Original Article

ASSESSMENT OF MERCURY LEVEL IN LOCAL POLISH AND IMPORTED HONEYS WITH USE OF DIRECT MERCURY ANALYZER

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Abstract

Mercury is a toxic metal which causes several serious health effects including kidney damage, anxiety depression and peripheral neuropathy, and because of its high volatility assessment in solid samples is problematic. In this study, forty-five samples of local Polish and imported honeys were analyzed by Direct Mercury Analyzer (DMA) for the determination of mercury content. The honey contamination was assessed regarding honey variety and country of origin. Hg level in all tested samples was found to not exceed the 0.81 µg/kg which was below 10% of the applicable law UE limit. Moreover, no statistically significant differences were found in the variety or geographical origin. The study concluded that the Direct Mercury Analyzer (DMA) instrument proved to be an excellent tool for screening mercury in honeys because of its simplicity, rapidity, low detection limit, accuracy and precision and no sample preparation.

Keywords: DMA, honey, mercury

INTRODUCTION

Honey is made by either bees from the nectar of plants or the secretions of plant-sucking insects foraging on trees. The raw material for honey production accumulates various chemical components, including heavy metals, and their level is closely related to their content, primarily in soil, but also in water and air (Murashova et al., 2019; Yaqub et al., 2020; Fisher et al., 2022). Consequently, when bees collect honeyflow, they retain most of the pollutants in their bodies, acting as a biofilter, but the remainder passes into the honey (Dżugan et al., 2018; Borsuk et al., 2021). As a result, not only bees, but also honey is used to monitor the cleanliness of the environment (Dżugan et al., 2017; Murashova et al., 2019; Yaqub et al., 2020).

Mercury (Hg), a heavy metal included in the top ten pollutants, has been rarely studied in honeys, but its occurrence can be dangerous for bees and consumers. Mercury cations are toxic, mainly due to their highly oxidizing nature and interaction with sulfhydryl groups which are a component of proteins including cell membranes proteins and enzymes. Such an interaction causes the loss of cell-membrane integrity and inhibition of most enzymatic processes in the body (Vieira et al., 2014). Mercury induces changes in cellular mitochondria, disrupts oxidative phosphorylation and electron transport, and leads to lipid peroxidation and cell death. It also reduces the concentration of glutathione, cysteine and homocysteine, causing oxidative stress, and lowers the activity of ion pumps and ATP synthesis, causing disturbances in ions distribution in the cellular and extracellular space (Brodziak-Dopierała et al., 2021). Methylmercury, the organic form, readily

crosses the blood-brain barrier through large transporters of neutral L-type amino acids and, due to its high affinity for lipid structures, accumulates in the brain where leads to neurotoxicity (Creed et al., 2019). Chronic mercury poisoning leads to various neurodegenerative diseases and death (Ferreira et al., 2015; Fischer et al., 2022), and for this reason it is important to control its concentration in foodstuff, including honey.

Hg content in honey is mostly determined by the use of the vapor generation technique coupled to atomic absorption spectrometry (CV AAS), fluorescence spectrometry (CV AFS), inductively coupled plasma optical emission spectrometry (CV ICP-OES) and inductively coupled plasma mass spectrometry (CV ICP-MS) (Ferreira et al., 2015). However, these techniques are solution-based, which means that if the sample is solid it has to be digested before it is introduced to the instrument, and because of the high volatility of mercury, this operation leads to a loss of analyte and poor recoveries if the sample is not digested correctly. The solution is to carry out measurements through direct mercury analysis, a technique for determining total mercury directly in solid, liquid and gas samples, using the principle of thermal decomposition, amalgamation, and atomic absorption (Ferreira et al., 2015). Thus, in this study the usefulness of the direct method of Hg determination with the Direct Mercury Analyzer (DMA) was checked using honeys of different botanical and geographical origin.

MATERIAL AND METHODS

Material

The research material consisting of forty-five honey samples (multifloral (22), linden (7), buckwheat (4), acacia (3), coniferous honeydew (3), rape (1), heather (1), leafy honeydew (1), thyme (1), eucalyptus (1) and coriander (1)) were available on the South-Eastern Polish market and originated from both local (n=20) and foreign (n=25) apiaries. Among the imported honey, the European (n=12) and blends from Europe and outside the Europe (n=13) were tested. All samples were packaged in tightly closed glass jars and opened directly before analysis. Depending on the variety, the honeys had a different color, from light yellow to brown, and their consistency was liquid or semi-liquid.

