



EURL-FV Multiresidue Method Using QuEChERS

by GC-QqQ/MS/MS & LC-QqQ/MS/MS for Fruits & Vegetables

UCT Products:

ECQUEU750CT 50 mL centrifuge tube contains: (4 grams MgSO₄, 1 gram NaCl, 0.5 grams Na Citrate Dibasic Sesquihydrate, 1 gram Na Citrate Tribasic Dihydrate)

ECMPS15CT 15 mL centrifuge tube contains: (900 mg MgSO₄ & 150 mg PSA)
(*other configurations are available*)

January 17, 2011

Introduction

This summary of the European Union Reference Laboratory Residue method describes a QuEChERS approach for the analysis of 138 pesticides included in the Coordinated Multiannual Community Control Programme for 2010, 2011 and 2012 (Commission Regulation (EC) No 901/2009). Analysis is developed for **avocado, carrot, orange and pepper**.

Samples are prepared according to the Quality Control procedure established in the "Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed" (Document No. SANCO/10684/2009)

Procedure

1) Sample Preparation (for pesticides analyzed by HPLC-MS/MS)

- a) Homogenize the sample using a food processor according the typical QuEChERS procedures
- b) Weigh 10 g \pm 0.1 g of sample into a 50 mL centrifuge tube
- c) Add 10 mL of acetonitrile
- d) Shake vigorously or vortex for 1 minute to disperse contents
- e) Centrifuge for 5 minutes @ 4000 rpm

2) Clean-Up

- a) Transfer 6 mL of the supernatant to product **ECMPS15CT**
- b) Shake vigorously or vortex for 1 minute
- c) Centrifuge @ 6000 rpm for 2 minutes
- d) Transfer 1 mL of extract to a test tube and add 220 µL of acetonitrile
- e) Using a 0.45µ Teflon syringe filter, transfer extract to an LC injection vial
- f) Sample is ready for HPLC analysis

1A) Sample Preparation (For pesticides analyzed by GC-MS/MS)

- a) Procedure is the same as for HPLC analysis through steps 2) c)
- b) Transfer 1 mL of extract to a test tube
- c) Evaporate to dryness
- d) Add 1 mL of cyclohexane:acetone (9:1) to the dried extract
- e) Shake or vortex until completely dissolved
- f) Filter extract using a 0.45µ Teflon syringe filter into a GC vial
- g) Sample is ready for analysis by GC-MS/MS

3) Instrumentation and Analytical Conditions for the LC/QqQ (MS/MS) System

- LC-MS/MS System 3200 Q TRAP, Applied Biosystems
- Column: Atlantis T3 2.1x100 mm, 3 µm
- Column temperature: 40 °C
 - Mobile phase A: H₂O, 2 mM ammonia formate, 0.1 1% formic acid
 - Mobile phase B: methanol
 - Injection volume: 10µL
 - Autosampler temperature: 10° C
 - Analysis time: 18 min.

HPLC Flow Rate and Elution Gradient Table

Time (min)	A (%)	B (5)	Flow (µL/min)
0.0	95	5	300
1.0	95	5	300
1.1	70	30	300
10.0	0	100	300
13.0	0	100	300
13.1	95	5	300

4) Instrumentation and Analytical Conditions for the GC/QqQ (MS/MS)

- GC: Agilent 7890 Series or equivalent
- Autosampler: Agilent 7683 Injector and sample tray
 - Inlet: Splitless
 - Carrier gas: He
 - Inlet pressure: 22.73 psi
 - Inlet temperature: 250°C
 - Injection volume: 1 µL
- Analytical column: Agilent J&W HP-5ms 30 m x 250 µm x 0.25 µm or equivalent
- Retention time locking: Chlorpyrifos methyl locked to 16.596 min
- Spectrometer: Agilent 7000B Series
- Source temperature: 280°C
- Quadrupole temperature: Q1 and Q2 = 150°C
- Collision gas flows: N₂ at 1.5 mL/min, He at 25 mL/min

