

**APPENDIX  
N  
GENERAL  
TRAINING  
PROGRAM**

**2015**

# APPENDIX N GENERAL TRAINING PROGRAM

## INDEX

FDA 2400 FORMS	1
APPENDIX N QC FORMS	2
APPENDIX N MEMOS	3
APPENDIX N REPORTING	4
PMO – APPENDIX N	5

# **FDA 2400 FORMS**

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
GENERAL REQUIREMENTS (REVISION 10/13)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
IDEXX-NEW SNAP® BETA-LACTAM TEST (raw commingled  
cow milk , raw commingled camel milk and raw commingled goats  
milk) (REVISION 10/13)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
CHARM SL (raw commingled cow, sheep, water buffalo and goat  
milk) AND SL-3 (raw commingled cow milk) BETA-LACTAM  
TESTS (REVISION 8/14)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
CHARM®FLUSLBL (raw commingled cow milk) FLUNIXIN AND  
BETA-LACTAM (REVISION 2/12)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
CHARM II BETA-LACTAM ASSAYS (REVISION 1/14)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
CHARM II COMPETITIVE ASSAYS FOR NON BETA-LACTAMS  
(REVISION 1/14)

APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
NEOGEN BETASTAR® (raw commingled cow milk) BETA-  
LACTAM TEST (REVISION 1/14)

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
GENERAL REQUIREMENTS**

**[Unless otherwise stated all tolerances  $\pm 5\%$ ]**

**1. Work Area** \_\_\_\_\_

- a. Ample working space and utilities \_\_\_\_\_
- b. Clean well ventilated, test kit used in temperature range specified by manufacturer, reasonably free from dust and drafts \_\_\_\_\_
- c. Adequate lighting, **[NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities, > 50 foot-candles at working surface (pref. 100)]** \_\_\_\_\_
- d. Eating and drinking not permitted in immediate testing area \_\_\_\_\_

**2. Storage Space** \_\_\_\_\_

- a. Cabinets, drawers, and shelves adequate \_\_\_\_\_
- b. Areas neat, clean and orderly \_\_\_\_\_

**3. Temperature Measuring Devices** \_\_\_\_\_

- a. National Institute of Standards and Testing (NIST) traceable thermometer or other temperature measuring device with certificate. Must be checked annually at ice point \_\_\_\_\_

1. Reference temperature measuring device identity: \_\_\_\_\_

Serial #	Date of Certificate	Ice Point Date
----------	---------------------	----------------

a: \_\_\_\_\_

b: \_\_\_\_\_

2. Graduation/recording interval not greater than 1.0°C **[NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities, 0.5°C]** \_\_\_\_\_

- b. Range of test temperature measuring device appropriate for designated use \_\_\_\_\_

1. Mercury-in-glass (MIG), alcohol/spirit-in-glass (AIG) or electronic/digital thermometers in degrees centigrade \_\_\_\_\_

2. Plastic lamination recommended for mercury thermometers \_\_\_\_\_

3. Graduation/recording interval not greater than 1.0°C **[NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities, 0.5°C]** \_\_\_\_\_

- c. Accuracy of all test temperature measuring devices checked before initial use and annually \_\_\_\_\_
  - 1. Checked against NIST traceable thermometer \_\_\_\_\_
  - 2. Accurate to  $\pm 1^{\circ}\text{C}$  when checked at temperature(s) of use \_\_\_\_\_
  - 3. Results recorded/documented and individual devices tagged \_\_\_\_\_
    - a. Tag includes identification/location, date of check, temperature(s) checked and correction factor(s), as applicable \_\_\_\_\_
- d. Temperature measuring devices are to be read to the nearest graduation/ recording interval, optionally labs may interpolate between graduations \_\_\_\_\_
- e. Temperature Monitoring Systems (wired/wireless) \_\_\_\_\_
  - 1. The software must record temperature reading from each sensor/probe in the piece of equipment being monitored at the same or greater frequency as stipulated for MIG or AIG thermometers. Optionally, set to register an alert/alarm when out of the acceptable temperature range \_\_\_\_\_
    - a. When temperature(s) are out of acceptable range for greater than two hours, event must be documented and corrective action taken as necessary; maintain records \_\_\_\_\_
  - 2. Optionally, a minimum two-day backup power source (battery/electrical) for the temperature monitoring system and/or all required sensors/probes, remote signal device and monitor/controller may be employed in case of power failure \_\_\_\_\_
  - 3. Temperature monitoring system records for each piece of equipment must be available/accessible for auditing as described in item 3.c above \_\_\_\_\_
- f. Automatic temperature recording instruments, if used, compared weekly against an accurate thermometer; maintain records \_\_\_\_\_
- g. Temperature measuring device(s) checked for accuracy at another location \_\_\_\_\_
  - 1. Location: \_\_\_\_\_
  - 2. Current and acceptable \_\_\_\_\_
  - 3. Copy of record on-site \_\_\_\_\_
- h. Dial thermometers not used in the laboratory \_\_\_\_\_

4. Refrigeration (Sample \_\_\_\_\_)  
 (Reagent \_\_\_\_\_)
- a. Size adequate for workload \_\_\_\_\_
  - b. Maintains samples at 0.0-4.5°C \_\_\_\_\_
  - c. Used for storage of milk or milk products, media and reagents only \_\_\_\_\_
    - 1. Not to be used to store food or drink for consumption \_\_\_\_\_
  - d. Record/download temperature (corrected) daily, from two temperature measuring devices with bulbs or sensor/probe immersed in liquid (in sealed containers) **[NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities, AM and PM]** \_\_\_\_\_
  - e. Temperature measuring devices located on upper and lower shelves of use \_\_\_\_\_
5. Freezer (\_\_\_\_\_)
- a. Size adequate for workload \_\_\_\_\_
  - b. Maintains -15°C or below \_\_\_\_\_
  - c. Used for storage of frozen milk products, controls, media and reagents only \_\_\_\_\_
    - 1. Not to be used to store food or drink for consumption \_\_\_\_\_
  - d. Record/download temperature (corrected) daily, from temperature measuring device with bulb or sensor/probe immersed in liquid (in sealed container) **[NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities, AM and PM]** \_\_\_\_\_
6. Balance, Electronic (if necessary) \_\_\_\_\_
- a. Weight capability appropriate for intended use \_\_\_\_\_
  - b. Appropriate sensitivity for accuracy check of pipetting devices within a tolerance of ±5% (0.001g sensitivity appropriate in most instances) \_\_\_\_\_
  - c. Checked monthly with Class S or S1, or equivalent ASTM 1, 2, or 3 weights corresponding to normal use of balance (At a minimum, Appendix N drug residue testing only laboratories must check the balance calibration within 30 days prior to the pipettor accuracy check) \_\_\_\_\_
    - 1. Certificate or other verification of authenticity \_\_\_\_\_
    - 2. Free from excessive wear, filth and corrosion \_\_\_\_\_

3. Weights within class tolerance \_\_\_\_\_

d. Checked annually by a qualified service representative \_\_\_\_\_

1. Date of Last Check: \_\_\_\_\_

e. Maintain records \_\_\_\_\_

**7. Pipettors, Calibrated, Fixed Volume or Electronic Only [Required for NCIMS Accredited Laboratories and Certified Industry Supervisor Facilities]** \_\_\_\_\_

a. Pipettors etched with identification (imprinted serial numbers acceptable) and tagged with date accuracy checked \_\_\_\_\_

b. Appropriate tips for pipettor(s) used \_\_\_\_\_

c. Follow manufacturer's instructions unless otherwise stated regarding proper technique for use \_\_\_\_\_

d. Pipetting devices accuracy checked on-site \_\_\_\_\_

e. Pipetting devices accuracy checked at another location \_\_\_\_\_

1. Location: \_\_\_\_\_

2. Current and acceptable \_\_\_\_\_

3. Copy of record on-site \_\_\_\_\_

f. Check accuracy with ten (10) consecutive measurements, by weight or by volume (>1.0 ml using a class A graduated cylinder), using separate tip for each measurement, every 6 months \_\_\_\_\_

g. Average of all 10 measurements must be  $\pm 5\%$  of specified delivery volume; maintain records \_\_\_\_\_

h. Or, check accuracy with 10 consecutive readings once every 6 months using the Artel PCS Pipette Calibration System, average of all 10 readings must be  $\pm 5\%$  of specified delivery volume; maintain records/printouts \_\_\_\_\_

