Macedonian pharmaceutical bulletin, **71** (1) 43 - 53 (2025)
ISSN: 1857-8969
DOI: 10.33320/maced.pharm.bull.2025.71.01.005
Professional paper

Development and validation of a GC-ECD method for pesticide residue detection in flour

Margarita Timofejeva[1*], Anita Najdenkoska[1], Biljana Bauer[2]

[1]*Institute for Public Health, Division for Contaminants and Ecotoxicology, Department for Chemical and Radiological testing, 50th Division 6, 1000 Skopje, Republic of North Macedonia*
[2]*Institute of Pharmacognosy, Faculty of Pharmacy, University Ss. Cyril and Methodius in Skopje, Mother Theresa 47, 1000 Skopje, Republic of North Macedonia*

Received: January 2025; Accepted: March 2025

Abstract

This study aimed to optimize and validate a rapid, reliable and accurate method for quantitative determination of pesticide residues in flour matrix. Pesticides were extracted using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) sample preparation technique for pesticide residue analysis in food matrices, followed by quantitative determination using GC-ECD. The method was validated for 26 pesticides at six calibration levels (0.005; 0.01; 0.02; 0.04; 0.1; 0.2 mg/L), using flour matrix. The method is accurate and precise, which can be concluded from the validation parameters: linearity, precision, limit of detection (*LOD)*, limit of quantification (*LOQ)*, accuracy and measurement uncertainty (*MU*). The calibration curves for all pesticides showed excellent linearity, with correlation coefficients (R^2) exceeding 0,99. The limit of detection (*LOD*) of the pesticides in the matrix varied from 0,0019 – 0,0083 mg/kg, while the limit of quantification (*LOQ*) from 0,0065 – 0,0276 mg/kg. The method demonstrated satisfactory accuracy and precision, with recovery values *(REC %)* within the acceptable range of 70–120% and relative standard deviation *(RSD %)* below 20% for all tested pesticides. The measurement uncertainty (*MU*) was also estimated based on the validation data and the values were found below the standard limit of 50%. The validated method proved to be reliable and suitable for routine pesticide residue analysis in flour matrices.

Key words: validation, GC-ECD, pesticide residues, QuEChERS

Introduction

The term 'pesticides' is commonly used to refer to products applied for plant protection. Based on their chemical composition pesticides are generally classified into four main groups: organochlorines (e.g., DDT), organophosphates (e.g., chlorpyrifos), carbamates (e.g., carbaryl), and pyrethroids (e.g., deltamethrin) (Akashe et al., 2018; Garud et.al., 2024; Kaur et al., 2019).

Cereals are one of the most intensively produced and consumed food products in the world. Cereals also provide important minerals, vitamins and other micronutrients that are necessary for maintaining optimal health (Rezaei et al., 2017). The protection of agricultural crops with the use of pesticides has a significant contribution to the growth of productivity. Taking this into account, special attention should be paid to pesticide residues. The negative effects of these pesticides occur only above a certain level of exposure. The toxicity of pesticides largely depends on their rational use in terms of proper application and correct dosage (WHO, 2022; Zanella et al., 2012).

International regulatory agencies emphasize the control of pesticides so that food products do not contain residues of individual pesticides that exceed the maximum residue levels (*MRL*) approved by regulatory agencies.

*email: mtimofejeva@yahoo.com

Table 1. Oven program

	Rate (^{0}C/min)	T (^{0}C/min)	Hold time (min)
(initial)	-	100.0	1.00
Ramp 1	20.0	140.0	2.00
Ramp 2	10.0	220.0	5.00
Ramp 3	5.0	310.0	1.0

The Regulation on General Requirements for Food Safety prescribes general food safety requirements in terms of maximum permitted concentrations of pesticide residues in food (FVA, 2021).

In this study, GC-ECD is used for the separation, identification and quantification of pyrethroid, organophosphate and organochlorine pesticide residues in flour.

