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Laboratory Methods for MRL Determination:

From Sampling to Results

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Safe Food Alliance / DFA of California

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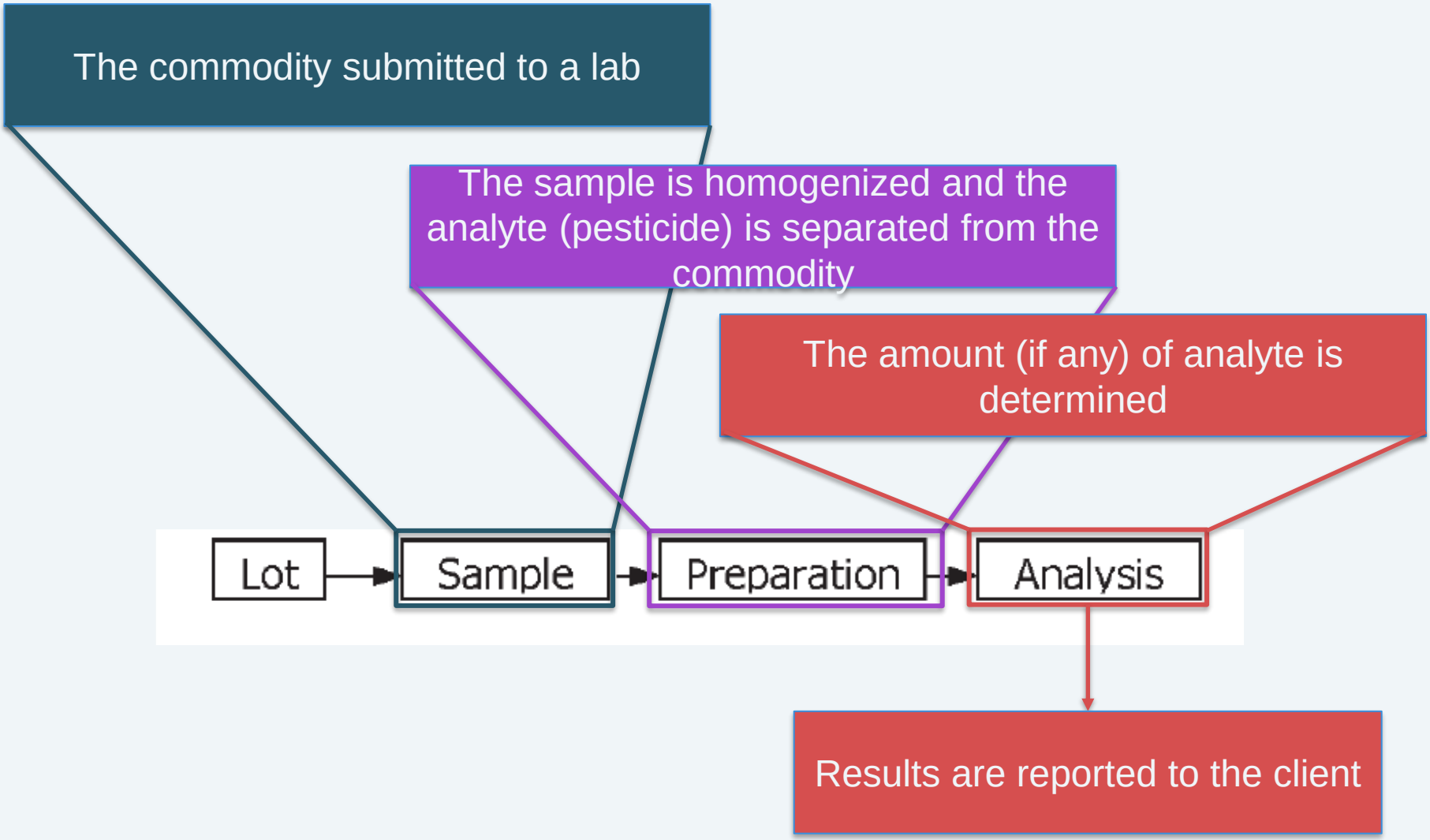
FOOD TESTING: AN OVERVIEW



Whitaker et al.: Journal of AOAC International Vol. 89, No. 4, 2006

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FOOD TESTING: WHERE DOES ERROR COME FROM?

