Cucumber | Spain | 0.18 |
| | Cucumber | Spain | 0.17 |
| | Cucumber | Spain | 0.17 |
| | Head lettuce | Belgium | 0.15 |
| | Cucumber | Spain | 0.14 |
| | Cucumber | Unknown | 0.14 |
| | Cucumber | Greece | 0.13 |
| | Cucumber | Spain | 0.11 |
| | Cucumber | Germany | 0.09 |
| | Cucumber | Spain | 0.04 |
| Propamocarb-N-desmethyl | Tomato | Spain | 0.11 |
| | Cucumber | Spain | 0.10 |
| | Head lettuce | Belgium | 0.07 |
| | Cucumber | Spain | 0.06 |
| | Cucumber | Spain | 0.06 |
| | Cucumber | Spain | 0.06 |
| | Cucumber | Spain | 0.05 |
| | Cucumber | Greece | 0.05 |
| | Iceberg lettuce | Germany | 0.05 |
| | Cucumber | Germany | 0.04 |
| | Tomato | Spain | 0.03 |
| | Cucumber | Spain | 0.03 |
| | Melon | Costa Rica | 0.03 |
| | Pickled cucumbers, preserved | Germany | 0.02 |
| | Cucumber | Unknown | 0.02 |

Table 6: Top 10 residue levels of less frequently found QuPPE-compounds (with < 50 findings in total)

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|--|----------------------------|-------------------|-----------------------|
| Ammelide <i>(Metabolite of some triazine herbicides as well as of melamine, see above)</i> | Cultivated mushroom, dried | China | 0.59 |
| | Pepper | Unknown | 0.57 |
| | Shiitake, dried | China | 0.36 |
| | Algae, dried | Germany | 0.30 |
| | Shiitake mushroom | Germany | 0.22 |
| | Pumpkin seed | China | 0.15 |
| | Pineapple, dried | Ghana | 0.13 |
| | Shiitake mushroom | Germany | 0.12 |
| | Shiitake mushroom | The Netherlands | 0.11 |
| | King oyster mushroom | Germany | 0.10 |

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|---|--------------------------------|----------------------|-----------------------|
| Ethephon | Tomato | Turkey | 1.03 |
| | Figs | Turkey | 0.99 |
| | Pineapple | Costa Rica | 0.91 |
| | Tomato | The Netherlands | 0.55 |
| | Figs | Turkey | 0.52 |
| | Figs | Turkey | 0.45 |
| | Grapes | Spain | 0.42 |
| | Grapes | South Africa | 0.36 |
| | Sour Cherry, frozen | Unknown | 0.34 |
| | Pineapple | Costa Rica | 0.33 |
| | Nicotine | Wild mushroom, dried | Turkey |
| Wild mushroom, dried | | Unknown | 0.38 |
| Triphala capsule | | Germany | 0.33 |
| Wild mushroom, dried | | Unknown | 0.19 |
| Black tea | | Unknown | 0.12 |
| Black tea | | Unknown | 0.10 |
| Black tea | | Unknown | 0.09 |
| Cumin seeds | | Turkey | 0.08 |
| Chili powder | | Unknown | 0.08 |
| Cumin seeds | | Unknown | 0.07 |
| Bromide | Turmeric | Unknown | 159 |
| | Cumin seeds | Unknown | 131 |
| | Moringa oleifera | Spain | 85 |
| | Cumin seeds | Germany | 43 |
| | Pepper | Unknown | 39 |
| | Pepper | Unknown | 37 |
| | Moringa oleifera | Germany | 35 |
| | Walnut | Germany | 34 |
| | Pepper | Unknown | 30 |
| Moringa oleifera | Germany | 29 | |
| Chlormequat chloride | Peanut, roasted with shell | Turkey | 0.81 |
| | Cordyceps capsules | Germany | 0.39 |
| | Rye flour | Unknown | 0.32 |
| | Rye flour | Germany | 0.30 |
| | Rye flour | Germany | 0.23 |
| | Rye flour | Germany | 0.19 |
| | Pasta | Unknown | 0.11 |
| | Coarse grains | Germany | 0.11 |
| | Vegan/vegetarian food, no milk | Unknown | 0.10 |
| | Rye flour | Germany | 0.09 |
| Trimesium <i>(Counter ion of glyphosate but also natural formation during drying process of crops)</i> <i>(Non-regulated metabolite of Ethephon)</i> | Parsley, dried | Unknown | 0.52 |
| | Black tea | Unknown | 0.39 |
| | Moringa oleifera | Germany | 0.12 |
| | Majoram dried | Unknown | 0.11 |
| | Moringa oleifera | Spain | 0.09 |
| | Black tea | Unknown | 0.09 |
| | Black tea | Sri Lanka | 0.09 |
| | Black tea | Unknown | 0.07 |
| | Moringa oleifera | Germany | 0.06 |
| | Moringa oleifera | Germany | 0.05 |
| | Chloridazon-desphenyl | Dill, frozen | Germany |
| Chives, frozen | | Unknown | 0.02 |
| Parsley, frozen | | Unknown | 0.02 |
| Dill leaves | | Germany | 0.02 |
| Dill, frozen | | Germany | 0.02 |
| Parsley | | Unknown | 0.02 |
| Chives, frozen | | Germany | 0.02 |