The QuEChERS (Quick Easy Cheap Effective Rugged and Safe multiresidue) method provides satisfactory results for a wide range of pesticides (Anastassiades et al., 2003). QuEChERS is a flexible method that can be easily modified based on the analyte properties, matrix composition and available equipment (Cho et al., 2013; Dórea et al., 2004). It involves two primary steps. The first step comprises liquid-liquid extraction with a 'salting out' process adding magnesium sulfate and sodium chloride to acetonitrile, which helps to separate the analytes from the matrix. The second step is a clean-up phase by dispersive solid-phase extraction (dSPE) with Primary Secondary Amine (PSA) and magnesium sulfate, which removes matrix compounds (such as fats, proteins, and chlorophyll) before chromatographic analysis (Saini et al., 2016).

The aim of this research was to develop and validate a new simple to use GC-ECD method for quantification of 26 pesticide residues in flour matrix according to SANTE guideline (SANTE, 2021). The various parameters that were studied for the development and validation of the method were linearity, precision, limit of detection (*LOD*), limit of quantification (*LOQ*), accuracy and measurement uncertainty (*MU*). Method validation is an essential technique for ensuring the reliability and accuracy of the analytical method.

Material and methods

The flour extraction procedure is in accordance with the standard - MKC EN 15662:2020 (ISRSM, 2020). This European Standard provides a method for the analysis of pesticide residues in foods of plant origin, such as fruits (including dried fruits), vegetables, cereals and other processed products thereof using GC, GC-MS and/or LC-MS/MS followed by Acetonitrile extraction and cleanup by dispersive SPE QuEChERS method.

Chemicals and standards

The following chemicals and standards were used: Acetonitrile (Carlo Erba, Pestipur-for pesticide analysis, ≥99.9%), Magnesium sulfate (Carlo Erba, pure for anhydrification, ≥99.9%), Sodium chloride (SRL, extrapure, ≥99.9%), Trisodium citrate dihydrate (SRL, extrapure, ≥99%), Sodium citrate dibasic sesquihydrate (Carlo Erba, according to pharmacopoeia, ≥98%), Formic acid (Carlo Erba, ≥99% for LC/MS), Certified reference material (CRM) pesticide stock standard - 25 components (CPA Chem) and Certified reference material (CRM) Hexachlorobenzene (CPA Chem). Certified reference materials (CRM) are a critical part of analytical chemistry as they will ensure analytical results are accurate and can be measured reliably.

Methods

Gas chromatography with electron capture detector (GC-ECD), model Agilent 8890 GC System equipped with an auto-sampler was used in this study. For efficient chromatographic separation and quantification of pesticides, a capillary column HP-5 (30 m × 0,25 mm × 0,25 μm), Agilent 19091J-433 was used. In order to achieve optimal separation and sensitivity of pesticides, the column, injector, and detector temperatures were maintained at 100, 250, and 300 °C, respectively. Nitrogen (N_2) as a carrier gas was set at a flow rate of 1 mL/min. GC was operated in splitless mode. Oven program values are given in Table 1.

Sample preparation

In the first step, 5 ± 0.5 g of a representative homogenized sample was weighed into a 50 mL tube, with the sample homogenized using a mortar and pestle. Then, 10 mL of redistilled water was added and the mixture was shaken vigorously. Then 10 mL of acetonitrile was added and mixed vigorously again. After that, 4 g of Magnesium sulfate, 1 g of Sodium chloride, 1 g of Trisodium citrate dihydrate and 0.5 g of Sodium sitrate dibasic sesquihydrate are added and mixed vigorously immediately. The mixture was then subjected to centrifugation at a speed of > 3000 rpm for 5 minutes.

8 mL of the extract was trasfered to a clean container and place the extract in the freezer for 2 hours to allow for the complete separation of the flour matrix components from the solvent.

In the second phase, the sample was purified by transferring 6 mL of the separated extract into a purification tube with 150 mg of PSA and 900 mg of $MgSO_4$ and centrifuged for 5 min. at a speed > 3000 rpm. To stabilize the extract, it was transferred to a vial and acidified with 5% formic acid in acetonitrile (10 µL of formic acid per 1 mL of extract).

Results and discussion

Gas chromatography with electron capture detector (GC-ECD) is an analytical technique frequently used for determination of pesticide residues. GC-ECD has good selectivity, linearity and sensitivity in the parts per billion (ppb) concentration range as main advantages. An internal standard was added to a known portion of the matrix in order to assess the correct performance of the testing procedure (Veiga-del-Baño et al., 2024). Method validation was performed according to SANTE guideline (SANTE, 2021), where parameters such as precision, accuracy, limit of detection (*LOD*), limit of quantification (*LOQ*), and linearity were evaluated. The method performance was fully validated according to the SANTE guideline (SANTE, 2021), with successful separation of the pesticides. Figure 1 shows a chromatogram of the spiked sample, where the distinct peaks correspond to the separation of individual pesticides tested in the study.

