

Residue Findings of QuPPE-Compounds in Samples of Plant Origin from the German Market in 2018

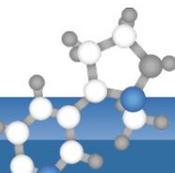
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The aim of this compilation is to give an overview to other labs as to which QuPPE-related compounds are currently encountered in food products of plant origin. This should help in the scope to be monitored. At the CVUA Stuttgart 34 compounds were routinely monitored by the QuPPE method in 2018. Table 1 shows a compilation of the scope.

Table 1: Scope of QuPPE-compounds that were routinely monitored by the CVUA Stuttgart in 2018

Compound	Notes on regulatory status	General remarks
Amitrole		Herbicide
Bialaphos	Not specifically regulated, MRL of 0.01 mg/kg applies	Herbicide (transforms to glufosinate)
Bromide		Metabolite of fumigant methylbromide, but also from soil
Chlorate	Currently new MRLs under discussion	Formerly used as herbicide, but nowadays mainly from irrigation or cleaning with chlorinated water
Chloridazon-desphenyl		Metabolite of chloridazon
Chlormequat		Growth regulator
Cyanuric acid	Non-regulated metabolite	Metabolite of cyromazine. But residues mainly originate from other sources. Cyanamide contained in fertilizers may convert to melamine through trimerization, which can further hydrolyze to cyanuric acid. Trichloroisocyanurate, that is used to retard the loss of chlorine in chlorinated water (e.g. in pools) also converts cyanuric acid.
Cyromazine		Fungicide
Difenzoquat	Not specifically regulated, MRL of 0.01 mg/kg applies	Herbicide
Ethephon		Growth regulator
HEPA	Non-regulated metabolite	Metabolite of ethephon
ETU	Non-regulated degradant	Degradant of ethylen-bis dithiocarbamates
Fosetyl		Fungicide (active component is phosphonic acid)
Phosphonic acid	Regulated with Fosetyl	Fungicide, also metabolite of fosetyl
Glufosinate		Herbicide
MPP (MPPA)		Metabolite of glufosinate
N-Acetyl Glufosinate		Metabolite of glufosinate
Glyphosate		Herbicide



AMPA	Non-regulated metabolite. Planned inclusion in RD of glyphosate	Metabolite of glyphosate
N-Acetyl-Glyphosate	Non-regulated metabolite. Planned inclusion in RD of glyphosate	Metabolite of glyphosate
N-Acetyl-AMPA	Non-regulated metabolite.	Metabolite of glyphosate
Maleic hydrazide		Sprouting inhibitor
Melamine	Non-regulated metabolite	Metabolite of Cyromazine, but residues mainly from other sources.(see cyanuric acid)
Mepiquat		Growth regulator
Mepiquat, 4-Hydroxy	Non-regulated metabolite	Metabolite of Mepiquat, mainly relevant for food of animal origin
Nereistoxine	Non-regulated multisource metabolite	Transformation product of various members of nereistoxin family of pesticides such as bensultap, cartap and thiocyclam
Nicotine*	MRLs set for rose hips, herbs and edible flowers, wild fungi, teas, herbal infusions and spices	Insecticide, contaminant from tobacco
Perchlorate	Regulated as a contaminant	Contaminant, e.g. from fertilizer
Propamocarb		Fungicide
Propamocarb N-desmethyl	Non regulated metabolite	Metabolite of propamocarb
Propamocarb-N-oxide	Non regulated metabolite	Metabolite of propamocarb
PTU	Regulated in infant- and baby food Reg. EC 125/2006 and 141/2006	Metabolite from propineb
Pymetrozine, 6-hydroxymethyl	Non-regulated metabolite	Metabolite of pymetrozine
Trimesium		Counter-ion of glyphosate

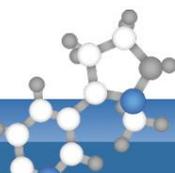
Residue Findings:

In 2018 a total of 2601 samples, mainly fruit and vegetables, but also cereals, pulses, processed goods, tea and others, were analyzed for QuPPE-amenable compounds at the CVUA Stuttgart. 2056 samples (79 %) contained quantifiable residues of one or more of the tested QuPPE compounds. Table 2 shows a compilation of the results.

