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FOOD AND DRUG ADMINISTRATION
COMPLIANCE PROGRAM GUIDANCE MANUAL

PROGRAM 7306.02

CHAPTER 6 - DAIRY INDUSTRY

Program Activity: Surveillance, Compliance

Data Control Code: BF-320-024

Problem Code: FH-18

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SUBJECT: SURVEY: POLYCHLORINATED BIPHENYLS IN MILK

PURPOSE

To provide guidance to the districts in conducting a survey of fluid milk for the polychlorinated biphenyls.

BACKGROUND

In 1970 PCB's were detected in milk samples from West Virginia. Follow up investigation revealed the apparent misuse of an electrical transformer fluid containing PCB as a vehicle for defoliant spraying operations adjacent to dairy pasturage as being responsible for the milk contamination. PCB's have also been found to be a component of sealants used to treat silos on dairy farms. Milk contamination has resulted from this source of PCB's.

Subsequent information derived from the total diet studies program, the general pesticide program, and other food surveys has shown the presence of PCB's in fish, milk, eggs, in grayboard used in cereal packaging materials and in the cereal product itself.

Surveys to date reveal that additional data is necessary to determine the extent of PCB occurrence in milk. Information is desired to determine the condition of milk used for bottling purposes and milk used for manufacturing purposes with respect to PCB contamination.

For the purpose of this survey "bottling milk" is milk which is destined for pasteurization and subsequent bottling for direct consumer use. "Manufacturing milk" is milk intended for use as a raw material in the production of a consumer dairy product (e.g., NFDM, condensed milk, cheese, butter, and ice cream).

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OBJECTIVE

To survey the milk supply on a state by state basis to determine the extent of PCB contamination of milk for bottling and manufacturing use.

ORIGINATOR: Bureau of Foods, Division of Compliance Programs, Program Development Branch, BF-323

APPROACH

1. Inspectionol

Districts are responsible for follow-up investigations including inspections if necessary to determine the source of any PCB contamination detected.

The Districts should work with the Regional Milk and Food Consultants and state and local officials to determine sampling sources and to facilitate follow-up activities.

There will be no CP-IP issuance by EDRO for this program.

2. Sample Collection

The attached district/state sampling schedule has been developed from available information regarding the milk production of each state for both bottling and manufacturing use. Districts should make every effort to collect the samples as specified and to distinguish between milk for bottling and milk for manufacturing use. It is recognized that in some instances the latter will not be possible.

Sampling sources should be selected on a random basis to reflect milk production and usage within each state. Samples may be collected from both interstate and intrastate sources.

District	State	Bottling Milk Samples	Manufacturing Milk Samples	Total Samples
		40	12	52
ATL-DO	Georgia	5	-	5
	Florida	7	-	7
	Alabama	3	1	4
	Mississippi	4	1	5
	Tennessee	6	3	9
	North Carolina	6	1	7
	South Carolina	3	2	5
	Kentucky	6	4	10
BLT-DO		13	3	16
	Maryland	6	1	7
	Virginia	6	1	7
	West Virginia	1	1	2

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District	State	Bottling Milk Samples	Manufacturing Milk Samples	Total Samples
BOS-DO		19	0	19
	Maine	2	-	2
	New Hampshire	1	-	1
	Vermont	9	-	9
	Rhode Island	1	-	1
	Connecticut	3	-	3
	Massachusetts	3	-	3
BUF-DO		46	0	46
	New York	46	-	46
CIN-DO		17	2	19
	Ohio	17	2	19
CHI-DO		9	4	13
	Illinois	9	4	13
DAL-DO		19	1	20
	New Mexico	1	-	1
	Oklahoma	5	1	6
	Texas	13	0	13
DEN-DO		9	11	20
	North Dakota	1	2	3
	South Dakota	1	5	6
	Montana	1	1	2
	Wyoming	1	1	2
	Colorado	3	1	4
	Utah	2	1	3
DET-DO		27	5	32
	Michigan	18	3	21
	Indiana	9	2	11
KAN-DO		22	23	45
	Iowa	6	13	19
	Nebraska	3	3	6
	Kansas	5	2	7
	Missouri	8	5	13

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District	State	Bottling Milk Samples	Manufacturing Milk Samples	Total Samples
LOS-DO		20	2	22
	California	18	2	20
	Arizona	2	0	2
MIN-DO		51	71	122
	Minnesota	10	32	42
	Wisconsin	41	39	80
NOL-DO		7	1	8
	Louisana	5	-	5
	Arkansas	2	1	3
NYK-DO		5	2	7
	New York	-	-	0
	New Jersey	3	-	3
	Puerto Rico	1	1 (if available)	2
	Virgin Island	1	1 (if available)	2
PHI-DO		30	1	31
	Pennsylvania	29	1	30
	Delaware	1	-	1
SAN-DO		20	2	22
	California	18	2	20
	Nevada	1	-	1
	Hawaii	1	-	1
SEA-DO		15	6	21
	Washington	9	-	9
	Idaho	2	5	7
	Oregon	3	1	4
	Alaska	1	-	1
	Totals	369	146	515

