Analytical Techniques for Glass Contamination of Food: A Guide for Administrators and Analysts

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ABSTRACT

An analytical scheme is presented to provide an overview of techniques applicable to glass contamination in a variety of consumer products. Procedures are referenced or presented in detail, with emphasis on quality assurance. References on forensic and analytical methods for the examination of glass are provided.

The glass packaging industry began in 1903 with the development of the first fully automated machinery for manufacturing glass containers. Glass rapidly gained acceptance as a packaging medium because it is transparent, chemically inert, odorless, tasteless, impermeable to moisture and gas, and readily adaptable to modern filling lines that are now capable of handling over 1,200 containers per minute. Contamination by glass fragments became critical with the advent of high-speed production methods. Despite the manufacturers' best efforts to produce contamination-free containers, however, some jars of food have been found to contain glass fragments. Glass contamination of food may occur in several ways: Containers may already be contaminated with glass when they are received by food processors from the manufacturer; finished food products may be contaminated by chipping or breakage of glass containers during processing, storage, shipping, and retail or consumer handling; glass fragments may also be introduced into food samples during preparation and analysis.

Consumers may also be a source of glass contamination of food. Although consumer tampering with food products or containers to acquire monetary awards or free replacement food items from a food manufacturer does not usually result in a public health threat, the monetary loss incurred in settling “consumer complaints” can be considerable.

This report presents a general discussion of analytical methods used to detect glass contamination. It provides analytical guidance to ensure maximum recovery of glass fragments, thorough characterization of the contaminant, and the best possible conclusions as to the probable sources of contamination.

PROCEDURES FOR GLASS EXAMINATION

A flow diagram (Fig. 1) of an analytical scheme provides administrators with the analytical options to be considered in drawing valid conclusions and gives analysts an overview of the tests to be performed.

Examination of glass containers for integrity

In cases of suspected tampering involving alleged unopened containers, it is essential to obtain the maximum amount of data on the suspect container and product to determine if the container had been previously opened. Details of the following tests are presented in Chapter 24 of the Food and Drug Administration’s Bacteriological Analytical Manual, 6th edition (44): closure and glass defects (24.34-A); vacuum (24.34-B); removal torque (cam-off) for press-on, twist-off, and lug-type twist closures (24.34-B2); security values (lug-tension) on lug-type twist cap (24.34-B3); pull-up (lug position) for lug-type twist cap (24.34-B4); gross and net weights; and headspace gas (24.30-B4).

Chemical measurements

Details of the following tests are presented in various sections (secs.) of the Official Methods of Analysis, 14th edition, of the Association of Official Analytical Chemists (AOAC) (1) (Caution: Jar lid must not be disturbed until all container integrity tests have been completed): pH of foods, secs. 32.010-32.018; total soluble solids: fruit products, sec. 22.024; sugar products, sec. 31.011; processed vegetable products, secs. 32.023-32.025; and titratable acidity, secs. 27.058-27.059. Additional procedures for inspecting food containers are provided by the U.S. Department of Agriculture (40-42) and the Food and Drug Administration (44).

External examination for glass fragments

In this examination all glass fragments are recovered from the external surfaces of the container. Glass fragments
Internal examination for glass fragments.

This examination is to recover all glass fragments inside the container, embedded sometimes in the label or more often boxes may adhere to the exterior surfaces of the product resulting from the breakage of filled containers in shipping. Some containers are tested to determine if they are in fact 

Equipment (listed in ref. 1): Puncture device (to release vacuum); sieves, sec. 44.002 (r); sedimentation equipment (see AOAC Methods of Analysis, 11th edition, sec. 40.036); bolting cloth, sec. 44.002 (d); spray device, sec. 44.002 (a); Hirsh funnel; vacuum flask; petri dish (plastic); forceps; probe; widefield stereoscopic microscope, sec. 44.002(o).

Procedure for opening container. If the lid is on the container and the area under the rim of the lid has not been rinsed to remove glass fragments, proceed as instructed under “External examination for glass fragments.” If the container is under vacuum and no container integrity tests are planned, release the vacuum with a puncture device. Remove the lid without disturbing the product adhering to its inner surface.