Cyanuric acid, phosphonic acid, perchlorate, nicotine, chlorate and melamine were found in more than 10 % of the samples.

Bromide, propamocarb, chlormequat, trimesium, propamocarb-N-oxide, chloridazon-desphenyl, HEPA, propamocarb N-desmethyl, mepiquat and ethephon at levels between 1 and 10 % of the samples.

The following compounds were not found in any of the samples: bialaphos, daminozide, difenzoquat, N-acetyl-AMPA, glufosinate, N-acetyl-glufosinate and pymetrozine-6-hydroxymethyl.


Table 2: Residue findings for SRM compounds (CVUA Stuttgart 2018)

Compound	# samples	# pos.	% pos.	Max (mg/kg)	Mean* (mg/kg)	Median* (mg/kg)	# >MRL	% >MRL	RL**
Cyanuric acid	2491	1067	43	6.1	0.055	0.010			0.005
Phosphonic acid	2601	736	28	175	3.6	0.79	15	0.6	0.05
Perchlorate	2601	655	25	2,4	0.047	0.012			0.005
Nicotine*	288	69	24	1.3	0.10	0.023	20	6.9	0.005
Chlorate	2600	572	22	4.2	0.056	0.012	288	11	0.005
Melamine	2600	402	15	5.1	0.12	0.026			0.01
Bromide	2601	87	3.3	97	16.2	12.1			5
Propamocarb	2600	79	3	6.7	0.33	0.039			0.005
Chlormequat	2600	76	2.9	11.1	0.30	0.046	11	0.5	0.005
Trimesium	2600	71	2.7	0.22	0.032	0.021	5	0.2	0.005
Propamocarb-N-oxide	2600	52	2	0.64	0.082	0.031			0.005
Chloridazon-desphenyl	2600	49	1.9	0.13	0.019	0.013			0.005
Ethephon, HEPA	2552	48	1.9	3.5	0.29	0.13			0.02
Propamocarb N-desmethyl	2600	38	1.5	2.3	0.10	0.016			0.005
Mepiquat	2600	32	1.2	0.3	0.031	0.018	1	0.04	0.005
Ethephon	2600	28	1.1	4.5	0.39	0.071	4	0.2	0.01
Maleic hydrazide	2494	21	0.8	10.9	4.4	4.5			0.01
Fosetyl	2601	16	0.6	4.6	0.53	0.083			0.01
ETU	2600	15	0.6	0.2	0.041	0.013			0.02
Glufosinat, MPP	2600	15	0.6	0.38	0.053	0.022	1	0.04	0.01
Cyromazine	2600	12	0.5	0.55	0.079	0.016	2	0.08	0.005
Glyphosate	2600	5	0.2	2.2	0.7	0.28	2	0.08	0.02
Nereistoxine	2600	5	0.2	0.11	0.038	0.022			0.005
Mepiquat, 4-Hydroxy	2600	3	0.1	0.087	0.060	0.084			0.005
AMPA	2600	2	0.08	0.032	0.032	0.032			0.005
Amitrol	2600	1	0.04	0.008					0.005
PTU	2600	1	0.04	0.011					0.005

* Mean and median of positives

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

MRL violations

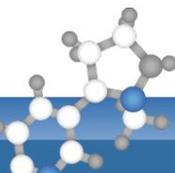
In 61 samples MRLs for different compounds were exceeded (see Table 3) and in additional 288 samples (not shown), the default MRL for chlorate was exceeded.

Table 3: Samples with QuPPE-compounds exceeding existing MRLs (CVUA Stuttgart 2018)

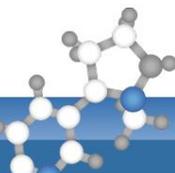
Compound	Commodity	Country of Origin	Conc. (mg/kg)	>MRL	>2xMRL *	Note
Chlormequat	Apple Juice	unknown	0.02	X		
Chlormequat	Avocado	Spain	0.64	X	X	
Chlormequat	Pear	Germany	0.72	X	X	
Chlormequat	Oyster mushrooms	Poland	11.1	X	X	
Chlormequat	Chilli peppers, dried	unknown	0.44	X	X	
Chlormequat	Paprica powder	unknown	0.76	X	X	
Chlormequat	Paprica powder	unknown	0.99	X	X	
Chlormequat	Ginger	China	0.05	X	X	
Chlormequat	Ginger	China	0.19	X	X	
Chlormequat	Red cabbage	unknown	0.03	X	X	