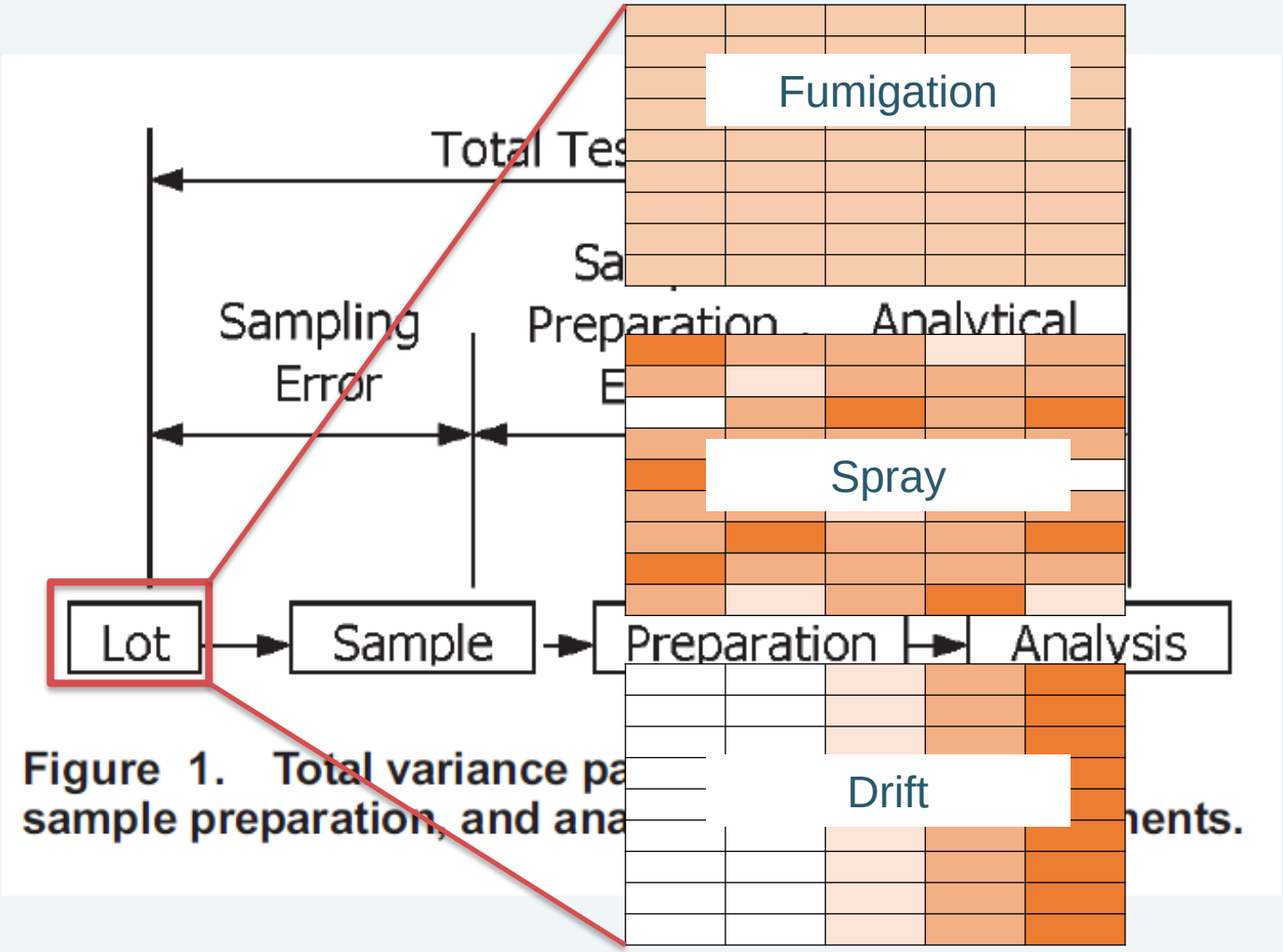

Figure 1. Total variance pa
sample preparation, and ana
ments.

Whitaker et al.: Journal of AOAC International Vol. 89, No. 4, 2006

FOOD TESTING: WHERE DOES ERROR COME FROM?

