

*Article*

# **False positive identification of pesticides in food using the European standard method and LC-MS/MS determination: examples and solutions from routine applications**

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**Supplementary Table S1:** Screening detection Limits (SDLs) established for 415 pesticides in olive oil under screening analysis.

Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)
1	Acephate	5	38	Buprofezin	5	75	Cyanazine	5
2	Acetamiprid	5	39	Butachlor	20	76	Cyanofenphos	5
3	Acetochlor	10	40	Butafenacil	5	77	Cyanophos	5
4	Acibenzolar-S-methyl	10	41	Butocarboxim	5	78	Cyazofamid	5
5	Alachlor	20	42	Butocarboxim-sulfoxide	5	79	Cybutrine	5
6	Aldicarb	5	43	Butoxycarboxim	5	80	Cyflufenamid	5
7	Aldicarb-sulfone	5	44	Buturon	5	81	Cymiazole	5
8	Aldicarb-sulfoxide	5	45	Cadusafos	50	82	Cymoxanil	5
9	Ametoctradin	5	46	Carbaryl	5	83	Cyproconazole	5
10	Ametryn	5	47	Carbendazim	5	84	Cyprodinil	5
11	Amidosulfuron	5	48	Carbetamide	5	85	Cyromazine	5
12	Aminocarb	5	49	Carbofuran	5	86	Demeton-S	10
13	Amitraz	5	50	Carboxin	5	87	Demeton-S-methyl	10
14	Anilofos	5	51	Carfentrazon	5	88	Demeton-S-methylsulphone	5
15	Atrazine	5	52	Carfentrazone-ethyl	5	89	Demeton-S-sulfone	5
16	Atrazine-desethyl	5	53	Chlorantranilprole	5	90	Demeton-S-sulfoxide	5
17	Atrazine-desisopropyl	5	54	Chlorbromuron	5	91	Desmedipham	5
18	Azaconazole	5	55	Chlorbufam	50	92	Desmetryn	5
19	Azamethiphos	5	56	Chlorfenvinphos	5	93	Diallát	50
20	Azinphos-ethyl	5	57	Chlorfluazuron	10	94	Diazinon	5
21	Azinphos-methyl	5	58	Chloridazon	5	95	Dichlorvos	10
22	Aziprotryne	5	59	Chlorotoluron	5	96	Diclobutrazol	5
23	Azoxystrobin	5	60	Chloroxuron	5	97	Dicrotophos	5
24	Benalaxyl	5	61	Chlorpyrifos	5	98	Diethofencarb	5
25	Bendiocarb	5	62	Chlorpyrifos-methyl	20	99	Difenoconazole	5
26	Benflutamid	5	63	Chlorsulfuron	5	100	Difenoxyuron	5
27	Bensulfuron-metil	5	64	Chlorthiophos	5	101	Diiflubenzuron	5
28	Benthiavalicarb-isopropyl	5	65	Chromafenozide	5	102	Diiflufenican	5
29	Benzolyp-prop-ethyl	5	66	Cinosulfuron	5	103	Dimefox	5
30	Benzoximate	5	67	Cirimidine	5	104	Dimefuron	5
31	Benzthiazuron	5	68	Clethodim	5	105	Dimethachlor	5
32	Bifenazate	5	69	Clofentezine	5	106	Dimethenamid	5
33	Bitertanol	5	70	Clomazone	5	107	Dimethirimol	5
34	Boscalid	5	71	Cloquintocet-mexyl	5	108	Dimethoate	5
35	Bromacil	5	72	Clothianidin	5	109	Dimethomorph	5
36	Bromucanazole	10	73	Coumaphos	5	110	Dimoxystrobin	5
37	Bupirimate	5	74	Crufomat	5	111	Diniconazole	10

Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)
112	Dinotefuran	5	149	Fenhexamid	5	186	Flutolanil	5
113	Dioxacarb	5	150	Fenitrothion	50	187	Flutriafol	5
114	Diphenamid	5	151	Fenobucarb	5	188	Fluxapyroxad	5
115	Dipropetryn	5	152	Fenoxaprop-P-ethyl	5	189	Fomesafen	25
116	Disulfoton	50	153	Fenoxycarb	5	190	Fonofos	10
117	Disulfoton-sulfone	25	154	Fenpropimorph	5	191	Foramsulfuron	5
118	Disulfoton-sulfoxide	25	155	Fenpyroximate	5	192	Forchlorfenuron	5
119	Diuron	5	156	Fensulfothion	5	193	Formetanate HCl	5
120	DMST	10	157	Fensulfothion-oxon	5	194	Fosthiazat	5
121	Dodemorph	5	158	Fensulfothion-oxon-sulfone	5	195	Fuberidazole	5
122	Dodine	10	159	Fensulfothion-sulfone	5	196	Furalaxyl	5
123	Doramectin	10	160	Fenthion	10	197	Furathiocarb	5
124	Emamectin-benzoate b1a	5	161	Fenthion-oxon-sulfone	5	198	Halofenozide	5
125	Emamectin-benzoate b1b	25	162	Fenthion-oxon-sulfoxide	5	199	Haloxifop	10
126	Epoxiconazole	5	163	Fenthion-sulfone	5	200	Haloxifop-2-ethoxyethyl	5
127	Etaconazole	5	164	Fenthion-sulfoxid	5	201	Haloxifop-P-methyl	5
128	Ethidimuron	5	165	Fenuron	5	202	Heptenophos	5
129	Ethiofencarb	5	166	Fipronil	25	203	Hexaconazole	5
130	Ethiofencarb-sulfone	5	167	Flamprop-isopropyl	5	204	Hexaflumuron	25
131	Ethiofencarb-sulfoxide	5	168	Flamprop-methyl	5	205	Hexazonon(Velpar)	5
132	Ethion	5	169	Flazasulfuron	5	206	Hexythiazox	5
133	Ethiprole	5	170	Flonicamid	5	207	Hydramethylnon	5
134	Ethirimol	5	171	Florasulam	5	208	Hydroxycarbofuran	5
135	Ethofumesate	5	172	Fluazifop	5	209	Imazalil	5
136	Ethoprophos	5	173	Fluazuron	5	210	Imazaquin	5
137	Ethoxyquin	5	174	Flubendiamide	5	211	Imidacloprid	5
138	Etoazole	5	175	Fludioxinil	25	212	Indoxacarb	5
139	Etrimfos	5	176	Flufenacet	5	213	Ipconazole	5
140	Famoxadone	25	177	Flufenoxuron	5	214	Iprobenfos	5
141	Fenamidone	5	178	Fluometuron	5	215	Iprodione	50
142	Fenamiphos	5	179	Fluopicolide	5	216	Iprovalicarb	5
143	Fenamiphos-sulfone	5	180	Fluopyram	5	217	Isocarbamide	5
144	Fenamiphos-sulfoxide	5	181	Fluoxastrobin	5	218	Isofenphos	5
145	Fenarimol	10	182	Fluquinconazole	5	219	Isofenphos-methyl	25
146	Fenazaquin	5	183	Fluridone	5	220	Isoprocab	5
147	Fenbuconazole	5	184	Flusilazole	5	221	Isoprothiolane	5
148	Fenfuram	5	185	Fluthiacet-methyl	5	222	Isoproturon	5

Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)
223	Isoxaflutole	5	262	Mevinphos	5	301	Phoxim	5
224	Isoxathion	5	263	Mexacarbate	5	302	Picoxystrobin	5
225	Ivermectin	10	264	Molinate	10	303	Pinoxaden	5
226	izoxaben	5	265	Monocrotophos	5	304	Piperonyl-butoxide	5
227	Kresoxim-methyl	20	266	Monolinuron	5	305	Piperophos	5
228	Linuron	5	267	Monuron	5	306	Pirimicarb	5
229	Lufenuron	20	268	Moxidectin	10	307	Pirimicarb-desmethyl	5
230	Malaoxon	5	269	Myclobutanil	5	308	Pirimiphos-ethyl	5
231	Malathion	5	270	Napropamide	5	309	Pirimiphos-methyl	5
232	Mandipropamid	5	271	Neburon	5	310	Pirimisulfuron-metil	5
233	Mecarbam	5	272	Nitenpyram	5	311	Prochloraz	5
234	Mefenacet	5	273	Nitralin	10	312	Prochloraz-desimidazole-amino	5
235	Mefenpyr-diethyl	10	274	Norflurazon	5	313	Profluralin	20
236	Mepanipyrim	5	275	Nuarimol	10	314	Promecarb	5
237	Mephosfolan	5	276	Omethoate	5	315	Prometon	5
238	Mepronil	5	277	Oxadiazon	10	316	Prometryne	5
239	Mesotrione	5	278	Oxadixyl	5	317	Propachlor	5
240	Metaflumizone	5	279	Oxamyl	5	318	Propamocarb	5
241	Metalaxyl	5	280	Oxamyl-oxime	5	319	Propanil	5
242	Metamitron	5	281	Oxycarboxin	10	320	Propargite	5
243	Metazachlor	5	282	Oxydemeton-methyl	5	321	Propetamphos	5
244	Metconazole	5	283	Paclobutrazol	5	322	Propham	50
245	Methabenzthiazuron	5	284	Paraoxon	5	323	Propiconazole1	5
246	Methacrifos	20	285	Paraoxon-ethyl	5	324	Propoxur	5
247	Methamidophos	5	286	Paraoxon-methyl	5	325	Propyzamide	5
248	Methidathion	5	287	Penconazole	5	326	Proquinazid	5
249	Methiocarb	5	288	Pencycuron	5	327	Prosulfocarb	5
250	Methiocarb-sulfone	5	289	Pendimethalin	20	328	Prosulfuron	5
251	Methomyl	10	290	Penthiopyrad	5	329	Prothioconazole	50
252	Methoprotryne	5	291	Pethoxamid	5	330	Prothiofos	20
253	Methoxyfenozide	5	292	Phenmedipham	5	331	Pymetrozine	5
254	Metobromuron	5	293	Phenothrin	20	332	Pyracarbolid	5
255	Metolachlor	5	294	Phenthoate	5	333	Pyraclostrobin	5
256	Metolcarb	10	295	Phenylthiourea	5	334	Pyraflufen-ethyl	5
257	Metosulam	5	296	Phorate-sulfone	5	335	Pyrazophos	5
258	Metoxuron	5	297	Phorate-sulfoxide	5	336	Pyrethrin	10
259	Metrafenon	10	298	Phosalone	5	337	Pyridaben	5
260	Metribuzin	5	299	Phosmet	5	338	Pyridafol	5

