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Evaluation of conventional and "organic" baby food brands for eight organochlorine and five botanical pesticides

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Abstract

Two lots of applesauce, pears, winter squash and carrots from each of two traditional commercials and one commercial "organic" brand of baby food were purchased from local retailers. These samples were analyzed for eight organochlorine (aldrin, dieldrin, *cis*-chlordane, *p,p′*-DDT, *p,p′*-DDE, *p,p′*-DDD, heptachlor, and hexachlorobenzene) and five botanical (nicotine, pyrethrin I, pyrethrin II, warfarin, and rotenone) pesticides in duplicate. The results indicated no detectable organochlorine pesticide residues at levels of detection between 4 and 11 pg/g, no detectable nicotine residues at the level of detection of 0.66 ng/g and no detectable pyrethrin I, pyrethrin II, warfarin and rotenone residues at levels of detection between 0.126 and 0.36 ng/g. Since no residues were found in any of the baby foods, there was no apparent distinction between the traditional commercial brands and the "organic" commercial brand of baby foods evaluated in this study.

1. Introduction

A growing concern for safer foods has led research into increased pesticide residue monitoring. The benefits of pesticide use to preserve the quality and quantity of foods available to the consumer is virtually unchallenged and infinitely valuable. One alternative to synthetic pesticide use is organic farming. However, this process is costly, labour intensive, and in some cases ineffective (Ware, 1996). In fact, in 1998 the estimated mark-up for "organic" foods was 20–50% higher than conventional foods (Mahoney, 1998). Still, the fear of pesticides has driven some of the public to seek organic alternatives for their grocery list. The "organic"-labelled foods, although assumed to contain lower levels of pesticides, are self regulated and tend to be more costly than traditional brands. The National Organic Program (NOP) is the first attempt, by the federal government, to regulate these sought-after "organic" labels. The Food and Drug Administration (FDA) has regulations for the use of synthetic pesticides and regularly monitors these residues in adult and infant foods (Food and Drug Administration [FDA], 1996). However, the use of botanical or "organic" pesticides in organic farming is largely not monitored and there are no provisions to do so, as specified by the NOP.

This study is one of the first efforts to clarify this pesticide issue. Evaluating traditional and "organic" brand name foods for high use botanical pesticides in a sensitive sub-population, such as infant foods, may give us a new view into food safety.

The FDA has been monitoring infant foods since the early 1960′s and has consistently found residue levels in these foods; however, the positive residues found were far below the tolerances and action levels set by the Environmental Protection Agency (EPA) (Pennington, Capar & Parfitt, 1996). The growing concern over infant food safety has led the FDA to increase infant food monitoring in their regulatory monitoring, incident/level monitoring and the total diet study programs (Yess et al., 1993). All the extended monitoring by FDA does not include analysis of "organic"-labelled foods for botanical pesticides.

Of the 20 plus botanical pesticides available to organic farmers, nicotine, pyrethins, rotenone and warfarin are among the most widely used (Ware, 1996). These pesticides were chosen for this study due to their popularity and because the NOP specifies that these botanical substances may be used in organic farming. Although they are from nature, these substances are

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unnaturally extracted and concentrated before use as pesticides. Due to their concentrations, many of these botanical pesticides are acutely toxic to humans and mammals, usually are broad spectrum poisons, and may be applied to crops closer to harvest and more often (Ware, 1996). These botanical pesticides are often more toxic than some of the organochlorine pesticides that have been banned since the 1970's (Ware, 1989). The FDA has monitored nicotine and pyrethrins, but these are the only botanical pesticides which have been included in the FDA studies (Food and Drug Administration [FDA], 1988).

Other chemicals which the FDA has included in their residue monitoring programs, since the start of residue monitoring, are organochlorine pesticides. Although many of these compounds have been banned for many years, these compounds are still persistent in our environment (Food and Drug Administration [FDA], 1998). A residual content of these compounds in foods today is still quantifiable and their avoidance in farming is largely impossible. The eight organochlorine pesticides chosen included: aldrin, dieldrin, *cis*-chlordane, DDT, DDE. DDD, heptachlor, and hexachlorobenzene. These compounds were chosen because of past high use in agriculture and their present persistence.

The objectives of the study are twofold. The first is to analyze traditional name brand baby foods and "organic" name brand baby foods that are consumed in large quantities by infants and children for persistent organochlorine pesticides. The second is to analyze these same samples for high-use botanical pesticides.

