

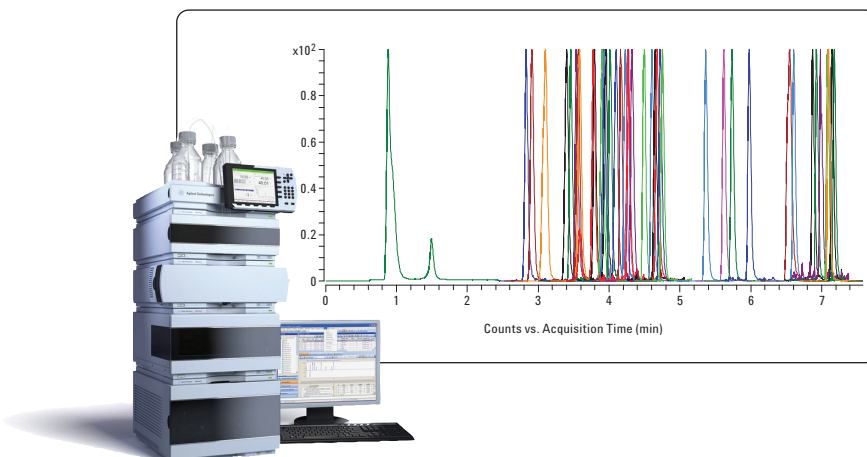
Determination of pesticides in baby food by UHPLC/MS/MS using the Agilent 1290 Infinity LC system and the Agilent 6460 triple quadrupole LC/MS

Application Note

Food

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Abstract

The qualitative and quantitative analysis of pesticides at trace levels in baby food matrices using UHPLC and triple quadrupole MS is demonstrated. Sample preparation is performed using an Agilent Bond Elut QuEChERS kit for extraction and dispersive SPE. The extracts are analyzed by LC/MS/MS on an Agilent 1290 Infinity LC system coupled to an Agilent 6460 triple quadrupole LC/MS using Dynamic MRM. The method and extraction performance were evaluated in terms of repeatability, linearity and sensitivity. Moreover the influence of the additional dispersive SPE cleanup was investigated. Detection limits were between 500 ng/kg and 10 ng/kg (ppt), which is much lower than the maximum residue level (MRL) of 10 µg/kg (ppb) imposed by the European Union.



Agilent Technologies

Introduction

Due to diversity of pesticides used in food protection and the globalization of the food industry, the monitoring of programs that cover a large number of pesticides is important. The application of UHPLC systems combined with the new generation triple quadrupole mass spectrometers facilitate the analysis of pesticides in challenging matrices such as food samples. As a result of the high sensitivity and the high scan rate capabilities of the Agilent 6460A triple quadrupole mass spectrometer, the simultaneous qualitative and quantitative multiresidue analysis of a large set of pesticides at trace levels can be performed.

The high sensitivity is essential for the analysis of these compounds in derived products, where the concentrations will be a fraction of the concentration in the raw material. In this respect, baby food is a challenging matrix. This application notes describes the quantitative analysis of 40 pesticides in baby food at levels below the maximum residue level (MRL) (10 µg/kg fruit or vegetable) specified in EC Regulation 396/2005 which was implemented in September 2008.¹ A QuEChERS extraction and dispersive SPE method was applied to isolate the pesticides from the baby food matrix. An Agilent 1290 Infinity LC was used to perform the separation on a Rapid Resolution High Definition (RRHD) ZORBAX Eclipse Plus column. The total analysis time was 10 min (including 1.5 min re-equilibration) and detection limits ranged from 10 to 500 ng/kg using Dynamic MRM and two transitions (quantifier and qualifier) per compound. Three different baby food compositions were analyzed. Extraction performance criteria such as repeatability, recovery (accuracy) and sensitivity were investigated.

Experimental

Instrumentation

An Agilent 1290 Infinity LC system and an Agilent 6460A triple quadrupole LC/MS with Agilent jet stream technology were used. The 1290 Infinity LC system was configured as follows:

Part number	Description
G4220A	Agilent 1290 Infinity Binary Pump with integrated vacuum degasser
G4226A	Agilent 1290 Infinity Autosampler
G1316C	Agilent 1290 Infinity Thermostatted Column Compartment
G4212A	Agilent 1290 Infinity Diode Array Detector

Method parameters:

Column	Agilent ZORBAX Eclipse Plus RRHD C18, 150 mm L × 2.1 mm id, 1.8 µm d _p	
Mobile phase	A = 0.05% (w/v) ammonium formate + 0.01% (v/v) formic acid in water B = Methanol	
Flow rate	0.5 mL/min	
Gradient	Min	% B
	0 to 5	10 to 65
	5 to 6.5	65 to 95
	6.5 to 8.5	95
	8.5 to 10	10
Temperature	45 °C	
Injection	2 µL, with needle wash (flushport, 5 s, water/methanol 1/1)	
Detection	MS/MS	
Ionization	Electrospray, positive ionization	

