



# 28

Chapter

## Extraneous Matter Examination

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## 28.1 INTRODUCTION

### 28.1.1 Background

Extraneous materials are any foreign substances in foods that are associated with objectionable conditions or practices in production, storage, or distribution of foods. Extraneous materials include (a) filth or objectionable matter contributed by animal contamination (rodent, insect, or bird matter) or unsanitary conditions, (b) decomposed material or decayed tissues due to parasitic or nonparasitic causes, and (c) miscellaneous matter (sand, soil, glass, rust, or other foreign substances). Bacterial contamination is excluded from these substances.

Filth is classified according to its extractability. Light filth is oleophilic and lighter than water (separated from product by floating it in an oil-aqueous mixture). Insect fragments, rodent hairs, and feather barbules are examples of light filth. Heavy filth is heavier than water and separated from the product by sedimentation based on different densities of filth, food particles, and immersion liquids ( $\text{CHCl}_3$ ,  $\text{CCl}_4$ , etc.). Examples of heavy filth are sand, soil, and nut-shell fragments. Sieved filth involves particles separated from the product by the use of selected mesh sizes. Whole insects, stones, sticks, and bolts are examples of sieved filth.

Various methods of isolation of extraneous matter from various food commodities can be found in the *Official Methods of Analysis* of the AOAC International and in the *Approved Methods of Analysis* of the AACC International. Presented here are a few procedures for some food commodities, with descriptions based on AOAC methods, but the quantities reduced to half.

### 28.1.2 Reading Assignment

Dogan, H., and Subramanyam, B. 2017. Extraneous matter. Ch 34, in *Food Analysis*, 5th ed. S.S. Nielsen (Ed.), Springer, New York.

### 28.1.3 Notes

Regulatory examination of samples by the Food and Drug Administration (FDA) would be based on replicate samples using official methods, including the specified sample size. However, for instructional purposes, the costs associated with adequate commercial 1-L Wildman trap flasks, reagents, and food samples specified in official methods may be prohibitive. Procedures given below are based on AOAC methods, but all quantities are reduced to half, and a 500-mL Wildman trap flask (vs. 1-L trap flask) is specified in most procedures. Commercially available 1-L trap flasks with the standard stopper rod would ideally be used (with all quantities in the procedures doubled). However, 500-mL trap flasks can be made for use in

this experiment. To do this, drill a hole through a rubber stopper of a size just too large for a 500-mL Erlenmeyer flask. Thread a heavy string through the hole in the stopper, and knot both ends of the string. Coat the sides of the rubber stopper with glycerin and *carefully* force it (with larger end of stopper pointed up) through the top of the flask. Note that the string could be a trap for contaminants such as rodent hair and insect fragments.

For the parts of this laboratory exercise that require filter paper, S&S #8 (Schleicher & Schuell, Inc., Keene, NH) is recommended. It meets the specifications set forth in the AOAC Method 945.75 Extraneous Materials (Foreign Matter) in Products, Isolation Techniques Part B(i), which suggests using “smooth, high wet strength, rapid acting filter paper ruled with oil-, alcohol-, and water-proof lines 5 mm apart.” The S&S #8 ruled filter paper is 9 cm in diameter and fits well into the *top* of standard 9-cm *plastic* petri dishes. The *bottom* of the plastic petri dish can be used as a protective cover over the sample filter paper in the top of the petri dish. The top of the plastic petri dish provides a 9 cm flat surface (as opposed to glass petri dishes) for examining the filter paper, making it easier to view the plate without having to continuously refocus the microscope. The 5-mm ruled lines provide a guide for systematically examining and enumerating contaminants on the filter paper at 30× magnification. To obtain a moist surface on which contaminants can be manipulated and observed, apply a small amount of glycerin: 60% alcohol (1:1) solution to the top of the petri dish before transferring the filter paper from the Buchner funnel. Using both overhead and substage lighting with the microscope will assist in identifying contaminants.

### 28.1.4 Objective

The objective of this laboratory is to utilize techniques to isolate the extraneous matter from various foods: cottage cheese, jam, infant food, potato chips, and citrus juice.

### 28.1.5 Principle of Methods

Extraneous materials can be separated from food products by particle size, sedimentation, and affinity for oleophilic solutions. Once isolated, extraneous materials can be examined microscopically.

## 28.2 EXTRANEEOUS MATTER IN SOFT CHEESE

### 28.2.1 Chemicals

	CAS no.	Hazards
Phosphoric acid ( $\text{H}_3\text{PO}_4$ )	7664-38-2	Corrosive

### 28.2.2 Reagents

- Phosphoric acid solution, 400–500 mL  
Combine 1 part phosphoric acid with 40 parts deionized distilled (dd) water (vol/vol).

