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Ensuring Food Safety through Comprehensive Pesticide Residue Monitoring and Regulatory Compliance in Infant Nutrition

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INTRODUCTION

The National Reference Centre for Pesticide Residues at the Public Health Authority of the Slovak Republic has established, validated, and applied analytical methods for the determination of more than 200 pesticide residues in baby food. The analyses of final extracts were carried out using high-performance liquid chromatography and gas chromatography coupled with tandem mass spectrometric detection (LC-MS/MS, GC-MS/MS), as well as gas chromatography with an electron capture detector (GC-ECD). Sample preparation was performed using the QuEChERS procedure [1]. Prior to routine use, all methods were validated for each pesticide residue according to the criteria outlined in the SANTE document (2011) [2, 3]. Selected pesticide determinations are accredited by the Slovak National Accreditation Service under the standard STN EN ISO/IEC 17025:2018. The performance of the methods is regularly verified, at least once every five years, through participation in international proficiency testing schemes, in which the laboratory consistently demonstrates reliable and satisfactory results.

DETAILS ABOUT LC-MS/MS (Q-TRAP) METHOD

Optimized LC conditions:

Column: Kinetex® 2.6 µm Polar C18 100 Å, 100 × 2.1 mm
Column temperature: 30 °C
Injection volume: 4, 6 µL
Autosampler temperature: 15 °C
Mobile phase A: 0.1% Formic acid in Methanol
Mobile phase B: 0.1% Formic acid in deionized water
Run time: 22 min (12.6 min)
Flow rate: 0.25 mL/min

Time (min)	MP A (%)	MP B (%)
0.00	5	95
5.20	45	55
10.70	95	5
11.80	95	5
15.00	100	0
15.10	100	0
22.00	5	95

Optimized MS/MS conditions:

Scan type: Scheduled MRM (Multiple Reaction Monitoring)
Target cycle time: 1 s
Ion source: Electrospray Ionization (ESI)
Curtain gas (CUR): 40 psi
Collision gas (CAD): Medium
Ion spray voltage (IS): 4500 (5500) V
Ion source temperature (TEM): 400 °C
Ion source gas 1 (GS1): 50 psi
Ion source gas 2 (GS2): 50 psi
Entrance potential (EP): 10 V

Time (min)	MP A (%)	MP B (%)
0.00	5	95
1.33	5	95
2.20	60	40
5.00	95	5
7.50	95	5
7.51	100	0
9.50	100	0
9.60	5	95
12.60	5	95

DETAILS ABOUT GC-MS/MS (IT), GC-ECD METHODS

GC-MS/MS (IT) method:

Capillary column: 5% phenyl-methylpolysiloxane stationary phase, 30 m × 0.25 mm i.d., 0.25 µm film thickness, equipped with a 10 m guard column

GC Conditions:

Oven temperature program:

Ramp Rate (°C/min)	Temp. (°C)	Hold Time (min)
–	75	3.5
20	190	0
2	220	0
10	300	5

Injector temperature program:

Ramp Rate (°C/min)	Temp. (°C)	Hold Time (min)
–	80	0.5
200	280	32.5
200	80	–

Carrier gas: Helium, constant flow at 1 mL/min
Injection volume: 7 µL

MS/MS Conditions:

Ion trap temperature: 220 °C
Transfer line temperature: 270 °C
Manifold temperature: 50 °C
Ionization mode: EI (Electron Ionization)

GC-ECD method:

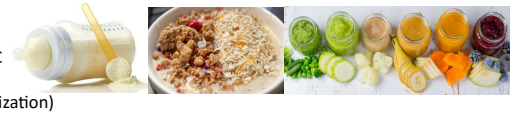
Capillary column: 100% dimethylpolysiloxane stationary phase, 60 m × 0.25 mm i.d., 0.25 µm film thickness

Primary GC Conditions:

Oven temperature program:

Ramp Rate (°C/min)	Temp.(°C)	Hold Time (min)
–	60	1
15	100	0
10	160	0
2.5	275	10

Injection mode: Splitless
Injector temperature: 250 °C
Detector temperature: 330 °C
Carrier gas: Helium, constant flow at 1 mL/min
Make-up gas: Nitrogen, constant flow at 30 mL/min
Injection volume: 1 µL



Matrices for Baby Food - Dairy, Cereal, Fruit, Vegetable, Meat Products, and Their Combinations

QuEChERS SAMPLE PRE-TREATMENT

Mix. 1: 8:2:2:1 ($MgSO_4$ (coarse) : NaCl : $Na_3Cit{\cdot}2H_2O$: $Na_2HCit{\cdot}1.5H_2O$). 6.5 g of Mix. 1 is added to10 mL ACN.
Mix. 2: 6:1 ($MgSO_4$ (fine) : PSA). 1.05 g of Mix. 2 is added to 6 ml of extract.
Mix. 3: 34.2:1.8:6 ($MgSO_4$ (fine) : GCB : PSA). 1.05 g of Mix. 3 is added to 6 ml of extract.
Mix. 4: 4:1 ($MgSO_4$ (coarse) : NaCl). 5 g of Mix. 4 is added to 6 ml of extract.
Mix. 5: 4:1 ($MgSO_4$: NaCl). For one analytical sample 5 g of Mix. 5 is added.
Mix. 6: 1:4 (NaCl : $MgSO_4$ (fine)). 2 g of Mix. 6 is added to 5 ml of extract.

QuEChERS – Basic Method: Sample preparation for pesticide analysis with extract clean-up using Mix. 2.
QuEChERS with GCB: Sample preparation for pesticide analysis in matrices with high carotenoid or chlorophyll content, with extract clean-up using Mix. 3.
QuEChERS acidified with H2SO4: Acidified method QuEChERS with 100 mL H_2SO_4 for Chlorotalonil, with extract clean-up using Mix. 4.
A-QuEChERS (QuA): Acidified method QuEChERS (10 mL 1% HCOOH in ACN), with extract clean-up using Mix. 5.
QuEChERS for matrices with high fat content: Sample preparation for pesticide analysis in matrices with high fat content, with extract clean-up using Mix. 6.

Procedure of QuEChERS (basic method or GCB)

(5 g powdered sample + 10-20 mL H_2O, let stand for 10 min) or (10 g liquid sample)

RESULTS & DISCUSSION

Analytical methods have been validated for a broad range of more than 200 pesticide active substances and metabolites in various infant food matrices, including fruit and vegetable purées, milk-based formulas, cereal products, and foods for special medical purposes. All baby food samples analyzed in recent years complied with the established pesticide safety standards.

Regarding selectivity and specificity, the blank response and the ratio of quantitation to qualification ions in the sample were not permitted to deviate by more than 30%. For linearity, deviations from the calibration curve were allowed up to 20%, and changes in response at calibration levels measured before and after the sample sequence had to remain within 30%. In terms of accuracy, acceptable recovery rates ranged between 60% and 140%, while residue reproducibility was required to be within 20%, and measurement uncertainty did not exceed 50%.

CONCLUSION

The results of official pesticide control and European monitoring of baby food conducted over the past ten years confirm its safety with respect to pesticide residues, as none of the analyzed samples exceeded the permitted limits.

FUTURE WORK / REFERENCES

[1] Anastassiades M, et al. Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce. J AOAC Int. 2003; 86(2):412–431.

[2] http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=URISERV:l13002&frontOfficeSuffix=%2Fa

[3] https://food.ec.europa.eu/plants/pesticides/maximum-residue-levels/guidelines-maximum-residue-levels_en