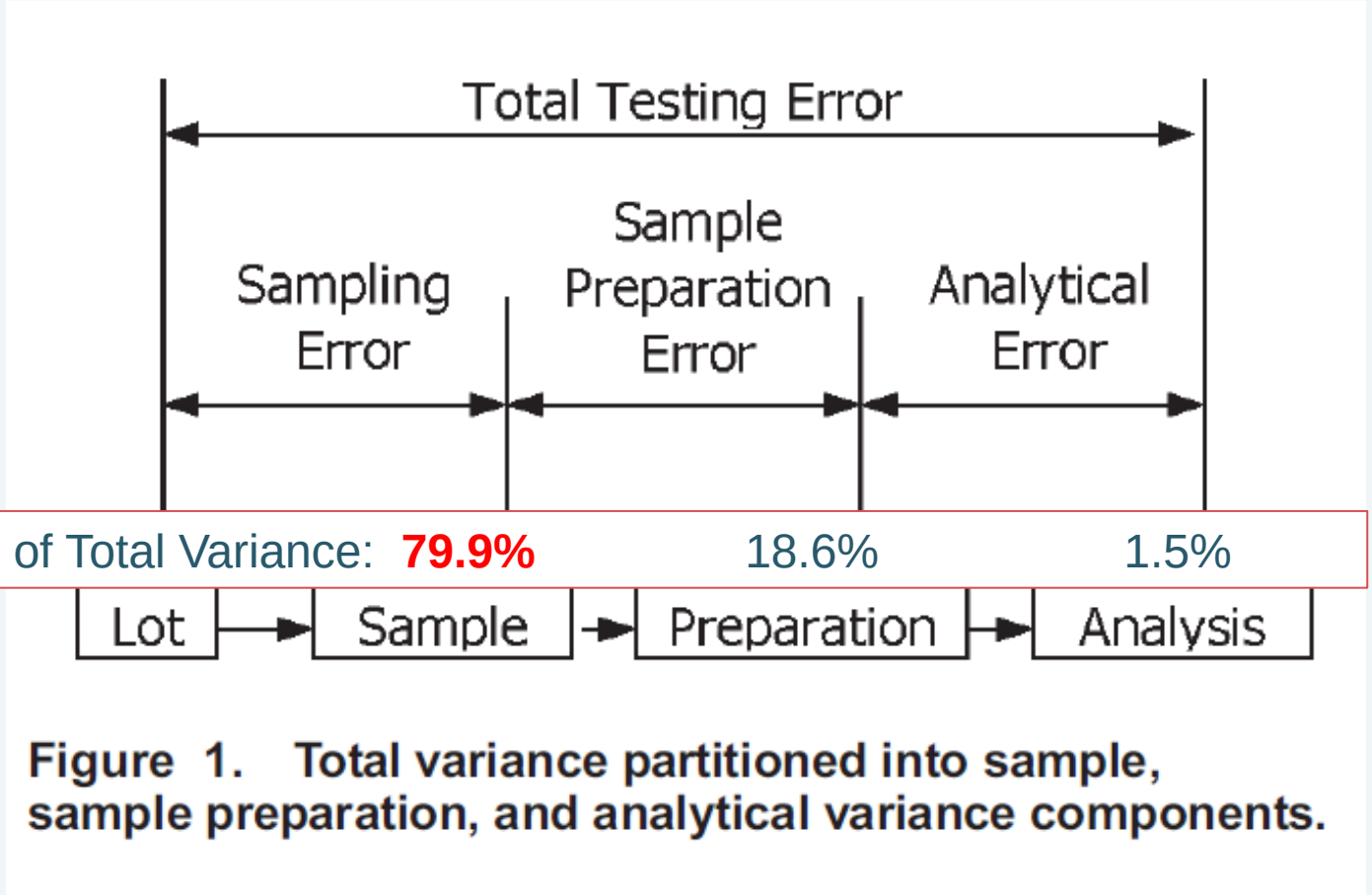

Figure 1. Total variance partitioned into sample, sample preparation, and analytical variance components.

Whitaker et al.: Journal of AOAC International Vol. 89, No. 4, 2006
Whitaker, T.; "PMIL Mycotoxin Webinar: Part 1 Designing Mycotoxin Sampling Plans". YouTube, 18/12/2014, https://www.youtube.com/watch?v=BgQ5bJCQX0s

SAMPLING ADVICE: THINGS TO CONSIDER

Recommended Sample Sizes:

INVESTIGATIONS OPERATIONS MANUAL 2020 — SAMPLE SCHEDULE CHART 3

Commodity classification	Examples	Nature of primary samples to be taken	Minimum sample size and number of units of each laboratory sample
1. PRIMARY FOOD COMMODITIES OF PLANT ORIGIN			
All fresh fruits, All fresh vegetables, Frozen bulk produce (not retail) except dry pulses			
Small sized products units generally < 25 g	Berries, peas, olives	whole units, or packages, or units taken with sampling device	1 kg (2.2 lbs)
Medium units gen			ast
Large si units gen			st 5
Pulses, Cere			
Tree nuts	(except coconuts)		1 kg (2.2 lbs)
	coconuts		5 units
Oilseeds	peanuts		0.5 kg (1.1 lb)
Seeds for beverages and sweets	See CP 7304.004		0.5 kg (1.1 lb)
Herbs *(for dried herbs see section 3 of this Table)*	fresh parsley others, fresh	whole units or units taken with sampling device	0.5 kg (1.1 lb) 0.2 kg (0.5 lb)
Spices	dried	whole units or units taken with sampling device	0.1 kg (0.25 lb)

If you have an FDA detention or similar regulatory issue, contact a lab for directions BEFORE collecting any samples!!!

FDA Investigations Operation Manual – Sampling Ch. 4, 2020

Pesticide regulations generally refer to the “edible portion of the crop. Avoid submitting twigs, leaves, soil and other possible sources of contamination

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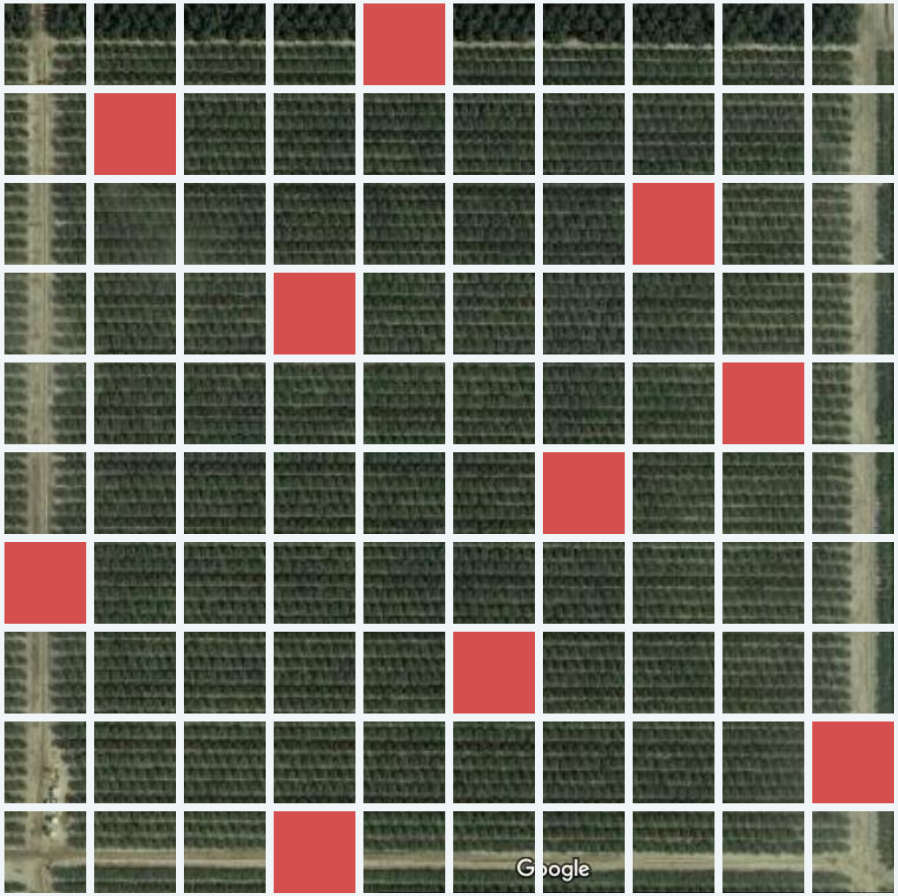
SAMPLING ADVICE: THINGS TO CONSIDER

