Residue Findings of QuPPe-Compounds in Samples of Plant Origin from the German Market in 2022

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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 analytes, on how to plan sampling, on which QuPPe compounds are worthwhile checking in the various samples. Ultimately, this contributes to a more targeted and efficient use of lab resources.
At CVUA Stuttgart, 44 QuPPe-compounds were routinely monitored in 2022 (see Table 1). Some of these compounds are not legally relevant, as they are not part of the legal residue definition.
Despite not being a QuPPe compound, a brief overview of the residue findings of 2-chloroethanol (formed from the reaction of the fumigant ethylene oxide with chloride) in 2022 is given at the end of this document. 2-CE findings in various products have been causing severe turbulences in the EU-food market since autumn 2020.

Table 1: Scope of QuPPe-compounds that were routinely monitored by the CVUA Stuttgart in 2022

Compound	Notes on legal limits	General notes
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, ametrin, cyanazine. Among those pesticides, 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 ammelide
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 fumigant methylbromide. Also originating from irrigation water and soil. Counter ion of certain quarternary ammonium compounds e.g. benzalkonium, Didecyl Dimethyl Ammonium (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 (which approval expired on 31/12/2018). Chloridazon-desphenyl is quite persistent in the environment, thus residues in succeeding crops, and in water, are encountered.
Chlormequat	MRLs refer to chlormequat chloride	EU-Approved growth regulator with a wide range of applications.
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, algaecides and bactericides. They are used in sanitation liquids and 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).

Compound	Notes on legal limits	General notes
Cyromazine	MRLs in food of plant origin are set at the LOQ with multiple exceptions (e.g. fruiting vegetables, lettuces, celery, fresh herbs, and sheep products).	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 dipyridone	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. Natural formation by bacteria under anaerobic conditions was reported. 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 needs to be checked.
ETU (ethylene thiourea)	Non-regulated degradant	Degradant of ethylen-bis-dithiocarbamates. Also impurity in formulations based on EBTCs. Also formed during food processing. Marker for EBTC-application history. Note: Most EBDTCs 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 did not receive EU-approval). Metiram is still approved (current approval period ends in Jan 24).
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.
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
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 *Sophora* 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 *Sophora* root extracts in fertilizers in Italy. Together with oxymatrine, often found in so-called "acacia honey" from China, which mostly originated from flowers of *Sophora* plants. *Sophora* extracts are also used in traditional Asian medicine and cosmetics.
Oxymatrine	MRL of 0.01 mg/kg applies (listed in EU-pesticide database)	Quinolizidine alkaloid present in *Sophora* 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 am-

Compound	Notes on legal limits	General notes
		meline 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
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 family, such as bensultap, sultap, cartap and thiocyclam
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 takes place in all stages of food production, through air, soil and human contact.
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		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)	Regulated in infant- and baby food Reg. EC 125/2006 and 141/2006	Degradant of propylen-bis-dithiocarbamates (essentially propineb). Also impurity in formulations based on propineb. Also formed during food processing. 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 take into account the 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).
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 2022, a total of 2714 samples, mainly fruit and vegetables, but also cereals, pulses, processed goods, tea and others, were analyzed for QuPPe-amenable compounds at the CVUA Stuttgart. 1966 samples (72 %) 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 2022)

Frequency of findings > respective RL	Compounds (pesticides and legally relevant metabolites shown in bold)
> 10 % of samples.	*Cyanuric acid, **Phosphonic acid**, Perchlorate, **Chlorate** and Melamine*
1 - 10 % of samples.	***2*-Chloroethanol**[1)], *Ammelide, **Thiocyanate**[2)], **Propamocarb**, Propamocarb-N-oxide, **Paraquat**, Propamocarb-N-desmethyl, Ethephon metabolite HEPA, **Trimetsium**, **Ethephon**, **Nicotine**, **Maleic hydrazide** and **Chlormequat-chloride***
0.1 -1% of samples	***Bromide**[3)], **Mepiquat-chloride**, **Diquat**, **Chloridazon-desphenyl**, **Glyphosate**, **Fosetyl**, Dimethoate-O-desmethyl, Morpholine, **Glufosinate met. MPPA**, Nereistoxin, Mepiquat- 4-hydroxy, **Amitrole**, **Glufosinate**, ETU and **Cyromazine***
Not detected above LOQ	*Ammeline, **Daminozide**, **Difenzoquat**, Diquat-dipyridone, Diquat met. TOPPS, Diquat-monopyridone, **Ethylene oxide** [1)], Glyphosate met. N-Acetylglyphosate, Glyphosate met. AMPA, **Matrine**, N-**Acetyl-glufosinate**, **Oxymatrine**, and PTU*