Method validation

A mixture of standard solutions containing a total of 26 pesticides, including Trifluralin, alpha HCH, HCB, beta HCH and others was added to the homogenized representative sample, with the concentration of the pesticides in the test sample for analysis ranging from 0.005 – 0.2 mg/L (sample equivalent to 0.01 – 0.4 mg/kg).

Linearity

Linearity was assessed using solvent and matrix matched standards (MMS), with calibration curves created ranging from 0.005; 0.01; 0.02; 0.04; 0.1; 0.2 mg/L obtaining a correlation coefficient that must meet or exceed $R^2 \geq 0.99$. The calibration curve demonstrated excellent linearity, with an R^2 value of 0.999, which indicates a very strong correlation between the concentration of pesticides and the response of the detector.

Repeatability

The repeatability of the method was assessed by calculating the relative standard deviation (*RSD*) for six measurements at two concentration levels, 0.005 mg/L and 0.04 mg/L. *RSD* is calculated using the formula: *RSD (%) = (SD / Xsr) * 100*, where *SD* is the standard deviation and *Xsr* is the mean concentration. Table 3 shows the repeatability *(RSD %)* for pesticides in a representative flour sample at the lowest concentration range of 0.005 mg/L, while Table 4 presents the *RSD* values at the medium concentration range of 0.04 mg/L.

The repeatability *(RSD %)* of the method was found to be satisfactory, with values falling within the SANTE guideline limit of ≤ 20% (SANTE, 2021).

Detection limits and quantification limits

The detection limit (*LOD*) and quantification limit (*LOQ*) were calculated using the following formulas: *LOD = 3.3 * σ / S* and *LOQ = 10 * σ / S*, where σ represents the standard deviation of the response and S is the slope of the calibration curve (EMA/CHMP/ICH/82072/2006, 2023; Wenzl et al., 2016). Table 5 provides detection (*LOD*) and quantification (*LOQ*) limits for each pesticide.

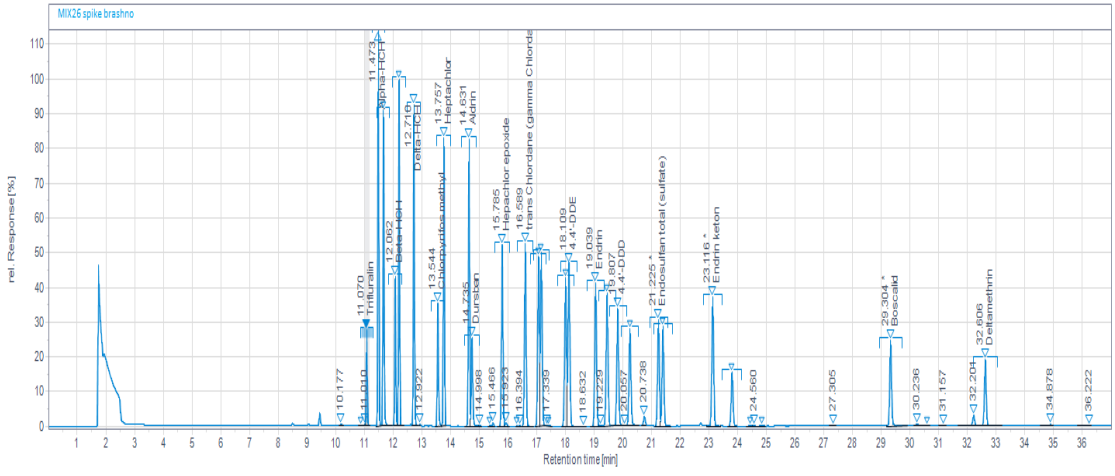

Fig. 1. Chromatogram obtained from spiked sample.