Compound	Commodity	Country of Origin	Conc. (mg/kg)	>MRL	>2xMRL *	Note
Chlormequat	Tomato	Germany	0.22	X	X	
Cyromazine	Ginger	China	0.26	X	X	
Cyromazine	Ginger	unknown	0.55	X	X	
Ethephon	Mango	Egypt	0.054	X		
Ethephon	Pineapple	Dominican Republic	3.4	X		
Ethephon	Pineapple	Ghana	4.5	X	X	
Ethephon	Chili peppers	Turkey	1.1	X	X	
Glufosinate	Lemon	South Africa	0.38	X	X	
Glyphosate	Millet	Ukraine	0.27	X	X	
Glyphosate	Millet	Ukraine	0.28	X	X	
Mepiquat	Mate	unknown	0.3	X	X	
Nicotine	Bean	China	0.013	X		
Nicotine	Mung beans	India	0.012	X		
Nicotine	Mandarine	Spain	0.015	X		
Nicotine	Pear	Italien	0.022	X	X	
Nicotine	Raspberry	Germany	0.012	X		
Nicotine	Wild mushrooms, dried	China	0.15	X		
Nicotine	Wild mushrooms, dried	Thailand	0.11	X		
Nicotine	Oyster mushrooms	Germany	0.023	X	X	
Nicotine	Oyster mushrooms	Germany	0.028	X	X	
Nicotine	White button mushroom	Poland	0.054	X	X	
Nicotine	Borecole	Germany	0.018	X		
Nicotine	Borecole	Germany	0.019	X		
Nicotine	Borecole	Germany	0.022	X	X	
Nicotine	Borecole	Germany	0.058	X	X	
Nicotine	Frisee lettuce	Germany	0.012	X		
Nicotine	Ginger	Peru	0.016	X		
Nicotine	Okra (Ladyfingers)	India	0.031	X	X	
Nicotine	Rucola	Germany	0.013	X		
Nicotine	Spinach	Germany	0.014	X		
Nicotine	Spinach	Italien	0.02	X		
Phosphonic acid	Baby and infant foods	DE-Öko	0.018	X		Based on pear and apple
Phosphonic acid	Baby and infant foods	DE-Öko	0.055	X	X	Based on pear and apple
Phosphonic acid	Baby and infant foods	DE-Öko	0.085	X	X	Based on pear
Phosphonic acid	Baby and infant foods	Producer Germany	0.057	X	X	Based on pear
Phosphonic acid	Blueberry	Germany	7.1	X	X	
Phosphonic acid	Currant	Germany	4.6	X	X	
Phosphonic acid	Gooseberry	Germany	5.1	X	X	
Phosphonic acid	Mirabelle	Germany	2.6	X		
Phosphonic acid	Papaya	Brazil	2.4	X		
Phosphonic acid	Plum	Republic of Moldova	2.3	X		
Phosphonic acid	Plum	Turkey	2.1	X		
Phosphonic acid	Pomegranate	Peru	2.5	X		
Phosphonic acid	Pomegranate	Turkey	6.6	X	X	
Phosphonic acid	Basil	Israel	138	X	X	
Phosphonic acid	Shallots	France	5.5	X	X	
Trimesium	White button mushroom	Germany	0.057	X		
Trimesium	White button mushroom	Poland	0.057	X		
Trimesium	Asparagus	Mexiko	0.054	X		
Trimesium	Asparagus	Mexiko	0.065	X		
Trimesium	Cucumber	Turkey	0.22	X	X	

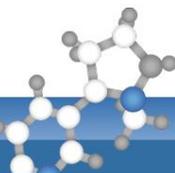
* >2xMRL means that the sample exceeded MRL even after deducting measurement uncertainty of 50%