2. Materials and methods

The samples were obtained from grocery stores located in the state of Michigan in the United States. Two traditional baby food name brands and one "organic"-labelled baby food name brand were purchased in lots of two. The samples analyzed were bought as processed "strained/junior (st/jr) second foods" baby foods, as sampled by the FDA in their total diet study (Pennington & Gunderson, 1987). Two of the four matrices analyzed included two fruits, apples and pears, which were listed as two of the eight adult food most eaten by infants and children by the FDA (Yess et al., 1993). The two remaining matrices were the vegetables, winter squash (for their availability) and carrots (chosen for their underground growth pattern).

Two lots within each brand of each matrix were randomly purchased to obtain a more accurate representation of the product. After acquisition the samples were stored in a dry, cool, and minimal light environment until opened and refrigerated immediately after opening. The two lots, in each name brand, of each matrix, were analyzed in triplicate to have unbiased representation. Percentage recoveries were run on each method for each matrix using standards with 98–99% purity and glass blanks were run periodically to ensure no cross contamination.

2.1. Organochlorine residues

The multiresidue method used in this study allowed for the extraction and separation of eight organochlorines. The compounds that were evaluated included, aldrin, *cis*-chlordane, DDD, DDE, DDT, dieldrin, heptachlor, and hexachloro-benzene (BHC). The method adapted for this study was taken from Nakamura et al. (1994), which was a multiresidue method developed for 48 pesticides in agricultural products. This method included an acetone extraction, liquid to liquid partition with ethyl acetate, florisil cleanup, eluting the pesticides with 30% ethyl acetate/70% n-hexane, and quantitation with electron capture gas chromatography (GC). The organochlorine pesticides in the method of Nakamura et al included heptachlor, *p,p′*-DDE, *p,p′*-DDD, aldrin, dieldrin, *o,p′*-DDT, and *p,p′*-DDT. The modified method was validated for the above six pesticides and for hexachlorobenzene and *cis*-chlordane. Since BHC and *cis*-chlordane are organochlorines, the compounds were easily added to the new multiresidue method.

The column used in the GC analyses was a fused-silica DB-5 type of, 30 m by 0.25 μm. Sample injection was automated. GC conditions included an injector port temperature of 220°C, an oven temperature of 210°C, and a detector temperature of 260°C. The limit of detection (LOD) for the eight organochlorines ranged from 4.4 to 10.2 pg/g. Specific LOD's for these pesticides were BHC 8.8 pg/g, Heptachlor, 4.4 pg/g, aldrin 5.6 pg/g, dieldrin 7.4 pg/g, *cis*-chlordane 7.2 pg/g, DDE 4.2 pg/g, DDD 9.2 pg/g and DDT 10.2 pg/g.

The percentage recoveries of triplicate samples spiked with 7.2–12 pg/g of organochlorine pesticides ranged from 75 to 120%. A representative organchlorine analysis chromatogram for a pear recovery is given in Fig. 1. Carrot and winter squash samples presented a slight problem with the orginal method and further modifications had to be included to acquire the percentage recoveries listed. After filtration, each carrot and winter squash sample was combined with 5 g of Celite 525 and set aside for 15 min. It was found that any time under 15 min did not remove the colour from the sample. Next, the sample was filtered again in a Buchner funnel, fitted with a 9 mm No. 5 Whatman filter. The remaining steps of the method remained the same.

2.2. Nicotine residues

To 10 g of apple and pear baby food, 30 ml of freshly prepared 20% NaOH was added and swirled about 1 min until blended. Nicotine was partitioned into ether

with the addition of 20 ml of 20% NaOH to force the nicotine out of the water. The collected ether layer was dried over anhydrous sodium sulfate and concentrated to about 1.0 ml under a stream of nitrogen. The sample was transferred and made up to volume with methanol in a 10 ml volumetric flask and covered with foil to prevent nicotine degradation by ultraviolet light.

High performance liquid chromatography (HPLC) was used to quantify the nicotine. The HPLC set up included an Altex pump (lower limit 0, upper limit 5), and ANS-3113 debubbler, a Linear UV-106 254 fixed wavelength detector (range 1.0 AUFS, rise time 0.3 s), and a HP 3300 integrator. The column used was a Phenomenex Develosil ODS-UG-5 unbonded silica gel with the following specifications: 17.2% total carbon, a median particle size of 5.6 μm, a surface area of 295 m^2/g, a pore volume of 1.10 ml/g, a median pore diameter of 137 A, and an internal diameter of 150×4.6 mm. Twenty microlitres of the sample were injected into the pump and moved at a flow rate of 0.4–0.6 ml/min. For squash and carrot samples, extraction was the same but the mobile phase for the HPLC was changed to 80/20 v:v methanol/water solution to eliminate an interfering peak. The limit of detection for the HPLC was 0.66 ng/g. Average percentage nicotine recoveries for triplicate analyses of apple, pear, squash and carrots spiked with 1.22, 1.22, 2.135 and 2.135 ng/g, respectively, were 111%, 101, 102 and 92%, respectively.