[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 the main focus being dry samples.*
[2)] *No MRLs were deemed necessary for thiocyanate.*
[3)] *Bromide is ubiquitous and virtually every sample is positive. The RL of 5 mg/kg represents the lowest MRL is food of plant origin.*

Table 3: Residue findings of QuPPe-compounds (CVUA Stuttgart 2022)

Compound	# samples	# pos.	% pos.	Max (mg/kg)	Mean[1] (mg/kg)	Median[1] (mg/kg)	# >MRL[2]	% >MRL	RL[3]
Cyanuric acid	2714	971	36	2.7	0.04	0.012	Not part of RD		0.005
Phosphonic acid	2720	622	23	138.0	3.4	0.97	**10**	0.4	0.05
Perchlorate	2714	461	17	0.62	0.04	0.013	***2***[4]	*0.07*	0.005
Chlorate	2715	359	13	10.1	0.07	0.013	**9**	0.3	0.005
Melamine	2714	309	11	1.6	0.12	0.034	*0*[4]	*0*	0.005
Thiocyanate	2714	198	7.3	80.6	9.1	3.8	0	0	0.1
Propamocarb	2716	101	3.7	21.7	0.38	0.027	**2**	0.07	0.005
Propamocarb-N-oxide	2715	80	2.9	0.43	0.05	0.024	0	0	0.005
Paraquat	285	7	2.5	0.50	0.12	0.089	**5**	1,8	0.01
Propamocarb-N-desmethyl	2714	62	2.3	0.71	0.04	0.012	Not part of RD		0.005
Ethephon metabolite HEPA	2714	61	2.2	5.5	0.23	0.095	Not part of RD		0.005
Ammelide	2714	48	1.8	0.50	0.10	0.067	Not part of RD		0.005
Trimesium	2714	47	1.7	1.2	0.09	0.031	**12**[5]	0.4	0.005
Ethephon	2714	41	1.5	1.7	0.21	0.11	**2**	0.07	0.01
Nicotine	2714	39	1.4	0.32	0.08	0.054	**12**	0.4	0.01
Chlormequat-chloride	2714	33	1.2	1.0	0.11	0.044	**5**	0.2	0.005
Maleic hydrazide	2714	32	1.2	19.3	6.3	6.2	**1**	0.04	0.01
Bromide[6]	2714	25	0.9	66.3	26.0	24.5	0	0	5
Mepiquat chloride	2714	23	0.8	0.18	0.04	0.013	**2**	0.07	0.005
Diquat	285	2	0.7	0.0	0.02	0.024	0	0	0.01
Chloridazon-desphenyl	2714	17	0.6	0.26	0.03	0.010	0	0	0.01
Fosetyl	2720	13	0.5	1.4	0.19	0.082	0	0	0.01
Glyphosate	2714	13	0.5	3.2	0.43	0.19	0	0	0.02
Dimethoate-O-desmethyl	2714	12	0.4	0.18	0.04	0.018	0	0	0.005
Glufosinate met. MPPA	2714	3	0.1	0.29	0.12	0.055	**1**	0.04	0.01
Morpholine	2715	3	0.1	0.48	0.25	0.17	additive		0.02
Amitrole	2714	2	0.07	0.20	0.10	0.10	**1**	0.04	0.005
Mepiquat, 4-hydroxy	2714	2	0.07	0.01	0.01	0.01	Not part of RD		0.005
Nereistoxin	2714	2	0.07	0.19	0.11	0.11	Not part of RD		0.005
Cyromazine	2714	1	0.04	0.08	-	-	0	0	0.005
ETU[7]	2714	1	0.4	0.14			Not part of RD		0.05
Glufosinate	2714	1	0.04	0.15	-	-	0	0	0.01