Table 2. List of 26 standard pesticide mixture showing retention time (RT), correlation coefficient (R^2) and calibration curves

Pesticides	R.T (min.)	R^2	Calibration curves
Trifluralin	11.067	0.99983	129400.7930 x -85.9190
alpha HCH	11.469	0.99977	476070.1101 x -911.2523
HCB	11.647	0.99981	434922.0783x+221.6561
beta HCH	12.057	0.99984	189140.7184 x +61.0342
gamma HCH	12.192	0.99949	392541.2975x-951.8057
delta HCH	12.704	0.99943	322618.7981x-853.8966
Chlorpyriphos methyl	13.537	0.99828	195869.4775x+740.0601
Heptachlor	13.753	0.99937	387607.8120x-834.1198
Aldrin	14.627	0.99958	502765.6111x-1077.2787
Dursban	14.727	0.99987	167530.7816x+161.8226
Heptachlor epoxide	15.782	0.99965	360792.2252x-601.0858
trans-Chlordane (gamma Chlordane)	16.584	0.99977	396103.0307x-630.9329
alpha Endosulfan	17.048	0.9999	385371.2400x-565.1596
cis-Chlordane (alpha Chlordane)	17.144	0.99976	378942.2834x-700.955
Dieldrin	17.982	0.99978	385969.9278x-819.0620
4.4'-DDE	18.105	0.9998	404845.1364x-829.5320
Endrin	19.035	0.99961	381515.6994x-817.4363
beta Endosulfan	19.431	0.99951	325562.1131x-767.2766
4,4'-DDD	19.800	0.99921	376717.0898x-1021.8495
Endrin aldehyde	20.227	0.99968	105446.5067x-215.0291
Endosulfan total	21.217	0.99988	16927.4672x-104.7942
4.4'-DDT	21.378	0.99947	110988.5834x-272.5193
Endrin ketone	23.110	0.99876	224375.4496x-502.6162
Methoxychlor (DMTD)	23.784	0.99974	89510.1270x-180.8050
Boscalid	29.303	0.99951	236464.2992x-663.0143
Deltamethrin	32.599	0.99959	55609.7964x-136.1222

Table 3. RSD (%) for pesticides in a representative flour sample in the lowest concentration range (0.005 mg/L)

Pesticides	Xsr (mg/L)	SD	RSD (%)
Trifluralin	0.0052	0.00040	7.710
alpha HCH	0.0050	0.00036	7.155
HCB	0.0053	0.00030	5.597
beta HCH	0.0055	0.00023	4.303
gamma HCH	0.0052	0.00038	7.215
delta HCH	0.0053	0.00040	7.610
Chlorpyriphos methyl	0.0055	0.00034	6.222
Heptachlor	0.0052	0.00056	10.857
Aldrin	0.0052	0.00041	7.896
Dursban	0.0049	0.00045	9.215
Heptachlor epoxide	0.0052	0.00047	9.169
trans-Chlordane (gamma Chlordane)	0.0053	0.00034	6.541
alpha Endosulfan	0.0049	0.00017	3.527
cis-Chlordane (alpha Chlordane)	0.0055	0.00029	5.338
Dieldrin	0.0049	0.00035	7.186
4.4'-DDE	0.0053	0.00026	4.996
Endrin	0.0052	0.00042	8.132
beta Endosulfan	0.0054	0.00031	5.853
4,4’-DDD	0.0049	0.00016	3.388
Endrin aldehyde	0.0052	0.00037	7.092
Endosulfan total	0.0054	0.00029	5.403
4.4'-DDT	0.0057	0.00024	4.225
Endrin ketone	0.0055	0.00026	4.814
Methoxychlor (DMTD)	0.0053	0.00059	11.285
Boscalid	0.0047	0.00031	6.593
Deltamethrin	0.0049	0.00048	9.899

Table 4. RSD (%) for pesticides in a representative flour sample in the mid-concentration range (0.04 mg/L)