1. PCS Calibration System Validation, upon receipt, validate the instrument by following the manufacturer's protocol \_\_\_\_\_

2. PCS Pipette System Quality Control \_\_\_\_\_

a. Following manufacturer's Procedure Guide and instrument prompts, perform an instrument calibration every 30 days or just prior to use \_\_\_\_\_

b. Record results and file Calibration Certificate (printout) \_\_\_\_\_

3. Store reagent kits and Instrument Calibrator kits at room temperature \_\_\_\_\_

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

4. Reagent Blanks and Sample Solutions are the same lot \_\_\_\_\_

5. PCS Pipette Calibration System Procedure, follow manufacturer's Procedure Guide and instrument prompts \_\_\_\_\_

i. Maintain records \_\_\_\_\_

**8. Deionized Water or Equivalent, or as specified by manufacturer** \_\_\_\_\_

### SAMPLES

**9. Sample Requirements** \_\_\_\_\_

a. Appendix N tanker sample(s) \_\_\_\_\_

1. Prevent contamination with disinfectants from hands or other sources \_\_\_\_\_

2. Ascertain temperature of bulk milk tanker; maintain records \_\_\_\_\_

3. Secure a representative sample for testing. If sample will not be tested without delay then a temperature control (TC) sample must be taken at the same time, transported, and maintained with the tanker sample(s) until it is tested \_\_\_\_\_

4. Tanker sample(s) tested promptly upon arrival at the testing location (date and time recorded) \_\_\_\_\_

a. Determine sample temperature by inserting a pre-cooled thermometer (pre-cooling of electronic/digital thermometer probes is not necessary) into temperature control \_\_\_\_\_

b. Temperature of bulk milk tanker may be used for temperature as received and tested if sample testing begins without delay \_\_\_\_\_

b. Appendix N Producer Trace Back Samples (Sample(s) not meeting the conditions outlined below may still be tested. The certified laboratory or CIS will document the condition of the sample(s)) \_\_\_\_\_

1. Samples should be accompanied by a temperature control (TC). If no TC, aliquot sample(s) for testing and measure temperature using one of the producer samples \_\_\_\_\_

2. Sample(s) should not be leaking \_\_\_\_\_

3. Tops of samples should be protected from direct contact with ice \_\_\_\_\_

- 4. Unprotected samples should not be submerged in water and/or ice or slush
- 

## PERFORMANCE TESTING

### 10. Performance Testing

---

- a. Run a positive and negative control before use on each new lot of kits, must give appropriate results; maintain records
  - b. Run a negative and positive control **DAILY** (on days testing), at each test site, must give appropriate results, if not, re-run controls (may be necessary to prepare new controls); if problem persists discontinue testing, contact State regulatory and seek technical assistance; maintain records
  - c. If available from manufacturer, check instrument calibration with check devices **DAILY** (on days testing), must give appropriate results, if not, discontinue testing and seek technical assistance; maintain records
  - d. If more than one analyst performs analysis, have different analyst run performance check on rotational basis
- 

### FOLLOW-UP ON TEST KIT POSITIVE RESULTS [Must comply with PMO Appendix N, current revision]

### 11. Verification of Initial Positive Tanker Samples

---

- a. The **SAME** sample is re-tested by the **SAME** analyst using the **SAME** test kit in **DUPLICATE** along with a positive and negative control
  - b. Positive and negative controls give the appropriate result(s)
    - 1. If positive and/or negative controls do not give appropriate results, re-run controls and samples. If problem persists seek technical assistance
  - c. If one or both duplicates is positive the tanker sample is **PRESUMPTIVE POSITIVE** and the sample is referred to the designated certified laboratory or Certified Industry Supervisor (CIS) as specified by the facility's protocol as per Agreement with the State Regulatory Agency
  - d. Presumptive positive samples must be forwarded to a certified laboratory, not tested by screening facility; producer samples must be tested by a certified laboratory
  - e. If both duplicates are negative milk may be received and processed, record and report as **NOT FOUND**
-

- f. Complete applicable section of Positive Report form and maintain records of all analyses  
\_\_\_\_\_
- 1. For Presumptive Positive samples, maintain a copy of the Positive Report form and forward the original to the certified laboratory or CIS  
\_\_\_\_\_

**12. Confirmation of Presumptive Positive Tanker Samples**  
**[Only in an accredited laboratory or by a CIS (refer to M-a-85 current revision for listing of test kits to assure equivalence)]**

- a. The **SAME** sample [or if it can be demonstrated that the original sample is suspect, a re-sample may be used at the State's discretion] is tested in **DUPLICATE** along with a positive and negative control  
\_\_\_\_\_
- b. Positive and negative controls give the appropriate result(s)  
\_\_\_\_\_
- 1. If positive and/or negative control do not give appropriate results, re-run controls and samples, if problem persists seek technical assistance  
\_\_\_\_\_
- c. If one or both duplicates is positive the tanker sample is **CONFIRMED POSITIVE**, milk may not be processed, contact State Regulatory  
\_\_\_\_\_
- d. Producer trace back performed on all producer samples from the load, see item 13  
\_\_\_\_\_
- e. If both duplicates are negative milk may be received and processed, record and report as **NOT FOUND**, producer trace back is not performed  
\_\_\_\_\_
- f. Complete applicable section of Positive Report form and maintain records of all analyses  
\_\_\_\_\_
- 1. For Confirmed Positive samples, maintain a copy of the Positive Report form and forward the original to the State Regulatory Agency  
\_\_\_\_\_

**13. Trace back of Producers on a Confirmed Positive Tanker**  
**[Only performed in an accredited laboratory or by a CIS (refer to M-a-85 current revision for listing of test kits to assure equivalence)]**

- a. Samples must be between 0.0 and 4.5°C. Maintain records  
\_\_\_\_\_
- b. Perform an initial single test on each producer sample  
\_\_\_\_\_
- c. Any producer sample that is positive must be re-tested  
\_\_\_\_\_
- d. The **SAME** sample is re-tested by the **SAME** analyst using the **SAME** test in **DUPLICATE** along with a positive and negative control  
\_\_\_\_\_
- e. Positive and negative controls give the appropriate result(s)  
\_\_\_\_\_
- 1. If positive and/or negative control do not give appropriate results, re-run controls and samples, if problem persists seek technical assistance  
\_\_\_\_\_

- f. If one or both duplicates is positive the producer sample(s) is (are) **POSITIVE** \_\_\_\_\_
- g. If both duplicates are negative record and report the appropriate producer sample(s) **NOT FOUND** \_\_\_\_\_
- h. Complete applicable section of Positive Report form and maintain records of all analysis \_\_\_\_\_
  - 1. For Confirmed Producer Positive samples, maintain a copy of the Positive Report form and forward the original to the State Regulatory Agency \_\_\_\_\_

**REPORTING AND RECORDS**

**14. Reporting and Records** \_\_\_\_\_

- a. Report as **Positive (+)** for beta-lactam, specific drug or inhibitor (when a non-specific microbial inhibitor test used without beta-lactamase) when demonstrated \_\_\_\_\_
- b. Report as **Not Found (NF)** when demonstrated \_\_\_\_\_
- c. Record test performed, interpretation of unknowns (samples) and controls \_\_\_\_\_
- d. Records, including all printouts, maintained for 2 years \_\_\_\_\_

**MISCELLANEOUS**

**15. Miscellaneous** \_\_\_\_\_

- a. Current Safety Data Sheets (SDS) accessible to analysts \_\_\_\_\_
- b. Current, applicable survey forms available in laboratory \_\_\_\_\_
- c. Positive Report forms available with instructions \_\_\_\_\_
- d. Personnel adequately trained \_\_\_\_\_
- e. Required split/check sample participation \_\_\_\_\_

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM**

**IDEXX - NEW SNAP® BETA-LACTAM TEST  
(Raw Commingled Cow, Raw Commingled Camel, and Raw Commingled Goat Milk)  
IMS # 9-11**

[Unless otherwise stated all tolerances are ±5%]