[1] Mean and median of positives
[2] Numerical MRL-exceedances
[3] RL= Reporting Limit (exemplary for fruits and vegetables)
[4] Perchlorate and melamine are legally contaminants. In two cases the ML of perchlorate, as defined in Reg.(EC) No. 1881/2006, was exceeded
[5] MRL-exceedances of trimesium are in most cases most likely due to the formation of trimesium during processing
[6] Bromide is ubiquitous and virtually every sample is positive. The RL of 5 mg/kg represents the lowest MRL is food of plant origin.
[7] Additional: 19 findings below LOQ used for dithiocarbamate screening, see also Table 6

MRL exceedances

In 78 samples (thereof 6 organic) MRLs of different compounds were numerically exceeded. In 26 of these samples (thereof 3 organic) the MRLs were exceeded even after deducting 50% measurement uncertainty. Table 4 gives an overview of these exceedances. In 5 samples (thereof 2 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 2022)

Compound	Commodity	Country of Origin	Conc. (mg/kg)	>2x MRL**	Note
Amitrole	Hop	Germany	0.2	X	
Chlorate *(herbicide. but chlorinated water used in irrigation or sanitation mostly responsible for levels found in food)*	Cherry	Italy	0.16	X	
	Cucumber	Germany	0.33		
	Nectarine	Unknown	0.054		
	Okra, frozen	Egypt	0.13		
	Spices, chili	Unknown	10.1	X	
	Sweet potato	Egypt	0.28	X	

Compound	Commodity	Country of Origin	Conc. (mg/kg)	>2x MRL**	Note
	Tomato	Unknown	0.64	X	
	Milk substitute	Unknown	2.4	X	
	Zucchini	Spain	0.22		
Chlormequat chloride	Ginger	China	0.039	X	
	Spices, chili	Unknown	0.12		
	Spices, chili	Unknown	0.15		
	Spices, chili	Unknown	0.18		
	Sunflower seed	Turkey	0.034		
Ethephon	Mango	Dom. Republic	0.38	X	
	Plum	Italy	0.099		
Fosetyl, sum *(phosphonic acid was the only detected compound in most cases)*	Asparagus	Germany	2.69		
	Asparagus	Germany	3.09		
	Asparagus	Germany	3.09		
	Avocado	South Africa	111.2		
	Button mushroom, dried	Poland	43.6	X	
	Plum jam	Germany	5.7	X	
	Green beans	Egypt	3.2		
	Lentil	Unknown	6.2	X	
	Mango	Dom. Republic	4.1		
	Mango	Peru	3.1		
	Mung bean	Myanmar	2.6		
	Quinoa	Unknown	3.5		organic
	Rucola	Italy	185.3	X	
	Sesame, roasted	Unknown	2.3		
Glufosinate (MPPA)	Nectarine	Unknown	0.29		
Maleic hydrazide	Green beans, frozen	Unknown	0.22		
Mepiquat chloride	Ginger	China	0.05	X	
	White button mushroom	Germany	0.015		
Nicotine *(insecticide, but tobacco-related contamination mostly responsible for levels found in food)*	Hops	Germany	0.017		
	Hops	Germany	0.021	X	
	Hops	France	0.016		
	Borecole	Germany	0.02		
	Borecole	Germany	0.031	X	
	Carob	Turkey	0.016		
	Flax seed, broken	India	0.014		organic
	Lambs lettuce	Germany	0.013		
	Pear	Turkey	0.18		organic
	Spices, chili	Unknown	0.21		
	Spices, chili	Unknown	0.012		
	Spinach	Germany	0.015		
Paraquat	Chia seeds	America	0.095	X	organic
	Chia seeds	Paraguay	0.064	X	
	Chia seeds	Paraguay	0.089	X	
	Sesame, roasted	Unknown	0.5	X	
	Spices, black pepper	Unknown	0.092		
Perchlorate *(contaminant in fertilizers)*	Gooseberry	Germany	0.17	X	ML=0.05 mg/kg
	Pomegranate	Peru	0.10		ML=0.05 mg/kg
Propamocarb	Green beans	Morocco	0,17		
	Strawberry, frozen	Egypt	0,026	X	
Trimesium *(Counter ion of glyphosate but also natural formation during drying process of crops)*	Black tea	India	0.11	X	organic
	Black tea	Sri Lanka	0.13	X	
	Black tea	Unknown	0.052		
	Black tea	Unknown	0.07		
	Black tea	Unknown	0.078		
	Black tea	Unknown	0.1		
	Black tea	Unknown	0.12	X	
	Spices, parsley	Unknown	1.09	X	
	Vegetable powder	Germany	1.2	X	organic
	Vegetable powder	Germany	0.098		
	White button mushroom	Germany	0.059		
	White button mushroom	Germany	0.086		

