

Title: RESIDUE IN HONEY – ACTUAL SITUATION

Author: *Dr. Cord Lüllmann*

Institution: Quality Services International GmbH, Bremen

Country: Germany

Phone: +49-421-594770

e-mail: info@qsi-q3.de

The means of Quality Management and Quality Control of bee products are analysing honey types from all over the world on origin and quality - hereby the main subjects are control of residues (antivarroa chemicals, antibiotics, pesticides); HMF, moisture, diastase, pollen, sugars, acid degree, proline, pH-value; sensory (taste, odour, appearance). Furthermore analysing bee products like royal jelly, pollen, propolis or beeswax, delivery of information and analysis of contents, adulteration and residues and give consultancy for all honey importers and packers in Europe concerning quality control of their products.

The European Commission is more and more focussing on the Quality Control of imported foodstuffs especially of animal origin.

Through this the regulation concerning the quality control of honey have strongly increased, the honey market is concerned by an increasing number of residue problems, the honey trade is influenced by scandals caused by residues of not permitted substances in animal food e.g. in the case of Chloramphenicol (CAP) this has led to a complete ban of imports from China. Recently the "Stiftung Warentest" - a German foundation, which is well known and their published results were accepted by consumers - criticized the appearance of Nitrofurane in honey.

The lecture will give a brief look on the regulations according to honey. The recent main changes will be given in short version as an overview on the latest regulations to point out the changes in regulations according to honey (changes are underlined) during the last time. The following regulations and directives were focused: 2001/110/EC, 96/23/EC (Annex IV), (EEC) No 2377/90, (EC) 178/2002, German Honey Directive and German Pesticide Directive as well as a recent Commission Decision 2002/657/EC to implement the Council Directive 96/23/EC.

Council Directive 2001/110/EC of 20. December 2001 relating to honey

Article 1 shows where the Directive shall apply to (Annex I: Names, Product Description and Definitions). The new definition of honey is:

§1. Honey is the natural sweet substance produced by *Apis mellifera* bees from the nectar of plants or from secretions of living parts of plants or excretions of plant-sucking insects on the living parts of plants, which the bees collect, transform by combining with specific substances of their own, deposit, dehydrate, store and leave in honeycombs to ripen and mature.

In §2b)vi. Filtered honey is mentioned: "Honey obtained by removing foreign inorganic or organic matter in such a way as to result in the significant removal of pollen."

Council Directive 96/23/EC of 29. April 1996

It is an directive on measures to monitor certain substances and residues thereof in live animals and animal products and repealing Directives 85/358/EEC and 86/496/EEC and Decisions 89/187/EEC and 91/664/EEC

Annex I:

Group A – Substances having anabolic effect and unauthorized substances

(6) Compounds included in Annex IV to Council Regulation (EEC) No 2377/90 of 26 June 1990 e.g. Chloramphenicol (CAP)

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GROUP B – Veterinary drugs ⁽¹⁾ and contaminants (see also (EEC) No 2377/90)

(1)Antibacterial substances, including sulphonamides, quinolones

(2)Other veterinary drugs (e.g. Antivarroa)

a)Anthelminitics

b)Anticoccidials, including Nitroimidazoles

c)Carbamates and Pyrethroids

d)Sedatives

e)Non-steroidal anti-inflammatory drugs (NSAIDs)

f)Other pharmacologically actives substances

(3)Other substances and environmental contaminants

a)Organochlorine compounds including PCBs

b)Organophosphorus compounds

c)Chemical elements, e.g. heavy metals

d)Mycotoxins

e)Dyes

f)Others

⁽¹⁾Including unlicensed substances which could be used for veterinary purposes

Regulation (EEC) No 2377/90 on veterinary drug residues in foodstuff of animal origin

The pharmacologically active substances are classified in 4 Annexes to the Regulation (positive Lists, which will be continually updated).

Annex I: List of pharmacologically active substances for which a MRL can be set, e.g. Amitraz (0,2 mg/kg) or Coumaphos (0,1mg/kg).

Annex II: List of substances for which there is no need to set a MRL, e.g. Lactic acid, Formic acid, Eucalyptus, Camphor, Menthol, Thymol (for all species used for food production, also bees), Fluvalinate and Phenol.

Annex III: List of pharmaceutically active substances for which a MRL cannot be set definitively but which may be given a provisional MRL for a defined period, e.g. Cymiazol (1mg/kg) until 01.07.2001.

Pharmaceutically active substances **not listed** in Annexes I, II or III are **not** allowed to be used in veterinary drugs, if the animal is used for food production (§ 14).

Annex IV: List of Substances for which it appear no MRL can be set because they pose a risk to human health in whatever quantity, e.g. Aristolochia spp. and preparations thereof, Chloramphenicol, Chloroform, Chlorpromazine, Colchicines, Dapson, Dimetridazol, Metronidazol, Nitrofurane (including Furazolidone), Ronidazol.