Methods

The mercury content in the honey samples was tested with the Direct Mercury Analyzer (DMA-80, Milestone Srl, Sorisole, BG, Italy) equipped with a built-in 40-position sample feeder in accordance with the internal procedure of the Food Hygiene and Nutrition Laboratory of the Provincial Sanitary and Epidemiological Station in Rzeszów (PB/HŻ/R-28 test procedure). The trueness of obtained results was confirmed by good z-scores (for vegetable product - 0.2 and for drink 0.0) in interlaboratory proficiency tests organized by the National Reference Laboratory in accordance with the principles contained in "The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories" (Thompson et al., 2006). Such a technique is based on the quantitative determination of the total mercury content in honey in the range from 0.0001 mg/kg (method limit of quantification) to 1.00 mg/kg. The detection limit was 0.00009 mg/kg, and prior the analysis, the new specific nickel cuvettes (boats) were roasted in a muffle furnace at 850°C and allowed to cool down. The boats were then cleaned in a DMA apparatus for sixty seconds according to the clean procedure, until the absorbance was ≤0.003. The Hg measurements were made using 100 mg of sample weighed directly into the cuvette, which was automatically transported to the oven where the sample was first dried at 200°C for 70 s and then thermally decomposed under a continuous oxygen flow under the pressure 4 bar (60 psi) with a speed of about 200 ml/min at a temperature of 650°C for three minutes. All mercury species are reduced to elemental mercury (Hg^o) and then carried along with reaction gases to a gold amalgamator where the mercury is selectively trapped. All non-mercury vapors and decomposition products are flushed from the system by the continuous flow of gas. The amalgamator is subsequently heated and releases for twelve seconds all trapped mercury to the double-beam fixed wavelength atomic absorption spectrophotometer. Finally, the

absorbance is measured at 253.7 nm as a function of mercury content within the next thirty seconds. The method was validated and used for the detection of Hg in honey and other foodstuff (Tab. S1). The following parameters were estimated: limit of detection (LOD), limit of quantification (LOQ), repeatability (precision), correctness (accuracy), selectivity, linearity (linear correlation coefficient), and the uncertainty of the method for determining the mercury content in foodstuffs as shown in Tab. S1.

Calibration was performed against an external standard Certipur® Certified Reference Material - Mercury ICP Standard 1000 mg/l Hg. According to the recommended calibration protocol, the DMA-80 device operates in three concentration ranges, so the corresponding calibration curves have been determined in three ranges: low (0-10 ng Hg), medium (10-20 ng Hg) and high (20-1000 ng Hg). For the performed curves, the R^2 coefficient was calculated and its values ranged from 0.9991 to 0.9999.

The limit of detection (LOD) was determined as the sum of the mean Hg concentration in blank samples - tap water (C_{avg}) and standard deviations from three measurements (SD), while the limit of quantification (LOQ) was C_{avg} +6 SD. In order to assess the repeatability of the method (precision under repeatability conditions), twenty individual analyses of mercury content in a selected food sample were performed (naturally contaminated fish Hg at the level of 0.0636 mg/kg).

The correctness of the test method, i.e. the degree of agreement between the mean value obtained from a series of test results and the adopted reference value, was determined through a full analytical procedure for samples of selected matrices enriched with a mixture of Hg standards at two or three levels of concentration. Six determinations were made for each concentration level. In addition, the Hg content in the matrix was determined in selected matrices without enrichment, in two repetitions. Empty boat analyzes were performed each time to check the purity of the system. Reference materials Squid T07319QC (Assigned Value Mercury (total) 17.0 µg/kg) and Powdered Rice T07419QC (Assigned Value Mercury (total) 106 µg/kg) - Fapas QC Material Fera Science Ltd (York, UK) were also analysed in order to control the correctness of the validated mercury determination method in selected foodstuffs. Moreover, fish muscle ERM® - BB422 (Certified value and uncertainty Hg: 0.601±0.030 mg/kg according to the European Commission Joint Research Center (JRC) Institute for Reference Materials and Measurements (Geel, Belgium)) was also tested.

