

**United States Department of Agriculture  
Agricultural Marketing Service, Science & Technology  
Pesticide Data Program**

SOP No.: LABOP 8		Page 1 of 6
Title: Sample Receipt, Storage, Archiving, and Disposal for Whole Milk		
Revision: Original	Replaces:	Effective: 1/1/96

**1. Purpose:**

To provide standard procedures for the receipt, storage, archiving and disposal of USDA-AMS Pesticide Data Program (PDP) whole milk samples.

**2. Scope:**

This SOP shall be used in all analytical laboratories which are conducting pesticide residue studies for PDP whole milk samples including support laboratories conducting stability or other types of pesticide residue which impact the program.

**3. Outline of Procedure:**

- 5.1 Sample Receipt
- 5.2 Sample Storage
- 5.3 Storage of Extracts
- 5.4 Storage of Reserve Samples
- 5.5 Disposal of Reserve Samples
- 5.6 Disposal of Extracts

**4. References:**

Good Laboratory Practice Standards 40CFR Parts 160.47, 160.51, and 160.81, EPA,  
August 17, 1989

Memorandum to State PDP Laboratories from Dr. Robert Epstein, Science Division,  
AMS, April 25, 1991

Memorandum to State PDP Laboratories from Dr. Robert Epstein, Science Division,  
AMS, May 22, 1991

USDA/AMS SOP LABOP-1 Sample Receipt, Storage, Archiving, and Disposal, rev. 3,  
effective 5/2/94

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**5. Specific Procedures:**

These operating procedures are presented as general procedures. Each laboratory shall have written operating procedures which shall provide specific details concerning how the procedures have been implemented in that laboratory.

**5.1 Sample Receipt**

- a. Milk samples shall be inspected upon arrival. Those samples received which are physically damaged, visually separated, and/or strong smelling shall be discarded and not analyzed.

Milk samples shall arrive at the laboratory in plastic containers which may be sealed in plastic bags by the collectors. If the containers or bags should split open during shipment, the laboratory shall document this on the Sample Information Form. The containers or bags shall not be opened, but shall be visually inspected for any deteriorating condition (e.g., plastic bags leaking, visually separated, discolored) which would make the sample inedible.

- b. Sample condition upon arrival (e.g., good, or entire sample spoiled - unable to analyze) shall be documented on the Sample Information Form. Documenting date and time received on the Sample Information Form is also acceptable.
- c. Each sample shall be assigned a unique laboratory identification number. This number shall be recorded in permanent non-smearing ink or waterproof, freezerproof stickers on the sample container and the accompanying paperwork.
- d. Each laboratory shall maintain a log of samples received. Suggested methods are:
1. Each sample shall be logged into a bound notebook with ink. Mistakes shall be crossed out, (one single line, no whiteout) and
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corrections dated and initialed. Minimum information for the logbook includes sample numbers, date and time received (unless documented on the Sample Information Form), and who received the sample. Other information may include commodity type, reference to analytical method, results and date when results were reported.

2. Computer logs are also acceptable. The laboratory shall assure that verified hard copies are generated on a routine basis, and that electronic storage of data follows acceptable practices. Refer to USDA/AMS SOP DATA-5.

## 5.2 Sample Storage

- a. All refrigerators and freezers used for PDP samples shall have controlled access. A logbook for each refrigerator and freezer shall be maintained that details sample traffic and periodic temperature checks. The temperature checks shall be made each working day, or the laboratory may use automatic temperature recording devices. All logbook entries are to be made in ink. Mistakes shall be crossed out, (one single line, no white-out) and corrections dated and initialed.
  - b. Samples shall be stored in refrigerators and freezers separate from standards.
  - c. Milk samples still sealed shall be refrigerated at 4°C or lower for a period not to exceed 240 hours from date of receipt until the sample is extracted.
  - d. Four pre-weighed sample aliquots for violative sample re-extraction should be frozen at -20°C or lower along with freezer spikes. Freezer spikes can be split into two groups (with one or more mixes) and each group spiked on alternating months or can all be spiked with every sample set.
  - e. If it is not possible to extract the sample within 240 hours from the date of receipt, then the pre-weighed aliquots, from section 5.2.d above, may be
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held for longer periods of time at -20°C or lower with the appropriate freezer spikes and analyzed at a later time, not to exceed 2 months.

5.3 Storage of Extracts

- a. Extracts shall be stored at 4°C or lower. In an internal SOP each laboratory shall establish procedures to assure or develop evidence that evaporation of the solvent since the last use is not occurring in sample extracts. Suggested procedures are weighing the extract plus the bottle/tube or recording/markings the volume of the extract using the calibration markings on the side of the bottle/tube.
- b. Injection vials shall be prepared immediately before injection or may be held at 4°C or lower before injection. Each laboratory shall in an internal SOP establish procedures for the number of re-injections per vial, as well as the maximum amount of time that a vial may be left at room temperature and still be injected.

5.4 Storage of Reserve Samples

- a. The frozen reserve portions of violative and non-violative milk samples shall be retained at -20°C or lower until final QA review.

5.5 Disposal of Reserve Samples

- a. The reserve sample may be discarded after time period(s) specified in section 5.4 have elapsed. Disposal shall be documented (e.g., in the freezer log or sample log) and shall contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.

5.6 Disposal of Extracts

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- a. Each laboratory shall establish the proper procedures for disposal (e.g., disposal by a licensed contractor) of its injection vials in an internal SOP.
  
  - b. The extracts may be discarded after time period(s) specified in the laboratory's internal SOP have elapsed. Disposal shall be documented (e.g. in the refrigerator/freezer log) and shall contain a minimum of date of disposal, initials of the individual who discarded the sample, and sample number(s) or set number(s). Each laboratory shall establish the proper procedures for disposal (e.g., disposal by a licensed contractor) of its extracts in an internal SOP.
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