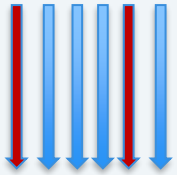
Field Samples:

- Superimpose a 10 x 10 grid
- Select 10 random areas to sample
- Combine into a single composite sample*
  *Do not do this if drift is suspected
- Standard GAP for harvesting and processing



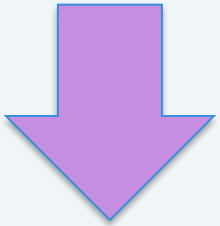
SAMPLING ADVICE: THINGS TO CONSIDER

Grapes



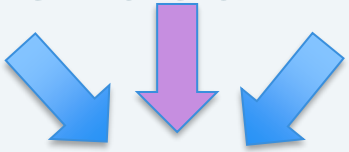
← Many Samples?
Easiest to Remove Violations?

Juice



←Fewer Samples?
Prior to Fermentation / Aging?

Unblended Wines



←Residue Reduction from Processing?

Finished Wines

←What Reg. Agencies might Test?
A lot of Time and Effort Already Invested

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“Hello, do you test for pesticides?”

“So what do you need us to look for?

“...Everything? Pesticides?

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"HELLO! DO YOU TEST FOR PESTICIDES?"

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	SULFUR	35	ALPHA-(PARA-NONYLPHENYL)-OMEGA-HYDROXYPOLY(OXYETHYLENE)*	69	BORIC ACID	102	ORYZALIN	136	MYCLOBUTANIL
2	MINERAL OIL	36	1,3-DICHLOROPROPENE	69	DIETHYLENE GLYCOL*	104	FLUOPYRAM	138	FLONICAMID
3	GLYPHOSATE	37	DIMETHOATE	71	CALCIUM HYPOCHLORITE	104	DISODIUM OCTABORATE TETRAHYDRATE	139	INDAZIFLAM
4	METHYLATED SOYBEAN OIL*	38	POTASSIUM N-METHYLDITHIOCARBAMATE	71	POLYACRYLAMIDE POLYMER*	106	COPPER ETHANOLAMINE COMPLEXES, MIXED	140	COPPER AMMONIUM CARBONATE
5	OXYFLUORFEN	39	CHLOROPICRIN	73	CAPTAN	106	PROPICONAZOLE	141	PYRAFLUFEN-ETHYL
6	COPPER HYDROXIDE	39	PHOSPHORIC ACID	74	PROPYLENE GLYCOL	108	GLYCEROL*	141	BACILLUS AMYLOLIQUEFACIENS STRAIN D747
7	AMMONIUM SULFATE	41	PERMETHRIN	75	CHLORINE	109	(S)-CYPERMETHRIN	143	ACETAMIPRID
8	PENDIMETHALIN	42	ABAMECTIN	76	[illegible]	109	BENSULIDE	143	PROPARGITE
9	PARAQUAT DICHLORIDE	42	CYPRODINIL	[illegible]	[illegible]	[illegible]	[illegible]BROMIDE	145	ALPHA-PINENE BETA-PINENE COPOLYMER*
10	PETROLEUM OIL, UNCLASSIFIED	44	UREA DIHYDROGEN SULFATE	[illegible]	[illegible]	[illegible]	[illegible]COHOL*	146	TALL OIL*
11	IMIDACLOPRID	44	CALCIUM HYDROXIDE	[illegible]	[illegible]	[illegible]	[illegible]DIOXIDE	147	CALCIUM CARBONATE*
11	LECITHIN*	46	KAOLIN	80	POLYALKENE [illegible] HEPTAMETHYL TRISILOXANE	114	TEBUCONAZOLE	148	ALPHA-TRIDECYL-OMEGA-HYDROXYPOLY(OXYETHANOL) PHOSPHATE
13	GLUFOSINATE-AMMONIUM	47	[illegible]	81	SPIROTETRAMAT	114	ALKYL(42%C12, 26%C18, 15%C14, 8%C16, 5%C10, 4%C8)1,3-PROPYLENEDIAMINE	148	FATTY ACIDS, METHYL ESTERS
14	MANCOZEB	47	CITRIC ACID	82	POTASSIUM BICARBONATE	116	FIPRONIL	150	CHLORTHAL-DIMETHYL
14	METHOXYFENOZIDE	47	HYDROTREATED PARAFFINIC SOLVENT	82	AMMONIUM NITRATE*	117	ESFENVALERATE	151	POTASH SOAP