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|-------------------------------|--------------------------------|-------------------|-----------------------|
| | Green beans, frozen | Unknown | 0.01 |
| | Cimarapa | Italy | 0.01 |
| | Green beans | Unknown | 0.01 |
| HEPA | Pineapple | Costa Rica | 0.54 |
| | White button mushroom | Germany | 0.39 |
| | White button mushroom | Germany | 0.31 |
| | Artichoke | Italy | 0.30 |
| | Grapes | South Africa | 0.10 |
| | Pineapple | Costa Rica | 0.10 |
| | Grapes | Italy | 0.07 |
| | Grapes | South Africa | 0.07 |
| | Grapes | Spain | 0.07 |
| | Tomato | Turkey | 0.06 |
| Maleic hydrazide | Potato | Germany | 27.7 |
| | Potato | France | 12 |
| | Potato products | Unknown | 7.7 |
| | Onion | Spain | 7.6 |
| | Shallots | Germany | 7.5 |
| | Potato | France | 6.6 |
| | Potato | Israel | 6.4 |
| | Potato products | Unknown | 6.2 |
| | Onion | Germany | 6.2 |
| | Potato products | Unknown | 5.3 |
| Thiram M1 | Bell peppers | Turkey | 0.05 |
| | Green beans | Senegal | 0.04 |
| | Green beans | Morocco | 0.04 |
| | Zucchini | Turkey | 0.02 |
| | Apricot | Turkey | 0.02 |
| | Strawberry | Spain | 0.01 |
| | Pear | Turkey | 0.01 |
| | Plum | Portugal | 0.01 |
| | Pear | Italy | 0.01 |
| | Mint | Unknown | 0.01 |
| Glyphosate | White beans | Brazil | 0.99 |
| | Black tea | Sri Lanka | 0.52 |
| | Brown lentil | Unknown | 0.49 |
| | White beans | Argentina | 0.34 |
| | Paprika | Unknown | 0.29 |
| | Kaki | Spain | 0.23 |
| | Kaki | Spain | 0.17 |
| | Pepper | Unknown | 0.10 |
| | Pomegranate | Turkey | 0.09 |
| | Carrot | Germany | 0.08 |
| Dimethoate-O-desmethyl | Onion | Germany | 0.20 |
| | Leek | Germany | 0.14 |
| | Orange | Italy | 0.13 |
| | Black-eyed pea | Madagascar | 0.10 |
| | Wine leaves, prepared in brine | Egypt | 0.09 |
| | Melon | Costa Rica | 0.05 |
| | Sweet potato | Egypt | 0.03 |
| | Raspberry, frozen | Unknown | 0.03 |
| | Avocado | Colombia | 0.02 |
| | Sweet potato | Egypt | 0.02 |
| Mepiquat chloride | Cordyceps capsules | Germany | 0.17 |
| | Rye flour | Unknown | 0.06 |
| | Sweet potato | Egypt | 0.04 |

| Compound | Commodity | Country of origin | Residue level (mg/kg) |
|--------------------------------|-----------------------|-------------------|-----------------------|
| | Sunflower seed | Unknown | 0.02 |
| | White button mushroom | Poland | 0.02 |
| | White button mushroom | Germany | 0.02 |
| | White button mushroom | Germany | 0.02 |
| | White button mushroom | Poland | 0.02 |
| | Rye flour | Germany | 0.02 |
| | Mango | Dom. Republic | 0.01 |
| 4-Methylimidazoline | Lime | Colombia | 0.02 |
| | Grapes | India | 0.02 |
| | Grapes | India | 0.02 |
| | Melon | Costa Rica | 0.01 |
| | Grapes | India | 0.01 |
| | Mango | Dom. Republic | 0.01 |
| | Grapes | India | 0.01 |
| | Lime | Colombia | 0.01 |
| | Grapes | India | 0.01 |
| | EU | Cumin seeds | Turkey |
| Basil dried | | Unknown | 0.18 |
| Wine leaves, prepared in brine | | Turkey | 0.12 |
| Parsley | | Germany | 0.11 |
| Papaya | | Ghana | 0.11 |
| Wine leaves, prepared in brine | | Turkey | 0.08 |
| Wine leaves, prepared in brine | | Turkey | 0.08 |
| Head lettuce | | Germany | 0.06 |
| Wine grapes | | Germany | 0.04 |
| Glufosinate MPPA | Shiitake, dried | China | 0.13 |
| | Lemon | South Africa | 0.03 |
| | Grapes | South Africa | 0.03 |
| | Grapes | South Africa | 0.02 |
| | Plum | South Africa | 0.02 |
| | Raspberry, frozen | Unknown | 0.02 |
| | Raspberry, frozen | Spain | 0.02 |
| | Plum | South Africa | 0.02 |
| | Raspberry, frozen | Unknown | 0.01 |
| Fosetyl | Melon | Honduras | 0.65 |
| | Hops | Germany | 0.47 |
| | Cucumber | Germany | 0.30 |
| | Grapes | South Africa | 0.08 |
| | Grapes | Italy | 0.04 |
| | Grapes | India | 0.02 |

Findings in organic samples

A look at the residues encountered in organic products gives an insight on compounds that potentially end up in food products from natural sources and on the matrices affected by such background contamination.