Lid examination. Examine the inside of the lid and surfaces of adhering product, using a widefield stereoscopic microscope. Keep the glass fragments found inside separate from those found outside the container. Take special care when examining lids whose bottom edge rolls inward. Glass fragments trapped in the rolled edge are extremely difficult to remove. Cutting the rolled edge at several locations around the lid aids in glass fragment removal. With respect to the lid, inside refers to all lid surfaces and the surface of adhering product that fall within the depression in the sealing compound made by the container rim. Outside refers to inner lid surfaces that fall outside the depression in the sealing compound made by the container rim. Fragments found in the rim depression in the sealing compound that can be matched with chips in the container rim should be reported separately.

Container rim examination. Before removing the product from the container, examine the container rim for chipped areas using the widefield stereoscopic microscope at 30-60X magnification. Also examine the product surface for glass fragments.

Product examination. Quantitatively transfer product residues on the internal lid surface and from the container to a nonglass beaker large enough to hold the net contents of the product container. Exercise care so as not to chip the container rim while transferring the product.

Liquid. Filter thin liquid food through a black bolting cloth or a ruled white filter paper, depending upon the color of the container glass.

Solids—dry or moist. Methods for determining heavy filth sedimentation are given in the AOAC Official Methods of Analysis (1). If a sedimentation method for a specific product is not available, use a method for a similar product. If the sedimentation approach is inappropriate, sieve the product either on a single sieve or on multiple nested sieves to separate it into particle size groups. Maximum sieve size for unretained residues (i.e., material that passes through the sieve and is discarded) is a USA Standard Sieve Series No. 40, with openings of 0.0165 inches or 0.425 mm. Examine residues on bolting cloth or filter paper of size-segregated product using a widefield stereoscopic microscope at 10-60X magnification.

Confirmation of fragment as glass. All suspect glass fragments must be tested to determine if they are in fact

Figure 1. Analytical scheme for examining glass.

resulting from the breakage of filled containers in shipping boxes may adhere to the exterior surfaces of the product container, embedded sometimes in the label or more often in product residues. Glass fragments are also frequently trapped between the jar threads and the lid or embedded in the exposed lid compound material.

Equipment (listed in ref. 1): No. 230 sieve - sec. 44.002 (r); spray device (aerator) - sec. 44.002 (a); black bolting cloth - sec. 44.002 (d); rubber policeman (nonglass rod); petri dishes (plastic).

Procedure. Wash the entire outside surface of product container with a spray of hot water while holding the container over a No. 230 sieve. Remove product residues, if present, with a rubber policeman while spraying with hot water; then spray with water until the exposed jar threads and lid are completely cleaned of product residues and adhering glass fragments. Transfer material retained on the sieve to either black or white filter paper for microscopic examination.

Internal examination for glass fragments

(Caution: Jar lid must not be disturbed until all container integrity tests have been completed. The purpose of this examination is to recover all glass fragments inside the container.)

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glass. Glass can be confirmed by optical-crystallographic methods to test for isotropism and refractive indices. Good laboratory quality assurance practices will ensure the integrity of analyses of suspect glass fragments.

**SHIPPING AND HANDLING**

If possible, the person who finds a suspect glass fragment should be instructed to seal it in a glassine or wax paper envelope and ship it in a well-padded cardboard package. Use of a glass container may compromise the integrity of the specimen, and plastic containers develop electrostatic charges that make removal of very small particles difficult. Great care and restraint must be exercised so as not to compromise the integrity of the item or, even worse, to permit the microscopic suspect fragment to flip out while a glassine container envelope is being opened. Unopened consumer products should be well padded in the shipping box to prevent breakage.

Opened containers of consumer products must be resealed to prevent product leakage and, if necessary, frozen or refrigerated to prevent microbial deterioration during shipment. When tampering of opened products is suspected, the original lid should not be used to reseal the container because evidence may be compromised. Either reseal with a clean lid from another container or use a strong plastic wrap, such as Saran Wrap™. Suspected glass fragments should never be affixed directly to adhesive tape of any type. Adhesive compounds interfere with microscopic analysis, and their removal from minute glass fragments involves difficult and tedious handling.

**WORK ENVIRONMENT, EQUIPMENT, AND CONDITIONS**

A work area free of extraneous glass fragments is a prerequisite to glass confirmation analyses. The work area must be wiped clean before each new sample is opened to prevent cross-contamination.

**Glass handling chamber**

The size of glass to be analyzed may range from a fraction of a millimeter to an intact container. Use a clear Plexiglas™ glass-handling chamber to contain fragments during initial sample opening or container chipping and when reducing fragment size or preparing slides. The handling chamber should be of adequate size, with a hinged top for introducing specimens and two minimum-sized hand holes. The bottom of the chamber should be lined with either white or black paper, whichever provides the best contrast with theglass specimen. The liner must be changed between examinations to prevent cross-contamination.