16	BIFENTHRIN	50	SULFURYL FLUORIDE	84	ALKYL (C8,C10) POLYGLUCOSIDE*	118	FLUBENDIAMIDE	152	FLUXAPYROXAD
17	OLEIC ACID, METHYL ESTER*	50	N,N-BIS-(2-OMEGA-HYDROXYPOLY(OXYETHYLENE)ETHYL)ALKYLAMINE, ALKYL DERIVED FROM TALLOW FATTY ACIDS	85	TRIFLURALIN	119	FOSETYL-AL	152	SODIUM CHLORATE
18	DIMETHYLPOLYSILOXANE*	52	METHYL BROMIDE	86	CARFENTRAZONE-ETHYL	119	RIMSULFURON	154	CAPRYLIC ACID
19	FATTY ACIDS, C16-C18 AND C18-UNSATURATED, METHYL ESTERS	53	ALPHA-ALKYL(C9-C11)-OMEGA-HYDROXYPOLY(OXYETHYLENE)*	87	PROPYLENE OXIDE	121	POTASSIUM HYDROXIDE*	155	POLYETHYLENE GLYCOL MONO(3-(TETRAMETHYL-1-(TRIMETHYLSILOXY)DISILOXANYL)PROPYL)ETHER
20	CHLORPYRIFOS	54	METHOMYL	88	2,4-D, DIMETHYLAMINE SALT	121	SODIUM CARBONATE PEROXYHYDRATE	156	ALPHA-2,6,8-TRIMETHYL-4-NONYLOXY-OMEGA-HYDROXYPOLY(OXYETHYLENE)*
21	CHLOROTHALONIL	55	LAMBDA-CYHALOTHRIN	88	SODIUM POLYACRYLATE*	123	QUINOXYFEN	157	BETA-CYFLUTHRIN
21	TALL OIL FATTY ACIDS*	56	ALPHA-(PARA-NONYLPHENYL)-OMEGA-HYDROXYPOLY(OXYETHYLENE)*	90	COPPER OXYCHLORIDE	124	[illegible]	[illegible]	[illegible]TILLATES, AROMATIC
23	ALPHA-(PARA-NONYLPHENYL)-OMEGA-HYDROXYPOLY(OXYETHYLENE), PHOSPHATE ESTER	57	FATTY ACIDS, MIXED*	91	MALATHION	[illegible]	[illegible]	[illegible]	[illegible]
24	POLYETHYLENE GLYCOL*	57	PETROLEUM DISTILLATES, REFINED	91	SAFLUFENACIL	[illegible]	[illegible]	[illegible]	[illegible]
25	PROPANIL	59	ALPHA-UNDECYL-OMEGA-HYDROXYPOLY(OXYETHYLENE)*	93	CRYOLITE	[illegible]	[illegible]	[illegible]	[illegible]
26	4-NONYLPHENOL, FORMALDEHYDE RESIN, PROPOXYLATED	60	COPPER SULFATE (PENTAHYDRATE)	93	TRIFLOXYSTROBIN	128	[illegible]	[illegible]	[illegible]
27	COPPER SULFATE (BASIC)	[illegible]	[illegible]	95	COPPER OXIDE (OUS)	129	METCONAZOLE	[illegible]	[illegible]
28	AZOXYSTROBIN	[illegible]	[illegible]	95	POLYETHER MODIFIED POLYSILOXANE*	130	AMMONIUM PROPIONATE*	[illegible]	[illegible]
29	PETROLEUM OIL, PARAFFIN BASED	[illegible]	[illegible]	97	BENZOIC ACID*	130	METHYLATED FATTY ACIDS FROM CANOLA OIL*	[illegible]	[illegible]
30	BOSCALID	[illegible]	[illegible]	98	S-METOLACHLOR	132	GIBBERELLINS	[illegible]	[illegible]
30	2-(3-HYDROXYPROPYL)-HEPTA-METHYL TRISILOXANE, ETHOXYLATED, ACETATE	65	PYRACLOSTROBIN	98	DIMETHYL ALKYL TERTIARY AMINES*	132	ALPHA-ALKYL(C9-C11)-OMEGA-HYDROXYPOLY(OXYETHYLENE)	[illegible]	[illegible]
32	ETHEPHON	66	ZIRAM	100	NALED	134	CORN STEEP LIQUOR*	[illegible]	[illegible]
33	VEGETABLE OIL*	67	SPINETORAM	100	MODIFIED PHTHALIC GLYCEROL ALKYD RESIN*	135	POLYOXYETHYLENE POLYOXYPROPYLENE*	∞	PUTRESCENT WHOLE EGG SOLIDS
34	IPRODIONE	67	THIOBENCARB	102	ETOXAZOLE	136	CARBARYL		