**GENERAL REQUIREMENTS**

- 1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

**SAMPLES**

- 2. See App. N GR item 9 \_\_\_\_\_

**APPARATUS & REAGENTS**

- 3. **Equipment** \_\_\_\_\_

- a. Heater block with SNAP insert thermostatically controlled at 45±5°C \_\_\_\_\_

- 1. Check temperature by placing standardized temperature measuring device in a tube containing liquid (bulb submersed); maintain records \_\_\_\_\_

- 2. Or, use 6-inch partial immersion thermometer placed directly into small thermometer well in middle of heating unit; maintain records \_\_\_\_\_

- 3. Temperature measuring device for each incubator (App. N GR item 3) \_\_\_\_\_

- b. IDEXX Readers for SNAP devices, with printer or data download capability \_\_\_\_\_

- 1. SNAPshot® Reader \_\_\_\_\_

- a. Check Set, Part Number 87-05856-01 (black skirt) \_\_\_\_\_

- 2. SNAPshot® DSR Reader \_\_\_\_\_

- a. Check Set, Part Number 87-14761-00 (blue skirt) \_\_\_\_\_

- c. Pipettor - 450 µL and disposable tips (see App. N GR item 7) \_\_\_\_\_

- d. Or single use 450 µL poly-pipet with indicator line to measure amount of sample, supplied by manufacturer (**screening only**) \_\_\_\_\_

- e. Timer \_\_\_\_\_

**4. Reagents**

a. SNAP Kit

Lot #: \_\_\_\_\_ Exp Date: \_\_\_\_\_

QC Date: \_\_\_\_\_ By: \_\_\_\_\_

1. Sample tubes containing reagent pellet

b. Positive Control

1. IDEXX Penicillin Positive Control

Lot #: \_\_\_\_\_ Exp Date: \_\_\_\_\_

c. Negative Control

1. Previously tested negative raw milk (item 5.d)

**5. Reagent stability**

- a. Kits must be received within 72 hours if shipped non-refrigerated; over 72 hours must be shipped refrigerated

- b. Store kits at 0-7°C, maintain no longer than manufacturer's expiration date

- c. Positive Control- Manufacturer supplied, maintain no longer than manufacturer's expiration date

1. Store according to label instructions
2. Reconstitute as per manufacturer's instructions with fresh or frozen previously screened beta-lactam negative raw milk.
3. Positive control must produce greater than 1.2 on the IDEXX reader; maintain records

Reader value: \_\_\_\_\_

4. Store reconstituted positive control at 0.0-4.5°C for no more than 24 hours

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

d. Negative Control - beta-lactam negative raw milk (fresh or frozen) \_\_\_\_\_

1. Negative control must produce less than 0.95 on the IDEXX reader; (SNAP Test Negative Control can be any of the approved species milk); maintain records \_\_\_\_\_

Sample ID: \_\_\_\_\_ Date Tested: \_\_\_\_\_

Reader value: \_\_\_\_\_

2. Store fresh negative control milk at 0.0-4.5°C for no more than 72 hours \_\_\_\_\_

3. Negative control milk frozen for later use \_\_\_\_\_

a. Aliquot within 24 hours and freeze at -15°C or colder in a non-frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

b. Thaw frozen milk at 0.0-4.5°C \_\_\_\_\_

c. Once thawed mix thoroughly, **Do Not** use if noticeable protein precipitation is present after thawing \_\_\_\_\_

d. Thawed negative control milk held at 0.0-4.5°C and used within 24 hours \_\_\_\_\_

4. Milk controls may not be refrozen \_\_\_\_\_

**6. Daily Performance and Operation Checks (see App. N GR item 10)** \_\_\_\_\_

a. Read Performance Check Set (Device #1 as Negative and Device #2 as Positive) \_\_\_\_\_

b. Both devices must read within the limits as indicated on the storage box label of the check set devices \_\_\_\_\_

Positive Range: \_\_\_\_\_ Negative Range: \_\_\_\_\_

c. If check sets fail, call IDEXX before proceeding \_\_\_\_\_

**TECHNIQUE**

**7. Test Procedure** \_\_\_\_\_

a. Set out required number of SNAP devices, sample tubes and pipets for the samples to be tested \_\_\_\_\_

1. Discard unused, un-refrigerated devices at the end of the day \_\_\_\_\_

- b. Pre-warm heater block(s) to  $45\pm 5^{\circ}\text{C}$ , and maintain  $45\pm 5^{\circ}\text{C}$  range for at least 5 min before beginning the test \_\_\_\_\_
- 1. Check initial pre-heating with a temperature measuring device (see App. N GR item 3); maintain records \_\_\_\_\_
- 2. Continuous use block heaters, check temperature daily with temperature measuring device (see App. N GR item 3); maintain records \_\_\_\_\_
- c. Label each device and sample tube \_\_\_\_\_
- d. Place device(s) on incubator block(s) \_\_\_\_\_
- e. Verify that blue reagent pellet is in bottom of tube before removing cap. If not in bottom, tap to bring down \_\_\_\_\_
- f. Remove and discard sample tube cap(s) \_\_\_\_\_
- g. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min (samples must be in appropriate containers to allow the use of vortexing) \_\_\_\_\_
- h. Add 450 uL of mixed sample/control to corresponding tube(s) \_\_\_\_\_
- 1. Using Pipettor (item 3.c) with a new tip for each sample/control draw up 450  $\mu\text{L}$  avoiding foam and bubbles \_\_\_\_\_
- a. Remove tip from liquid \_\_\_\_\_
- b. While holding the pipettor vertically, expel test portion to sample tube \_\_\_\_\_
- 2. Using a new manufacturer provided single-use 450  $\mu\text{L}$  poly-pipet (item 3d.) for each sample/control (**Screening Only**) \_\_\_\_\_
- a. Draw up 450 uL of sample to indicator line, avoiding foam and bubbles \_\_\_\_\_
- b. Remove tip from liquid \_\_\_\_\_
- c. While holding poly-pipet vertically, expel test portion to sample tube \_\_\_\_\_
- i. Agitate sample tube(s) to dissolve reagent pellet \_\_\_\_\_
- j. Place tube(s) in heater block next to device with the corresponding ID \_\_\_\_\_
- k. Incubate tube(s) for 5 min (use timer) at  $45\pm 5^{\circ}\text{C}$  \_\_\_\_\_
- l. After incubation, pour contents of each tube into sample well of corresponding device \_\_\_\_\_

- m. Watch blue activation circle, as it begins to disappear push the activator firmly until it "snaps" flush with the body of the SNAP device (device remains on heater block) \_\_\_\_\_
- n. Incubate device for 4 min (use timer) at  $45\pm 5^{\circ}\text{C}$  \_\_\_\_\_
- o. At the end of incubation, visually inspect the control and test spots. The test is invalid and the same sample should be retested with a new SNAP device if:
  - 1. The control spot fails to develop color \_\_\_\_\_
  - 2. Blue streaking occurs in the background or the background is the same color as the sample or control spots \_\_\_\_\_
  - 3. The sample or control spots are not uniform in color or exhibit poor spot quality \_\_\_\_\_
- p. Insert only valid tests in the reader **IMMEDIATELY (no longer than 30 sec)** after completion of incubation \_\_\_\_\_

**8. Interpretation with Idexx Reader for SNAP Devices** \_\_\_\_\_

- a. IDEXX Reader for SNAP devices automatically prints results as Positive or Negative (NF) \_\_\_\_\_

**9. Verification of Initial Positive Tanker Samples (see App. N GR item 11); Confirmation of Presumptive Positive Tanker Samples (see App. N GR item 12); and Traceback of Producer(s) on a Confirmed Positive Tanker (see App. N GR item 13)** \_\_\_\_\_

**10. Reporting (see App. N GR item 14)** \_\_\_\_\_

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM**

**CHARM® SL (Raw Commingled Cow, Sheep, Water Buffalo and Goat Milk), IMS #9-C13  
AND  
Charm 3 SL3 (Raw Commingled Cow Milk), IMS #9-C15**

