Results of the surveillance and control plans on **pesticide residues in honey** for 2014 and 2015

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Abstract
Plans for the surveillance and control of the contamination of foodstuffs of animal origin are organised each year by the Directorate General for Food (DGAL) in accordance with European regulations. For the beekeeping sector, samples are collected at the preliminary stage from French beekeepers. Pesticide residues (veterinary drugs and plant protection products) are analysed in honey using gas chromatography (GC) and liquid chromatography coupled with electrospray tandem mass spectrometry (LC-MS/MS). The results of the 2014 and 2015 plans show low levels of contamination below the maximum residue limits (MRLs).

Keywords
Honey, Pesticides, Residues, Surveillance and control plans

Résumé
Les plans de surveillance et de contrôle de la contamination des denrées alimentaires d'origine animale sont mis en place chaque année par la Direction générale de l'Alimentation (DGAL) en application de la réglementation européenne. Pour la filière apicole, les prélèvements sont réalisés au stade de la production primaire chez les apiculteurs français. Les résidus de pesticides (médicaments vétérinaires et phytosanitaires) sont recherchés dans les miels par chromatographie en phase gazeuse (GC) et par chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (LC-MS/MS). Les résultats des plans de 2014 et 2015 ont montré des taux de contamination très faibles inférieurs aux limites maximales en résidus (LMR).

Mots-clés
Miel, pesticides, résidus, plan de surveillance, plan de contrôle

For many years now, France has implemented surveillance and control plans (PSPC) for chemical residues in foodstuffs of animal origin. Since 1997, these plans have been carried out in accordance with the requirements of Directive 96/23/EC, under which Member States of the European Union (EU) must test for chemical residues (more specifically pesticides) in their products of animal origin. The main objective of these plans is to assess the level of contamination of foodstuffs placed on the national market with a view to protecting public health and identifying and removing possible sources of pollution. In the beekeeping sector, a sampling programme for honey is carried out each year by the DDecPPs at beekeeper holdings in France. Analyses of pesticide residues are performed at the ANSES Sophia Antipolis Laboratory.

In France, the beekeeping sector is made up of professional beekeepers, who generate 63% of honey production, and multi-activity or recreational beekeepers. Concerning organic beekeeping, the estimated production of certified honey ranged between 1200 and 1500 tonnes in 2014, and accounted for about 10% of national honey production [1]. Three regions represent more than 40% of production: Provence-Alpes-Côte d'Azur (PACA), Midi-Pyrénées and Rhône-Alpes (Figure 1). In 2014, 13,200 tonnes of honey were produced, including about 10,000 by beekeepers with more than 50 hives (Table 1). Beekeepers collect honey in the supers in late spring to late summer depending on the region and transhumance routes. Production can be multi-flower honey (or polyfloral honey) or monofloral honey, when one type of flower is the main source of the product. The breakdown of honeys sampled by floral origin is shown in Figure 2 for 2014 and 2015.

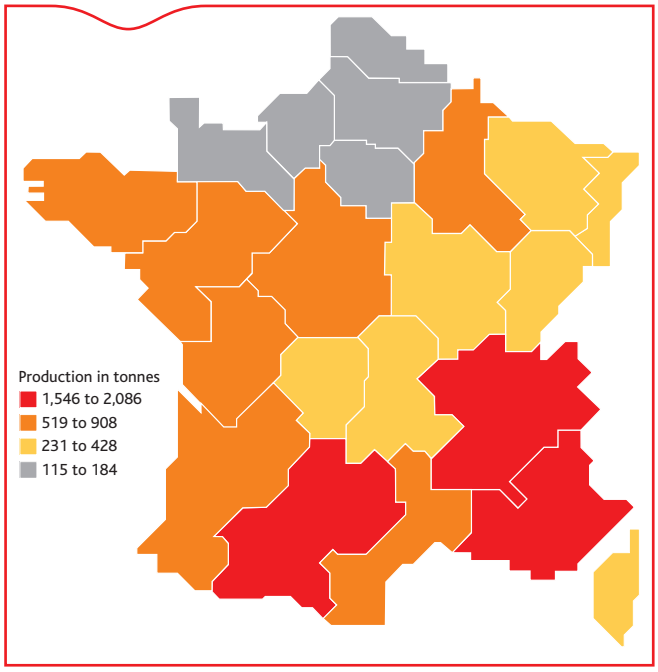

Figure 1. Regional distribution of estimated honey production in 2014 [1]