Citric Acid

Vegetable Oil

Putrescent Whole Egg Solids

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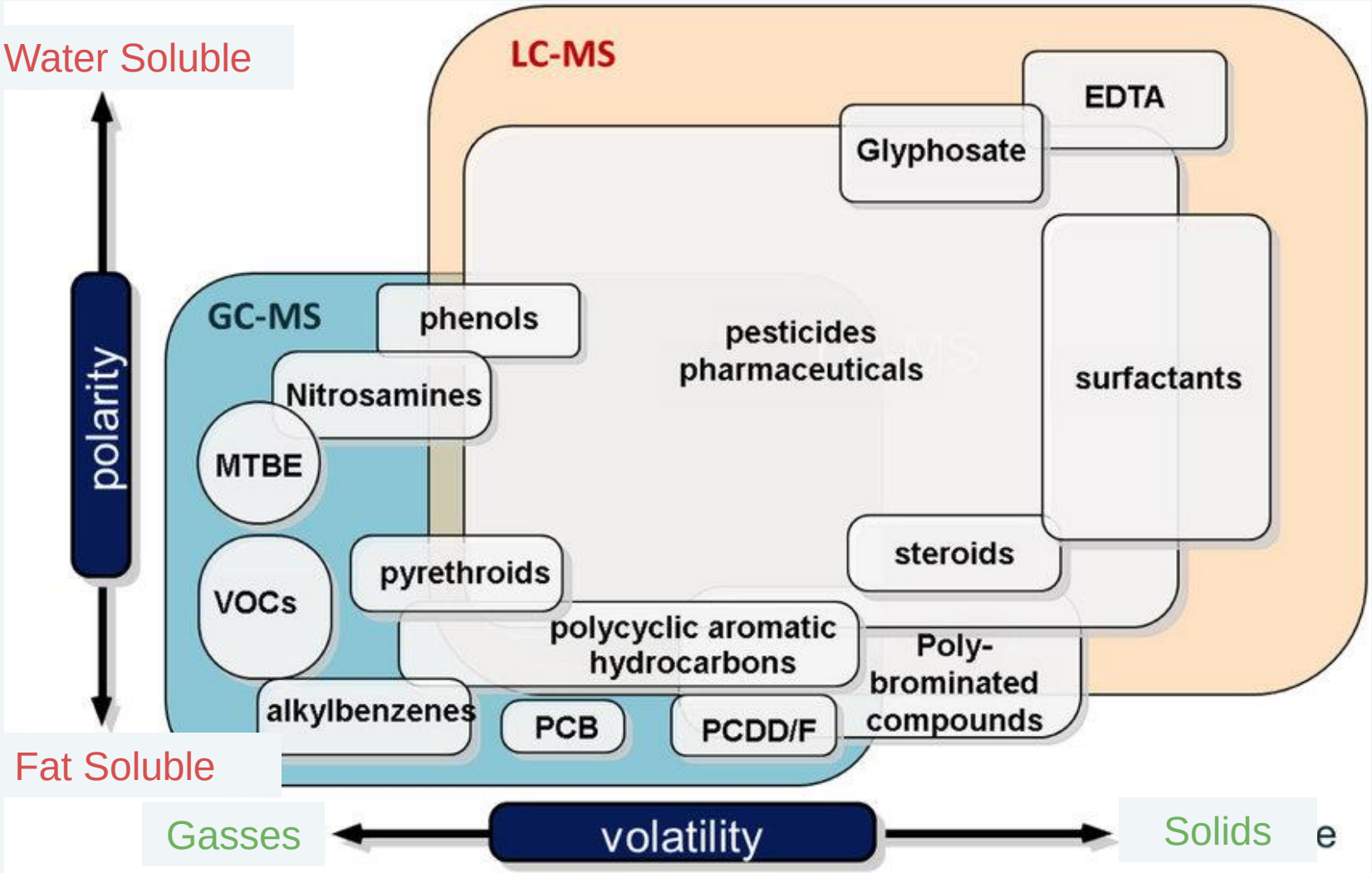
THE "TOP" CA PESTICIDES