Jet Stream parameters

Drying gas temperature	250 °C
Drying gas flow	10 L/min
Nebulizer pressure	30 psig
Sheath gas temperature	340 °C
Sheath gas flow	11 L/min
Capillary voltage	4500 V
Nozzle voltage	500 V

Acquisition

Dynamic MRM	See Table 1
Delta EMV	50
Cycle time	200 ms

Compound		Precursor ion (<i>m/z</i>)	Product ion (<i>m/z</i>)	Fragmentor (V)	Collision energy (V)	Retention time (min)	Retention time window (min)
Cyromazine	Q	167.1	85.1	100	25	1.20	1.5
Cyromazine	q	167.1	125.1	100	25	1.20	1.5
Flonicamid	Q	230.1	203.1	80	15	2.85	0.8
Flonicamid	q	230.1	174.1	80	15	2.85	0.8
Thiamethoxam	Q	292.2	211.0	85	4	2.92	0.8
Thiamethoxam	q	292.2	181.0	85	16	2.92	0.8
Monocrotofos	Q	224.1	127.0	85	10	3.11	0.8
Monocrotofos	q	224.1	193.0	85	5	3.11	0.8
Dicrotofos	Q	238.1	112.1	90	5	3.41	0.8
Dicrotofos	q	238.1	127.0	90	15	3.41	0.8
Ethiofencarb-sulfone	Q	258.1	107.1	80	10	3.47	0.8
Ethiofencarb-sulfone	q	258.1	201.1	80	10	3.47	0.8
Imidacloprid	Q	256.1	175.1	90	20	3.55	0.8
Imidacloprid	q	256.1	209.0	90	15	3.55	0.8
Clothianidin	Q	250.0	169.1	90	7	3.58	0.8
Clothianidin	q	250.0	132.1	90	15	3.58	0.8
Ethiofencarb-sulfoxide	Q	242.1	107.1	80	15	3.60	0.8
Ethiofencarb-sulfoxide	q	242.1	185.1	80	15	3.60	0.8
Methiocarb-sulfoxide	Q	242.0	185.1	80	10	3.79	0.8
Methiocarb-sulfoxide	q	242.0	170.0	90	15	3.79	0.8
Thiofanox-sulfone	Q	251.1	57.1	100	15	3.80	0.8
Thiofanox-sulfone	q	251.1	76.1	100	15	3.80	0.8
Trichlorfon	Q	256.9	109.0	100	15	3.92	0.8
Trichlorfon	q	256.9	221.0	100	15	3.92	0.8
Vamidothion	Q	288.1	146.1	80	10	3.94	0.8
Vamidothion	q	288.1	118.1	80	20	3.94	0.8
Acetamiprid	Q	223.1	126.0	100	15	3.94	0.8
Acetamiprid	q	223.1	56.0	100	15	3.94	0.8
Carbofuran-3-OH	Q	238.1	163.1	85	5	3.96	0.8
Carbofuran-3-OH	q	238.1	181.1	85	5	3.96	0.8
Fenthion-oxon-sulfoxide	Q	279.0	104.1	125	30	4.03	0.8
Fenthion-oxon-sulfoxide	q	279.0	121.1	125	30	4.03	0.8
Carbendazim	Q	192.1	160.1	100	15	4.11	0.8
Carbendazim	q	192.1	132.1	100	25	4.11	0.8
Fenthion-oxon-sulfone	Q	295.0	217.1	125	25	4.18	0.8
Fenthion-oxon-sulfone	q	295.0	104.1	125	25	4.18	0.8
Cymoxanil	Q	199.2	128.0	65	5	4.24	0.8
Cymoxanil	q	199.2	111.0	100	20	4.24	0.8
Oxycarboxin	Q	268.1	175.0	100	10	4.27	0.8
Oxycarboxin	q	268.1	146.9	100	25	4.27	0.8
Chlothiamid	Q	205.9	189.0	85	20	4.29	0.8
Chlothiamid	q	205.9	172.0	85	20	4.29	0.8

Table 1
Dynamic MRM data acquisition parameters for the compounds under investigation. Q = quantifier, q = qualifier.