There is no EC legislation to pesticide residues limits in honey up to now although a proposal is in process. Some countries have limits e.g. Germany, the Netherlands or Italy.

Although there is no EC legislation on limits of pesticide residues, the **Rapid Alert System for Food and Feed (RASFF)** Regulation (EC) No 178/2002 is able to protect from great danger: e.g. excessive concentration of pesticide residues (results of the tests: e.g. 0.36 mg/kg Carbofurane) This system has proofed oneself. But this is not sufficient, it is necessary to have clear definition on limits for the entire Europe.

Commission Decision 2002/657/EC of 12 August 2002

Implementing the Council Directive 96/23/EC concerning the performance of analytical methods and interpretation of results. The main subjects of this Decisions were ensuring the quality and comparability of the analytical results generated by laboratories approved for official residue control, which have to be accredited. The results of positive samples shall be published immediately for more transparency. It is necessary to establish specific rules e.g. to

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determine common criteria for the interpretation of test results of official control laboratories or to provide for the progressive establishment of **minimum required performance limits (MRPL)** of analytical methods for substances for which no permitted limit has been established and in particular for those substances whose use is not authorised, or is specifically prohibited in the Community in order to ensure a harmonised implementation of Directive 96/23/EC. If no permitted limit has been established for a substance, the decision limit is the lowest concentration level at which a method can discriminate with a statistical certainty of 1-a that the particular analyte is present.

In our practical work in the laboratory, we analyse for example bee pharmaceuticals like Coumaphos, Brompropylate, Dibrombenzophenon, Fluvalinate; veterinary drugs and antibiotics like Streptomycin, Tetracyclines, Sulphonamides, Chloramphenicol or Nitrofurane. For all parameters we had increased sample frequency compared to last year data. According to the **bee pharmaceuticals** we saw that no Brompropylate and Dibromobenzophenon was detected during the sampling period. The compound mostly found was Coumaphos, a few samples showed appearance of Fluvalinate (Figure 1).

Compound	total	n.n.	<=0,01	<=0,05	<=0,1	>0,1
Coumaphos	1520	1290 84,9%	132 8,3%	93 6,1%	3 0,2%	2 0,2%
Brompropylate	1520	1520 100%				
Dibromobenzo-phenon	1520	1520 100%				
Fluvalinate	1520	1511 99,4%	9 0,6%			

Figure 1: Bee pharmaceuticals Overview 2001 to August 2002

The **vetinary drugs/antibiotics** showed the following development:

Streptomycin showed a significant increase of samples where no residue was detected (cp. 2001/2002). During 2003 the number of samples with no Streptomycine contamination were held on a constant high level (Figure 2).

The samples with no detection of Oxytetracycline decreased slightly between 2001 and 2002 and increased up to 2003 (Figure 3), the ones with no Tetracycline increased constantly up to nearly 100% (Figure 4).

Sulphonamides – e.g. Sulphathiazol (Figure 5) - showed a slight increase in samples with no residues detected. For Sulphamethacine we registered after a slight increase (2001 to 2002) of samples with no Sulphamethacine contamination more positive samples in 2003 (Figure 6). The comparison of 2002 and 2003 according to detection of Chloramphenicol showed that the number of CAP-free samples increased.

For Nitrofurane resp. the four detected metabolites (Figure 8) it is not possible to compare due to the fact that there is data only for a complete time frame, but they show that it was possible to identify contaminated samples.

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Find Figures 2-8 in the following:

year 2000	total	n.n.	<0,02	<0,1	<0,2	>0,2
number	1.991	1.473	216	212	23	66
share	100 %	74 %	10,9 %	10,6 %	1,2 %	3,3 %
year 2001	total	n.n.	<0,02	<0,1	<0,2	>0,2
number	1.988	1085	92	701	58	52
share	100 %	54,6 %	4,6 %	35,3 %	2,9 %	2,6 %
year 2002	total	n.n.	<0,02	<0,1	<0,2	>0,2
number	2.664	2.171	77	341	37	37
share	100 %	81,5 %	2,9 %	12,8 %	1,4 %	1,4 %
year 2003	total	n.n.	<0,02	<0,1	<0,2	>0,2
number	4.217	3.795	72	236	59	51
share	100 %	90,0 %	1,7 %	5,6 %	1,4 %	1,2 %