**BETA-LACTAM TESTS**

**[Unless otherwise stated all tolerances are ±5%]**

**GENERAL REQUIREMENTS**

1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

**SAMPLES**

2. See App. N GR item 9 \_\_\_\_\_

**APPARATUS & REAGENTS**

3. Equipment \_\_\_\_\_

- a. Charm Sciences Strip Incubator:  
56±1°C 8 min timer - SL beta-lactam test;  
56±1°C 3 min with internal timer – SL3 beta-lactam test  
56±1°C Charm EZ display when message “Add milk to strip and close door” \_\_\_\_\_

1. Clean and level. Check temperature daily (day of use);  
maintain records \_\_\_\_\_

- a. Charm EZ printout acceptable for daily temperature check  
(annual accuracy check required); maintain records \_\_\_\_\_

2. Temperature measuring device for each incubator  
(App N. GR item 3) \_\_\_\_\_

3. Lid closed (slightly sprung so that timer not active)  
when not running tests \_\_\_\_\_

4. Incubator Temperature: \_\_\_\_\_

5. Timer if not included in incubator  
Incubation Time of internal timer: \_\_\_\_\_

- b. ROSA® Reader, ROSA Pearl Reader (with or without ROSA Barcode  
option), Charm EZ or Charm Sciences equivalent with print out or  
download of data; manual available

Serial Number: \_\_\_\_\_

1. SL beta-lactam test - ROSA Reader V1.03 or higher  
(or if ROSA Pearl Reader or Charm EZ see 3.b.2) \_\_\_\_\_
  - a. Calibrators - 2 line for SL beta-lactam \_\_\_\_\_
 

Two Line Range(s):	Result	
Low: _____	_____	_____
High: _____	_____	_____
  - b. Maintain records \_\_\_\_\_
2. SL3 beta-lactam test - ROSA Pearl Reader V3.00 or higher or  
Charm EZ \_\_\_\_\_
  - a. Calibrators - Low and High for use in all assay channels \_\_\_\_\_
 

Range(s) Solid Color Ranges:	Result	
Low: _____ (darker magenta)	_____	_____
High: _____ (lighter pink)	_____	_____
  - b. Maintain records \_\_\_\_\_
3. Calibrator serial numbers match reader SN \_\_\_\_\_
4. **Do not proceed if out of range.** Manufacturer should be contacted  
for corrective actions \_\_\_\_\_
5. Printer or computer link for hardcopy download \_\_\_\_\_
- c. Pipettor - 300  $\mu$ L and disposable tips (see App. N GR item 7) \_\_\_\_\_
- d. Or single use 300  $\mu$ L ROSA-pipet with overflow bulb to accurately  
measure amount of sample, supplied by manufacturer (**screening only**) \_\_\_\_\_
- e. Optional Centrifuge (Not applicable to SL3 beta-lactam test) - mini  
or equivalent (1200-2000 x g) for frozen controls \_\_\_\_\_

**4. Reagents**

- a. Test Strips (EZ Compatible for Charm EZ)

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

QC Date: \_\_\_\_\_ By: \_\_\_\_\_

- b. Positive Control

- 1. Lyophilized or tablet 5 ppb Penicillin G beta-lactam tests

Lot #: \_\_\_\_\_ Exp Date: \_\_\_\_\_

- c. Negative Control

- 1. Previously negative tested raw milk (item 5.d)

**5. Reagent stability**

- a. SL3 reagents must be received within 72 hours if shipped non-refrigerated; over 72 hours must be refrigerated. (Not applicable to the SL reagents)

- b. Store reagents at 0.0-4.5°C, desiccant blue, maintain no longer than manufacturer's expiration date

- 1. **Do not use if desiccant indicator is white or pink**

- c. Positive Control - Manufacturer supplied, maintain no longer than manufacturer's expiration date

- 1. Reconstitute with Negative Control (raw milk), tested +400 or more positive, used within 48 hours when maintained at 0.0-4.5°C

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

- 2. Or, aliquot within 24 hours and freeze at -15°C or colder in a non frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

- a. Thaw slowly overnight in refrigerator or more rapidly in cold water. Mix well until sample is homogeneous

- 1. **Do not use if there is visible protein precipitation**

- b. Store at 0.0-4.5°C and use within 24 hours; do not refreeze

- c. For **SL ONLY**, centrifuge 3 min and cool \_\_\_\_\_
  - 1. Test portion below fat layer without mixing \_\_\_\_\_

3. Day of use, must produce +400 or greater reading; maintain records \_\_\_\_\_

Test Value: \_\_\_\_\_

**Do not proceed if out of range** \_\_\_\_\_

- d. Negative Control - raw milk tested –600 or more negative; (SL Test Negative Control can be any of the approved species milk) \_\_\_\_\_

Sample ID: \_\_\_\_\_ Test Value: \_\_\_\_\_

Date tested: \_\_\_\_\_

- 1. Use within 72 hours when maintained at 0.0-4.5°C \_\_\_\_\_
- 2. Or, aliquot within 24 hours and freeze at –15°C or colder in a non frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

- a. Thaw slowly overnight in refrigerator or more rapidly in cold water. Mix well until sample is homogeneous \_\_\_\_\_

- 1. **Do not use if there is visible protein precipitation** \_\_\_\_\_

- b. Store at 0.0-4.5°C and use within 24 hours; do not refreeze \_\_\_\_\_

- c. For SL **ONLY**, centrifuge 3 min and cool \_\_\_\_\_

- 1. Test portion below fat layer without mixing \_\_\_\_\_

3. Day of use must produce –600 or more negative; maintain records \_\_\_\_\_

**Do not proceed if out of range** \_\_\_\_\_

## TECHNIQUE

### 6. Daily Performance and Operation Check

- a. See App. N GR items 10.b-d
- b. If using ROSA reader Versions 1.05 and higher, or ROSA-Pearl, use ESC 5 reader function to enter performance monitoring mode of reader; if using Charm EZ, use Menu to enter Performance Monitoring mode and "Perf Mon" to enter daily performance check; refer to manual for directions
- c. Check Calibrators; items 3.b.1 or 3.b.2
- d. Positive and negative controls must give appropriate readings prior to any sample analysis (see App. N GR item 10.a)
- e. Controls in-range when in performance monitoring mode, ROSA reader version 1.05 and higher, ROSA Pearl or Charm EZ
- f. **Do not proceed if out of range**

### 7. Test Procedure

- a. Set out required number of test strips and place them in a dry labeled container at room temperature, or take out strips as needed
  - 1. Discard unused test strips at the end of the day
- b. Label test strips, one for each test sample and each control. Avoid crushing sample compartment(s)
- c. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min (samples/controls must be in appropriate containers to allow the use of vortexing)
  - 1. Centrifuge sheep milk sample(s)/controls that have been previously frozen; refer to 5.c.2.a-c and 5.d.2.a-c
- d. Place strip into appropriate incubator
- e. While holding strip flat, peel back plastic (to 'peel to here' line) to expose sample pad compartment. Avoid lifting the wick and sponge under tape
  - 1. For multiple samples, complete steps 7.d-g for each sample/control, before starting test of next sample

- 2. Complete all samples within 2 min (1 min 15 sec for SL3 test) of placing first strip in incubator \_\_\_\_\_
  
- f. Add 300  $\mu$ L of mixed sample/control to corresponding strip \_\_\_\_\_
  - 1. Using pipettor (item 3.c) with new tip for each sample/control, draw up 300  $\mu$ L avoiding foam or bubbles \_\_\_\_\_
    - a. Remove tip from liquid \_\_\_\_\_
    - b. While holding the pipettor vertically, expel test portion slowly into either side well of appropriate strip \_\_\_\_\_
  - 2. Using new manufacturer-provided ROSA-pipet (item 3.d) for each sample/control **[Screening only]** \_\_\_\_\_
    - a. Squeeze top bulb while holding vertically with bulb and overflow reservoir side pointing down, draw up test portion avoiding foam and bubbles. Sample should completely fill pipet shaft and overflow into the bottom half of the overflow reservoir \_\_\_\_\_
    - b. Remove tip from liquid \_\_\_\_\_
    - c. While holding the ROSA-pipet vertically, expel test portion slowly into either side well of appropriate strip. Excess portion should remain in reservoir \_\_\_\_\_
  