Compound		Precursor ion (<i>m/z</i>)	Product ion (<i>m/z</i>)	Fragmentor (V)	Collision energy (V)	Retention time (min)	Retention time window (min)
Thiacloprid	Q	253.1	126.0	100	20	4.34	0.8
Thiacloprid	q	253.1	186.0	100	10	4.34	0.8
Florasulam	Q	360.0	129.1	100	20	4.51	0.8
Florasulam	q	360.0	191.9	100	10	4.51	0.8
Tricyclazole	Q	190.1	163.2	100	20	4.62	0.8
Tricyclazole	q	190.1	136.2	100	25	4.62	0.8
Butocarboxim	Q	213.1	75.1	110	15	4.66	0.8
Butocarboxim	q	213.1	156.1	110	5	4.66	0.8
Thiabendazole	Q	202.0	175.0	120	25	4.69	0.8
Thiabendazole	q	202.0	131.0	120	35	4.69	0.8
Aldicarb	Q	208.0	116.0	70	0	4.73	0.8
Aldicarb	q	208.0	89.1	70	5	4.73	0.8
DMSA	Q	201.0	92.1	85	15	4.76	0.8
DMSA	q	201.0	137.1	85	10	4.76	0.8
Propoxur	Q	210.1	111.1	50	10	5.36	0.8
Propoxur	q	210.1	93.0	50	20	5.36	0.8
Carbaryl	Q	202.1	145.1	50	2	5.62	0.8
Carbaryl	q	202.1	127.0	50	20	5.62	0.8
Monolinuron	Q	215.2	126.0	100	20	5.75	0.8
Monolinuron	q	215.2	148.1	100	20	5.75	0.8
Fluazifop	Q	328.1	282.1	120	20	5.99	0.8
Fluazifop	q	328.1	254.1	120	20	5.99	0.8
Spiroxamine	Q	298.4	144.2	100	10	6.54	0.8
Spiroxamine	q	298.4	100.2	100	10	6.54	0.8
Pyrimethanil	Q	200.1	107.1	100	25	6.61	0.8
Pyrimethanil	q	200.1	82.0	100	30	6.61	0.8
Fenhexamid	Q	302.1	97.0	120	10	6.88	0.8
Fenhexamid	q	302.1	142.1	100	5	6.88	0.8
Fenbuconazole	Q	337.2	125.0	120	15	6.94	0.8
Fenbuconazole	q	337.2	194.1	120	15	6.94	0.8
Iprodion	Q	330.0	244.9	110	10	6.98	0.8
Iprodion	q	330.0	287.9	110	5	6.98	0.8
Kresoxim-methyl	Q	314.2	116.0	70	10	7.08	0.8
Kresoxim-methyl	q	314.2	222.0	70	10	7.08	0.8
Penconazole	Q	284.1	69.9	85	15	7.11	0.8
Penconazole	q	284.1	158.8	85	30	7.11	0.8
TPP	Q	327.1	77.0	180	40	7.14	0.8
TPP	q	327.1	151.9	180	40	7.14	0.8
Pyraclostrobin	Q	388.2	194.1	100	10	7.18	0.8
Pyraclostrobin	q	388.2	296.2	100	10	7.18	0.8

Table 1
Dynamic MRM data acquisition parameters for the compounds under investigation. Q = quantifier, q = qualifier. (continued)

Solutions and Samples

Stock solutions of the pesticides were prepared in acetonitrile. These solutions were diluted to the appropriate concentration (range 0.05 ppb to 1 ppm) in 1% v/v acetic acid in acetonitrile. An internal standard solution of triphenylphosphate (TPP, 20 µg/mL) was prepared in the same solvent.

Sample Preparation

Three baby food products were obtained from a local supermarket. According to the labels, the samples were composed of the following ingredients:

- Sample 1: carrots (40%), potatoes (18%), tomatoes (18%), beans (13%), beef (10%)
- Sample 2: water (37%), potatoes (30%), spinach (17%), chicken (10%)
- Sample 3: carrots (54%), potato (23%), water (16%), rice (7%)

The sample preparation was performed using Agilent Bond Elut QuEChERS AOAC kits for extraction and dispersive SPE cleanup. The procedure is described below.

Extraction

1. Weigh 15 g of sample into a 50-mL centrifuge tube.
2. Add 100 µL TPP solution.
3. Add spiking solution, if necessary.
4. Vortex for 30 s.
5. Add 15 mL of 1% v/v acetic acid in acetonitrile and the Bond Elut AOAC extraction salt (p/n 5982-5755).
6. Cap tubes and shake vigorously by hand for 1 min.
7. Centrifuge at 4,000 rpm for 5 min.
8. Filter 1 mL of sample through a syringe filter (0.2 µm pore size, regenerated cellulose, p/n 5061-3366) and analyze directly (no SPE) or (additional clean-up).

Dispersive SPE

1. Transfer 8 mL from the centrifuged extract into a 15-mL Bond Elut AOAC dispersive SPE tube for fatty samples (p/n 5982-5158).
2. Vortex for 30 s.
3. Centrifuge at 13,000 rpm for 2 min.
4. Filter 1 mL through a syringe filter (0.2 µm pore size, regenerated cellulose, p/n 5061-3366) and analyze.