The selectivity of the method was obtained through appropriate preparation of the food sample (mixing, grinding, homogenization) and mineralization after drying in the apparatus at 650˚C. Moreover, the use of an appropriate spectrometer detector and a low-pressure mercury lamp as a radiation source (wavelength 253.65 nm) limits an interference from other components in the mixture.

The uncertainty of the validated test method was estimated taking into account the following elements: determination of the measurand, identification and quantification of sources of uncertainty, calculation of the combined standard and expanded uncertainties. The calculations were performed in accordance with the rules for assessing and expressing uncertainty in quantitative chemical analyzes contained in the Eurachem/CITAC guide, Quantifying Uncertainty in Analytical Measurement (Ellison & Williams, 2012), and in the guidelines by Konieczka and Namieśnik (2009).

Statistical analysis

The statistical analysis of the obtained test results was carried out with the use of the Statistica ver. 13.0 (Statsoft, Krakow, Poland) and Microsoft Excel. The results were analyzed as the arithmetic mean of three determinations from each honey sample. Statistically significant differences between individual groups were assessed on the basis of one-way analysis of variance followed by Tukey's test at the significance level $p<0.05$).

RESULTS

The results of the analysis of Hg content in the tested honey samples measured with a DMA-80 automatic mercury analyzer (Direct Mercury

Table 1.

Mercury content in individual honey samples

No. sample	Honey variety/country of origin	Mean± SD [µg/kg]	No. sample	Honey variety	Mean± SD [µg/kg]
1	MT/Poland	n.d.	24	L/Europe	0.1157±0.0817
2	MT/Poland	n.d.	25	L/Europe	n.d.
3	MT/Poland	0.1273±0.0734	26	L/Europe	0.1112±0.0786
4	MT/Europe and abroad	n.d.	27	L/Europe and abroad	0.1159±0.0818
5	MT/Europe and abroad	n.d.	28	L/Poland	0.1041±0.0735
6	MT/Europe	0.2636±0.1520	29	L/Poland	n.d.
7	MT/Poland	n.d.	30	B/Poland	n.d.
8	MT/Poland	0.2743±0.1582	31	B/Poland	0.2031±0.1434
9	MT/Europe and abroad	0.1136±0.0655	32	B/Europe and abroad	n.d.
10	MT/Ukraine and Bulgaria	n.d.	33	B/Poland	n.d.
11	MT/Poland	n.d.	34	A/Ukraine and Italy	n.d.
12	MT/Poland	n.d.	35	A/Poland	0.1309±0.0925
13	MT/Poland	n.d.	36	A/Europe and abroad	0.5383±0.3802
14	MT/Europe and abroad	0.2958±0.2088	37	CH/Italy	0.8119±0.4683
15	MT/Europe and abroad	n.d.	38	CH/Poland	0.5773±0.3330
16	MT/Europe and abroad	0.1823±0.1287	39	CH/Poland	n.d.
17	MT/Europe and abroad	n.d.	40	R/Poland	0.6731±0.4754
18	MT/Europe and abroad	0.5915±0.4178	41	H/Spain	0.2390±0.1689
19	MT/Poland	0.3263±0.2306	42	LH/Europe	n.d.
20	MT/Europe and abroad	0.1367±0.0966	43	T/Spain	0.5214±0.3007
21	MT/Ukraine	n.d.	44	E/Uruguay	0.4339±0.2502
22	MT/Poland	n.d.	45	C/Italy	0.3441±0.1985
23	L/Poland	0.1159±0.0818			

n.d. - not detected

MT-multifloral, L-linden, B-buckwheat, A-acacia, CH-coniferous honeydew, R-rape, H-heather, LH-leafy honeydew, T-thyme, E- eucalyptus, C-coriander

Analyzer) are presented in Tab. 1. Among the tested samples in 21 honeys (46%) mercury was not detected. The Hg level in tested honey samples was strongly diversified and ranged from 0.1041 to 0.8119 µg/kg.

The level of mercury contamination of tested samples in terms of honey variety was also observed to highly varied (Fig. 1). Among all tested varieties, coniferous honeydew honey exhibited the highest mercury content (0.463 µg/kg) and buckwheat honey the lowest (0.051 µg/kg on average). However, the observed varietal differences were not statistically significant ($p>0.05$) probably due to a great variation within a single variety.