Pesticides	Xsr (mg/L)	SD	RSD (%)
Trifluralin	0.041	0.0025	6.043
alpha HCH	0.042	0.0017	4.027
HCB	0.041	0.0020	4.804
beta HCH	0.041	0.0023	5.634
gamma HCH	0.043	0.0015	3.392
delta HCH	0.044	0.0031	7.011
Chlorpyriphos methyl	0.037	0.0038	10.034
Heptachlor	0.043	0.0015	3.400
Aldrin	0.038	0.0014	3.556
Dursban	0.041	0.0012	3.027
Heptachlor epoxide	0.040	0.0027	6.646
trans-Chlordane (gamma Chlordane)	0.041	0.0042	10.293
alpha Endosulfan	0.040	0.0018	4.330
cis-Chlordane (alpha Chlordane)	0.040	0.0020	5.093
Dieldrin	0.038	0.0016	4.144
4.4'-DDE	0.040	0.0027	6.641
Endrin	0.040	0.0027	6.816
beta Endosulfan	0.042	0.0035	8.377
4,4’-DDD	0.037	0.0025	6.819
Endrin aldehyde	0.036	0.0049	13.485
Endosulfan total	0.043	0.0022	5.040
4.4'-DDT	0.048	0.0063	13.118
Endrin ketone	0.048	0.0052	10.951
Methoxychlor (DMTD)	0.048	0.0059	12.228
Boscalid	0.038	0.0022	5.670
Deltamethrin	0.045	0.0028	6.243

Table 5. Method detection limits and quantification limits

Pesticides	LOD (mg/кg)	LOQ (mg/кg)
Trifluralin	0.0038	0.0126
alpha HCH	0.0040	0.0134
HCB	0.0032	0.0107
beta HCH	0.0034	0.0114
gamma HCH	0.0033	0.0111
delta HCH	0.0030	0.0101
Chlorpyriphos methyl	0.0038	0.0128
Heptachlor	0.0030	0.0098
Aldrin	0.0038	0.0127
Dursban	0.0027	0.0091
Heptachlor epoxide	0.0036	0.0120
trans-Chlordane (gamma Chlordane)	0.0040	0.0134
alpha Endosulfan	0.0031	0.0103
cis-Chlordane (alpha Chlordane)	0.0034	0.0113
Dieldrin	0.0040	0.0134
4.4'-DDE	0.0045	0.0150
Endrin	0.0041	0.0138
beta Endosulfan	0.0024	0.0080
4,4’-DDD	0.0046	0.0152
Endrin aldehyde	0.0033	0.0111
Endosulfan total	0.0055	0.0183
4.4'-DDT	0.0055	0.0182
Endrin ketone	0.0022	0.0075
Methoxychlor (DMTD)	0.0019	0.0065
Boscalid	0.0083	0.0276
Deltamethrin	0.0080	0.0268

Table 6. Accuracy of the method

Pesticides	REC (%) 0.005(mg/L)	REC (%) 0.04(mg/L)
Trifluralin	103.333	102.750
alpha HCH	100.000	105.458
HCB	106.000	102.333
beta HCH	109.000	102.500
gamma HCH	104.333	107.500
delta HCH	105.667	110.917
Chlorpyriphos methyl	109.000	93.458
Heptachlor	103.333	107.833
Aldrin	104.333	95.292
Dursban	98.667	101.375
Heptachlor epoxide	103.000	99.833
trans-Chlordane (gamma Chlordane)	105.333	103.167
alpha Endosulfan	97.667	101.125
cis-Chlordane (alpha Chlordane)	109.667	100.500
Dieldrin	98.000	95.958
4.4'-DDE	105.667	100.292
Endrin	103.667	99.750
beta Endosulfan	107.333	104.917
4,4'-DDD	97.000	92.750
Endrin aldehyde	104.000	91.125
Endosulfan total	108.333	107.458
4.4'-DDT	114.667	119.667
Endrin ketone	109.667	119.208
Methoxychlor (DMTD)	105.000	120.042
Boscalid	93.333	94.833
Deltamethrin	97.667	112.917

Accuracy

Accuracy of the method was evaluated by calculating the recoveries *(*REC *%)* of known amounts of analyte added to the blank flour matrix. The recoveries were calculated using the formula: *REC (%) = (Cs / C) * 100*, where *Cs* is the measured concentration of the analyte in the spiked sample and *C* is the concentration added to the sample. According to SANTE/11312/2021 guidelines, recovery values should be between 70 and 120%. The obtained results showing adequate accuracy at two spike levels, 0.005 mg/L and 0.04 mg/L, are summarized in Table 6.