Results and discussion

State-of-the-art LC/MS/MS equipment enables fast multiresidue analysis of pesticides at low levels in complex matrices. The Agilent 1290 Infinity LC provides the necessary power to perform analysis of the 40 selected pesticides within the 10-min total analysis time (run time and equilibration time). A 15 cm column was preferred above a 10 or 5 cm column because of the higher resolving power. This is useful to minimize ion suppression or response enhancement due to matrix effects. Methanol was chosen as an organic modifier because of the significantly improved sensitivity compared to acetonitrile for this analysis.

During the analysis, a total of 82 transitions (2 per solute + 2 for IS) had to be performed. The dynamic MRM function allows MRM transition lists to be built based on a retention time window specified for each analyte. Consequently, the pesticides are only monitored during that elution window in the analytical run. This approach leads to equivalent or better results in terms of sensitivity and quantification (data points) compared to the traditional time segment based methods². With the Dynamic MRM enabled, the maximum number of concurrent MRMs was 32. Using an MRM cycle time of 200 ms, the minimal and maximal transition dwell times were 2.75 and 96.50 ms

(values given by MassHunter acquisition software), respectively. The resulting number of data points across the peaks was above 20 for all compounds which is largely sufficient for quantitation purposes.

The performance of the LC/MS/MS method was tested by the analysis of standard solutions. The chromatogram (overlaid MRMs of quantification ions) for a 10 ppb solution is shown in Figure 1. Figures of merit are summarized in Table 2. The injection precision was tested at two concentration levels (1 and 10 ppb). The standard solutions were each injected five consecutive

times. The linearity of the method was evaluated between 0.05 and 20 ppb at eight levels (0.05, 0.10, 0.20, 0.50, 1, 2, 10, and 20 ppb). Each solution was injected once. The lowest level is below the detection limit for some compounds. For these analytes, the calibration curve was started at the limit of detection.

The sensitivity was excellent and all compounds could be analyzed at the sub-ppb level. An example of the ion traces (quantification ion transition and qualifier ion transition) and the corresponding calibration curves for fluazifop

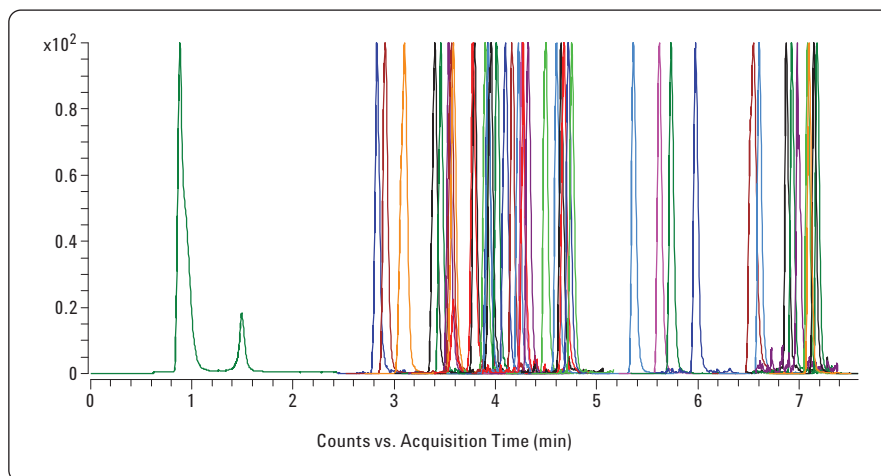


Figure 1
MRM of a 10 ppb standard solution (only quantifier transitions are shown).