In the current study, the mercury level was also analyzed taking into account the origin of the sample, and such data clustering was presented in Fig. 2. Honey was divided into Polish (n=20), European (n=12) and blends of honeys from Europe and outside the Europe (n=13). Such grouping of the results showed that honeys from Poland were characterized by the lowest levels of mercury while honeys from European countries (Spain, Italy, Bulgaria, Ukraine) by the highest. Nevertheless, due to the great inner-group

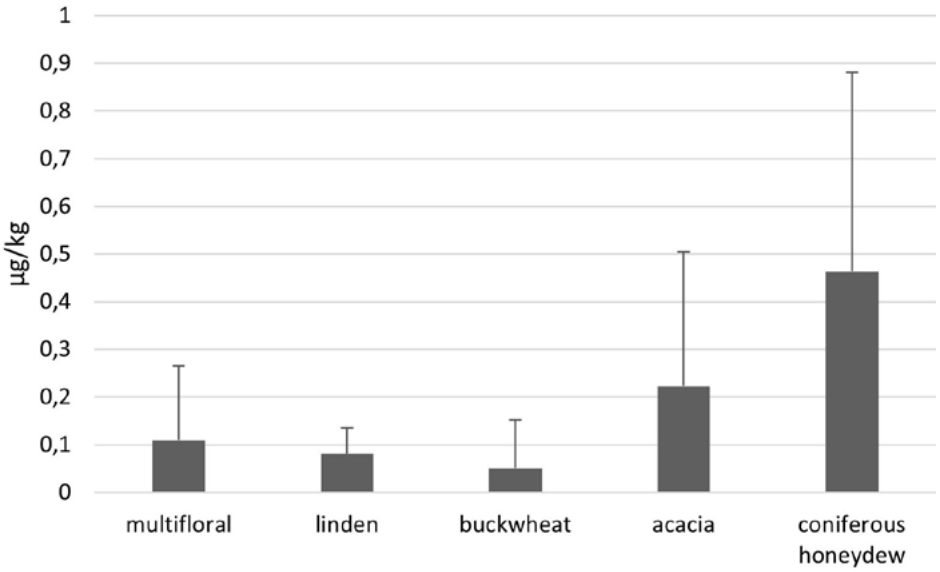

Fig. 1. Mercury content (µg/kg) in individual honey varieties (average ± SD; $p>0.05$).

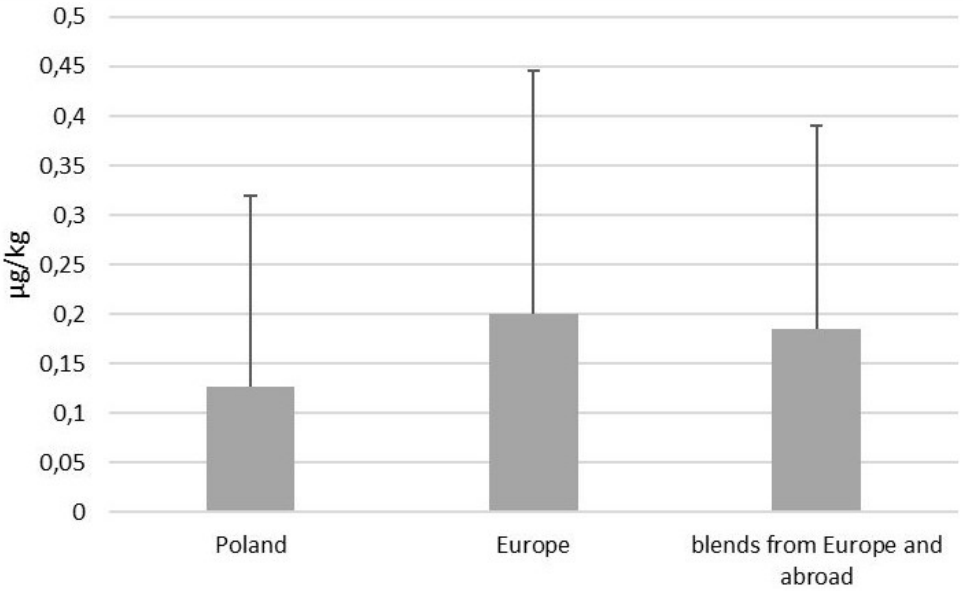

Fig. 2. Mercury content (µg/kg) in samples regarding the origin (average ± SD; $p>0.05$).

variation, no statistically significant differences were found between the groups ($p>0.05$).