Table 1. Honey production in 2014 by class of hives [1]

Number of hives held	Number of beekeepers	Honey production (in tonnes)
0-10	25,304	1277
10-50	8,721	1,956
50-150	1,451	1,550
150-450	1,362	4,962
>450	355	3,461
Total	**37,193**	**13,206**
>50	3,168	9,973

Source: AND International 2014/2015 survey

Sampling

In accordance with the instructions in the guidance notes DGAL/SDSPA/SDPA/N2013-8214 [2] and DGAL/SDSPA/2014-999 [3], honey sampling is conducted in a targeted manner from beekeepers in France. In 2014 and 2015, the number of honeys sampled was established by product type on the basis of production size, using a distribution key fixed at the EU level (Directive 96/23/EC) and the results of plans carried out in previous years. Sampling was random

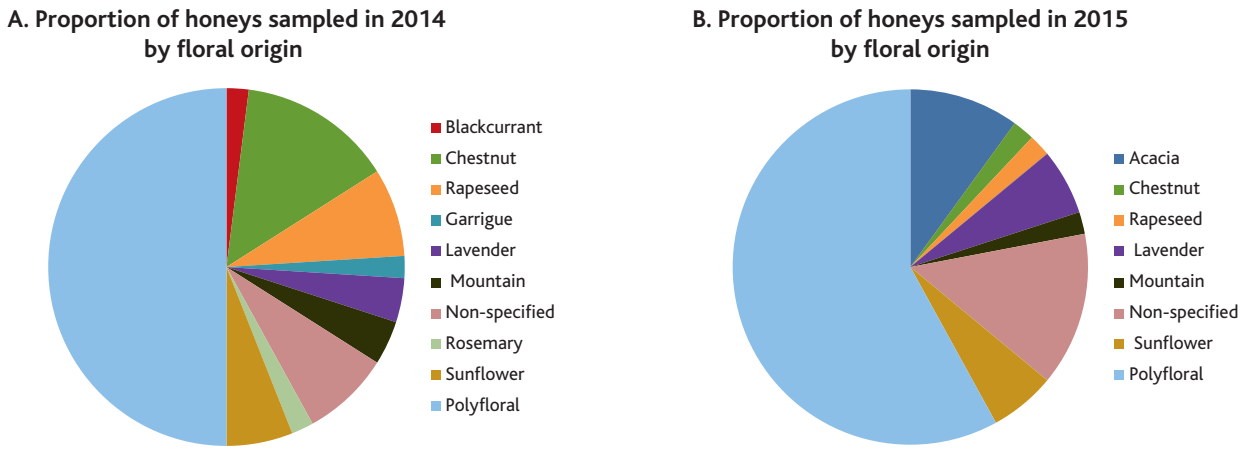

Figure 2. Proportion of honeys sampled by floral origin as part of the 2014 control plan (A) and the 2015 control plan (B)