Compound	Repeatability of injection (% RSD)		Linearity (R ²)	Detection limit (ppb)	
	1 ppb	10 ppb		Q	q
Acetamiprid	2.20	1.62	0.9999	0.02	0.02
Aldicarb	4.82	2.03	0.9999	0.01	0.02
Butocarboxim	19.93	2.36	0.9996**	0.20	0.50
Carbaryl	1.70	1.73	0.9996	0.01	0.01
Carbendazim	2.93	1.28	0.9997	0.01	0.05
Carbofuran-3-OH	14.68	2.50	0.9996*	0.10	0.10
Chlothiamid	20.64	7.28	0.9979*	0.20	1.00
Clothianidin	7.69	2.14	0.9999*	0.10	0.20
Cymoxanil	7.30	3.88	0.9998*	0.10	0.50
Cyromazine	2.02	1.08	0.9993	<0.50 ¹	0.50
Dicrotofos	3.69	1.01	0.9994	0.01	0.02
DMSA	5.13	2.36	0.9996	0.05	0.20
Ethiofencarb-sulfone	2.69	2.25	0.9998	0.05	0.20
Ethiofencarb-sulfoxide	6.24	2.02	0.9991*	0.10	0.10
Fenbuconazole	11.29	1.24	0.9994**	0.20	1.00
Fenhexamid	5.19	4.93	0.9988**	0.20	1.00
Fenthion-oxon-sulfone	13.96	7.10	0.9988	0.05	0.05
Fenthion-oxon-sulfoxide	13.13	2.90	0.9986	0.05	0.10
Flonicamid	10.87	2.55	0.9980	0.05	0.20
Florasulam	9.51	3.25	0.9999	0.05	0.20
Fluazifop	5.77	3.28	0.9998**	0.20	0.50
Imidacloprid	3.31	1.15	0.9998	0.05	0.05
Iprodione	24.53	4.28	0.9984***	0.50	5.00
Kresoxim-methyl	4.46	1.16	0.9999	0.01	0.05
Methiocarb-sulfoxide	4.02	2.85	0.9991*	0.10	0.20
Monocrotofos	1.71	1.45	0.9996	0.01	0.02
Monolinuron	0.67	0.27	0.9999	0.05	0.05
Oxycarboxin	5.92	1.93	0.9991	0.05	0.05
Penconazole	2.02	1.60	0.9997	0.01	0.02
Propoxur	0.70	0.94	0.9998	0.01	0.01
Pyraclostrobin	1.23	0.93	0.9996	0.01	0.02
Pyrimethanil	5.55	0.60	0.9997	0.02	0.05
Spiroxamine	0.91	0.87	0.9997	<0.01	<0.01
Thiabendazole	2.99	0.96	0.9999	0.02	0.02
Thiacloprid	1.57	1.10	0.9995	0.02	0.05
Thiamethoxam	1.38	1.89	0.9998	0.01	0.05
Thiofanox-sulfone	5.13	2.14	0.9998	0.05	0.10
Trichlorfon	6.34	4.31	0.9988	0.05	0.20
Tricyclazole	1.66	0.85	0.9999	0.02	0.02
Vamidothion	4.56	1.16	0.9997	0.01	0.01

* Detection limit is 0.10 ppb, calibration starts at 0.10 ppb.

** Detection limit is 0.20 ppb, calibration starts at 0.20 ppb.

*** Detection limit is 0.50 ppb, calibration starts at 0.50 ppb.

1 High due to interference of a system peak.

Table 2
Method performance results.

(a compound with relatively low sensitivity) and for propoxur (a compound with relatively good sensitivity) are shown in Figures 2 and 3, respectively. Most of the compounds have detection limits below 0.05 ppb. The sensitivity for spiroxamine is below the lowest level injected (0.01 ppb) which is significantly better compared to the other pesticides. No accurate detection limit could be determined for cyromazine due to a system peak that interfered at low levels.

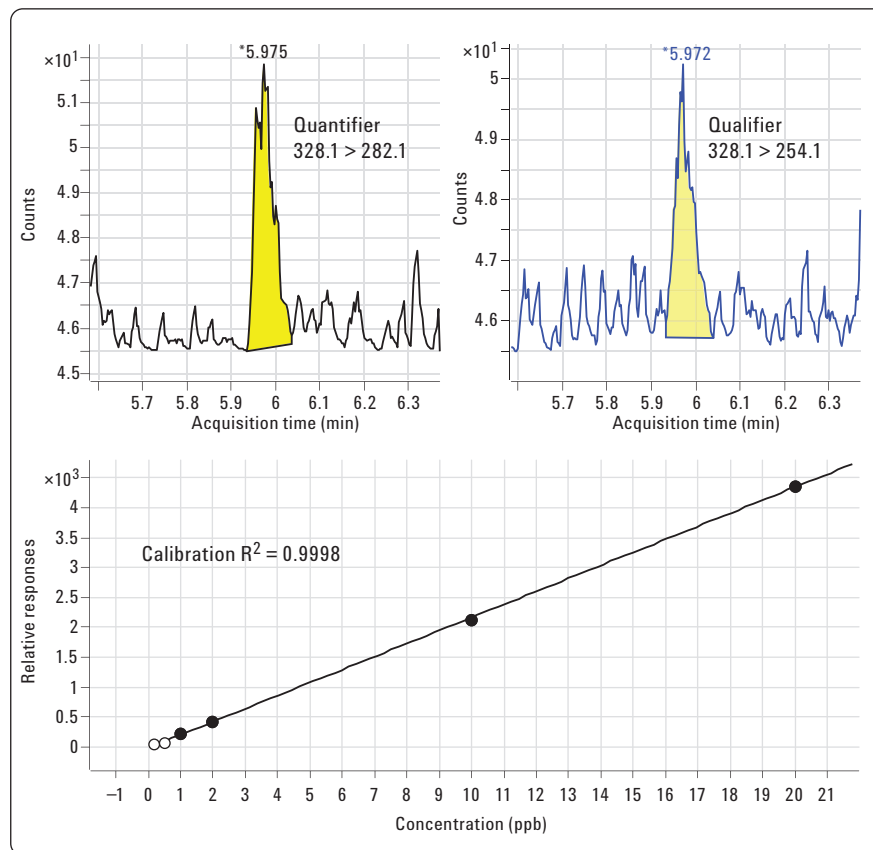


Figure 2
Ion traces for two transitions at the LOD (0.5 ppb standard solution) and calibration curve for fluazifop.