- g. Re-seal plastic firmly around sample pad compartment \_\_\_\_\_
  
- h. ROSA Reader and Charm EZ (read only mode) \_\_\_\_\_
  - 1. Close lid and latch ROSA incubator to start automatic timer in the incubator. If no automatic timer in incubator, set external timer for 8 min for SL. For SL test, incubate 8 min not to exceed 9 min. For SL3 test, incubate 3 min not to exceed 3 min and 30 sec \_\_\_\_\_
  - 2. At end of incubation visually inspect C (Control) line An absent C line, a partial C line or an indistinct C line indicates an invalid test; and the sample/control must be re-tested \_\_\_\_\_
  - 3. Insert only valid test(s) in reader \_\_\_\_\_
    - a. ROSA reader set to appropriate channel \_\_\_\_\_
      - 1. SLBL slow blink for SL beta-lactam test \_\_\_\_\_
      - 2. SLBL solid (no blink) for SL3 beta-lactam test \_\_\_\_\_

3. Press ENTER, reading and interpretation appear in 5 sec, read strips within 5 min (3 min with SL3) of completion of incubation. Strips may be held vertically, sample compartment down while waiting to be read

---

b. Charm EZ automatically sets channel when color coded strip inserted

---

1. Close door; reading and interpretation appear in 5 sec, read strips within 5 min (3 min with SL3) of completion of incubation. Strips may be held vertically, sample compartment down while wait to read

---

i. Charm EZ (incubate and read mode)

---

1. Charm EZ automatically sets channel and incubator temperature when color coded strip inserted. Optionally enter sample ID

---

2. Peel strip (7.e) and add milk (7.f)

---

3. Close door to begin

---

4. Charm EZ automatically prompts for further testing when positive

---

## 8. Interpretation with Reader

---

a. If there is a negative or zero reading on the reader, sample is a **Negative (NF)**

---

b. If there is a positive reading on the reader, sample is **an Initial Positive**

---

## 9. Verification of Initial Positive Tanker Samples (see App. N GR item 11); Confirmation of Presumptive Positive Tanker Samples (see App. N GR item 12); and Traceback of Producer(s) on a Confirmed Positive Tanker (see App. N GR item 13)

---

## 10. Reporting (see App. N GR item 14)

---

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM**

**CHARM® FLUSLBL (raw commingled cow milk)  
FLUNIXIN and BETA-LACTAM TEST  
[Unless otherwise stated all tolerances are ±5%]**

**GENERAL REQUIREMENTS**

1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

**SAMPLES**

2. See App. N GR item 9 \_\_\_\_\_

**APPARATUS & REAGENTS**

3. Equipment \_\_\_\_\_

- a. Charm Sciences Strip Incubator: 56±1°C 8 min timer \_\_\_\_\_

1. Clean and level. Check temperature daily (day of use); maintain records \_\_\_\_\_

2. Temperature measuring device for each incubator (App N. GR item 3) \_\_\_\_\_

3. Lid closed (slightly sprung so that timer not active) when not running tests \_\_\_\_\_

4. Incubator Temperature: \_\_\_\_\_

- b. ROSA® Reader, ROSA Pearl Reader (with or without ROSA Barcode option) or Charm Sciences equivalent with print out or download of data; manual available

Serial Number: \_\_\_\_\_

1. ROSA Reader V1.07 or higher (or if ROSA Pearl Reader see 3.b.2) \_\_\_\_\_

- a. Calibrators - 3 lines for SL-6 beta-lactam \_\_\_\_\_

Three Line Range(s): Result

Low: \_\_\_\_\_

High \_\_\_\_\_

- b. Maintain records

- 2. ROSA Pearl Reader V3.00 or higher \_\_\_\_\_
  - a. Calibrators - Low and High for use in all assay channels \_\_\_\_\_
 

Range(s)	Solid color Ranges:	Result
Low calibrator: _____ (darker magenta)		_____
High Calibrator: _____ (lighter pink)		_____
  - b. Maintain records \_\_\_\_\_
- 3. Calibrator serial numbers match ROSA reader SN \_\_\_\_\_
- 4. **Do not proceed if out of range.** Manufacturer should be contacted for corrective actions \_\_\_\_\_
- 5. Printer or computer link for hardcopy download \_\_\_\_\_
- c. Pipettor - 300 µL and disposable tips (see App. N GR item 7) \_\_\_\_\_
- d. Or single use 300 µL ROSA-pipet with overflow bulb to accurately measure amount of sample; supplied by manufacturer (**screening only**) \_\_\_\_\_

**4. Reagents** \_\_\_\_\_

- a. Test Strips \_\_\_\_\_
 

Lot #: _____	Exp. Date: _____
QC Date: _____	By: _____
- b. Positive Control \_\_\_\_\_
  - 1. Positive Control labeled as "2 ppb Flunixin and 5 ppb Penicillin G Standard" \_\_\_\_\_
 

Lot #: _____	Exp Date: _____
--------------	-----------------
  - 2. Lyophilized 2 ppb Flunixin \_\_\_\_\_
 

Lot # _____	Exp. Date: _____
-------------	------------------
  - 3. Or alternative to 4.b.1-4.b.2, 5 ppb Penicillin G and 2 ppb Flunixin tablet \_\_\_\_\_

4. Preparation

- a. Add 10.0 mL negative raw milk (item 5.d) to Flunixin control (item 4.b.2), and allow to rehydrate for 5 min \_\_\_\_\_
- b. Add 8.0 mL of reconstituted Flunixin control (item 4.b.4.a) to Positive Control (item 4.b.1), and allow to rehydrate for 5 min \_\_\_\_\_
- c. Or, alternative to 4.b.4.a and 4.b.4.b, add 5.0 mL negative milk (item 5.d) to Flunixin and Penicillin G tablet (item 4.b.3), and allow to rehydrate for 5 min \_\_\_\_\_

c. Negative Control \_\_\_\_\_

- 1. Previously negative tested raw milk (item 5.d) \_\_\_\_\_

5. Reagent stability \_\_\_\_\_

- a. FLUSLBL reagents received refrigerated \_\_\_\_\_
- b. Store reagents at 0.0-4.4°C, desiccant blue, maintain no longer than manufacturer's expiration date \_\_\_\_\_
  - 1. **Do not use if desiccant indicator is white or pink** \_\_\_\_\_
- c. Positive Control - Manufacturer supplied; maintain no longer than manufacturer's expiration date \_\_\_\_\_
  - 1. Reconstituted Control (4b4), tested +400 or more positive; use within 48 hours when maintained at 0.0-4.4°C  
Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_ \_\_\_\_\_
  - 2. Or, aliquot within 24 hours and freeze at -15°C or colder in a non frost-free freezer or in an insulated foam container in a frost-free freezer; use within 3 weeks  
Lab Date prep: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_ \_\_\_\_\_
    - a. Thaw slowly overnight in refrigerator or more rapidly in cold water. Mix well until sample is homogeneous \_\_\_\_\_
      - 1. **Do not use if there is visible protein precipitation** \_\_\_\_\_
    - b. Store at 0.0-4.4°C and use within 24 hours; do not refreeze \_\_\_\_\_

3. Day of use, must produce +400 or greater reading; maintain records

Test Value: \_\_\_\_\_

**Do not proceed if out of range**

- d. Negative Control - raw milk tested -400 or more negative with FLUSLBL test

Sample ID: \_\_\_\_\_ Test Value: \_\_\_\_\_

Date tested: \_\_\_\_\_

1. Used within 72 hours when maintained at 0.0-4.4°C
2. Or, aliquot within 24 hours and freeze at -15°C or colder in a non frost-free freezer or in an insulated foam container in a frost-free freezer; use within 3 weeks

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

- a. Thaw slowly overnight in refrigerator or more rapidly in cold water. Mix well until sample is homogeneous

**1. Do not use if there is visible protein precipitation**

- b. Store at 0.0-4.4°C and use within 24 hours; do not refreeze

3. Day of use must produce -400 or more negative with FLUSLBL test; maintain record

**Do not proceed if out of range**

## TECHNIQUE

### 6. Daily Performance and Operation Check

- a. See App. N GR items 10.b-d
- b. Use ESC 5 reader function to enter performance monitor mode of reader; refer to manual for directions
- c. Check Calibrators, items 3.b.1 & 3.b.2