Table 4: 15 Highest residue findings for the compounds with findings >50

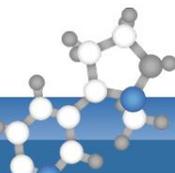
Compound	Country of origin	Commodity	Residue level (mg/kg)
Cyanuric acid	Mexico	Asparagus	6.1
	Mexico	Asparagus	4.7
	Unknown	Food contact material, bamboo cutting board	3.4
	Costa Rica	Pineapple	2.7
	Unknown	Paprika powder	1.5
	Israel	Pomegranate	1.1
	Unknown	Chilli, dried	0.79
	Poland	Oyster mushrooms	0.71
	Poland	Oyster mushrooms	0.68
	Serbia	Stinging nettle seed	0.62
	Costa Rica	Pineapple	0.62
	Costa Rica	Pineapple	0.58
	Germany	Oyster mushrooms	0.54
	China	Mu Err mushrooms, dried	0.52
	Costa Rica	Pineapple	0.51
Phosphonic acid	Germany	Hops	174.6
	Israel	Basil	138
	Unknown	Paprika powder	64.5
	Germany	Parsley	52.7
	Italy	Rucola	48.9
	Germany	Parsley	46.4
	Germany	Head lettuce	43.9
	Germany	Blackberry	35.5
	Germany	Oakleaf lettuce	34.9
	Germany	Parsley	32.9
	Italy	Pear	32.8
	Germany	Beer ingredients	31.4
	Germany	Strawberry	30.3
	Germany	Strawberry	30.1
	Germany	Blackberry	29.5
Perchlorate	Germany	Borecole	2.4
	Spanien	Moringa	2.2
	Chile	Oregano	1.7
	Tansania	Moringa	1.6
	Unknown	German spearmint	1.4
	China	Oolong tea	1.3
	Serbia	Stinging nettle seed	0.86
	Unknown	Rooibos	0.79
	Unknown	Cumin seed	0.59
	Tansania	Euphorbia hirta	0.45
	China	Tea, unfermented	0.39
	Unknown	Kale, frozen	0.38
	Unknown	Peppermint	0.29
	Tansania	Euphorbia hirta	0.28
	Unknown	Rooibos	0.28
Nicotine	Unknown	Food contact material, disposable plate from plant leaves	1.3
	Unknown	Food contact material, disposable plate from plant leaves	1.1
	Unknown	Wild mushrooms, dried	0.82
	Bosnia	Wild mushrooms, dried	0.53
	Unknown	Food contact material, disposable plate from plant leaves	0.46
	Unknown	Food contact material, disposable plate from plant leaves	0.39
	Unknown	Food contact material, disposable plate from plant leaves	0.21
	China	Mu Err mushrooms, dried	0.15
	Unknown	Chamomile	0.12
	Serbia	Stinging nettle seed	0.12
	Unknown	Food contact material, disposable plate from plant leaves	0.11
	Thailand	Mu Err mushrooms, dried	0.11



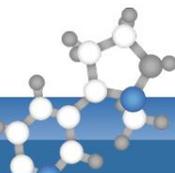
Compound	Country of origin	Commodity	Residue level (mg/kg)
	Unknown	Chamomile	0.11
	Spain	Moringa	0.1
	China	Tea, unfermented	0.094
Chlorate	China	Mu Err mushrooms, dried	4.2
	Unknown	Paprika powder	3.3
	Unknown	Food contact material, disposable plate from plant leaves	2.3
	Peru	Asparagus	2
	Unknown	Spices, sweet pepper	1.2
	Unknown	Bell pepper, frozen	0.74
	Germany	Lamb's lettuce	0.72
	Unknown	Chilli, dried	0.68
	Unknown	German spearmint	0.63
	Thailand	Coriander	0.62
	Israel	Basil	0.44
	Italy	Asparagus	0.32
	Belgium	Aubergine	0.31
	Belgium	Green beans, frozen	0.28
	Belgium	Bell peppers	0.28
Melamine	Germany	Oregano	5.1
	Germany	Shiitake mushroom	4
	Germany	Parsley	3.8
	Germany	Lamb's lettuce	3.4
	Germany	Shiitake mushroom	3.04
	Germany	Celeriac	2
	Spain	Parsley	1.5
	Germany	Parsley	1.3
	Germany	Celeriac	1.1
	Germany	Chives	1.1
	Germany	Lamb's lettuce	1.1
	Germany	Potato	0.67
	China	Mu Err mushrooms, dried	0.58
	Germany	Hops	0.58
	Germany	Rucola	0.53
Bromide	Tanzania	Euphorbia hirta	97
	Thailand	Coriander	48.5
	Spain	Moringa	46.4
	Turkey	Lime-blossom	44.2
	Unknown	Chamomile	41.8
	Tanzania	Euphorbia hirta	41.7
	Thailand	Coriander	36.7
	Unknown	Cumin seed	36.4
	Unknown	Pepper black	35.9
	Unknown	Chamomile	26.5
	Italy	Rucola	25.4
	Italy	Head lettuce	24.6
	Unknown	Rooibos	24
	Germany	Barley grass powder	22.5
	Unknown	Chamomile	20.7
Propamocarb	Spain	Spinach	6.7
	Spain	Spinach	5.6
	Italy	Head lettuce	3.9
	Belgium	Endive	0.795
	Spain	Cucumber	0.76
	Spain	Cucumber	0.71
	Spain	Cucumber	0.7
	Turkey	Cucumber	0.62
	Spain	Cucumber	0.48
	Spain	Cucumber	0.41
	Italy	Spinach	0.41