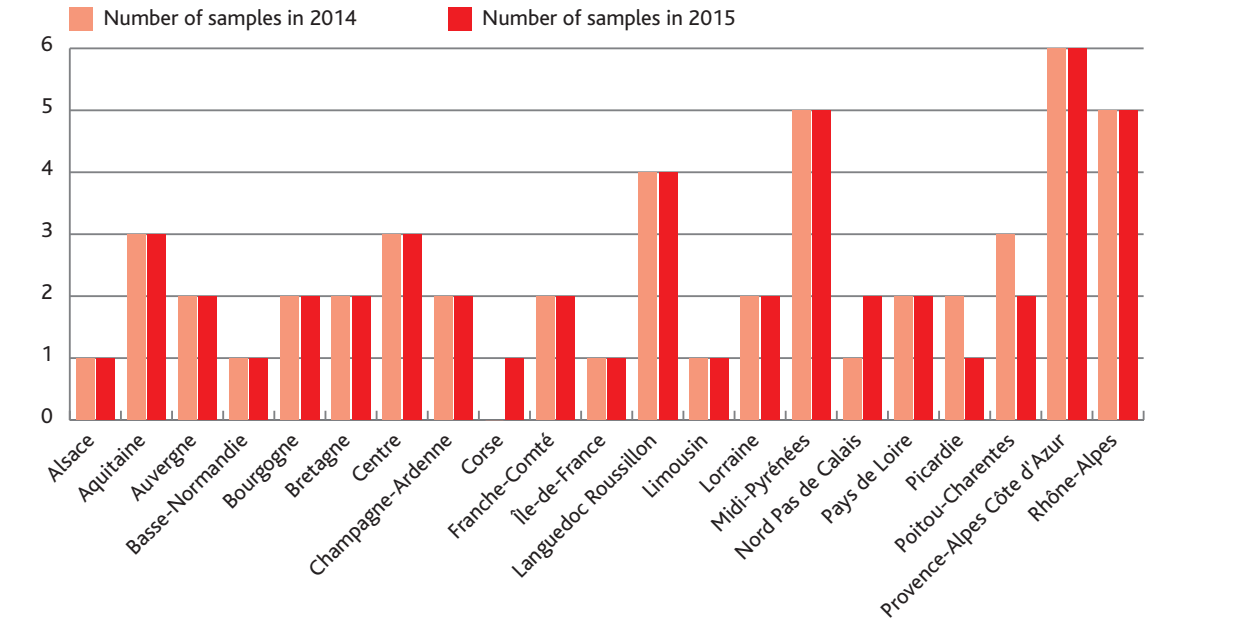

Figure 3. Distribution of samples by region as part of the 2014 and 2015 PSPC

(surveillance plan) for 54% of honeys in 2014, and 56% of honeys in 2015. For other honeys, sampling was targeted (control plan). The plans covered 20 regions (Figure 3). For 2014 and 2015, the control plan involved the collection of 50 samples of monofloral or polyfloral honey, excluding mixed honeys. Different flower varieties were represented among the 50 honeys sampled in 2014 and 2015 (Figure 2). Polyfloral honeys were the most commonly sampled variety (at least half of the samples over the two years).

Testing for pesticide residues

The main compounds tested for are acaricides used to control *Varroa destructor*, a mite parasite of bees. Residues of acaricides can be found in honey. Apivar® (amitraz) and Apistan® (tau-fluvalinate) are the main veterinary drugs used and they have been granted marketing authorisations (MAs) for the treatment of bee colonies. Other products are also authorised such as Thymovar®, Apilife Var® or Apiguard® (thymol), or more recently, MAQs (formic acid) or Api-Bioxal® (oxalic acid) and Apitraz® (amitraz). On the sampling forms, the DDecPPs indicate the beekeepers' use of these various products, which must be mentioned in the beekeeping registers maintained at the production holdings. Some beekeepers also use other veterinary products with MAs for other species, such as Taktic® for ruminants (amitraz). A few years ago, Asuntol® (coumaphos), which has an MA for species other than bees, was used by certain beekeepers. Residues of coumaphos accumulated in beeswax and could lead to contamination of honey. This product is no longer authorised.

Insecticides belonging to the neonicotinoid class (imidacloprid, clothianidin, acetamiprid, thiacloprid and thiamethoxam) are also tested for in honey. They are water-soluble compounds that may be found in honey. These insecticides are used in agriculture either for seed coating or foliar spray of crops. It should be noted that the EU suspended the use of imidacloprid, clothianidin and thiamethoxam in four field crops (maize, rapeseed, sunflower and cotton) as of late 2013.

All analyses for these pesticide residues are performed by the ANSES Sophia Antipolis Laboratory, which is accredited in accordance with Standard NF EN ISO/IEC 17025. The methods used have been validated in line with SANCO/12571/2013 [4] and are accredited by the French Accreditation Committee (COFRAC). Quantitative analyses are carried out using gas chromatography coupled with

electron capture detector (GC-ECD) and nitrogen-phosphorus detector (GC-NPD) to assay acaricides, and liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) to assay neonicotinoids in honey.