**Equipment and reagents**

Glass microscope slides and coverslips must be used to mount suspect fragments for examination. Both the slide and coverslip should be thoroughly cleaned to remove intrinsic glass fragments. Glass slides and coverslips used in the index determination should have a refractive index of 1.515, which is the refractive index of most commercial slides and coverslips. Refractive indices of standard liquids should be checked with a refractometer because they may vary with the age, use, and storage conditions of the liquids. Newly purchased refractive index liquids should be certified (Cargille Laboratories, Inc., or equivalent) and free of PCBs.

**Temperature correction**

All refractive index determinations must be temperature-corrected (3). The working temperature should be determined with a calibrated thermometer placed directly on the microscope stage. This will account for heat generated by the microscope light source and indicate the actual temperature at which the refractive index was determined.

**Analyst qualifications**

Glass confirmation analyses should be performed only by trained, qualified analysts. Analysts who do not routinely perform these tests should refamiliarize themselves with the procedure by reviewing the appropriate literature cited in the bibliography and by performing the polarized light microscopy on known specimens of glass, plastic, sand, and similar materials.

**ANALYTICAL PROCEDURE**

**Physical description**

Before analysis of suspect fragments or containers, describe each item completely. Sketch or photograph suspect fragments, adding descriptive features such as color, transparency, fracturing details, and dimensions (in millimeters) and weights (in grams or milligrams, as appropriate) to the illustration. Examine product containers for chipped areas and other defects and describe or note on the illustration. If both a suspect fragment and an associated container are to be analyzed, check each fragment for fit to all chipped areas of the container.

**Preparing fragments for analysis**

If the glass sample is contaminated by product material, label adhesive, lid compound, or other residue that would interfere with the analysis, clean it by using the appropriate procedures before beginning the tests. Obtain particles approximately 120 mesh (125μm or 0.005 inch) from suspect fragments or containers for mounting to determine isotropism and refractive index; save a single unaltered piece of each fragment for other possible tests. Large fragments and whole containers can be partially reduced in size by breaking off small particles with sturdy forceps or needle-nosed pliers inside the handling chamber. Size can be reduced further by wrapping a small piece in glassine paper, placing it on a steel backing plate of suitable size, and breaking it with a small flat-faced hammer. Prepare microscope slide mounts of the powdered material in refractive index liquids inside the specimen handling chamber.
Test for isotropism

For details of this test, see An Introduction to the Method of Optical Crystallography (3). To determine refractive index, see Official Methods of Analysis, sec. 36.280 (1).

Characterization of glass

Once the suspect material is known to be a glass substance, the next phase in a forensic investigation is to determine its source. (For additional information on the forensic examination of glass fragments, see 25,26,29-31,34,35,37,45.) No single attribute should be used alone to establish the source of contamination. Both physical and chemical properties of the contaminating fragment and the alleged contributing material must be determined. Because of the cost and complexity of many of the examinations, the results of each analysis (e.g., refractive index of the suspect fragment and the possible source) should be compared before proceeding to the next test. Some properties and characteristics of glass to be considered are the shape (physical match or jig-saw fit); surface features, such as flatness and fracturing (9,14,21,24); color; density (2,6,18,19,23,33); refractive index (3,4,7,8,10-13,15,17,22,27,32,36,38,39,43); elemental composition (2,6,18,19,23,33); and dispersion properties (13,15,36).

COMMENTS

Although glass contamination of consumer products on a national scale is a rare event, the experiences gained in the 1986 nationwide incident of baby foods contaminated with glass have contributed to analytical improvements. At the onset of the crisis, many problems surfaced and new and unfamiliar situations were encountered; administrative protocols and analytical methods were either incomplete or entirely lacking. In short, no comprehensive source of information or guidance existed. Within a matter of days, however, representatives from all levels of government met in a cooperative effort to develop a working plan to deal with the problem. Over the course of 3 months, samples were collected and analyzed, and the problems involved in developing the necessary analytical techniques were solved. The procedures described in this paper, therefore, provide a proven basis for improved glass analysis in foods.

REFERENCES

36. Slater, D. P., and W. Fong. 1982. Density, refractive index, and dis-