Compound	Country of origin	Commodity	Residue level (mg/kg)	
Chlormequat	Spain	Cucumber	0.4	
	Spain	Zucchini	0.39	
	Unknown	Spinach, frozen	0.36	
	Spain	Cucumber	0.34	
	Poland	Oyster mushroom	11.1	
	Unknown	Cereal flakes & porridge	1.2	
	Unknown	Paprika powder	0.99	
	Unknown	Paprika powder	0.76	
	Germany	Pear	0.72	
	Spain	Avocado	0.65	
	Germany	Wheat flour	0.52	
	Unknown	Wheat flour	0.49	
	Unknown	Cereal flakes & porridge	0.49	
	Unknown	Spices, chilli	0.44	
	Unknown	Cereal flakes & porridge	0.36	
	China	Mu Err mushrooms, dried	0.27	
	Germany	Tomato	0.22	
China	Ginger	0.19		
Unknown	Wheat flour	0.18		
Propamocarb-N-oxide	Spain	Spinach	0.64	
	Turkey	Cucumber	0.32	
	Spain	Cucumber	0.29	
	Spain	Cucumber	0.28	
	unknown	Kale, frozen	0.25	
	Germany	Cucumber	0.24	
	Spain	Cucumber	0.21	
	Spain	Spinach	0.2	
	Spain	Cucumber	0.2	
	Spain	Cucumber	0.16	
	Spain	Zucchini	0.16	
	Spain	Cucumber	0.15	
	Italy	Head lettuce	0.11	
	Spain	Cucumber	0.1	
	Spain	Zucchini	0.076	
	Trimesium	Turkey	Cucumber	0.22
		unknown	Peppermint	0.17
China		Oolong tea	0.087	
Turkey		Lime-blossom infusion	0.082	
Germany		Barley grass powder	0.069	
Germany		Wheatgrass powder	0.068	
Turkey		Mandarine	0.067	
Mexico		Asparagus	0.065	
Tanzania		Euphorbia hirta	0.058	
Poland		White button mushroom	0.057	
Germany		White button mushroom	0.057	
Mexico		Asparagus	0.054	
unknown		Paprika powder	0.052	
Germany		White button mushroom	0.05	
Republic of Korea		King oyster mushroom	0.048	