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

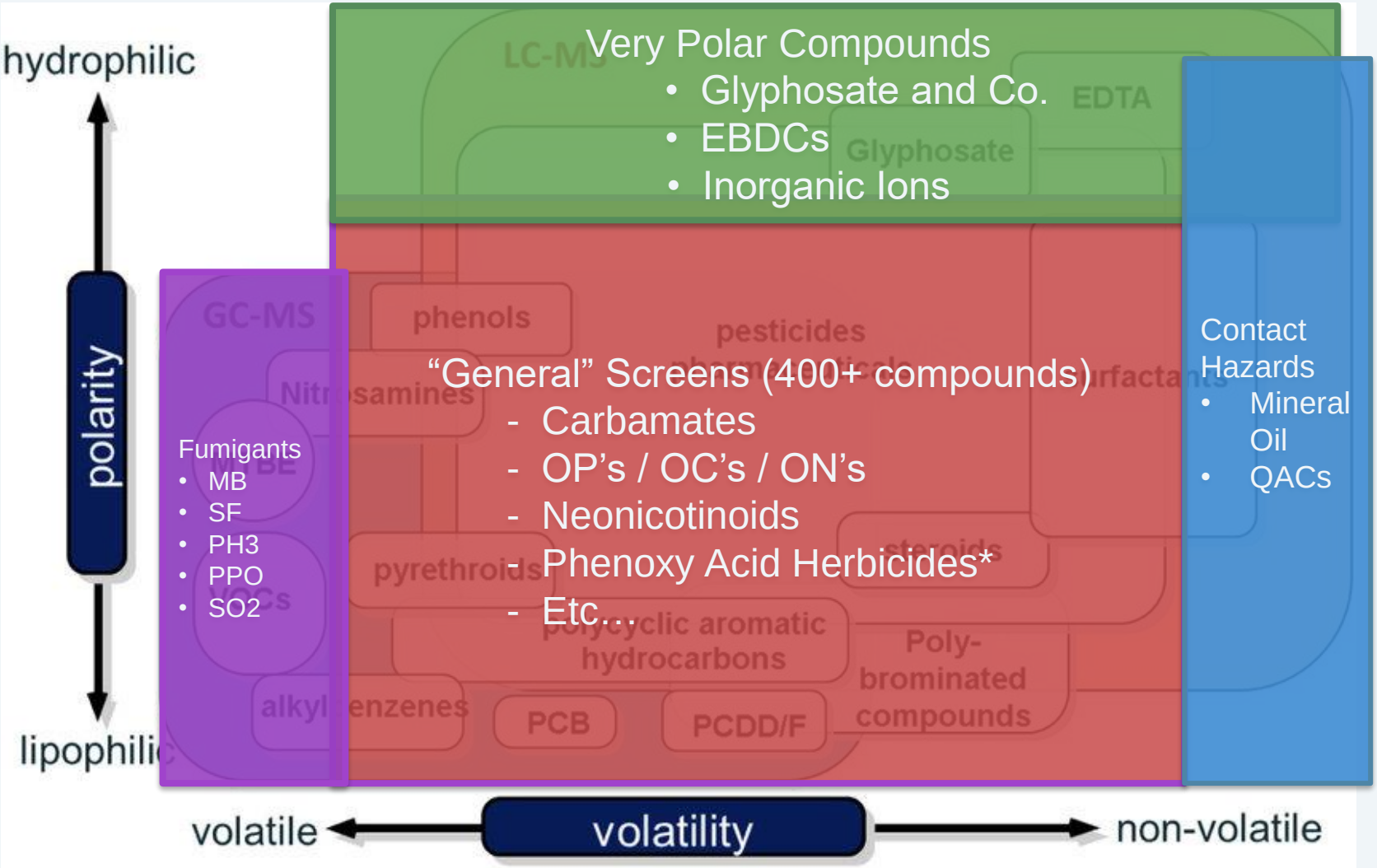
RESIDUE ANALYSIS: THE (2-D) UNIVERSE OF CHEMICALS

(Almost) All pesticide residue testing is done using GC-MS and / or LC-MS



https://www.researchgate.net/figure/Domains-of-GC-MS-and-LC-MS-techniques-in-the-two-dimensions-of-hydrophobicity-and_fig10_290221006

RESIDUE ANALYSIS: THE (2-D) UNIVERSE OF CHEMICALS



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Qu.E.Ch.E.R.S.:
Quick Easy Cheap Effective Rugged and Safe

- Extract with water and acetonitrile
- Add salts, buffers, etc... to separate water and ACN and drive **most** compounds into the organic phase
- Clean-up ACN layer with various combinations of salts (water) and sorbents (fats, pigments, etc...)
- Analyze (GC-MS and / or LC-MSMS)

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THE "TOP" CA PESTICIDES

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

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THE "TOP" CA PESTICIDES: "GENERAL" SCREENS

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3							DIFENOCONAZOLE
4	PARA						EPTC
5							THIAMETHOXAM
6	GLUF						DIURON
7							METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

- Phenoxy Acid Herbicides, like 2,4-D, can be analyzed with the same method as the general screen, but need a slightly different clean-up step

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THE "TOP" CA PESTICIDES: POLAR COMPOUNDS

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
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21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

THE "TOP" CA PESTICIDES: POLAR COMPOUNDS

Quick Method for the Analysis of numerous Highly Polar Pesticides in Foods of Plant Origin via LC-MS/MS involving Simultaneous Extraction with Methanol (QuPPe-Method)

- **Version 9.2** (October 2016, Document History, see page **65**)

Authors: M. Anastassiades; D. I. Kolberg; A. Benkenstein; E. Eichhorn; S. Zechmann; D. Mack; C. Wildgrube; I. Sigalov; D. Dörk; A. Barth

Method 1.1 "Glyphosate & Co. AS 11"

Method 1.2 "Glyphosate & Co. AS 11-HC"

Method 1.3 "Glyphosate & Co. Hypercarb"

Method 1.4 "PerChloPhos"

Method 2 "Fosetyl and Maleic Hydrazide"

Method 3 "Amitrole & Co"

Method 4.1 "Quats & Co Obelisc R"

Method 4.2 "Quats & Co BEH Amide"

Method 5 "Quats & Co. MonoChrom MS"

Method 6 "Streptomycin and Kasugamycin"

Method 7 "Morpholine, Diethanolamine and Triethanolamine"

Method 8 "Triazole derivative metabolites (TDMs)"

- Extract with acidified water / methanol
- Centrifuge, filter and analyze (LC-MSMS)

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THE "TOP" CA PESTICIDES: POLAR COMPOUNDS

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

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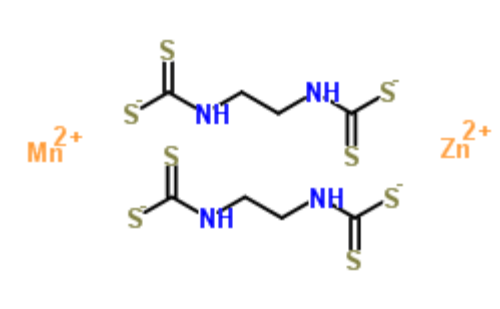
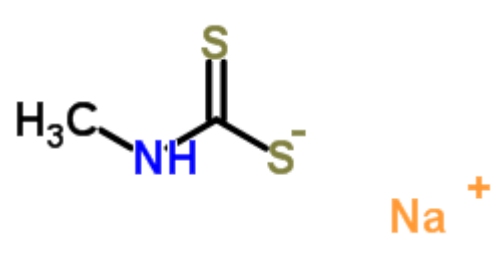
THE "TOP" CA PESTICIDES: EBDC COMPOUNDS