- d. Positive and negative controls must give appropriate readings prior to any sample analysis (see App. N GR item 10.a) \_\_\_\_\_
- e. Controls in-range when in performance monitoring mode \_\_\_\_\_
  - 1. If out of range, manufacturer should be contacted for corrective action, 800-343-2170 \_\_\_\_\_
- f. **Do not proceed if out of range** \_\_\_\_\_

**7. Test Procedure** \_\_\_\_\_

- a. Set out required number of test strips for samples to be tested in one day and place them in a dry labeled container at room temperature, or take out strips as needed \_\_\_\_\_
  - 1. Discard unused test strips at the end of the day \_\_\_\_\_
- b. Label test strips, one for each test sample and each control. Avoid crushing sample compartment(s) \_\_\_\_\_
- c. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min (samples/controls must be in appropriate containers to allow the use of vortexing) \_\_\_\_\_
- d. Place strip into appropriate incubator \_\_\_\_\_
- e. While holding strip flat, peel back plastic (to 'peel to here' line) to expose sample pad compartment. Avoid lifting the wick and sponge under tape \_\_\_\_\_
  - 1. For multiple samples, complete steps 7.d-g for each sample/control, before starting test of next sample \_\_\_\_\_
  - 2. Complete all samples within 2 min of placing first strip in incubator \_\_\_\_\_
- f. Add 300 µL of mixed sample/control to corresponding strip \_\_\_\_\_
  - 1. Using pipettor (item 3.c) with new tip for each control/sample, draw up 300 µL avoiding foam or bubbles \_\_\_\_\_
    - a. Remove tip from liquid \_\_\_\_\_
    - b. While holding the pipettor vertically, expel test portion slowly into either side well of appropriate strip \_\_\_\_\_
  - 2. Using new manufacturer-provided ROSA-pipet (item 3.d) for each control/sample **[Screening only]** \_\_\_\_\_

- a. Depress top bulb while holding vertically with bulb and overflow reservoir side pointing down, draw up test portion avoiding foam and bubbles. Sample should completely fill pipet shaft and overflow into the bottom half of the overflow reservoir \_\_\_\_\_
- b. Remove tip from liquid \_\_\_\_\_
- c. While holding the ROSA-pipet vertically, expel test portion slowly into either side well of appropriate strip. Excess portion should remain in reservoir \_\_\_\_\_

g. Re-seal plastic firmly around sample pad compartment \_\_\_\_\_

h. Close and latch incubator cover to start 8 min. automatic timer in the incubator. Incubate 8 min, not to exceed 9 min \_\_\_\_\_

i. At end of incubation, visually inspect C (Control) line. An absent C line or a partial C line or an indistinct C line indicates an invalid test and the sample/control must be re-tested \_\_\_\_\_

j. Insert only valid test(s) in reader (set to appropriate channel) \_\_\_\_\_

1. SLBL rapid blink for FLUSLBL Test \_\_\_\_\_

k. Press ENTER, reading and interpretation appear in 5 sec, read strips within 5 min of completion of incubation \_\_\_\_\_

**8. Interpretation with ROSA Reader** \_\_\_\_\_

a. If there is a negative or zero reading on the reader, sample is a **Negative (NF)** \_\_\_\_\_

b. If there is a positive reading on the reader, sample is an **Initial Positive** \_\_\_\_\_

**9. Verification of Initial Positive Tanker Samples (see App. N GR item 11); Confirmation of Presumptive Positive Tanker Samples (see App. N GR item 12); and Traceback of Producer(s) on a Confirmed Positive Tanker (see App. N GR item 13)** \_\_\_\_\_

**10. Reporting (see App. N GR item 14)** \_\_\_\_\_

# CHARM® II BETA-LACTAM ASSAYS

## APPENDIX N BULK MILK TANKER SCREENING TEST FORM

Competitive (Raw Commingled Cow Milk and Pasteurized White Milks) IMS #9-C2  
Sequential (Raw Commingled Cow and Goat Milk) IMS #9-C3  
Quantitative (Raw Commingled Cow Milk) IMS #9-C4  
Cloxacillin (Raw Commingled Cow Milk) IMS #9-C9

### GENERAL REQUIREMENTS

1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

### SAMPLES

2. See App. N GR item 9 \_\_\_\_\_

### APPARATUS & REAGENTS

3. **Equipment** \_\_\_\_\_

a. Analyzer heater for 13 x 100 mm tubes \_\_\_\_\_

1. 85±2°C for Competitive Assay \_\_\_\_\_

2. 65±2°C for Sequential Assay \_\_\_\_\_

3. 55±2°C for Quantitative Assay \_\_\_\_\_

4. 35±2°C for Cloxacillin Assay \_\_\_\_\_

5. Temperature checked by electronic display, or by placing accuracy checked temperature measuring device in tube containing liquid (bulb submersed) in heating unit; maintain records \_\_\_\_\_

6. Or, use 6 inch partial immersion thermometer placed directly into small thermometer well in middle of heating unit; maintain records \_\_\_\_\_

7. Temperature measuring device for each incubator (App. N item 3) \_\_\_\_\_

b. Mixer, Maxi-mixer II or equivalent \_\_\_\_\_

c. Centrifuge, Whisperfuge® or Heraeus® (3400 rpm) or equivalent \_\_\_\_\_

d. Scintillation counter, Charm II or equivalent \_\_\_\_\_

e. Scintillation fluid dispenser, set to dispense 3 mL \_\_\_\_\_

1. Check every six (6) months with Class A graduated cylinder and record; maintain records \_\_\_\_\_

f. Cotton swabs \_\_\_\_\_

- g. Borosilicate test tubes, 13 x 100 mm \_\_\_\_\_
- h. Plastic stoppers for tubes \_\_\_\_\_
- i. Pipettors - Fixed Volume or Electronic (see App. N GR item 7) \_\_\_\_\_
  - 1. 300 µL and appropriate tips \_\_\_\_\_
  - 2. 5.0 mL and appropriate tips \_\_\_\_\_
- j. Timer \_\_\_\_\_

**4. Reagents** \_\_\_\_\_

- a. Scintillation fluid – Optifluor or equivalent supplied by manufacturer of test kits \_\_\_\_\_
- b. Competitive, Sequential or Quantitative Assay \_\_\_\_\_
  - 1. Reagent blister packages: microbial binder (green) tablet, tracer reagent (yellow) tablet \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 2. 0.008 IU/mL Penicillin G standard \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 3. Zero control standard \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
- c. Cloxacillin Assay \_\_\_\_\_
  - 1. Reagent blister packages: microbial/antibody binder (white) tablet, tracer reagent (blue) tablet \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 2. 10 ppb Cloxacillin standard \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 3. Zero control standard \_\_\_\_\_
    - Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

**5. Reagent stability** \_\_\_\_\_

- a. All tablet reagents stored at -15°C or below \_\_\_\_\_

- b. Positive Control – Lyophilized 0.008 IU/mL penicillin G or 10 ppb cloxacillin standard for Cloxacillin assay \_\_\_\_\_
1. Reconstitute with 100 mL (measured) Negative Control (allow to sit 15 min prior to use or aliquotting) \_\_\_\_\_  
 Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_
  2. For Quantitative Only: Dilute reconstituted 0.008 IU/mL Penicillin G standard 1:4 with Zero Control Standard \_\_\_\_\_
  3. Use within 48 hours when stored at 0.0-4.5°C \_\_\_\_\_
  4. Or, aliquot within 24 hours and freeze at -15°C or colder in a non-frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_  
 Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_
    - a. Thaw and use within 24 hours. Store at 0.0-4.5°C \_\_\_\_\_
- c. Negative Control – Lyophilized Zero Control Standard (ZCS) or alternatively, raw milk qualified to test similar to ZCS \_\_\_\_\_
- Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_
1. Reconstitute ZCS according to manufacture instructions (allow to sit 15 min prior to use or aliquotting) \_\_\_\_\_
    - a. To qualify raw milk, test sample 3 times and average results. Average must be within +/-10% of ZCS \_\_\_\_\_  
 Lab Prep. Date: \_\_\_\_\_ Lab Exp Date: \_\_\_\_\_
  2. Use within 48 hour when stored at 0.0-4.5°C \_\_\_\_\_
  3. Or, aliquot within 24 hours and freeze at -15°C or colder in a non-frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_  
 Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_
    - a. Thaw and use within 24 hours. Store at 0.0-4.5°C \_\_\_\_\_
- d. Scintillation fluid expires six (6) months after opening \_\_\_\_\_
- Date Opened: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