The QuEChERS sample preparation procedure was applied to three baby food samples. Extracts were analyzed with and without additional dispersive SPE cleanup. There were no target compounds detected above the LOD in non-spiked samples. The resulting chromatogram, shown as an overlay of quantification transitions for a sample spiked at 1-ppb level with all 40 pesticides, is depicted in Figure 4. The signals for the quantifier and qualifier transitions for fluazifop and propoxur in the spiked sample at 1-ppb level are shown in Figure 5. From these traces it is clear that excellent selectivity and sensitivity are obtained. The relative response of the quantification transition and qualifier transition are clearly within the limits for positive identification.

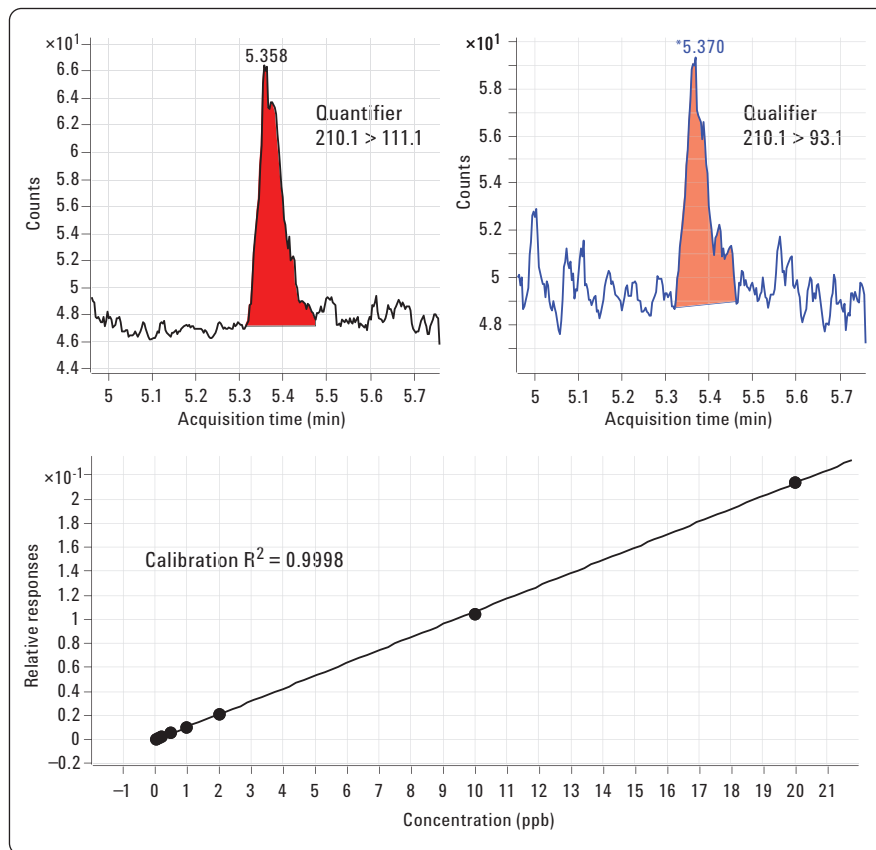


Figure 3
Ion traces for two transitions at the LOD (0.01 ppb standard solution) and calibration curve for propoxur.

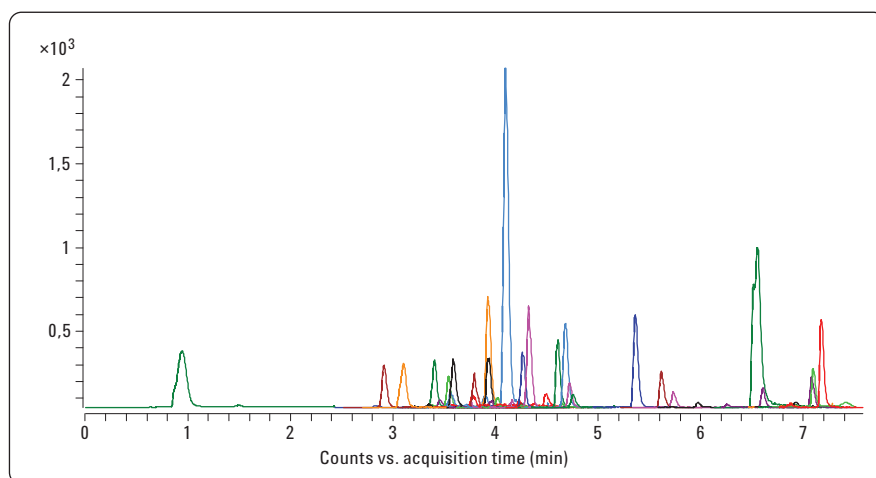


Figure 4
MRM of an extract of sample 2 spiked with 1 ppb (only quantifier transitions are shown). No dispersive SPE performed on the sample. The transition for the internal standard is not shown.