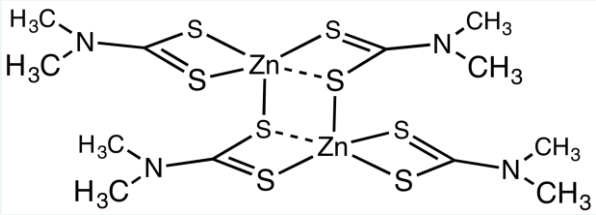
Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

THE "TOP" CA PESTICIDES: EBDC COMPOUNDS

EBDC = Ethylene BisDithioCarbamates







EBDCs are analyzed by acid conversion to carbon disulfide (CS_2) and headspace analysis

0.05 ppm

European Union

Commodity Note

MRL applies to whole product after removal of shell. Other products to which the EU Almonds MRL applies are Apricot kernels, Bitter almonds, Canarium nuts/galip nuts, and Pili nuts as designated in Annex I to Regulation (EC) No 396/2005.

Residue Definition

Dithiocarbamates (dithiocarbamates expressed as CS2, including maneb, mancozeb, metiram, propineb, thiram and ziram)

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THE "TOP" CA PESTICIDES: EBDC COMPOUNDS

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

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THE "TOP" CA PESTICIDES: VOLATILES

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

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THE "TOP" CA PESTICIDES: VOLATILES

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
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12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
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14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
19	POTASSIUM N-METHYLDITHIOCARBAMATE	38	SPIROTETRAMAT	56	FLUBENDIAMIDE		
20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

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THE "TOP" CA PESTICIDES: "GENERAL" SCREENS

Rank	Compound	Rank	Compound	Rank	Compound	Rank	Compound
1	GLYPHOSATE	22	ABAMECTIN	42	2,4-D, DIMETHYLAMINE SALT	59	RIMSULFURON
2	OXYFLUORFEN	22	CYPRODINIL	43	MALATHION	60	QUINOXYFEN
3	PENDIMETHALIN	23	METAM-SODIUM	43	SAFLUFENACIL	61	DIFENOCONAZOLE
4	PARAQUAT DICHLORIDE	24	SULFURYL FLUORIDE	44	TRIFLOXYSTROBIN	61	EPTC
5	IMIDACLOPRID	25	METHYL BROMIDE	45	S-METOLACHLOR	62	THIAMETHOXAM
6	GLUFOSINATE-AMMONIUM	26	METHOMYL	46	NALED	63	DIURON
7	MANCOZEB	27	LAMBDA-CYHALOTHRIN	47	ETOXAZOLE	64	METCONAZOLE
8	METHOXYFENOZIDE	28	CHLORANTRANILIPROLE	47	ORYZALIN	65	CARBARYL
9	BIFENTHRIN	29	POTASSIUM PHOSPHITE	48	FLUOPYRAM	65	MYCLOBUTANIL
10	CHLORPYRIFOS	30	PYRACLOSTROBIN	49	COPPER ETHANOLAMINE COMPLEXES	66	FLONICAMID
11	CHLOROTHALONIL	31	ZIRAM	49	PROPICONAZOLE	67	INDAZIFLAM
12	PROPANIL	32	SPINETORAM	50	(S)-CYPERMETHRIN	68	PYRAFLUFEN-ETHYL
13	AZOXYSTROBIN	32	THIOBENCARB	50	BENSULIDE	69	ACETAMIPRID
14	BOSCALID	33	CALCIUM HYPOCHLORITE	51	SODIUM BROMIDE	69	PROPARGITE
15	ETHEPHON	34	CAPTAN	52	SULFUR DIOXIDE	70	CHLORTHAL-DIMETHYL
16	IPRODIONE	35	CHLORINE	53	TEBUCONAZOLE	71	FLUXAPYROXAD
17	1,3-DICHLOROPROPENE	36	SODIUM HYPOCHLORITE	54	FIPRONIL	71	SODIUM CHLORATE
18	DIMETHOATE	37	HYDROGEN CYANAMIDE	55	ESFENVALERATE	72	BETA-CYFLUTHRIN
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20	CHLOROPICRIN	39	TRIFLURALIN	57	FOSETYL-AL		
20	PHOSPHORIC ACID	40	CARFENTRAZONE-ETHYL	57	RIMSULFURON		
21	PERMETHRIN	41	PROPYLENE OXIDE	58	QUINOXYFEN		

- General Screen
- Polar Screen
- EBDC Screen
- Volatiles
- Inorganics

HOW DID I GET THIS RESIDUE???

- Mistakes
  - Wrong bottle
  - Sprayer drove too fast or too slowly
  - Equipment malfunction
- Drift
  - Neighboring Fields
  - Other land use: right-of-way's, golf course, residential
  - Animal intrusion
- Other (breakdown, reaction, etc…)
- Laboratory error

THANK YOU!

Wiley Hall
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wileyh@safefoodalliance.com

QUESTIONS?