Results

Because the laboratory received all the planned samples, the effective sampling rate for 2014 and 2015 was 100%. For certain compounds, maximum residue limits (MRLs) in honey have been defined [5, 6 and 7]: amitraz (200 µg/kg), coumaphos (100 µg/kg), acetamiprid (50 µg/kg) and thiacloprid (200 µg/kg). The analytical methods used can detect the presence of residues below these values. The limits of quantification (LOQs) are 1 µg/kg for imidacloprid, acetamiprid and thiacloprid, 4 µg/kg for bromopropylate, chlorfenvinphos, thiamethoxam and clothianidin, 5 µg/kg for tau-fluvalinate, 6 µg/kg for amitraz, and 8 µg/kg for coumaphos. The results of the 2014 and 2015 plans are shown in Figures 4 and 5. Results obtained for 2014 show traces of residues for acetamiprid, thiacloprid, coumaphos, tau-fluvalinate and chlorfenvinphos. Certain honey samples (8%) contained two pesticide residues. Chlorfenvinphos, a plant protection product, is tested for in honey because it may be used outside this scope as a treatment in hives against *Varroa*. Traces of chlorfenvinphos (<LOQ) were found in one polyfloral honey from the Aveyron *département* in 2014. As a reminder, its use has been prohibited in France and the European Union since 31 December 2007. Residues of acetamiprid and thiacloprid are mainly found in spring-time honeys (rapeseed), polyfloral honeys, and those from sunflowers, blackcurrants and lavender. Honeys containing coumaphos and tau-fluvalinate residues came from colonies for which the treatments were not indicated in the sampling forms. However, these pesticide residues came from either application of the corresponding veterinary drugs by the beekeeper, or contact of the honey with contaminated beeswax. This is because certain liposoluble pesticides (e.g. amitraz, tau-fluvalinate and coumaphos) tend to accumulate in beeswax and some of them are stable and persistent in this matrix.

Conclusion - Outlook

All of the pesticide residues found in honey as part of the 2014 and 2015 control plans were at very low levels, below the MRL for coumaphos, acetamiprid and thiacloprid. However, one prohibited plant protection product, chlorfenvinphos, was found in a honey sample from the 2014 control plan. It is important to monitor this pesticide in future control plans for honey in France. In such cases,