* >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)*	Spices, chilli	Unknown	2.7
	Garlic, wild (Ramson)	Bulgaria	2.1
	Oyster mushroom	Germany	1.4
	Lemon	Spain	0.92
	Pineapple	Costa Rica	0.79
	Pineapple	Costa Rica	0.68
	Pineapple	Costa Rica	0.63
	Avocado	Israel	0.54
	Oyster mushroom	Poland	0.5
	Oyster mushroom	Poland	0.47
	Pineapple	Costa Rica	0.46
	Spices, chilli	Unknown	0.46
	Oyster mushroom	Germany	0.45
	Pineapple	Costa Rica	0.4
	Avocado	Israel	0.39
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)*	Rucola	Italy	138
	Hops	Germany	91.3
	Avocado	South Africa	82.8
	Strawberry	Germany	63.7
	Hops	Germany	63
	Blackberry	Germany	53.2
	Hops	Germany	43.6
	Avocado	Chile	40.5
	Rucola	Italy	39.3
	Strawberry	Germany	37.1
	Button mushroom, dried	Poland	32.5
	Pear	Italy	29.2
	Chicory	Germany	25.1
	Pear	Italy	22.5
	Blackberry	Germany	22.4
Perchlorate *(Regulated as contaminant)*	Vegetable powder	Germany	0.62
	Spices, parsley	Unknown	0.48
	Spices, parsley	Unknown	0.47
	Spices,cumin	Unknown	0.42
	Spices, cinnamon	Unknown	0.38
	Lambs lettuce	Italy	0.37
	Spices, cinnamon	Unknown	0.35
	Parsley	Spain	0.31
	Spices, cinnamon	Unknown	0.28
	Hops	Germany	0.27
	Spices, cumin	Unknown	0.26
	Spices, dill	Unknown	0.24
	Spices, cinnamon	Unknown	0.23
	Spices, oregano	Unknown	0.22
	Spices, cinnamon	Turkey	0.2
Chlorate *(herbicide, but chlorinated water used in irrigation or sanitation mostly responsible for levels found in food)*	Spices, chilli	Unknown	10.1
	Milk substitute	Unknown	2.4
	Spices, chilli	Unknown	0.72
	Tomato	Unknown	0.64
	Button mushroom, dried	Poland	0.42
	Spices, chilli	Unknown	0.39
	Spices, parsley	Unknown	0.36
	Cucumber	Germany	0.33
	Spices, chilli	Unknown	0.32
	Cultivated mushroom , dried	China	0.29
	Sweet potato	Egypt	0.28
	Spices, sweet pepper	Unknown	0.27
	Celery	Spain	0.24
	Spices, chilli	Unknown	0.22
	Zucchini	Spain	0.22