Uncertainty

Measurement uncertainty (*MU*) is inherently associated with each measurement. Intralaboratory validation data were used to estimate the standard uncertainty and then expressed at the 95% level of confidence. Three significant sources: repeatability (precision), recovery and uncertainty of the standard were considered as the main contributors to measurement uncertainty (Ellison et al., 2000; Eppe et al., 2017). The combined standard uncertainty of each pesticide was estimated using the following formula: $U(C) = \sqrt{(U^2_{rep} + U^2_{bias} + U^2_{st})}$.

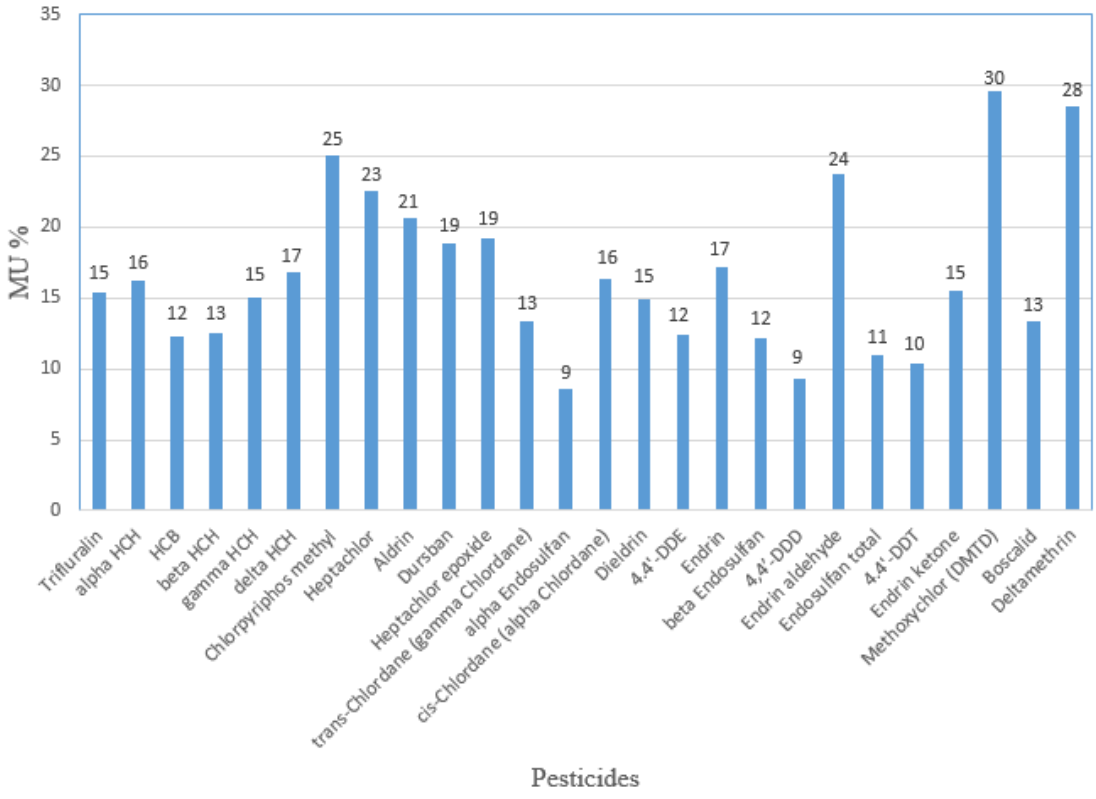

Fig. 2. Measurement uncertainty MU (%) for each pesticide.

The uncertainty due to bias (*Ubias*) was estimated using recovery *(REC %)*, which represents how accurately the method recovers known amounts of analyte from a sample. The repeatability uncertainty (*Urep*) was derived from the relative standard deviation *(RSD %)*, while the uncertainty of the standard (*Ust*) depends on the purity of the certified reference material used. The combined standard uncertainty *U(C)* is calculated by summing the squared values of uncertainty sources: repeatability (*Urep*), bias (*Ubias*) and the uncertainty related to the standard (*Ust*). The expanded uncertainty (*U*) is obtained by multiplying the combined uncertainty by a coverage factor (*k=2*), which provides a 95% confidence level using the formula: $U = 2 * U(C)$.

Figure 2 shows the measurement uncertainty (*MU* (%)) for each pesticide. The values for each pesticide were calculated based on the combined uncertainties, with the expanded uncertainty satisfying the acceptance criterion of less than 50% as required by SANTE/11312/2021 guidelines.