261	Metsulfuron-methyl	5	300	Phosphamidon	5	339	Pyridaphenthion	5
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Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)	Number	Compound	SDL (ng/g)
340	Pyrifenoxy	5	366	Sulfosulfuron	5	392	Thiofanox-sulfoxide	20
341	Pyrimethanil	5	367	Sulfotep (Tetraethyldithiopyrophosphate)	20	393	Thionazin(zinophos)	50
342	Pyriproxyfen	5	368	Tau-fluvalinate	5	394	Thiophanate	5
343	Pyroquilon	5	369	Tebuconazole	5	395	Thiophanate-methyl	5
344	Quinalphos	5	370	Tebufenozide	5	396	Tolclofos-methyl	20
345	Quinoclamine	5	371	Tebufenpyrad	5	397	Tolfenpyrad	5
346	Quinoxifen	5	372	Tebutam	5	398	Tralkoxydim	5
347	Quizalofop	10	373	Tebuthiuron	5	399	Triadimefon	5
348	Resmethrin	10	374	Teflubenzuron	50	400	Triadimenol	10
349	Rimsulfuron	10	375	Tembotrion	5	401	Triallate	50
350	Rotenone	5	376	Temephos	5	402	Triasulfuron	5
351	Sebuthylazin	5	377	Terbufos-sulfone	5	403	Triazophos	5
352	Secbumeton	5	378	Terbufos-sulfoxide	5	404	Trichlorfon	5
353	Silthiofam	5	379	Terbumeton	5	405	Tricyclazole	5
354	Simazine	5	380	Terbuthylazine-desethyl	5	406	Trietazin	5
355	Simetryn	5	381	Terbutryn	5	407	Trifloxystrobin	5
356	Spinetoram	5	382	Tetrachlorvinphos	5	408	Triflumizole	5
357	Spinosad (Spinosyn A)	5	383	Tetraconazole	5	409	Triflumuron	5
358	Spinosad (Spinosyn D)	5	384	Tetrametrin	20	410	Triflusulfuron-methyl	5
359	Spirodiclofen	10	385	Thiabendazole	5	411	Trimethylphenyl-methylcarbamate	5
360	Spiromesifen	10	386	Thiacloprid	5	412	Triticonazole	5
361	Spirotetramat	5	387	Thiamethoxam	5	413	Vamidothion	5
362	Spirotetramat-cisenol	20	388	Thidiazuron	5	414	Vamidothion-sulfoxide	5
363	Spiroxamine	5	389	Thifensulfuron-methyl	5	415	Zoxamide	5
364	Sulfentrazone	5	390	Thiodicarb	5			
365	Sulfometuron-methyl	5	391	Thiofanox-sulfone	5			

**Supplementary Table S2:** Recovery% (n = 10) and precision (RSD%) data calculated for 23 compounds under confirmatory validation. Sample matrix was olive oil. The first and second data correspond to 0.010 mg/kg and 0.100 mg/kg spiking levels, respectively.

Compound	Recovery%	RSD%
Atrazin	103-106	3.8-5.4
Benalaxyl	98.1-103	3.6-5.2
Bromacil	101-104	1.5-3.0
Carbofuran	98.9-103	1.0-8.4
Chlorotoluron	99.2-104	3.1-7.1
Cyprodinil	104-113	7.8-8.4
Dimethomorph Isomer 1	98.9-103	2.0-6.1
Dimethomorph Isomer 2	98.8-101	1.9-10.5
Sum of dimethomorph isomers	98.8-102	1.7-7.8
Famoxadone	101-103	2.4-9.8
Fenamidone	98.7-105	1.6-7.5
Fludioxinil	92.8-103	3.7-3.8
Imazaquin	96.4-107	4.4-5.2
Iprovalicarb	99.6-106	1.4-5.2
Mepanipyrim	95.7-102	6.0-6.1
Metazachlor	93.9-100	1.1-9.6
Metsulfuron-methyl	92.0-103	1.7-10.2
Monocrotophos	96.4-102	2.4-7.9
Oxadiazon	94.0-103	6.6-9.2
Pendimethalin	89.1-99.6	2.7-6.6
Pyraclostrobin	102-105	4.4-6.3
Spiroxamine	96.5-103	4.7-13.3
Thiamethoxam	96.7-99.6	1.2-11.4
Tralkoxydim	104-107	6.8-6.9