In 2023 the vast majority of the findings in organic products concerned compounds known to contaminate food products either naturally, or from applications other than the use of pesticides. Thiocyanate is naturally occurring in various crops of the brassica and allium family. Bromide is ubiquitous and is always contained in living organisms. Chlorate is often a result of using chlorinated water for irrigation or for sanitation purposes in packing and processing facilities (e.g. for washing surfaces or the products themselves). The contaminant perchlorate is often contained in fertilizers, including guano, which is used in organic agriculture. Melamine, ammeline and cyanuric acid may also originate from fertilizers. Trimesium is naturally formed when certain products are dried, and can be thus considered a processing contaminant. Phosphonic acid may originate from past applications as it is quite

persistent both in perennial plants and the soil. Still some findings raise the suspicion of a recent misuse and require further investigation. Chloridazon-desphenyl is a metabolite of chloridazon, which is quite persistent in the environment, thus residues in succeeding crops, and in water, are encountered.

Table 7: Overview of findings in 356 organic samples (no. of findings ≥ 5); (CVUA in 2023)

| Compound Name | No. of samples | RL (mg/kg) | No. of findings >RL | Percentage of positives | Mean (mg/kg) | Max (mg/kg) | Remarks |
|------------------------------|----------------|------------|---------------------|-------------------------|--------------|-------------|--|
| Ammelide | 353 | 0.005 | 12 | 3,4 | 0,086 | 0,30 | May originate from fertilizers (incl. urea fertilizers) |
| Bromide | 353 | 5 | 5 | 1,4 | 29,2 | 85,3 | Ubiquitous element |
| Chlorate | 353 | 0.005 | 52 | 14,7 | 0,034 | 0,41 | May end up in organic products through irrigation or sanitation procedures in the processing facilities |
| Chloridazon-desphenyl | 353 | 0.01 | 13 | 3,7 | 0,013 | 0,027 | Metabolite of chloridazon quite persistent in the environment, thus residues in succeeding crops, and in water, are encountered. Findings concerned in the following commodities: Green beans frozen 4x, parsley frozen 2x, dill 2x, chives frozen 2x, carrot, pumpkin, basil |
| Cyanuric acid | 353 | 0.005 | 111 | 31,4 | 0,097 | 4,9 | May originate from fertilizers (incl. urea fertilizers) |
| Melamine | 353 | 0.005 | 24 | 6,8 | 0,050 | 0,182 | May originate from fertilizers (incl. urea fertilizers) |
| Perchlorate | 353 | 0.005 | 56 | 15,9 | 0,108 | 4,2 | May originate from fertilizers inclusion guano |
| Phosphonic acid | 353 | 0.05 | 8 | 2,3 | 10,4 | 79,2 | Phosphonic acid used to be employed by organic farmers in the past as it was marketed as a "leaf fertilizer" suitable for organic farming. As the compound is quite persistent, residues are still found especially in crops of perennial plants such as berries. The findings >LOQ concerned the following matrices: Almond (2x), Wine grapes (2x), Lemon, Kiwi, Avocado, Zucchini. Contamination through manure is also conceivable source especially for in vegetables. |
| Thiocyanate | 353 | 0.1 | 22 | 6,2 | 9,6 | 43,9 | Naturally occurring in various <i>allium</i> and <i>brassica</i> products, such as onions and kale |
| Trimesium | 353 | 0.005 | 5 | 1,4 | 0,117 | 0,39 | Trimesium was shown to be formed naturally during the drying process of various products of Black tea, Sweet potato, Moringa 3x. |

Residues of Ethylene oxide / 2-Chloroethanol

In 2023 a total of 131 samples, mainly highly processed products from third countries or unknown origin, were analyzed for residues of a fumigation with ethylene oxide. Ethylene oxide fumigations are not approved within the EU but are not uncommon elsewhere. The main purpose is disinfestation and disinfection. Since ethylene oxide is carcinogenic, mutagenic and reprotoxic and since 2-chloroethanol, the main a reaction product of ethylene oxide, is a suspected mutagenic, EU-MRLs are set at the analytical limit of determination (practical zero tolerance).

2-chloroethanol was encountered in quantifiable levels in only four of the 131 samples (in food supplements (3x) and in sesame (1x)). The food supplements with positive findings were based on Triphala, Cordyceps and Moringa. Residues of the highly volatile and reactive ethylene oxide were not found in any of the tested samples.

Is seems as if the official border controls as well the controls of the food business operators themselves seem to be very successful in preventing the import of ethylene oxide fumigated food products into EU market.