2.3. Multiresidue analysis of pyrethrin I and II, warfarin, and rotenone residues

The multiresidue analyses used were developed for water (Vandervoort, 1999). The availability of multiresidue analysis of botanical pesticides is limited. Five grams of baby food were acidified with 10 ml of freshly prepared HCl solution (pH 3), after which 20 ml of saturated NaCl were added. The first liquid to liquid partition used hexane. After drying over anhydrous sodium sulfate, a second liquid to liquid extraction was conducted using methylene chloride and then drying over sodium sulfate. Finally, to the contents of the turbo vap tube, 1 ml of DI water was added and the contents reduced in volume to about l ml under a slow nitrogen stream. This 1 ml extract was made up to volume with methanol. HPLC conditions were the same as indicated for nicotine. The column used was a Waters Nova-Pak C_{18} column, with an internal diameter of 150×3.9 mm. The HPLC flow rate varied between 0.4 and 0.7 ml/min. Pyrethrins I and II were quantified at a wavelength of 235 nm and warfarin and rotenone at the wavelength of 280 nm. Limits of detection for pyrethrin I, pyrethrin II, rotenone and warfarin, respectively, were 0.126, 0.126, 0.30 and 0.36 ng/g.

Percentage recovery and standard deviation variability were larger for this group of botanical pesticides. Table 1 gives the percentage recovery and standard deviation for pyrethrin I, pyrethrin II, rotenone, and warfarin, representative of triplicate 5 g samples spiked with 120 ng/g pyrethrin I and II, 0.130 ng/g rotenone and 133 ng/g warfarin. Values for extraction from water are given for contrast. Obviously the method worked much better on water than on the baby foods. Some method variations were tried to obtain the recoveries listed but the results of this section of the study can only be considered exploratory and further method development and refinement is needed if botanical pesticide residues are to be quantitated in a variety of foods.

3. Results and discussion

Three samples of each of two lots of two traditional commercial manufacturers and one organic commercial manufacturer were analyzed for eight organochlorine pesticide residues. Baby foods used included apples, pears, squash and carrots. The results of these analyses for aldrin, dieldrin, *cis*-chlordane, *p,p′*-DDE, *p,p′*-DDD, *p,p′*-DDT, heptachlor, and hexachlorobenzene are presented in Table 2. It is evident that the brands and lots evaluated were free of the eight organochlorine pesticides analyzed in this study. These findings are consistent with past analyses of traditional commercially processed baby foods and also indicate that "organic" commercial baby food used in this study had non-detectable residues of these compounds. The organic industry sells its products by claiming that they have lower pesticide residues; however, no comparison had

Table 1
Percentage recovery[a] for pyrethrin I, pyrethrin II, rotenone and warfarin

		Percentage recovery and standard deviation of analyses of spiked samples			
Botanical pesticide	Water[b]	Apple	Pear	Squash	Carrot
Pyrethrin I	71.0	28.0±30.6	62.6±30.6	57.9±46.1	58.1±39.4
Pyrethrin II	84.5	19.3±16.1	59.0±16.0	56.1±26.6	58.6±42.1
Rotenone	67.0	44.5±27.3	57.0±40.5	69.8±37.1	68.1±45.1
Warfarin	83.2	66.0±47.6	70.7±38.3	148±31.9	177±28.9

[a] Based on the results of three spiked samples.
[b] Vandervoort, 1999.

Table 2
Residues (ng/g) of organochlorine pesticides in apple, pear, squash and carrot baby food samples from three manufacturers

		Organochlorine pesticides							
Food sample	Manufacture type[a]	Aldrin	Dieldrin	*cis*-Chlordane	DDD	DDE	DDT	Heptachlor	BHC
Apple	TCM 1	nd[b]	nd	nd	nd	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd	nd	nd	nd	nd
	OCM	nd	nd	nd	nd	nd	nd	nd	nd
Pear	TCM 1	nd	nd	nd	nd	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd	nd	nd	nd	nd
	OCM	nd	nd	nd	nd	nd	nd	nd	nd
Squash	TCM 1	nd	nd	nd	nd	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd	nd	nd	nd	nd
	OCM	nd	nd	nd	nd	nd	nd	nd	nd
Carrot	TCM 1	nd	nd	nd	nd	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd	nd	nd	nd	nd
	OCM	nd	nd	nd	nd	nd	nd	nd	nd