**TECHNIQUE**

**6. Control Point and Negative Control Average to be determined for each new lot of reagents. Steps 6, 7, and 8 are for the various Charm beta-lactam screening methods and it is operator choice which method is followed**

\_\_\_\_\_

a. Competitive Assay Control Point (CP) and Negative Control Average

\_\_\_\_\_

1. Run six 0.008 IU/mL Pen G

2. Run three Negative Controls

\_\_\_\_\_

Penicillin G

Negative Control

1. \_\_\_\_\_

1. \_\_\_\_\_

2. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

Av. \_\_\_\_\_

5. \_\_\_\_\_

6. \_\_\_\_\_

Av. \_\_\_\_\_

+15% \_\_\_\_\_

CP \_\_\_\_\_

\_\_\_\_\_

b. Sequential Assay Control Point (CP) and Negative Control Average

\_\_\_\_\_

1. Run six 0.008 IU/mL Pen G

2. Run three Negative Controls

\_\_\_\_\_

Penicillin G

Negative Control

1. \_\_\_\_\_

1. \_\_\_\_\_

2. \_\_\_\_\_

2. \_\_\_\_\_

3. \_\_\_\_\_

3. \_\_\_\_\_

4. \_\_\_\_\_

Av. \_\_\_\_\_

5. \_\_\_\_\_

6. \_\_\_\_\_

Av. \_\_\_\_\_

+25% \_\_\_\_\_

CP \_\_\_\_\_

\_\_\_\_\_

c. Quantitative Assay Control Point (CP) and Negative Control Average

1. Run six Negative Controls

2. Run three 0.002 IU/mL Pen G (1 part 0.008 IU/mL and 3 parts Negative Control)

Negative Control

Penicillin G

1. \_\_\_\_\_  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
4. \_\_\_\_\_  
5. \_\_\_\_\_  
6. \_\_\_\_\_  
Av. \_\_\_\_\_  
-15% \_\_\_\_\_  
CP \_\_\_\_\_

1. \_\_\_\_\_  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
Av. \_\_\_\_\_

d. Cloxacillin Assay Control Point (CP) and Zero Control Average

1. Run six 10 ppb Cloxacillin

2. Run three Negative Controls

Cloxacillin

Negative Control

1. \_\_\_\_\_  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
4. \_\_\_\_\_  
5. \_\_\_\_\_  
6. \_\_\_\_\_  
Av. \_\_\_\_\_  
+15% \_\_\_\_\_  
CP \_\_\_\_\_

1. \_\_\_\_\_  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
Av. \_\_\_\_\_

7. Acceptability of Control Point Determinations

a. If any of the 6 control point determinations deviate from the average, redo that determination

- 1. For Competitive Assay cannot deviate by more than  $\pm 15\%$
- 2. For Sequential Assay cannot deviate by more than  $\pm 25\%$
- 3. For Quantitative Assay cannot deviate by more than  $\pm 15\%$
- 4. For Cloxacillin Assay cannot deviate by more than  $\pm 15\%$

b. If the re-determined value is within the allowed deviation recalculate the average and proceed with testing

c. If the value is not within allowed deviation, run another set of 6 standards

- d. A common control point for multiple analysts may be used \_\_\_\_\_
- 1. Control point determination performed by one analyst only \_\_\_\_\_
- 2. Control point determination rotated and inclusive of all certified/approved analysts \_\_\_\_\_
- 3. If daily performance check fails and is not resolved by using fresh controls, technique should be reviewed for consistency and corrective action taken as necessary \_\_\_\_\_

**8. Daily Performance and Operation Check (also see App. N GR item 10)** \_\_\_\_\_

- a. The negative control tests  $\pm 20\%$  ( $\pm 15\%$  for Quantitative Assay) established for each new kit lot \_\_\_\_\_
- b. The positive control tests less than or equal to the control point \_\_\_\_\_
- c. If these conditions are not met re-determine control point(s) \_\_\_\_\_
  - 1. Conditions met, proceed with testing \_\_\_\_\_
  - 2. Conditions not met, discontinue testing and seek technical assistance \_\_\_\_\_

**9. Beta-lactam (all except Cloxacillin) Test Procedures** \_\_\_\_\_

- a. Label test tubes, one for each test sample \_\_\_\_\_
- b. Add 1 green tablet to each tube \_\_\_\_\_
- c. Add 300  $\mu\text{L}$  water to each tube \_\_\_\_\_
- d. Breakup tablets in tubes by mixing tubes 10 times on mixer in a rise and fall motion in 10 sec, if necessary continue mixing, green tablets must be completely suspended before proceeding \_\_\_\_\_
- e. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting, use within 3 min (samples must be in appropriate container to allow the use of vortexing) \_\_\_\_\_
- f. Add 5.0 mL of mixed sample/control to corresponding tube \_\_\_\_\_
  - 1. Using pipettor (item 3.i.2) with new tip for each sample/control, draw up 5 mL avoiding foam or bubbles \_\_\_\_\_
  - 2. Remove tip from liquid \_\_\_\_\_
  - 3. Expel test portion into appropriate tube \_\_\_\_\_

g. Competitive Assay

1. The following steps must be completed within 40 sec (all sample tubes being assayed)
  - a. Add yellow tablet to each tube
  - b. Vortex tubes 10 times in a rise and fall motion in 10 sec (yellow tablets do not breakup)
2. Incubate tubes for 3 min at  $85\pm 2^{\circ}\text{C}$
3. Remove tubes and centrifuge for 3 min, optionally for 5 min (same time used to determine control point)
4. Skip to item 11

h. Sequential Assay

1. Vortex tubes 10 times in a rise and fall motion in 10 sec
2. Incubate tubes for 2 min at  $65\pm 2^{\circ}\text{C}$
3. The following steps must be completed within 40 sec (all sample tubes being assayed)
  - a. Add yellow tablet to each tube
  - b. Vortex tubes as in item 9.h.1 above
4. Incubate tubes for 2 min at  $65\pm 2^{\circ}\text{C}$
5. Remove tubes and centrifuge for 3 min, optionally for 5 min (same time used to determine control point)
6. Skip to item 11

i. Quantitative Assay

1. Vortex tubes 10 times in a rise and fall motion in 10 sec
2. Incubate tubes for 7 min at  $55\pm 2^{\circ}\text{C}$
3. The following steps must be completed within 40 sec (all sample tubes being assayed)
  - a. Add yellow tablet to each tube
  - b. Vortex tubes as in item 1 above
4. Incubate tubes for 2 min at  $55\pm 2^{\circ}\text{C}$

5. Remove tubes and centrifuge for 3 min, optionally for 5 min (same time used to determine control point) \_\_\_\_\_
6. Skip to item 11 \_\_\_\_\_

**10. Cloxacillin Test Procedure** \_\_\_\_\_

a. Competitive Assay \_\_\_\_\_

1. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting, use within 3 min (samples must be in appropriate containers to allow the use of vortexing) \_\_\_\_\_
2. Fill identified test tubes  $\frac{3}{4}$  full with milk samples, avoiding foam and bubbles, and centrifuge for 5 min \_\_\_\_\_
3. Cool tubes to 0.0-4.5°C \_\_\_\_\_
4. Label empty test tubes, one for each test sample \_\_\_\_\_
5. Add 1 white tablet to each new empty tube \_\_\_\_\_
6. Add 300  $\mu$ L water to each tube \_\_\_\_\_
7. Breakup tablets in tubes by vortexing tubes 10 times on mixer in a rise and fall motion in 10 sec, if necessary continue vortexing, white tablets must be completely suspended before proceeding \_\_\_\_\_
8. Draw up 5.0 mL of centrifuged sample/control from below the fat layer \_\_\_\_\_
  - a. Use new tip for each sample/control \_\_\_\_\_
  - b. Remove tip from liquid \_\_\_\_\_
  - c. Expel test portion into appropriate tube \_\_\_\_\_
9. The following steps must be completed within 40 sec (all sample tubes being assayed) \_\_\_\_\_
  - a. Add blue tablet to each tube \_\_\_\_\_
  - b. Vortex tubes 10 times in a rise and fall motion in 10 sec (blue tablets do not breakup) \_\_\_\_\_
10. Incubate tubes for 3 min at 35 $\pm$ 2°C \_\_\_\_\_
11. Remove tubes and centrifuge for 5 min \_\_\_\_\_