Compound	Commodity	Country of origin	Residue level (mg/kg)
Melamine *(Metabolite of cyromazine, but also contaminant originating from multiple sources; regulated as contaminant)*	Hops	Germany	1.6
	Parsley	Germany	1.5
	Potato	Germany	1.4
	Potato	Germany	1.4
	Parsley	Unknown	1.2
	Potato	Germany	1.1
	Hops	Germany	1,0
	King oyster mushroom	Germany	1,0
	Chives	Germany	0.84
	Potato	Germany	0.81
	Hops	Germany	0.69
	Ginger	China	0.64
	Food contact material	Unknown	0.61
	Lambs lettuce	Italy	0.61
	Potato	Germany	0.59
Thiocyanate *(mostly of natural origin)*	White cabbage	Germany	80.6
	Brussels sprout	The Netherlands	52.6
	Savoy cabbage	Italy	47.7
	Brussels sprout	Italy	43.2
	Brussels sprout	The Netherlands	43.2
	Cereal sprouts	Germany	37.2
	Brussels sprout	The Netherlands	34.2
	Brussels sprout	The Netherlands	34.2
	Savoy cabbage	Germany	31.9
	Brussels sprout	The Netherlands	30.8
	White cabbage	Germany	29.6
	White cabbage	Germany	29.6
	Brussels sprout	The Netherlands	29
	Broccoli	Italy	28.5
	Brussels sprout	The Netherlands	27.6
Propamocarb	Spinach	Spain	21.7
	Spinach, frozen	Unknown	4.2
	Head lettuce	Germany	1.7
	Spinach	Belgium	1.7
	Cucumber	Spain	0.76
	Cucumber	Spain	0.52
	Cucumber	Spain	0.5
	Lollo	France	0.5
	Cucumber	Spain	0.49
	Cucumber	Austria	0.4
	Cucumber	Spain	0.39
	Cucumber	Spain	0.33
	Cucumber	Unknown	0.28
	Cucumber	Unknown	0.27
	Cucumber	The Netherlands	0.26
Propamocarb-N-oxide	Brussels sprout	The Netherlands	0.43
	Spinach	Spain	0.30
	Brussels sprout	The Netherlands	0.27
	Cucumber	Spain	0.27
	Brussels sprout	The Netherlands	0.19
	Cucumber	Spain	0.16
	Cucumber	Spain	0.14
	Brussels sprout	The Netherlands	0.13
	Cucumber	Unknown	0.1
	Cucumber	Spain	0.099
	Cucumber	Spain	0.098
	Cucumber	Spain	0.088
	Cucumber	Spain	0.081
	Cucumber	Spain	0.076
	Cucumber	Spain	0.072
Propamocarb-N-desmethyl	Spinach	Spain	0.71
	Spinach, frozen	Unknown	0.36
	Spinach	Belgium	0.092

Compound	Commodity	Country of origin	Residue level (mg/kg)
	Brussels sprout	The Netherlands	0.078
	Cucumber	Spain	0.064
	Head lettuce	Germany	0.048
	Cucumber	Spain	0.045
	Cucumber	Spain	0.043
	Cucumber	Unknown	0.041
	Brussels sprout	The Netherlands	0.037
	Cucumber	Spain	0.036
	Potato	Germany	0.031
	Cucumber	Spain	0.027
	Brussels sprout	The Netherlands	0.025
	Zucchini	Spain	0.025
HEPA *(Non-regulated metabolite of Ethephon)*	Button mushroom, dried	Poland	5.5
	Wild mushroom, frozen	Russian Federation	0.59
	White button mushroom	Poland	0.59
	Spices	Unknown	0.58
	White button mushroom	Poland	0.47
	White button mushroom	Germany	0.38
	White button mushroom	Poland	0.33
	King oyster mushroom	Germany	0.32
	Wild mushroom, frozen	Unknown	0.31
	White button mushroom	Poland	0.29
	White button mushroom	Poland	0.28
	White button mushroom	The Netherlands	0.26
	White button mushroom	Poland	0.26
	White button mushroom	Poland	0.26
	White button mushroom	Germany	0.24

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 *(Metabolite of some triazine herbicides as well as of melamine, see above)*	King oyster mushroom	Germany	0.5
	Pineapple, dried	Ghana	0.42
	Shiitake mushroom	Ukraine	0.27
	King oyster mushroom	Germany	0.24
	Hops	Germany	0.22
	Hops	France	0.21
	King oyster mushroom	Unknown	0.2
	Hops	Germany	0.19
	Pineapple	Ecuador	0.17
	King oyster mushroom	Germany	0.17
Trimesium *(Counter ion of glyphosate but also natural formation during drying process of crops)*	Vegetable powder	Germany	1.2
	Spices, parsey	Unknown	1.1
	Spices, dill	Unknown	0.25
	Vegetable powder, rosebuds	Unknown	0.15
	Black tea	Sri Lanka	0.13
	Black tea	Unknown	0.12
	Black tea	India	0.11
	Black tea	Unknown	0.1
	Vegetable powder	Germany	0.098
	White button mushroom	Germany	0.086
Ethephon	Figs	Turkey	1.7
	Pineapple	Costa Rica	0.73
	Sour cherry preserves	Unknown	0.64
	Grapes	Brazil	0.5
	Spices	Unknown	0.43
	Grapes	South Africa	0.38
	Grapes	South Africa	0.32
	Grapes	Peru	0.29