DISCUSSION

Hg content in all tested honey samples was far below the maximum residues permitted in obligatory regulation. The EU Commission Regulation 2018/73 from January 16, 2018 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and Council specifies that the maximum mercury content in honey in must be 10 µg/kg. Brodziak-Dopierała et al. (2021) who assessed the Hg content in the Polish honeys using DMA found that for all tested samples the range of changes was 0.02-1.55 µg/kg and the arithmetic mean of all samples, regardless of the variety, was 0.37 µg/kg. These results are in line of our observations, and more importantly in our previous study Hg concentrations in all samples of local honey measured by means of the ICP-OES method were found to be under the detection level (<1 ppm) (Dżugan et al., 2018). However, Madras-Majewska et al. (2014) conducted similar research with ICP-OES and the Hg concentration in honeys from Polish apiaries was on average 0.27 µg/kg, which was similar to the values obtained in the present research.

Brodziak-Dopierała et al. (2021) used DMA and determined similarly to the present study that the Hg highest concentration in Polish honeys was 1.02 µg/kg for honeydew honeys successively 0.52 µg/kg for linden honeys, rape, acacia, multifloral and buckwheat honeys. Nevertheless, the levels measured by these authors were higher than in the present study. Moreover, in contrast to present study, differences in Hg content among individual honey types were statistically significant ($p<0.05$). In our previous research, we found exceptionally high Al levels in honeydew honeys compared to other varieties (Dżugan et al., 2017) and speculate that it is due to its specific origin: the production of honeydew by plant sucking insects, or the specificity of the cellular tree saps from which honeydew comes. However, the Hg values obtained for honeydew honey were still about twenty times lower than the applicable limits.

The Hg contamination of honeys has been reported by foreign authors. Astolfi et al. (2021) tested the mercury level in Italian honeys with the same technique and had values ranging between 0.91 and 3.37 µg/kg. Vieira et al. (2014) compared the usefulness of CV AAS method and DMA in the determination of Hg concentration in Brazilian honeys and found that all thirty-five tested honey samples exhibited Hg concentrations below the LOQ for both methods (2.5 µg/kg for DMA, and 60.0 µg/kg for CV AAS). Toth et al. (2016) studied the Hg content in honeys from eastern Slovakia by AMA mercury analyzer and learnt that honey from the city of Košice had 0.081 µg Hg/kg and honey from the rural areas of Rozhanovce had 0.079 µg Hg/kg. Moreover, no statistically significant differences between the concentration of Hg in honeys from urban and rural areas were found. All these studies confirm that the Hg content in honey measured by DMA is well below the limits and poses no risk to the health of consumers (Fisher et al., 2022).

The above results confirm the usefulness of DMA for Hg determination in honey. The possibility to feed the DMA device a “raw” sample of the tested material significantly improves and speeds up the analysis, as it neither requires a large amount of sample and nor generates high final dilution factors. The cold mercury vapor generated by the pyrolytic decomposition of the sample is captured by the gold sorbent, forming an amalgam. Decomposition products, such as nitrogen or sulfur oxides, halides and others, which may interfere with the absorbance measurement, are previously eliminated on the catalyst. The remaining products of combustion are washed away by the carrier gas (oxygen), and mercury vapor is released into the optical path once by the heating of the amalgamator furnace. This solution allows increased selectivity and removal of possible interference (Vieira et al., 2014; Brodziak-Dopierała et al., 2021).

The main advantage of DMA analysis is the avoidance of a sample preparation. Microwave-assisted digestion is most commonly used for preparing honey for mineral composition determination (Ferreira et al., 2015; Astolfi et al., 2021), but this requires large sample masses

and reagent volumes, often leading to high final dilution factors and a consequent increase in the method detection limits (Astolfi et al., 2020; Grainger et al., 2020). In literature, many studies have quantified Hg concentrations in honey matrices with atomic absorption spectroscopy and inductively coupled plasma-mass (ICP-MS) or optical emission spectrometry (ICP-OES) (Dos et al., 2010; Nawrocka et al., 2016; Oroian et al., 2016; Dżugan et al., 2017; Fisher et al., 2022), while the direct mercury analyzer (DMA) is a rather new method rarely used in honey analysis, despite being fast and requiring little operator input (Vieira et al., 2014; Astolfi et al., 2021; Brodziak-Dopierała et al., 2021).