The performance criteria of the sample preparation and analysis method are summarized in Table 3. The extraction repeatability is calculated on sample 1, spiked at the 10-ppb level and repeated (extraction + analysis) five times. Most RSDs are below 10%, with the exception of iprodione and fluazifop, where higher values are obtained after SPE. The average recovery (response spike sample / response calibration sample) for the three different samples was between 70% and 110% at 1 and 10 ppb spike level in most cases. No significant differences were observed between the different matrices. The recovery is satisfactory even at the 1-ppb level and in most cases there is no significant difference between extracts that have been subjected to the additional SPE procedure and those that have not. For cyromazine, better values are obtained after SPE. For fluazifop, on the other hand, very low recoveries (and high RSD) are obtained when additional dispersive SPE is used. In this case, analysis without additional SPE is recommended.

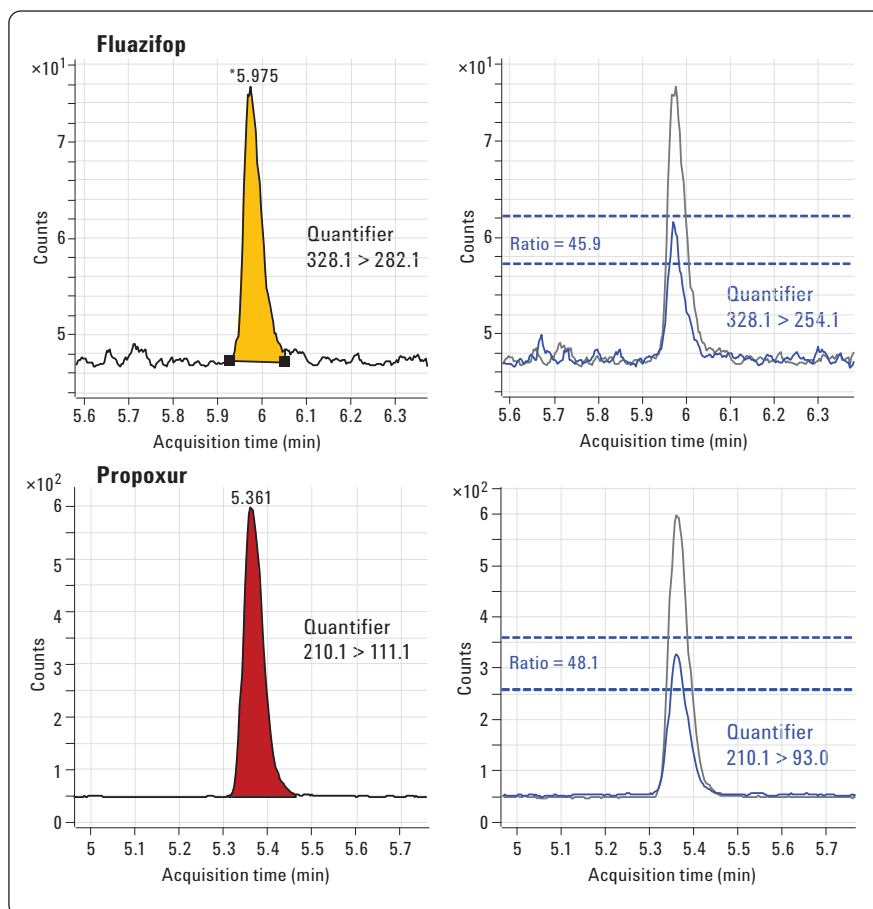


Figure 5
Ion traces for 2 transitions for fluazifop and propoxur in an extract of sample 2 spiked with 1 ppb. No dispersive SPE performed on the sample. The uncertainty was set at 20% (dotted lines).