Conclusion

The developed method demonstrates a robust and reliable approach for pesticide residue quantification in flour. The parameters of the analysis instrument were also optimized accordingly for the analysis of pesticide residues in the flour matrix. The method allows quantification at typical MRL concentrations of 0.01 mg/kg in accordance with the guidelines of Document Nº SANTE/11312/2021 for linearity, precision, accuracy, *LOD*, *LOQ* and measurement uncertainty i.e. the obtained data were within the acceptable range of the validation limit. It is internationally recognized that a developed method should necessarily be validated as these validation methods also show the qualification and competency of the analytical laboratory. Future studies should focus on expanding the method's scope to include a broader range of pesticides and other cereal matrices.

This method can be further expanded by incorporating inter-laboratory validation data, broadening the scope by including additional pesticides and testing the effectiveness of the method on other cereals such as oats, barley, corn, etc.

References

Akashe, M.M., Pawade, U.V., Nikam A.V., 2018. Classification of Pesticides A Review, Int. J. Res. Ayurveda Pharm. 9(4). doi: 10.7897/2277-4343.094131

Anastassiades, M., Lehotay, S.J., Stajnbaher, D., Schenck, F.J., 2003. 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. 86(2), 412-431. doi: 10.1093/jaoac/86.2.412

Cho, T.H., Park, Y.H., Park, H.W., Hwang, L.H., 2013. Evaluation of QuEChERS Method for Determination of pesticide Residues Using GC/NPD and GC/ECD. Korean. J. Pestic. Sci. 17(1), 65-71. Doi: 10.7585/kjps.2013.17.1.65

Dórea, H.S., Sobrinho, L.L., 2004, Analysis of pesticide residues in rice using Matrix Solid-Phase Dispersion (MSPD), J. Braz. Chem. Soc. 15(5). doi:10.1590/S0103-50532004000500013

Ellison, S., Roesslein, M., Williams, A., Berglund, M., 2000. Eurachem/CITAC Guide CG4. Quantifying Measurement Uncertainty in Analytical Measurement, 2nd edition. 2000. Available from: https://www.researchgate.net/publication/236884859_EurachemCITAC_Guide_CG4_Quantifying_Measurement_Uncertainty_in_Analytical_Measurement

EMA/CHMP/ICH/82072/2006, 2023. Committee for Medicinal Products for Human Use ICH Q2(R2) guideline on validation of analytical procedures. Available from: https://www.ema.europa.eu/en/documents/scientific-guideline/ich-q2r2-guideline-validation-analytical-procedures-step-5-revision-1_en.pdf

Eppe, G., Schaechtele, A., Haedrich, J., Fernandes, A., 2017. Guidance Document on Measurement Uncertainty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry. 2017. doi: 10.13140/RG.2.2.35446.09285.

FVA (Food and Veterinary Agency), 2021. Правилник за општите и посебните барања за безбедност на храната во однос на максимално дозволените нивоа на резидуи од пестициди во или врз храната од растително и од животинско потекло. Available from: https://fva.gov.mk/mk/zakon-bezbednost-khranata/pravilnik/2021-95-pravilnik-opshtite-i-posebnite-barana-bezbednost-khranata-odnos-maksimalno-dozvolenite-nivoa-rezidui

Garud, A., Pawar, S., Patil, M.S., Kale, S.R., Patil, S., 2024. A Scientific Review of Pesticides: Classification, Toxicity, Health Effects, Sustainability, and Environmental Impact. The Cureus Journal of Medical Science. 16(8). doi: 10.7759/cureus.67945

ISRSM (Standardization Institute of the Republic of North Macedonia). MKC EN 15662, 2020. Foods of plant origin - Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE - Modular QuEChERS-method 2020. Available from: https://isrsm.gov.mk/mk/project/show/isrsm:proj:68188

Kaur, R., Mavi, G.K., Raghav, S., 2019. Pesticides Classification and Its Impact on Environment. Int. J. Curr. Microbiol. App. Sci. 8(3), 1889-1897. doi:10.20546/ijcmas.2019.803.224