Table 5 Highest residue 10 findings for the compounds with findings <50

Compound	Country of origin	Commodity	Residue level (mg/kg)
Chloridazon desphenyl	Germany	Parsley	0.13
	Spain	Romaine lettuce	0.12
	Germany	Rucola	0.055
	unknown	Romaine lettuce	0.048
	Germany	Beetroots	0.031
	Germany	Parsley	0.03
	unknown	Parsley	0.025
	Italy	Chard	0.024
	Germany	Parsley	0.024
	unknown	Kale, frozen	0.022
Ethephon, Hepa	Bosnia	Boletus, dried	3.5
	Germany	Apple Juice Concentrate	2.4
	Germany	Apple Juice Concentrate	1.7
	Turkey	Chili peppers	0.65
	Dom. Republik	Pineapple	0.55
	Germany	Oyster mushroom	0.55
	Ghana	Pineapple	0.46
	Poland	White button mushroom	0.27
	unknown	Chilli, dried	0.24
	China	Shiitake mushroom	0.23
Propamocarb-N-desmethyl	Spain	Spinach	2.3
	Spain	Spinach	0.45
	Italy	Head lettuce	0.27
	Spain	Cucumber	0.099
	Turkey	Cucumber	0.09
	unknown	Kale, frozen	0.09
	unknown	Spinach, frozen	0.087
	Spain	Cucumber	0.066
	Spain	Tomato	0.047
	Spain	Cucumber	0.044
Mepiquat	unknown	Mate	0.3
	unknown	Cereal flakes & porridge	0.081
	unknown	Paprika powder	0.057
	Poland	White button mushroom	0.046
	unknown	Paprika powder	0.04
	Germany	White button mushroom	0.034
	unknown	Spices, sweet pepper	0.032
	Germany	White button mushroom	0.031
	unknown	Green spelt	0.031
	unknown	Green spelt	0.028
Ethephon	Ghana	Pineapple	4.5
	Dom. Republic	Pineapple	3.4
	Turkey	Chili peppers	1.1
	Brazil	Figs	0.3
	South Africa	Grapes	0.22
	South Africa	Grapes	0.16
	Peru	Grapes	0.15
	Panama	Pineapple	0.15
	unknown	Spices, sweet pepper	0.14
	Costa Rica	Pineapple	0.12
Maleic hydrazide	Germany	Potato	10.9
	Germany	Potato	9.2

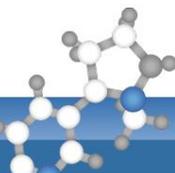


Compound	Country of origin	Commodity	Residue level (mg/kg)
	unknown	Potato	8.8
	Germany	Potato	7.4
	New Zealand	Onion	6.2
	France	Potato	5.6
	France	Potato	5.4
	Germany	Potato	5.3
	France	Shallots	5.3
	unknown	Onion	5.1
Fosetyl	Germany	Hops	4.6
	Germany	Hops	1.4
	Germany	Blackberry	0.72
	Italy	Rucola	0.69
	Italy	Head lettuce	0.52
	Italy	Rucola	0.14
	Germany	Oakleaf lettuce	0.1
	Italy	Grapes	0.097
	Italy	Rucola	0.068
	Germany	Beer ingredients	0.053
ETU	Germany	Parsley	0.2
	Turkey	Wine leaves, prepared in brine	0.14
	Germany	Parsley	0.092
	Germany	Rucola	0.07
	Spain	Cucumber	0.023
	Brazil	Papaya	0.021
	Kenia	Green beans	0.013
	Italy	Radish, small	0.013
	Spain	Zucchini	0.009
	Spain	Cucumber	0.008
Glufosinate MPP	South Africa	Lemon	0.38
	Spain	Strawberry	0.068
	South Africa	Peach	0.062
	Belgium	Kiwi	0.062
	South Africa	Grapes	0.031
	South Africa	Plum	0.031
	Spain	Strawberry	0.023
	Peru	Grapes	0.022
	South Africa	Plum	0.02
	Spain	Strawberry	0.019
Cyromazine	unknown	Ginger	0.55
	China	Ginger	0.26
	Spain	Aubergine	0.026
	Brazil	Melon	0.019
	The Netherlands	Melon	0.018
	Brazil	Melon	0.017
	Turkey	Cucumber	0.015
	Spain	Melon	0.01
	Brazil	Melon	0.01
	Brazil	Melon	0.009
Glyphosate	Canada	Lentil	2.2
	China	Mu Err mushrooms, dried	0.49
	Ukraine	Millet	0.28
	Ukraine	Millet	0.27
	unknown	Bean	0.25
Nereistoxine	Marocco	Tomato	0.11



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EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

Compound	Country of origin	Commodity	Residue level (mg/kg)
	Italy	Tomato	0.032
	unknown	Coriander	0.022
	Spain	Zucchini	0.016
	Turkey	Zucchini	0.009