Compound	Commodity	Country of origin	Residue level (mg/kg)
	Sour cherry, frozen	Unknown	0.28
	Grapes	Chile	0.27
Nicotine	Black tea	India	0.32
	Spices, oregano	Turkey	0.27
	Spices, cinnamon	Sri Lanka	0.25
	Spices, chilli	Unknown	0.21
	Spices, chilli	Unknown	0.18
	Spices, cinnamon	Unknown	0.17
	Black tea	Unknown	0.16
	Spices, cinnamon	Unknown	0.15
	Spices, cinnamon	Unknown	0.14
	Spices, cinnamon	Unknown	0.14
Chlormequat chloride	Hops	France	0.044
	Cereal flakes & porridge	Unknown	0.072
	Cereal flakes & porridge	Unknown	0.36
	Oat	Germany	0.022
	Oat	Germany	0.15
	Oat	Germany	1.03
	Wheat	Germany	0.064
	Wheat flour	Unknown	0.052
	Soy bean	Unknown	0.008
	Sunflower seed	Turkey	0.022
Maleic hydrazide	Potato	Germany	19.3
	Potato	Germany	14.6
	Potato	Germany	13.7
	Potato	Germany	10.9
	Potato	France	10.4
	Potato	France	9.6
	Onion	Germany	9.3
	Potato	Germany	9.1
	Potato	Germany	8.8
	Potato	Germany	8.6
Bromide	Spices, cinnamon	Unknown	66.3
	Spices, cumin	Unknown	57.6
	Spinach	Italy	42.9
	Spices, cumin	Turkey	42.4
	Spices, pepper black	Unknown	40.7
	Rucola	Unknown	37
	Food contact material	Unknown	36.5
	Vegetable powder	Germany	31.6
	Spices, basil	Unknown	31.4
	Spices, basil	Germany	31.3
Mepiquat chloride	Cereal flakes & porridge	Unknown	0.013
	Cereal flakes & porridge	Unknown	0.056
	Sunflower seed	Unknown	0.081
	Button mushroom, dried	Poland	0.063
	King oyster mushroom	Germany	0.022
	King oyster mushroom	Germany	0.024
	Oyster mushroom	Germany	0.18
	White button mushroom	Germany	0.008
	White button mushroom	Germany	0.009
	White button mushroom	Germany	0.009
Chloridazon-desphenyl	Chamomile tea	Unknown	0.26
	Dill leaves	Germany	0.094
	Coriander	Germany	0.045
	Parsley, frozen	Poland	0.023
	Parsley	Germany	0.016

Compound	Commodity	Country of origin	Residue level (mg/kg)
	Spinach	Belgium	0.014
	Celeriac	The Netherlands	0.011
	Green beans, frozen	Unknown	0.01
	Spinach, frozen	Unknown	0.01
	Kale, frozen	Unknown	0.009
Fosetyl	Beer ingredients	Germany	1.4
	Zucchini	Spain	0.2
	Grapes	Italy	0.16
	Rucola	Italy	0.14
	Wine	Germany	0.095
	Rucola	Italy	0.086
	Wine	France	0.082
	Wine	Germany	0.076
	Wine	Germany	0.054
	Cucumber	Spain	0.048
Glyphosate	Lentil	Unknown	3.2
	Lentil	Canada	0.66
	Flax seed	Unknown	0.41
	Lentil	Turkey	0.29
	Lentil	Unknown	0.29
	Spices, chilli	Unknown	0.21
	Flax seed, broken	Unknown	0.19
	Lentil	Unknown	0.12
	Lentil	Turkey	0.11
	Pomegranate	Turkey	0.044
Dimethoate-O-desmethyl	Teas and tea-like products	Albania	0.18
	Leek	Germany	0.13
	Garlic	Spain	0.081
	Quince	Turkey	0.022
	Leek	Germany	0.022
	Bell peppers	Hungary	0.021
	Leek	Germany	0.015
	Avocado	Colombia	0.013
	Melon	Brazil	0.012
	Leek	Unknown	0.01
ETU	Vine leaves	Turkey	0.14
	Tomato	Spain	0.019
	Wine, red	Portugal	0.008
	Black currant, nectar	Unknown	0.008
	Radish	Italy	0.006
	Green beans, preserved	Kenya	0.006
	Black currant, nectar	Unknown	0.005
	Pulses	Turkey	0.005
	Pears	Italy	0.005
	Wine, red	France	0.005

Findings in organic samples

A look at residue finding in organic products gives an insight on compounds that potentially end up in food products from natural sources and the matrices affected.

