Determination of lead and cadmium residues in French honeys

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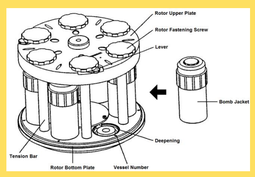
• Introduction

The chemical, metal industries and incinerators as well as the dynamic growth of automobile traffic (lead in gasoline was only suspended in 2000) have resulted in an increase of the heavy metals contamination of the environment. Honey is a "pure nature product" by definition, so the consumer does not expect any contamination with heavy metals. The environmental pollution with heavy metals implies a risk of contamination of honey through the pollen, nectar and water collected by bees [1-4]. Then, various methods [5, 6] have been developed for the control of honeys quality because the presence of heavy metals residues in honey is undesirable from the standpoint of food sanitation.
In EEC, no maximum residue limits (MRL's) are fixed for heavy metals in honey. In France, the admissible lead and cadmium contents in honey should not exceed 100 µg/kg and 50 µg/kg respectively. Each year, honey produced by beekeepers is submitted to a surveillance program [Directive 96/23/CE]. A rapid spectrophotometric method is described for evaluation of lead and cadmium residue levels in honeys. Results obtained from samples since 2002 from different areas of France are presented.

• Materials and method

Sample preparation :



1 g of honey in 3 ml HNO_3 (65% min) in pressure vessel MF 100

⬇

Mineralization using the Microwave Sample Preparation System (Anton Paar) :

15 min	0 min	0 min	
200 W ⟶	600 W ⟶	1000 W ⟶	0 W
0 min	5 min	5 min	15 min

⬇

Extract into 25 ml of ultrapure water

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Injection volume (20 µl) for analysis

Analysis by graphite furnace atomic absorption spectrometry :

- AAnalyst 100 (Perkin Elmer) equipped with the graphite furnace HGA-800 and the AS-72 Autosampler was used in the experiments.
- The matrix modifier was composed by a mixture of $Pd(NO_3)_2$ and $Mg(NO_3)_2$ at 2 g/l in ultrapure water.
- The autosampler sequence was the following one :
  pipet diluent (ultrapure water) + 3 µl of matrix modifier + 20 µl of sample/standard and each sample/standard was analyzed in 3 replicates.
- 582 honey samples collected were analyzed.

• Results

This method requires a rapid mineralization of the samples (time of the cycle = 40 minutes).The system performances of mineralization were checked with matrix of honey harvested in the apiary of the laboratory (a blank and a spiked honey at 100 and 20 µg/kg for Pb and Cd respectively). Standard solutions of Pb (50 µg/l) and Cd (2 µg/l) were used for the calibration of the spectrometer. The limits of detection and quantification were respectively 31 and 61 µg/kg for Pb and 9 and 18 µg/kg for Cd.
For each year of sampling, different variety of honeys were analyzed and results are presented in figures 1 and 2. Investigations with Veterinary Services were made in the apiaries where highly contaminated honeys were detected to try to know the origin of the contamination (traffic, industrial activities, incinerator factories or inappropriate materials used by beekeepers on production sites). For example, the highest value of Pb detected in honey was 1744 µg/kg. Then, the contamination of the honey from this area (in the "Département" of the Côte d'Or) was controlled each year and the level was 925 µg/kg in the year 2008. For residues of Cd, the levels were below of the limit (50 µg/kg) except for one sample collected in the year 2003 (68 µg/kg).

Figure 1 : Residues of lead detected in honeys sampled since 2002

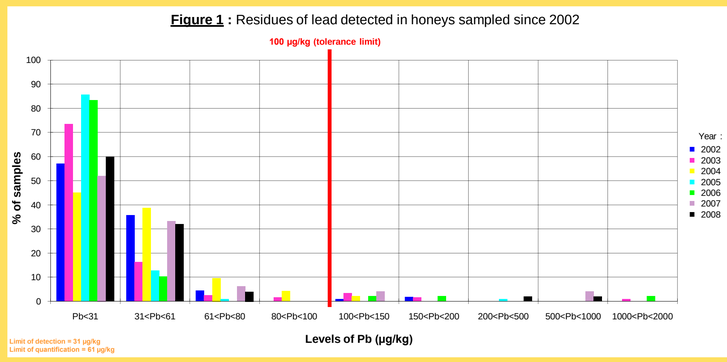
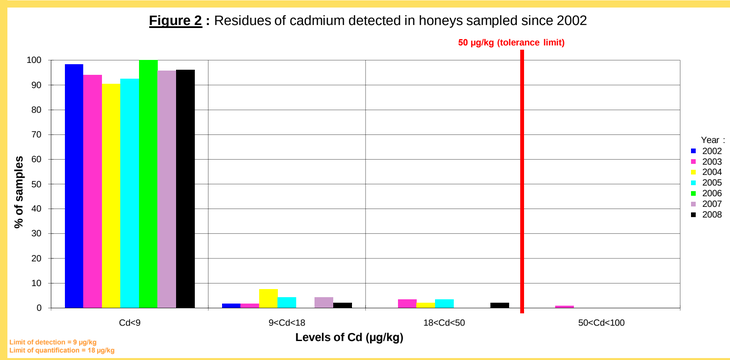


Figure 2 : Residues of cadmium detected in honeys sampled since 2002



• Conclusion

The method used allows the determination of lead and cadmium contents of 582 honey samples originating from different regions of France collected during several years in the frame of annual surveillance programs (Ministry of Agriculture). The quantification limit in honey was 61 and 18 µg/kg for lead and cadmium respectively. Results show that residue levels detected in honey are generally low in honey and are well below the current limits. Sometimes, contaminations of honey were detected which were investigated to determine the origin of the contamination and to propose corrective actions. The highest values of lead (above 100 µg/kg) were found in honeys from polluted area in the vicinity of apiaries or in honey samples extracted with inappropriate materials used on production sites.

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• **References :**

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