Rezaei, M., Shariatifar, N., Shoeibi, S., Ahmadi, M.A., Khaniki, G.J., 2016. Determination of Residue from 58 Pesticides in the Wheat Flour Consumed in Tehran, Iran by GC/MS. Iran J. Pharm. Res. 16(3), 1048-1058. doi: 29201093

Saini, M., Mishra, S., Alam, S., Thakur, L.K., Singh, O.P., Raza, S., 2016. Method Development and Validation of Multiclass Pesticide Residues and Metabolites in Wheat by GC-ECD and GC-MS. Asian Journal of Research in Chemistry 9(1), 13. doi: 10.5958/0974-4150.2016.00003.1

SANTE/11312/2021, 2021. Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed, European Commission, 2021. Available from: https://food.ec.europa.eu/system/files/2023-11/pesticides_mrl_guidelines_wrkdoc_2021-11312.pdf

Veiga-del-Baño, J.M., Oliva, J., Cámara, M., Martinez, P.A., Motas, M., 2024. Matrix-Matched Calibration for the Quantitative Analysis of Pesticides in Pepper and Wheat Flour: Selection of the Best Calibration Model. Agriculture 14(7), 1014. doi:10.3390/agriculture14071014

Wenzl, T., Haedrich, J., Schaechtele, A., Robouch, P., Stroka, J., 2016. Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food, EUR 28099 EN, Publications Office of the European Union, Luxembourg, 2016, ISBN 978-92-79-61768-3. doi:10.2787/8931

WHO (World Health Organization), 2022. Pesticide residues in food. Geneva. Available from: https://www.who.int/news-room/fact-sheets/detail/pesticide-residues-in-food

Zanella, R., Damian, O., Amaral Friggi, C., Leonardo, M., Bohrer, M., 2012. An Overview About Recent Advances in Sample Preparation Techniques for Pesticide Residues Analysis in Cereals and Feedstuffs. Pesticides - Recent Trends in Pesticide Residue Assay. 2012. doi:10.5772/48752

Резиме

Развој и валидација на GC-ECD метода за детекција на остатоци од пестициди во брашно

Маргарита Тимофејева[1*], Анита Најденкоска[1], Билјана Бауер[2]

[1]Институт за јавно здравје, Одделение за контаминенти и екотоксикологија при Одделот за хемиско и радиолошко тестирање, ул. 50-та Дивизија бр. 6, 1000 Скопје, Република Северна Македонија
[2]Институт за фармакогнозија, Фармацевтски Факултет, Универзитет ,,Св. Кирил и Методиј", Мајка Тереза 47, 1000 Скопје, Република Северна Македонија

Клучни зборови: валидација, GC-ECD, резидуи од пестициди, QuEChERS

Оваа студија имаше за цел да оптимизира и да валидира брз, сигурен и прецизен метод за квантитативно определување на остатоци од пестициди во матрицата на брашно. Пестицидите беа екстрахирани со употреба на QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) техника за подготовка на примероци за анализа на остатоци од пестициди во матрици на храна, проследена со квантитативно определување користејќи GC-ECD.

Методот беше валидиран за 26 пестициди на шест калибрациони нивоа (0,005; 0,01; 0,02; 0,04; 0,1; 0,2 mg/L), користејќи матрица од брашно. Методот е точен и прецизен, што може да се заклучи од параметрите за валидација: линеарност, прецизност, лимит на детекција (*LOD*), лимит на квантификација (*LOQ*), точност и мерна неодреденост (*MU*). Кривите на калибрација за сите пестициди покажаа одлична линеарност, со коефициенти на корелација (R^2) кои надминуваат 0,99. Лимитот на детекција (*LOD*) на пестицидите во матрицата вариираше од 0,0019 - 0,0083 mg/kg, додека лимитот на квантификација (*LOQ*) од 0,0065 - 0,0276 mg/kg. Методот покажа задоволителна точност и прецизност, со вредности на процентот на обновено количество во прифатлив ранг од 70 - 120% и релативна стандардна девијација (*RSD%)* под 20% за сите тестирани пестициди. Мерната неодреденост исто така беше проценета врз основа на податоците за валидација, а добиените вредности беа под стандардната граница од 50%.

Валидираниот метод се покажа како доверлив и соодветен за рутинска анализа на резидуи од пестициди во матрица на брашно.