[a] TCM 1 = traditional commercial manufacture 1; TCM 2 = traditional commercial manufacture 2; OCM = organic commercial manufacture.
[b] nd = Indicates the compound was not detected in the three samples run in each of the two lots.

been made with processed foods. Yess et al. (1993) indicated that 99% of pesticide residues can be eliminated by washing with soap and water. Moreover, many processors have required, for many years, that their suppliers adhere to strict pesticide application reporting requirements and that applications are made in accordance with registration standards (Chin, 1991). Focusing on the prevention of illegal and unnecessary pesticides starts with the fields in which the commodity is grown. Since 1960, the food processing industry has had in place a program known as the NFPA Protective Screen Program. This program detailed the source of raw produce and pesticide chemicals that are permitted for crop production.

The pesticide issue is further diminished with processed foods. It is known that processing fruits and vegetables largely eliminates residues, so the justification for buying processed "organic" foods due to an expected lower pesticide level is unfounded. In the processed baby foods tested, there was no distinction between "organic" and traditional baby foods.

The evaluation of foods for botanical pesticides is one of the major current issues in food residue monitoring

Table 3
Residues (ng/g) for the botanical pesticide nicotine in apple, pear, squash and carrot baby food samples from three manufacturers

		Botanical pesticide
Food sample	Manufacture type[a]	Nicotine
Apple	TCM 1	nd[b]
	TCM 2	nd
	OCM	nd
Pear	TCM 1	nd
	TCM 2	nd
	OCM	nd
Squash	TCM 1	nd
	TCM 2	nd
	OCM	nd
Carrot	TCM 1	nd
	TCM 2	nd
	OCM	nd

[a] TCM 1 = traditional commercial manufacture 1; TCM 2 = traditional commercial manufacture 2; OCM = organic commercial manufacture.
[b] nd = Indicates the compound was not detected in the three samples run in each of the two lots.

Table 4
Residues (ng/g) for the botanical pesticides pyrethrin I, pyrethrin II, rotenone and warfarin in apple, pear, squash and carrot baby food samples from three manufacturers

		Botanical pesticide			
Food sample	Manufacture type[a]	Pyrethrin I	Pyrethrin II	Rotenone	Warfarin
Apple	TCM 1	nd[b]	nd	nd	nd
	TCM 2	nd	nd	nd	nd
	OCM	nd	nd	nd	nd
Pear	TCM 1	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd
	OCM	nd	nd	nd	nd
Squash	TCM 1	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd
	OCM	nd	nd	nd	nd
Carrot	TCM 1	nd	nd	nd	nd
	TCM 2	nd	nd	nd	nd
	OCM	nd	nd	nd	nd

[a] TCM 1 = traditional commercial manufacture 1; TCM 2 = traditional commercial manufacture 2; OCM = organic commercial manufacture.
[b] nd = Indicates the compound was not detected in the three samples run in each of the two lots.

today. The increased popularity of organic foods necessitates the inclusion of botanical pesticides in residue monitoring programs. Organic foods are assumed to be free of pesticides by the public, but what the organic food industry does not advertise is that botanical pesticides are allowed for the use on these foods, with only self-regulation by the organic food industry.

The three samples of each of two lots of two traditional commercial manufacturers and one organic commercial manufacturer were also analyzed for the following botanical pesticides: nicotine, pyrethrin I, pyrethrin II, rotenone, and warfarin. Nicotine analyses were conducted separately and the results of these analyses are given in Table 3. Observing the data it is clear that no residues of nicotine were found at the level of detection in any of the samples tested. A modification for a botanical multiresidue analyses was used to determine the remaining botanical pesticides. This method needs further refinement for food matrices; although the limits of detection were between 0.1 and 2.0 ng/g, the recovered concentrations were much higher. Nevertheless, the preliminary data obtained in this study also indicated that none of these botanical pesticides residues occurred at levels above the limits of detection given (Table 4).

The increase in public concern over safer foods has led to the study presented here. The FDA has excellent monitoring programs to monitor our food supply. These programs ensure the safest foods possible. Still, the public's fear of synthetic pesticides has opened a growing market of "organically" produced foods. The industry has been largely self-regulated and neither government nor industry residue programs monitor foods for botanical pesticides. These botanical pesticides are allowed for use in organic farming and some of the botanicals are more toxic than persistent pesticides. In this study, neither the traditionally manufactured nor the "organic" manufactured commercial baby food samples had detectable levels of the eight organochlorine or four botanical pesticides; however, organic foods sell for 2–3 times as much as traditionally processed foods. The need for the monitoring of botanical pesticides in the "organically"-labelled foods, to justify the increased purchase price, should be a part of future residue monitoring programs.

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