In 2022 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 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, ammelide and cyanuric acid may also originate from fertilizers. Nicotine contamination may occur through a multitude of pathways, e.g. when crops are grown on contaminated soil, when products are exposed to nicotine-containing smoke or air, or when products are touched by smokers (e.g. during harvest or processing). The origins of HEPA (degradant of ethephon) need to be investigated further, but a natural formation is possible. 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.

Table 7: Overview of findings in organic samples (no. of findings ≥5); (CVUA in 2022)

Compound Name	No. of samples	RL (mg/kg)	No. of findings >RL	Percentage of positives	Mean (mg/kg)	Max (mg/kg)	Remarks
Ammelide	394	0.005	8	2.0	0.10	0.42	May originate from fertilizers (incl. urea fertilizers)
Bromide	394	5	5	1.3	18.8	31.6	Ubiquitous element
Chlorate	394	0.005	66	16.8	0.023	0.24	May end up in organic products through irrigation or sanitation procedures in the processing facilities
Cyanuric acid	394	0.005	164	41.6	0.041	0.38	May originate from fertilizers (incl. urea fertilizers)
Ethephon met. HEPA	394	0.005	5	1.3	0.087	0.24	May indicate the use of ethephon (e.g. in the case of cultivated mushrooms the use of substrate such as hay and manure containing ethephon or HEPA). Ruminant manure is suspected to naturally contain HEPA. It needs to be clarified whether HEPA findings in other types of samples, has also natural sources or whether it is a result of ethephon use. Findings concerned in the following commodities: Cinnamon, Millet, Lupin flour, Apple Juice, White button mushroom
Melamine	394	0.005	28	7.1	0.089	1	May originate from fertilizers (incl. urea fertilizers)
Nicotine	394	0.01	5	1.3	0.16	0.32	Contamination with nicotine can occur at various stages of food production and affect conventional and organic products alike. The findings >LOQ concerned the following matrices:Cinnamon, linseed, parsley leaves, Tea and Chili.
Perchlorate	394	0.005	77	19.5	0.028	0.2	May originate from fertilizers inclusion guano
Phosphonic acid	399	0.05	17	4.3	0.53	2.6	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: Quinoa (3x), Rucola (3x), Ginger (2x), Wine (2x), Lentil (2x) Kumquat, Clementine, Strawberry, Passion fruit.
Thiocyanate	394	0.1	27	6.9	12.0	80.6	Naturally occurring in various *allium* and *brassica* products, such as onions and kale
Trimesium	394	0.005	11	2.8	0.16	1.2	Trimesium was shown to be formed naturally during the drying process of various products of plant origin. . Findings concerned in the following commodities: Basil, dried (2x), Beetroot, processed (2x), Vegetable powder, Wheatgrass powder, Flax seeds, Black tea, Berries (dried), Sunflower seeds (shelled), Rosehips, powder.

Residues of Ethylene oxide / 2-Chloroethanol

In 2022 a total of 253 samples, mainly highly processed products, 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 is a suspected mutagenic, EU-MRLs are set at the analytical limit of determination (practical zero tolerance).

In 7.5 % of the samples 2-chloroethanol, a reaction product of ethylene oxide, could be detected and quantified. In many cases, the residues concerned processed products and mixtures, which made it difficult to legally evaluate the findings. 2-Chloroethanol was mainly found in food supplement and Asian instant soups. Residues of the highly volatile and reactive ethylene oxide were not found in any of the tested samples.

Table 8: Overview of products analyzed for ethylene oxide/ 2-chloroethanol (CVUA in 2022)

Product group	No. of samples analysed	No. of samples with findings	No. of samples with levels>RL (0.05 mg/kg)	Max level mg/kg	Mean level mg/kg	>MRL
Baby and infant foods	8	0				
Cereals and cereal products	32	0				
Dry fruits and seeds	13	0				
Food additives	3	0				
Food supplements	88	8	6	117	29.4	
Fruit, processed	2	1	1	0.38	0.38	
Soups, Sauces	57	20	12	17.1	6.3	1
Spices, seasonings	21	0				
Vegan/vegetarian substitute products	11	0				
Other	18	0				
Total	253	30	19 (7.5%)	117	13.3	1 (0.4%)