GC Oven Temperature Program

	Rate (°C/min)	Value (°C)	Hold Time (min)	Run Time (min)
Initial		70	2	2
Ramp 1	25	150	0	5.2
Ramp 2	3	200	0	21.9
Ramp 3	8	280	10	41.9

Spike Level with Method Validation Results

Pesticide	Mean	RSD	Mean	RSD	Technique w MS/MS
	0.01 mg/Kg	0.01 mg/Kg	0.1 mg/Kg	0.1 mg/Kg	
Acephate	88	7	89	3	HPLC
Acetamiprid	84	10	102	4	HPLC
Acrinathrin	103	12	91	13	GC
Aldicarb(RD)	105	13	98	5	HPLC
Amitraz(RD)	89	8	80	7	HPLC
DMPF	84	7	92	5	HPLC
DMF	75	5	103	7	HPLC
Azinphos-methyl	86	17	100	5	HPLC
Azoxystrobin	87	6	104	4	HPLC
Bifenthrin	95	9	92	10	GC
Bitertanol	118	11	103	8	HPLC
Boscalid	98	8	100	4	HPLC
Bromopropylate	95	11	94	12	GC
Bupirimate	96	6	94	10	GC
Buprofezin	87	8	89	8	GC
Cadusafos	88	16	96	6	HPLC
Captan	76	11	85	9	GC
Carbaryl	110	9	97	5	HPLC
Carbendazim(rd)	96	6	95	5	HPLC
Carbofuran(rd)	91	14	103	4	HPLC
Chlorfenvinphos	99	8	86	27	GC
Chlorotalonil	56	4	67	3	GC
Chlorpropham(RD)	99	7	95	7	GC
Chlorpyrifos	98	6	92	7	GC
Chlorpirifos-methyl	96	5	93	7	GC
Clofentezin(RD)	93	27	88	9	HPLC
Clothianidin	109	8	98	7	HPLC
Cyfluthrin(RD)	104	13	97	16	GC
Cypermethrin(RD)	109	11	109	16	GC
Cyproconazole	97	13	95	5	HPLC
Cyprodinil	97	7	91	9	GC
Deltamethrin	106	14	95	16	GC
Diazinon	99	7	91	7	GC
Dichlofluanid	68	5	71	2	GC
Dichlorvos	85	9	98	6	HPLC
Dicloran	111	17	97	8	GC
Difenoconazole	94	9	99	5	HPLC
Dimethoate(RD)	86	11	99	5	HPLC
Dimethomorph	107	10	97	5	HPLC

Diphenylamine	101	8	86	7	GC
Endosulfan(rd)	92	10	89	10	GC
Epoxiconazole	86	8	101	3	HPLC
Ethion	114	9	102	10	GC
Etofenprox	95	13	94	14	GC
Etoprofos	98	4	92	6	GC
Fenarimol	98	12	90	13	GC
Fenazaquin	89	9	88	11	GC
Fenbutatin oxide		-	78	4	HPLC
Fenbuconazole	106	12	100	5	HPLC
Fenhexamid	79	9	94	5	HPLC
Fenitrothion	116	10	103	7	GC
Fenoxycarb	105	24	98	8	HPLC
Fenpropathrin	99	10	96	11	GC
Fenpropimorph	81	7	99	3	HPLC
Fenthion(RD)	92	7	90	8	GC
Fenthion sulfoxide	86	15	92	10	HPLC
Fenvalerate	96	15	93	15	GC
Fipronil(RD)	98	5	91	11	GC
Fludioxonil	132	3	96	2	HPLC
Flufenoxuron	83	18	110	10	HPLC
Fluquinconazole	94	21	99	7	HPLC
Flusilazole	98	8	93	10	GC
Flutriafol	99	8	102	5	HPLC
Folpet	59	21	65	13	GC
Formetanate(RD)	87	10	95	4	HPLC
Fosthiazate	79	20	114	8	HPLC
Hexaconazole	95	12	96	4	HPLC
Hexythiazox	104	15	97	12	HPLC
Imazalil	88	6	97	6	HPLC
Imidacloprid	96	17	100	6	HPLC
Indoxacarb(RD)	90	40	113	9	HPLC
Iprodione	106	13	94	13	GC
Iprovalicarb	105	8	99	4	HPLC
Kresoxim-methyl	103	14	107	5	HPLC
Lambda-cyhalothrin(RD)	108	12	96	13	GC
Linuron	97	29	99	6	HPLC
Lufenuron	132	16	110	12	HPLC
Malathion(RD)	107	6	100	9	GC
Mepanipyrim(RD)	103	12	95	12	GC
Metalaxyl(rd)	111	11	94	8	GC
Metconazole	101	9	95	4	HPLC
Methamidophos	91	13	89	6	HPLC
Methidathion	115	6	101	9	GC
Methiocarb(RD)	109	33	104	15	HPLC
Methomyl(RD)	109	7	106	5	HPLC