Figure 2: Streptomycine (mg/kg) Overview 2000-2003

year 2000	total	n.n.	<=0,01	<=0,1	<=0,2	>0,2
number	1.438	1.385	19	30	2	2
share	100 %	96,4 %	1,3 %	2,1 %	0,1 %	0,1 %
year 2001	total	n.n.(= <0,01)		<=0,1	<=0,2	>0,2
number	1.331	1.293		32	1	4
share	100 %	97,2 %		2,4 %	0,1 %	0,3 %
year 2002	total	n.n.(= <0,01)		<=0,1	<=0,2	>0,2
number	2.415	2.241		152	10	12
share	100 %	92,8 %		6,3 %	0,4 %	0,5 %
year 2003	total	n.n.(= <0,01)		<=0,1	<=0,2	>0,2
number	3.834	3.604		215	12	4
share	100 %	94,0 %		5,6 %	0,3 %	0,1 %

Figure 3: Oxytetracycline (mg/kg) Overview 2000-2003

year 2000	total	n.n.	<=0,01	<=0,1	<=0,2	>0,2
number	1.438	1.328	25	72	7	6

share	100 %	92,3 %	1,7 %	5,0 %	0,5 %	0,5 %
year 2001	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	1.331	1.097		220	8	5
share	100 %	82,5 %		16,5 %	0,6 %	0,4 %
year 2002	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	2.415	2.152		191	53	19
share	100 %	89,1 %		7,9 %	2,2 %	0,8 %
year 2003	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	3.837	3.791		38	4	4
share	100 %	98,8 %		1,0 %	0,1 %	0,1 %

Figure 4: Tetracycline (mg/kg) Overview 2000-2003

year 2000	total	n.n.	$\leq 0,01$	$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	2.777	2.253	48	295	54	55
share	100 %	83,8 %	1,7 %	10,6 %	1,9 %	2,0 %
year 2001	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	2.955	2.435		377	52	91
share	100 %	82,4 %		12,8 %	1,8 %	3,1 %
year 2002	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	4.403	3.901		409	48	44
share	100 %	88,6 %		9,3 %	1,1 %	1,0 %
year 2003	total	n.n. ($\leq 0,01$)		$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	5.288	4.828		386	42	37
share	100 %	91,3 %		7,3 %	0,8 %	0,7 %

Figure 5: Sulphathiazol (mg/kg) Overview 2000-2003

year 2000	total	n.n.	$\leq 0,01$	$\leq 0,1$	$\leq 0,2$	$> 0,2$
number	2.777	2.591	28	111	19	28

share	100 %	93,3 %	1,0 %	4,0 %	0,7 %	1,0 %
year 2001	total	n.n.		<=0,1	<=0,2	>0,2
number	2.957	2.820		85	21	31
share	100 %	95,4 %		2,9 %	0,7 %	1,0 %
year 2002	total	n.n.		<=0,1	<=0,2	>0,2
number	4.401	4.238		136	13	13
share	100 %	96,3 %		3,1 %	0,3 %	0,3 %
year 2003	total	n.n.		<=0,1	<=0,2	>0,2
number	5.292	4.932		312	21	27
share	100 %	93,2 %		5,9 %	0,4 %	0,5 %

Figure 6: Sulphamethacine (mg/kg) Overview 2000-2003

total 2002	n. n.	= < 0,3	> 0,3	> 1	>2	>5
2.181	1.830	98	120	61	57	15
100 %	83,9 %	4,5 %	5,5 %	2,8 %	2,6 %	0,7 %
total 2003	n. n.	<=0,3	<= 1	<= 2	<= 5	> 5
2.215	2.020	53	95	22	18	5
100 %	91,2 %	2,4 %	4,3 %	1,0 %	0,8 %	0,2 %

Figure 7: Chloramphenicol (µg/kg = ppb) 2002-2003

Nitrofurane- Semicarbazid total	n. n.	<= 0,5	>= 1	>= 2	>= 5	>5
1.914	1.858	1	26	18	7	4
100 %	97,1 %	0,1 %	1,4 %	0,9 %	0,4 %	0,2 %
Nitrofurane- AOZ total	n. n.	<= 0,5	>= 1	>= 2	>= 5	>5
1.914	1.737	22	83	43	21	8
100 %	90,8 %	1,1 %	4,3 %	2,2 %	1,1 %	0,4 %
Nitrofurane- AH total	n. n.	<= 0,5	>= 1	>= 2	>= 5	>5

1.914 100 %	1.912 99,9 %	0 0 %	2 0,1 %	0 0 %	0 0 %	0 0 %
Nitrofurane- AMAZ total	n. n.	<= 0,5	>= 1	>= 2	>= 5	>5
1.914 100 %	1.908 99,7 %	0 0 %	3 0,2 %	1 0,1 %	1 0,1 %	1 0,1 %

Figure 8: Nitrofurane – four metabolites ($\mu\text{g/kg}$ = ppb) from 01.01. 2002 - 12.05.2004

These data refer to the fact that the situation has not changed to better produce of honey in general but the control lead to the conscience that the products were analysed and contaminations by residues were detected and reported. The great demand for analyses to verify the quality of food products is documented by the continuous increased sample amounts. The maxim shall be furthermore: "control leads to confidence".