According to the obtained results, all analyzed honeys contained Hg in an amount not exceeding the applicable mercury standard of 10 µg/kg and posed no risk to the health of consumers. Moreover, no statistically significant differences depending on the variety or geographical origin were found. Finally, it was proved that DMA could be used as a quick, precious, and time-saving technique for Hg determination in honey in contrast to the ICP MS/OES methods which require specific sample preparation through wet mineralization which involves high dilution, generating excessively high levels of quantification.

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REFERENCES

Astolfi, M.L., Conti, M.E., Marconi, E., Massimi, L., Canepari, S. (2020). Effectiveness of different sample treatments for the elemental characterization of bees and beehive products. *Molecules, 25*(18), 4263. https://doi.org/10.3390/molecules25184263

Astolfi, M.L., Conti, M.E., Ristorini, M., Frezzini, M.A., Papi, M., Massimi, L., Canepari, S. (2021). An Analytical Method for the Biomonitoring of Mercury in Bees and Beehive Products by Cold Vapor Atomic Fluorescence Spectrometry. *Molecules, 26*(16), 4878. https://doi.org/10.3390/molecules26164878

Borsuk, G., Sulborska, A., Stawiarz, E., Olszewski, K., Wiącek, D., Ramzi, N., … Jedryczka, M. (2021). Capacity of honeybees to remove heavy metals from nectar and excrete the contaminants from their bodies. *Apidologie, 52*, 1098-1111. https://doi.org/10.1007/s13592-021-00890-6

Brodziak-Dopierała, B., Mendak-Oleś, P., Fischer, A. (2021). Występowanie rtęci w różnych rodzajach miodów. *Medycyna Środowiskowa, 23*(1-4), 39-43. https://doi.org/10.26444/ms/138310 (in Polish)

Creed, J.H., Peeri, N.C., Anic, G.M., Thompson, R.C., Olson, J.J., LaRocca, R.V., … Egan, K.M. (2019). Methylmercury exposure, genetic variation in metabolic enzymes, and the risk of glioma. *Scientific Reports, 9*, 10861. https://doi.org/10.1038/s41598-019-47284-4

Dos, F., Depoi, S., Bentlin, F., Pozebon, D. (2010). Methodology for Hg determination in honey using cloud point extraction and cold vapor-inductively coupled plasma optical emission spectrometry. *Analytical Methods, 2*, 180-185. https://doi.org/10.1039/B9AY00189A

Dżugan, M., Wesołowska, M., Zaguła, G., Kaczmarski, M., Czernicka, M., Puchalski, C. (2018). Honeybees (*Apis mellifera*) as a biological barrier for contamination of honey by environmental toxic metals. Environmental *Monitoring and Assessment, 190*, 101. https://doi.org/10.1007/s10661-018-6474-0

Dżugan, M., Zaguła, G., Wesołowska, M., Sowa, P., Puchalski, C. (2017). Levels of toxic and essential metals in varietal honeys from Podkarpacie. *Journal of Elementology, 22*(3), 1039-1048. https://doi.org/10.5601/jelem.2016.21.4.1298

Ellison, S., & Williams, A. (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, 3, 2012. https://www.eurachem.org/index.php/publications/guides/quam#translations (accessed: 15.06.2019).

EU Commission Regulation 2018/73 of 16 January 2018 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and Council as regards maximum residue levels for mercury compounds in and on certain products.

