

Chlorinated Pesticide Residues in Honey

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Samples of commercially available honey, labeled as shown in Table 1, were examined for pesticide residues to 1) determine if honeys produced in areas where no pesticides or fertilizers were used were free of residues 2) compare the residue content, if any, of "organic," "natural," or "pure" products with processed products 3) determine the presence, if any, of polychlorobiphenyls (PCB's) that may have been derived from plastic foundations occasionally used in bee hives (KOOVER 1972) or from plastic containers that are sometimes used to package the final product. Subsequent to the initiation of this study, Consumer Reports (ANONYMOUS 1972) reported that no detectable pesticide residues were found in six samples of honey purchased in supermarkets and health-food stores.

Twenty-gram samples of honey were mixed with 50 ml of water in a 1-liter separatory funnel. Acetonitrile (100 ml) was added and the funnel was shaken vigorously for 2 minutes. Hexane (50 ml) was added and the funnel was again shaken vigorously for 2 minutes. The sample was allowed to stand until the phases separated. Water (600 ml) was added and the funnel was gently shaken for 30 seconds. The acetonitrile-water phase was removed and discarded. The hexane phase was washed with two 25 ml portions of water and then concentrated to a small volume in a flash evaporator. The concentrated residue was cleaned up by means of the KADOUM (1967) procedure. All reagent solvents were Nanograde quality. All glassware used in the analytical procedure was heat-treated prior to use for 16 hours at 200° C. as previously described (BEVENUE et al. 1971). The first fraction (hexane) from the Kadoum procedure contained the PCB's, aldrin, heptachlor, and a portion of the DDE. The second fraction (benzene:hexane, 70:30) contained the remainder of the DDE, plus lindane, dieldrin, and DDT; this fraction would also have

Journal Series No. 1519 of the Hawaii Agricultural Experiment Station.

TABLE 1

Source and Description of Honey Samples

<u>Source and Sample No.</u>	<u>Label Description</u>
<u>Hawaii</u>	
2	Organic, Kona coffee
4	Lehua, raw
12	Moanalua, comb pack
17	Lehua
18	Kiawe, North Kona area, 1971 crop
<u>California</u>	
1	Orange, 100% natural
3	Raw
5	Sage, 100% natural
6	Safflower
7	100% natural
8	Pure
9	Wild, 100% natural
10	Clover, "Whipt, " 100% natural
11	Comb pack, natural
13	Unprocessed, raw
14	Alfalfa-Safflower blend, unprocessed, raw
15	Clover
16	Processed
<u>Iowa</u>	
19	Comb pack (plastic container)
<u>Mainland China, North</u>	
20	Chinese milk vetch
21	Non-descriptive

contained heptachlor epoxide, DDD, and chlordane. The cleaned-up fractions were concentrated to 1 ml under nitrogen and 5 μ l aliquots were injected into the gas chromatograph. The gas chromatograph was a Varian Aerograph Model 1200 with an electron capture detector. Column temperature was 190^o C, injection port temperature 210^o C, and detector temperature 200^o C. Flow rate of nitrogen carrier gas was 25 ml/min. Two 1/8 inch x 6 feet borosilicate columns were used; one column contained 4% SE-30, 6% QF-1 silicones on Chromosorb W, HP, 80/100 mesh and the other column contained 1.5% OV-17, 1.95% QF-1 on

Supelcoport 80/100 mesh. Recovery values obtained from fortified samples are given in Table 2.

TABLE 2

Recovery of Pesticides from Fortified Honey Samples

<u>Pesticide</u>	<u>p.p.b. added</u>	<u>p.p.b. recovered</u>	<u>% Recovery</u>
Lindane	0.25	0.19	76
Heptachlor	0.50	0.41	82
Aldrin	0.50	0.42	84
Heptachlor Epoxide	0.50	0.35	70
DDE	0.50	0.43	86
DDD	0.50	0.39	78
DDT	0.50	0.47	94
Dieldrin	0.50	0.42	84
Aroclor 1254	20.0	18.0	90

All but two of the 21 samples (or 90% of the samples) contained chlorinated pesticide residues in the low parts per billion (p.p.b.) range (Table 3). One sample, labeled "natural, comb pack," indicated the presence of a contaminant in the low p.p.b. range with somewhat similar characteristics to Aroclor 1254; this sample also contained lindane, DDE and DDT. An interesting fact about the two samples which contained no detectable residues was the label designation of each of the two samples; one was labeled "pure," and the other was labeled "comb," and the latter was packed in a plastic container. The samples of Kiawe and Lehua honeys produced in areas where no known pesticides or fertilizers were used, contained very low but detectable amounts of DDT. The so-called "organic" or "natural" labeled products were not, as implied by some health-food stores, free from residues. The lindane, DDE and DDT residues observed in the honeys by gas chromatography were confirmed by mass spectrometry data obtained from a Finnigan Model 3000 Gas Chromatograph Peak Identifier.

The levels of residue present in the honey, as illustrated in Table 3, were quite low and they were probably introduced into the honey by bees that fed on nectar from contaminated blossoms. This has been demonstrated in studies with Sevin (carbaryl), where the bees have carried Sevin along with the nectar and pollen into their hives; it was also shown that Sevin residues in honey and bee bread persisted for periods of 6 to 9 months (SOLOV'YEVA 1971). Federal laws do not allow any pesticides in honey. Therefore, the legal tolerance for pesticides in honey is "zero." However, since bees may not be capable of discriminating between contaminated and non-contaminated blossoms and because

TABLE 3

Chlorinated Pesticide Residues in Honey
(parts per billion)

<u>Sample No.</u>	<u>Lindane</u>	<u>Dieldrin</u>	<u>DDE</u>	<u>p,p'-DDT</u>	<u>Total</u>
<u>Hawaii</u>					
17				0.10	0.10
4				0.18	0.18
18				0.20	0.20
2				0.48	0.48
12	0.15	0.22		0.36	0.73
				Average	0.34
<u>California</u>					
8					N.D. *
16				0.10	0.10
13				0.17	0.17
5			0.05	0.31	0.36
7				0.42	0.42
10			0.06	0.40	0.46
3	0.08			0.45	0.53
9				0.83	0.83
1			0.05	0.80	0.85
14			0.31	0.61	0.92
11	0.08		0.15	1.40	1.63
6			0.57	1.12	1.69
15			1.04	2.60	3.64
				Average	0.97
<u>Iowa</u>					
19					N.D. *
<u>Mainland China, North</u>					
21	0.26			0.32	0.58
20	0.78		0.06	1.46	2.30
				Average	1.44

* N.D. = None detectable. Limits of detection 0.05 p.p.b. for chlorinated pesticides, 2 p.p.b. for Aroclor 1254.

analytical instrumentation has developed to a high degree of sophistication, the probability of finding honeys which contain no ("zero") pesticide residues is low; based on this study, at least, the probability would be about 10 per cent of any samplings.

Acknowledgment

We acknowledge with thanks the assistance of Karl Yanagihara for acquiring the mass spectra data.

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