### 28.2.3 Hazards, Precautions, and Waste Disposal

Adhere to normal laboratory safety procedures. Wear safety glasses at all times. Waste likely may be put down the drain using a water rinse, but follow good laboratory practices outlined by environmental health and safety protocols at your institution.

### 28.2.4 Supplies

- Beaker, 1 L (for phosphoric acid solution)
- Beaker, 600 mL (to boil water)
- Buchner funnel
- Cottage cheese, 115 g
- Filter paper
- Heavy gloves
- Pipette, 10 mL (to prepare phosphoric acid solution)
- Pipette bulb or pump
- Spoon
- Sidearm flask, 500 mL or 1 L
- Stirring rod
- Tap water, ca. 500 mL (boiling)
- Tweezers
- Volumetric flask, 500 mL (to prepare phosphoric acid solution)
- Weighing boat

### 28.2.5 Equipment

- Hot plate
- Microscope
- Top loading balance
- Water aspirator system

### 28.2.6 Procedure

(Based on AOAC Method 960.49, Filth in Dairy Products)

1. Weigh out 115-g cottage cheese and add it to 400–500-mL boiling phosphoric acid solution (1+40 mixture) in a 1-L beaker, stirring with a glass stirring rod continuously to disperse the cottage cheese.
2. Filter the mixture through filter paper in a Buchner funnel, using a vacuum created by a water aspirator. Do not let the mixture accumulate on the paper, and continually wash filter with a stream of hot water to prevent clogging. Make sure the cheese mixture is hot as it is filtered. When filtration is impeded, add hot water or phosphoric acid solution (1+40 mixture) until

the paper clears. [May also use dilute (1–5%) alkali or hot alcohol to aid in filtration.] Resume addition of sample and water until sample is filtered.

3. Examine filter paper microscopically.

## 28.3 EXTRANEEOUS MATTER IN JAM

### 28.3.1 Chemicals

	CAS no.	Hazards
Heptane (12.5 mL)	142-82-5	Harmful, highly flammable, dangerous for the environment
Hydrochloric acid, concentrated (HCl) (5 mL)	7647-01-0	Corrosive

### 28.3.2 Hazards, Precautions, and Waste Disposal

Heptane is an extremely flammable liquid; avoid open flames, breathing vapors, and contact with skin. Otherwise, adhere to normal laboratory safety procedures. Wear safety glasses at all times. Dispose of heptane waste as hazardous waste. Other waste may be put down the drain using a water rinse.

### 28.3.3 Supplies

- 2 Beakers, 250 mL (for weighing jam and heating water)
- Buchner funnel
- Filter paper
- Glass stirring rod
- Graduated cylinder, 100 mL
- Ice water bath (to cool mixture to room temperature)
- Jam, 50 g
- Graduated pipette, 10 mL (for heptane)
- Pipette bulb or pump
- Sidearm flask, 500 mL or 1 L
- Spoon
- Thermometer
- Tweezers
- Volumetric pipette, 5 mL (for conc. HCl)
- Waste jar (for heptane)
- Water, dd, 100 mL (heated to 50 °C)
- Wildman trap flask, 500 mL

### 28.3.4 Equipment

- Hot plate
- Microscope
- Top loading balance
- Water aspirator system

### 28.3.5 Procedure

(Based on AOAC Method 950.89, Filth in Jam and Jelly)

1. Empty contents of jam jar into beaker and mix thoroughly with glass stirring rod.
2. Weigh 50 g of jam into a beaker, add ca. 80 mL dd water at 50 °C, transfer to a 500-mL trap flask (use the other ca. 20 mL dd water to help make transfer), add 5 mL conc. HCl, and boil for 5 min.
3. Cool to room temperature (with an ice water bath).
4. Add 12.5 mL heptane and stir thoroughly.
5. Add dd water to a level so heptane is just above rubber stopper when in the "trap" position.
6. Trap off the heptane, and filter the heptane through filter paper in a Buchner funnel using vacuum created by a water aspirator.
7. Examine filter paper microscopically.

## 28.4 EXTRANEOUS MATTER IN INFANT FOOD

### 28.4.1 Chemicals

	CAS no.	Hazards
Light mineral oil (10 mL)	8012-95-1	

### 28.4.2 Hazards, Precautions, and Waste Disposal

Adhere to normal laboratory safety procedures. Wear safety glasses at all times. Waste may be put down the drain using water rinse.

### 28.4.3 Supplies

- Baby food, ~113 g (one jar)
- Buchner funnel
- Filter paper
- Glass stirring rod
- Graduated cylinder, 10 or 25 mL
- Pipette bulb or pump
- Sidearm flask, 500 mL or 1 L
- Spoon
- Tweezers
- Volumetric pipette, 10 mL
- Water, deaerated, 500 mL
- Wildman trap flask, 500 mL

### 28.4.4 Equipment

- Microscope
- Water aspirator system

### 28.4.5 Procedure

(Based on AOAC Method 970.73, Filth in Pureed Infant Food, A. Light Filth)

1. Transfer 113 g (one jar) of baby food to a 500-mL trap flask.
2. Add 10 mL of light mineral oil and mix thoroughly.
3. Fill the trap flask with deaerated water (can use dd water) at room temperature.
4. Let stand 30 min, stirring four to six times during this period.
5. Trap off mineral oil in a layer above the rubber stopper and then filter the mineral oil through filter paper in a Buchner funnel using vacuum created by a water aspirator.
6. Examine filter paper microscopically.