	Repeatability of extraction (% RSD)1		Average recovery (%)2				Lowest level detected in extract (ppb)3
Compound	No IS SPE	No IS No SPE	1 ppb SPE	No SPE	10 ppb SPE	No SPE	
Acetamiprid	1.50	1.55	107.0	83.4	99.3	92.2	0.10
Aldicarb	1.74	1.69	91.9	82.8	92.4	81.8	0.10
Butocarboxim	4.41	4.17	95.4	79.6	91.8	86.9	1.00
Carbaryl	1.84	1.30	92.3	75.4	93.2	80.1	0.10
Carbendazim	1.87	1.23	88.6	79.4	90.5	78.1	0.10
Carbofuran-3-OH	6.13	4.62	85.8	114.4	100.5	98.2	1.00
Chlothiamid	4.59	9.54	87.5	105.1	91.8	70.1	1.00
Clothianidin	3.01	1.70	87.0	117.7	103.7	103.2	1.00
Cymoxanil	5.27	5.11	101.5	72.1	99.0	98.1	1.00
Cyromazine	1.70	0.54	108.7	87.1	73.0	57.8	<10.00 ⁴
Dicrotofos	2.73	1.98	102.9	83.0	93.5	83.5	0.10
DMSA	2.13	1.73	96.3	109.9	103.0	108.1	0.10
Ethiofencarb-sulfone	1.52	2.96	92.1	85.6	91.8	84.2	1.00
Ethiofencarb-sulfoxide	2.33	0.74	90.8	88.9	92.1	83.2	0.10
Fenbuconazole	3.77	6.28	107.5	80.6	90.8	99.8	1.00
Fenhexamid	5.22	7.29	74.0	100.8	91.5	83.5	1.00
Fenthion-oxon-sulfone	6.08	4.49	91.4	78.4	89.8	89.8	1.00
Fenthion-oxon-sulfoxide	2.63	0.90	122.8	96.5	100.8	90.0	0.10
Flonicamid	2.77	3.02	94.4	86.1	94.3	91.8	0.10
Florasulam	5.98	3.76	72.2	103.3	73.8	115.9	0.10
Fluazifop	20.71	1.45	14.4	117.7	18.8	92.0	1.00
Imidacloprid	2.98	2.35	115.2	114.9	111.5	117.6	0.10
Iprodion	14.30	4.37	87.0	90.8	89.8	91.0	1.00
Kresoxim-methyl	4.16	3.82	74.0	80.6	71.8	80.7	0.10
Methiocarb-sulfoxide	3.06	1.63	94.5	98.5	93.1	87.1	0.10
Monocrotofos	1.54	0.72	90.2	81.6	90.9	83.1	0.10
Monolinuron	1.66	0.71	90.0	80.9	92.9	84.3	0.10
Oxycarboxin	2.04	1.74	89.8	107.4	101.0	105.6	0.10
Penconazole	4.25	2.77	73.4	78.2	76.9	83.6	0.10
Propoxur	1.61	0.25	94.7	83.0	95.6	84.9	0.10
Pyraclostrobin	3.62	4.59	86.1	89.3	84.8	90.9	0.10
Pyrimethanil	1.99	2.22	85.5	84.4	86.8	78.3	0.10
Spiroxamine	3.91	1.50	79.6	91.7	78.4	85.8	0.10
Thiabendazole	1.29	1.52	92.7	74.5	91.1	78.7	0.10
Thiacloprid	2.51	1.74	96.7	90.7	94.3	86.4	0.10
Thiamethoxam	2.09	1.12	104.8	108.5	112.4	108.7	0.10
Thiofanox-sulfone	2.70	1.48	99.0	93.3	91.5	84.1	0.10
Trichlorfon	6.94	1.93	86.9	86.5	99.7	92.4	1.00
Tricyclazole	0.90	1.58	91.3	72.0	90.9	75.6	0.10
Vamidothion	1.75	2.63	92.4	79.2	90.0	81.8	0.10

1 Sample 1, spiked with 10 ppb, extracted 5 times. 1 injection per extract.

2 Average of samples 1 to 3, spiked at 1 ppb and at 10 ppb and extracted once. 1 injection per extract.

3 Samples were spiked at 0.1, 1, and 10 ppb level. Lowest detected level is reported.

4 High due to interference of a system peak.

Table 3
Extraction performance.

Conclusion

The multiresidue LC/MS/MS method enabled the analysis of 40 pesticides at low levels in baby food. Sample preparation was performed using an Agilent Bond Elut QuEChERS AOAC kit. The total analysis time using the Agilent 1290 Infinity LC system and the Agilent 6460A triple quadrupole LC/MS was 10 min. All compounds could be detected at 0.5 µg/kg or lower in the samples, which is 20 times lower than the MRL for these compounds in baby food according to EU regulation. The extraction repeatability and recovery were good. No difference on extraction and analytical performance due to differences in sample matrix were observed. The optional dispersive SPE cleanup procedure can be applied but for some solutes larger standard deviation and lower recoveries were observed after SPE.

References

1. European Parliament and Council Regulation No. 396/2005 on Maximum Residue Levels of Pesticides In or On Food and Feed of Plant and Animal Origin and Amending Council Directive 91/414/EEC, 23 February 2005.
2. P. Stone, T. Glauner, K. Kuhlmann, T. Schlabach, K. Miller, "New Dynamic MRM Mode Improves Data Quality and Triple Quad Quantitation in Complex Analyses," Agilent Technologies, Technical Overview, 5990-3595EN, June 2009.

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