Ferreira, S.L.C., Lemos, V.A., Silva, L.O.B., Queiroz, A.F.S., Souza, A.S., da Silva, E.G.P., … das Virgens, C.F. (2015). Analytical strategies of sample preparation for the determination of mercury in food matrices - A review. *Microchemical Journal, 121*, 227-236. http://dx.doi.org/10.1016/j.microc.2015.02.012

Fischer, A., Brodziak-Dopierała, B., Bem, J., Ahnert, B. (2022). Analysis of Mercury Concentration in Honey from the Point of View of Human Body Exposure. *Biological Trace Elements Research, 200*, 1095-1103. https://doi.org/10.1007/s12011-021-02744-9

Grainger, M.N.C., Hewitt, N., French, A.D. (2020). Optimized approach for small mass sample preparation and elemental analysis of bees and bee products by inductively coupled plasma mass spectrometry. *Talanta, 214*, 120858. https://doi.org/10.1016/j.talanta.2020.120858

Konieczka, P., Namieśnik, J. (2009). Quality Assurance and Quality Control in the Analytical Chemical Laboratory: A Practical Approach, Boca Raton. London 2009, New York, CRC Press.

Murashova, E.A., Tunikov, G.M., Nefedova, S.A., Karelina, O.A., Byshova, N.G., Serebryakova, O.V. (2019). Major factors determining accumulation of toxic elements by bees and honey products. *International Transaction Journal of Engineering, Management, & Applied Sciences & Technologies, 11*(3), 11A03N. https://doi.org/10.14456/ITJEMAST.2020.54

Nawrocka, A., Durkalec, M., Szkoda, J., Kmiecik, M. (2016). Determination of trace and essential elements in honey by quadrupole inductively coupled plasma-mass spectrometry. *Euroreference, 1*, 52-57.

Oroian, M., Prisacaru, A., Hretcanu, E.C., Stroe, S.G., Leahu, A., Buculei, A. (2016). Heavy Metals Profile in Honey as a Potential Indicator of Botanical and Geographical Origin. *International Journal of Food Properties, 19*(8), 1825-1836. https://doi.org/10.1080/10942912.2015.1107578

Thompson, M., Ellison, S.L.R., Wood, R. (2006). The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories (IUPAC Technical Report). *Pure Applied Chemistry, 78*(1), 145-196.

Toth, T., Kopernicka, M., Sabo, R., Kopernicka, T. (2016). The evaluation of mercury in honey bees and their products from eastern Slovakia. *Animal Science and Biotechnology, 49*(1), 257-260.

Vieira, H.P., Nascentes, C.C., Windmoller, C.C. (2014). Development and comparison of two analytical methods to quantify the mercury content in honey. *Journal of Food Composition and Analysis, 34*(1), 1-6. http://dx.doi.org/10.1016/j.jfca.2014.02.001

Yaqub, G., Khalid, M., Ikram, A., Sohail, A. (2020). Monitoring and risk assessment due to presence of metals and pesticides residues in honey samples from the major honey producing forest belts and different brands. *Food Science and Technology, 40*(1), 331-335. https://doi.org/10.1590/fst.01919

Table S1.

Validation parameters of the used method and evaluation criteria

Parameter			Analyte	Evaluation criterion
			Mercury	
Linearity- correlation coeficient R^2			0.9998	≥ 0.998
Limit of detection (LOD) (mg/kg)			0.00009	
Limit of quantification (LOQ) (mg/kg)			0.0001	
Spiked sample analysis	Foodstuff	Level of enrichment (mg/kg)	Recovery* (%)	
	honey	0.00142	98.45	
		0.00948	90.37	
		0.9480	105.35	
	poultry meat	0.00095	109.00	
		0.9480	105.52	
	butter	0.00948	104.56	
	red currant	0.00948	98.81	
	celery	0.00948	94.41	
	Dietary supplement	0.09480	101.25	
	red shrimp	0.47400	104.42	
	zander fillet with skin fresh	0.56880	94.95	
Repeatability RSD_r(%) relative standard deviation of repeatability fish - level Hg: 0.0636 mg/kg			0.05	$HORRAT_r$< 2
Accreditation scope (mg/kg)			0.001 - 1.00	-
The lower reading range of the results for the purposes of this work: from LOQ of the method				

Commission Regulation (UE) No 2021/705

*The recovery for the method was calculated according to the following formula:

$$Recovery\,[\%] = \frac{C_w - C_m}{C_d} \cdot 100\%$$

where:
C_d - the concentration of the substance added to the sample,
C_m - the concentration of the substance in the sample where analyte has not been added,
C_w - the concentration of the substance measured in the spiked sample.