## 28.5 EXTRANEOUS MATTER IN POTATO CHIPS

### 28.5.1 Chemicals

	CAS no.	Hazards
Ethanol, 95 %	64-17-5	Highly flammable
Heptane (9 mL)	142-82-5	Harmful, highly flammable, dangerous to environment
Petroleum ether (200 mL)	8032-32-4	Harmful, highly flammable, dangerous to environment

### 28.5.2 Reagents

- Ethanol, 60%, 1 L  
Use 95% ethanol to prepare 1 L of 60% ethanol; dilute 632 mL of 95% ethanol with water to 1 L.

### 28.5.3 Hazards, Precautions, and Waste Disposal

Petroleum ether, heptane, and ethanol are fire hazards; avoid open flames, breathing vapors, and contact with skin. Otherwise, adhere to normal laboratory safety procedures. Wear safety glasses at all times. Heptane and petroleum ether wastes must be disposed of as hazardous wastes. Other waste may be put down the drain using a water rinse.

### 28.5.4 Supplies

- Beaker, 400 mL
- Buchner funnel
- Filter paper
- Glass stirring rod
- Graduated cylinder, 1 L (to measure 95% ethanol)
- Ice water bath

- Potato chips, 25 g
- Sidearm flask, 500 mL or 1 L
- Spatula
- Wildman trap flask, 500 mL
- Tweezers
- Volumetric flask, 1 L (to prepare 60% ethanol)
- Waste jars (for heptane and petroleum ether)

### 28.5.5 Equipment

- Hot plate
- Microscope
- Top loading balance
- Water aspirator system

### 28.5.6 Procedure

(Based on AOAC Method 955.44, Filth in Potato Chips)

1. Weigh 25 g of potato chips into a 400-mL beaker.
2. With a spatula or glass stirring rod, crush chips into small pieces.
3. In a hood, add petroleum ether to cover the chips. Let stand 5 min. Decant petroleum ether from the chips through filter paper. Again add petroleum ether to the chips, let stand 5 min, and decant through filter paper. Let petroleum ether evaporate from chips in hood.
4. Transfer chips to a 500-mL trap flask, add 125 mL 60% ethanol, and boil for 30 min. Mark initial level of ethanol on flask. During boiling and at the end of boiling, replace ethanol lost by evaporation as a result of boiling.
5. Cool in ice water bath.
6. Add 9 mL heptane, mix, and let stand for 5 min.
7. Add enough 60% ethanol to the flask so that only the heptane layer is above the rubber stopper. Let stand to allow heptane layer to form at the top, trap off the heptane layer, and filter it through filter paper in a Buchner funnel.
8. Add 9 mL more heptane to solution. Mix and then let stand until heptane layer rises to the top. Trap off the heptane layer, and filter it through filter paper (i.e., new piece of filter paper, not piece used in Parts 3 and 4) in a Buchner funnel.
9. Examine the filter paper microscopically.

## 28.6 EXTRANEOUS MATTER IN CITRUS JUICE

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### 28.6.1 Supplies

- Beaker, 250 mL
- Buchner funnel
- Cheesecloth
- Citrus juice, 125 mL
- Graduated cylinder, 500 mL or 1 L
- Sidearm flask, 250 mL
- Tweezers

### 28.6.2 Equipment

- Microscope
- Water aspirator system

### 28.6.3 Procedure

[Based on AOAC Method 970.72, Filth in Citrus and Pineapple Juice (Canned), Method A. Fly Eggs and Maggots]

1. Filter 125 mL of juice through a Buchner funnel fitted with a double layer of cheesecloth. Filter with a vacuum created by a water aspirator. Pour the juice slowly to avoid accumulation of excess pulp on the cheesecloth.
2. Examine material on cheesecloth microscopically for fly eggs and maggots.

## 28.7 QUESTIONS

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1. Summarize the results for each type of food analyzed for extraneous materials.
2. Why are contaminants such as insect fragments found in food, when the Pure Food and Drug Act prohibits adulteration?

## RESOURCE MATERIALS

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AOAC International (2016) Official methods of analysis, 20th edn. (On-line). AOAC International, Rockville, MD  
Dogan H, Subramanyam B. (2017) Extraneous matter. Ch 34. In: Nielsen SS (ed) Food analysis, 5th edn. Springer, New York