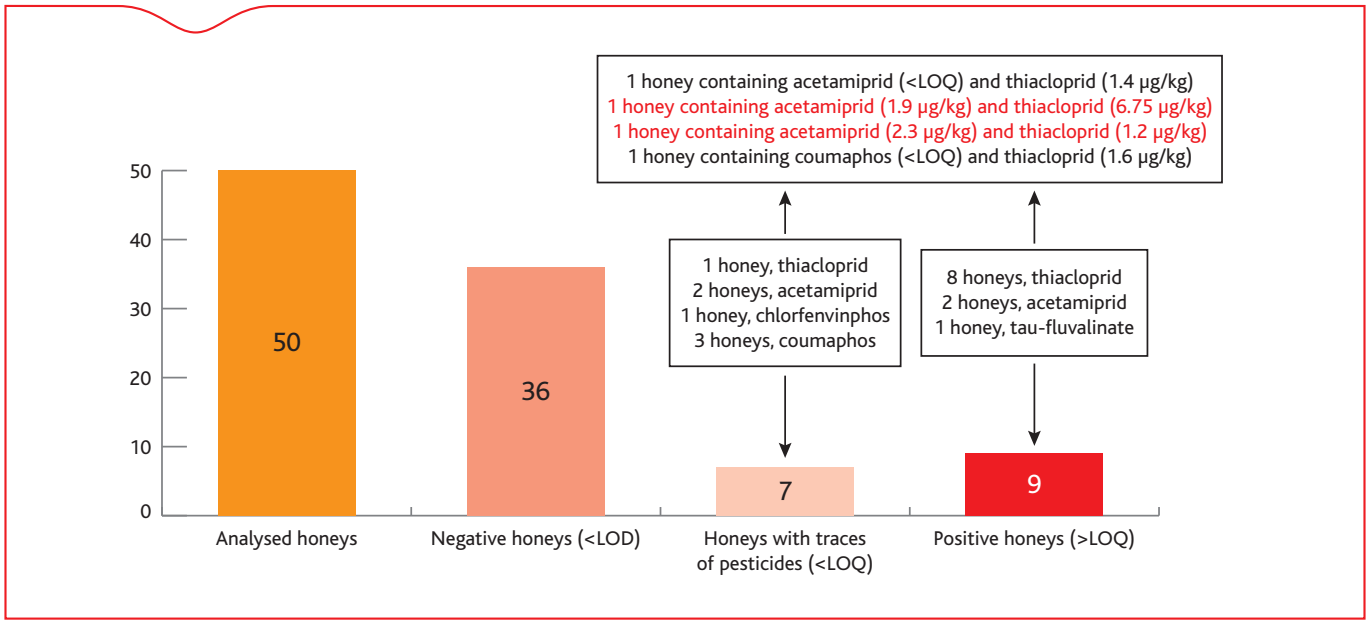

Figure 4. Results of pesticide residue analyses in honey in the 2014 plan

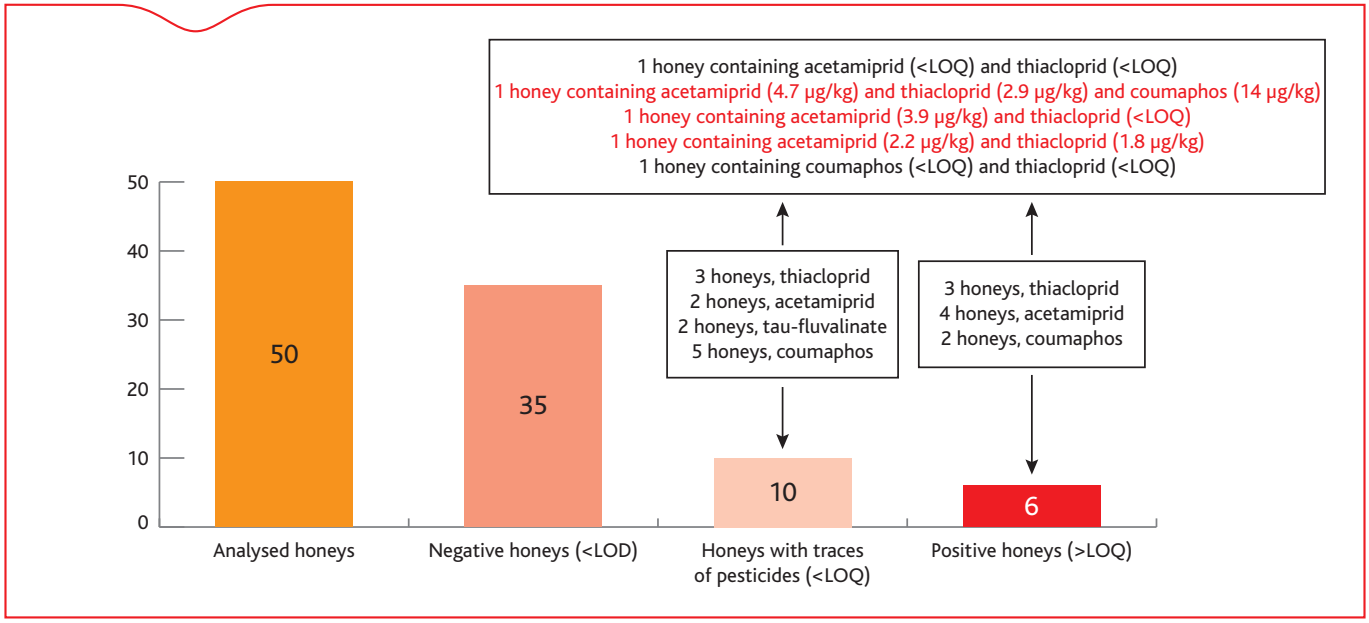

Figure 5. Results of pesticide residue analyses in honey in the 2015 plan

the veterinary services carry out investigations to determine the source of honey contamination. Another important point is that it is crucial to mention on the sampling forms the treatment schedules shown in the beekeepers' registers.

It would also be beneficial to have the harvesting date of the sampled honey, to better determine the possible source of contamination. Depending on the apiary's environment, plant protection products applied to neighbouring crops can be found in the nectar and pollen collected by the bees, and thus contaminate the honey.

References

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[5] Commission Regulation (EU) No 37/2010 of 22 December 2009 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin. OJ, 2010, 1-72.

[6] Commission Regulation (EU) No 87/2014 of 31 January 2014 amending annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for acetamiprid, butralin, chlorotoluron, daminozide, isoproturon, picoxystrobin, pyrimethanil and trinexapac in or on certain products. https://www.fsai.ie/uploadedFiles/Legislation/Food_Legisation_Links/Pesticides_Residues_in_food/Reg87_2014.pdf.

[7] Commission Regulation (EU) No 364/2014 of 4 April 2014 amending annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for fenpyroximate, flubendiamide, isopyrazam, kresoxim-methyl, spirotetramat and thiacloprid in or on certain products. https://www.fsai.ie/uploadedFiles/Legislation/Food_Legisation_Links/Pesticides_Residues_in_food/Reg364_2014.pdf.