Methoxyfenozide	125	12	99	13	HPLC
Monocrotophos	79	10	98	6	HPLC
Myclobutanil	89	11	91	11	GC
Oxadixyl	94	12	88	9	GC
Oxamyl	96	8	96	5	HPLC
Oxydemeton-methyl(RD)	94	7	97	4	HPLC
Paclobutrazole	91	13	100	5	HPLC
Parathion	119	6	101	9	GC
Parathion-methyl(RD)	109	6	100	7	GC
Pencycuron	100	18	101	6	HPLC
Penconazole	97	11	97	5	HPLC
Pendimethalin	116	8	96	8	GC
Permethrin(rd)	98	12	93	13	GC
Phenthoate	109	7	99	8	GC
Phosalone	111	14	99	14	GC
Phosmet(RD)	100	10	99	7	HPLC
Pyraclostrobin	95	11	110	4	HPLC
Pirimicarb(RD)	92	6	94	7	GC
Pirimiphos-methyl	111	8	94	9	GC
Prochloraz(RD)	88	8	95	5	HPLC
Procymidone	94	8	93	8	GC
Profenofos	100	10	97	10	GC
Propamocarb(RD)	68	9	69	6	HPLC
Propargite	112	10	104	7	HPLC
Propiconazole	94	9	91	10	GC
Propyzamide	99	5	96	7	GC
Prothioconazole	78	24	33	12	HPLC
Pyridaben	95	14	92	13	GC
Pyrimethanyl	119	13	95	6	GC
Pyriproxyfen	103	16	95	13	GC
Quinoxifen	114	8	87	10	HPLC
Spinosad(RD)	97	11	98	4	HPLC
Spiroxamine	124	15	80	15	HPLC
Taufluvallinate	102	13	96	17	GC
Tebuconazole	113	11	95	11	GC
Tebufenozide	124	45	97	13	HPLC
Tebufenpyrad	92	11	94	12	GC
Teflubenzuron	96	31	103	14	HPLC
Tefluthrin	89	5	89	7	GC
Tetraconazole	107	10	91	8	GC
Tetradifon	87	12	90	13	GC
Thiabendazole	92	9	93	8	HPLC
Thiamethoxam(RD)	84	16	101	5	HPLC
Thiacloprid	86	8	105	4	HPLC
Thiophanate-mehyil	69	13	104	6	HPLC
Tolclofos-methyl	91	4	97	5	GC

Tolyfluanid(RD)	69	9	72	10	GC
Triadimenol(RD)	-	-	105	29	HPLC
Triazophos	117	7	102	11	GC
Trichlorfon	75	19	106	7	HPLC
Trifloxystrobin	93	13	103	7	HPLC
Triflumuron	-	-	121	6	HPLC
Trifluralin	92	3	88	6	GC
Triticonazole	104	14	97	5	HPLC
Vinclozolin(RD)	97	7	95	7	GC
Zoxamide	79	17	112	5	HPLC

Summarized and adapted from "EURL-FV Multiresidue Method using QuEChERS followed by GC-QqQ/MS/MS and LC-QqQ/MS/MS for Fruits and Vegetables," European Reference Laboratory in Pesticide Residue, 2009

DCN-117110-201