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Samples should consist of 7 quarts of fluid milk collected from route collection trucks (bulk farm milk pick-up) on completion of pickup or from collection station points prior to further dilution of the milk at the processing plant. If milk is received in cans at the processor, samples should be collected from the holding tanks prior to process.

Plastic containers or containers in which the contents come into contact with rubber or cardboard closures or inserts should not be used in the collection of samples for PCB analysis. One quart Mason jars, which have been washed to rinse out cardboard particles or other foreign matter which may interfere with PCB analysis, may be used. Tin foil should be inserted beneath the jar lid and screw top to prevent milk contact with the rubber gasket.

Aseptic technique (sterile ladles) should be used in collecting samples for this survey to avoid adding to the bacterial load of the bulk milk from which the sample is collected.

Samples may be official or investigational. Collection reports should be flagged "PCB - Milk Survey" and should include the following information.

1. State where collected.
2. Point of collection (e.g., route truck, holding tank, etc.).
3. Volume of milk sampled.
4. Number of producers represented.
5. Type of milk (e.g., bottling, manufacturing).
6. Milk route number or patron(s) can number.
7. Route truck number or license number.

See Inspector Operations Manual, Chapter 4, Sections 443.2, 452.2, 454, and 454.3 for guidance in sample handling, shipment, and notification of receiving laboratory.

3. Sample Analysis

Analyze samples for PCB, organochlorine and organophosphorus pesticide residues using appropriate PAM Vol. I methodology for extraction, cleanup, and GLC determination: extraction 211.13h(1) (4/1/71 revision) cleanup, 211.14a,d (4/1/71 revision); EC-KCLTD GLC, 321. Inject approximately 3 mg milk fat equivalent for GLC. Analyze 20% of the District sample quota (with a minimum of four samples) for the chlorophenoxy acid residues.

Determine residues of pesticides in the usual manner. Evaluate EC-GLC chromatogram of 6% Florisil eluate for presence of PCB and pesticides. If presence of PCB is indicated, complete analysis using the necessary treatment of extract prior to GLC determination.

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In most instances when both PCB and DDT and analogs are present, the PCB and organochlorine pesticides require separation by chromatography on silicic acid [Armour and Burke, JAOAC 53, 761 (1970)] prior to determination. In some instances dehydrochlorination of DDT and TDE to their respective olefins [Sawyer and Krause, LIB 1249 (Jan. 8, 1971)], or a combination of dehydrochlorination followed by oxidation of olefins to the respective dichlorobenzophenone [Mulhern, et al, JAOAC 54, 548 (1971)] will permit quantitation of PCB without column chromatographic separation.

Appropriate treatment will depend on the PCB [Aroclor(s)] indicated present, the particular pesticide(s), and the relative amounts of PCB and pesticides. Aroclors 1221, 1232, and 1242 divide between the petroleum ether and polar solvent eluates from the silicic acid column, but residues of these Aroclors can possibly be quantitated in the presence of DDT and analogs, and would not necessarily require column chromatographic separation from these pesticides.

Previous experience indicates that PCB residues in cows' milk will be more similar in GLC pattern to Aroclors 1254 or 1260.

Quantitate PCB residues by electron capture GLC or by use of halogen specific microcoulometric or electrolytic conductivity GLC detectors. Compare the total area response from the PCB residue to the total area response from a known weight of the Aroclor with most similar GLC pattern. However, if the GLC curve shows that the PCB residue clearly consists of components of more than one Aroclor, if possible quantitate the residues separately using the appropriate Aroclor reference for the respective portion of the GLC curve.

When PCB residue is 5 ppm or above in the fat, confirm residue identity by GLC with halogen specific microcoulometric or electrolytic conductivity detectors and/or demonstration of the residue's stability to boiling alkali. Additional confirmatory analyses are left to discretion of the Laboratory Director.

Limit of detection for PCB residue in milk fat is approximately 1.5 ppm (as Aroclor 1254), when using a 3 mg sample injection and EC-GLC operated at the above conditions.

Report PCB residue in terms of Aroclor(s) used in quantitation (e.g., 2 ppm Aroclor 1242 and 3 ppm Aroclor 1260).

PCB residue levels in milk fat should be rounded off to the nearest 0.1 ppm.