**11. After Centrifugation Step in Beta-Lactam (9.g.3, 9.h.5, and 9.i.5) and Cloxacillin (10.a.11) Test Procedures** \_\_\_\_\_

- a. Immediately pour off milk \_\_\_\_\_

- b. While still draining tubes, remove fat ring with 2 or more cotton swabs, continue until dry, do not touch pellet (do not go much below the fat ring) \_\_\_\_\_
- c. Add 300 µL of water to tubes and break up pellets using vortex mixer \_\_\_\_\_
- d. Pellets must be completely suspended before proceeding to next step \_\_\_\_\_
- e. Add 3 mL of scintillation fluid to each tube, cap and vortex until uniformly mixed \_\_\_\_\_
- f. Count tubes on scintillation counter for 1 min using [14C] channel \_\_\_\_\_
- g. Record counts as counts per minute (CPM) \_\_\_\_\_

**12. Interpretation** \_\_\_\_\_

- a. If the beta-lactam assay (not applicable to Cloxacillin Assay) result the analyzer is at least 50 points greater than the control point, then the sample result is Negative (NF) \_\_\_\_\_
- b. If Cloxacillin assay result is greater than the control then the sample is Negative (NF) \_\_\_\_\_
- c. If the beta-lactam assay result in the analyzer is less than or equal to the control point then the sample is Presumptive Positive \_\_\_\_\_
- d. If the beta-lactam assay (not applicable to Cloxacillin Assay) result in the analyzer is less than 50 points greater than the control point, then the sample must be re-counted \_\_\_\_\_
  - 1. If on re-count the result is greater than the control point, then the sample is Negative (NF) \_\_\_\_\_
  - 2. If on re-count the result is equal to or less than the control point, then the sample is Presumptive Positive \_\_\_\_\_

**13. Verification of Initial Positive Samples (see App. N GR item 11); Confirmation of Presumptive Positive Samples (see App. N GR item 12); and Producer Traceback (see App. N GR item 13). For Quantitative Assay: PROMPTLY retest the SAME sample using the Sequential Assay or Competitive Assay, and when these beta-lactam assays give Not Found [NF] the Cloxacillin Assay is required** \_\_\_\_\_

**14. Reporting (see App. N GR item 14)** \_\_\_\_\_

**15. Handling of Exempt Quantities of Radioactive Materials** \_\_\_\_\_

- a. No mouth pipetting \_\_\_\_\_
- b. No smoking, eating or use of cosmetics while reagents are being handled \_\_\_\_\_

- c. NRC (Nuclear Regulatory Commission) licensed facilities must meet requirements as they relate to the use of gloves, other protective measures, and handling of wastes \_\_\_\_\_
- d. Wash hands thoroughly after handling reagents \_\_\_\_\_
- e. Wipe up spills immediately and thoroughly \_\_\_\_\_
- f. Properly dispose of all contaminated waste \_\_\_\_\_

**CHARM® II COMPETITIVE ASSAYS**

**FOR SULFONAMIDES (IMS #9C-10), TETRACYCLINES (IMS #9C-12)  
AND CHLORAMPHENICOL (IMS #9C-11)**

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM  
(Raw Commingled Cow Milk)**

**GENERAL REQUIREMENTS**

1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

**SAMPLES**

2. See App. N GR item 9 \_\_\_\_\_

**APPARATUS & REAGENTS**

3. **Equipment** \_\_\_\_\_

- a. Analyzer heater for 13 x 100 mm tubes \_\_\_\_\_

1. 85±2°C for Sulfonamide Assay \_\_\_\_\_

2. 35±2°C for Tetracycline Assay \_\_\_\_\_

3. Check temperature by electronic display, or by placing accuracy checked temperature measuring device in tube containing liquid (bulb submersed) in heating unit; maintain records \_\_\_\_\_

4. Or, use 6 inch partial immersion thermometer placed directly into small thermometer well in middle of heating unit; maintain records \_\_\_\_\_

5. Temperature measuring device for each incubator (App. N GR item 3) \_\_\_\_\_

- b. Ice-water bath, 0.0-4.5°C for Chloramphenicol Assay \_\_\_\_\_

- c. Mixer, Maxi-mixer II or equivalent \_\_\_\_\_

- d. Centrifuge, Whisperfuge® or Heraeus® (3400 rpm) or equivalent \_\_\_\_\_

- e. Scintillation counter, Charm II or equivalent \_\_\_\_\_

- f. Scintillation fluid dispenser, set to dispense 3 mL \_\_\_\_\_

1. Checked every six (6) months with Class A graduated cylinder and record; maintain records \_\_\_\_\_

- g. Cotton swabs (not applicable for Chloramphenicol Assay) \_\_\_\_\_

- h. Borosilicate test tubes, 13 x 100 mm \_\_\_\_\_

- i. Plastic stoppers for tubes \_\_\_\_\_
- j. Pipettors – Fixed Volume or electronic (see App. N GR item 7) \_\_\_\_\_
  - 1. 300 µL and appropriate tips \_\_\_\_\_
  - 2. 5.0 mL and appropriate tips \_\_\_\_\_
  - 3. 1.0 mL and appropriate tips (not applicable Sulfa Drug Assay) \_\_\_\_\_
- k. Timer \_\_\_\_\_

**4. Reagents** \_\_\_\_\_

- a. Scintillation fluid – Optifluor or equivalent supplied by manufacturer of test kits \_\_\_\_\_
- b. Sulfonamide Assay (Competitive Assay) \_\_\_\_\_
  - 1. Reagent blister packages: microbial/antibody binder (white) tablet, tracer reagent (pink) tablet \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 2. 10 ppb Sulfamethazine standard or multi-standard \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 3. Zero control standard \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
- c. Chloramphenicol Assay (Chloramphenicol and other Amphenicols) \_\_\_\_\_
  - 1. Reagent blister packages: reagent (white tablet), tracer reagent (green tablet) and Charcoal (black tablet) \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 2. 1 ppb Chloramphenicol standard or multi-standard \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
  - 3. Zero control standard \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_
- d. Tetracycline Assay (Competitive Assay) \_\_\_\_\_
  - 1. Reagent blister packages: microbial/antibody binder (white) tablet, tracer reagent (orange) tablet \_\_\_\_\_
 

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

2. 30 ppb Oxytetracycline standard or multi-standard \_\_\_\_\_

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

3. Zero control standard \_\_\_\_\_

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

**5. Reagent stability** \_\_\_\_\_

a. All tablet reagents stored at  $-15^{\circ}\text{C}$  or below \_\_\_\_\_

b. Positive Control – Lyophilized 10 ppb Sulfamethazine, 30 ppb Oxytetracycline and 1 ppb Chloramphenicol standards \_\_\_\_\_

1. Reconstitute with 100 mL (measured) Negative Control (allow to sit 15 min prior to use or aliquotting); use within 48 hours at  $0.0-4.5^{\circ}\text{C}$  \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

2. Or, aliquot within 24 hours and freeze at  $-15^{\circ}\text{C}$  or colder in a non-frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

a. Thaw and use within 24 hours. Store at  $0.0-4.5^{\circ}\text{C}$  \_\_\_\_\_

c. Negative Control – Lyophilized Zero Control Standard (ZCS) or alternatively raw milk qualified to test similar to ZCS \_\_\_\_\_

1. Reconstitute ZCS according to manufacture instructions. (Allow to sit 15 min prior to use or aliquotting) \_\_\_\_\_

a. To qualify raw milk, test sample 3 times and average results. Average must be within  $\pm 10\%$  of ZCS \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

2. Use within 48 hours when stored at  $0.0-4.5^{\circ}\text{C}$  \_\_\_\_\_

3. Or, aliquot within 24 hours and freeze at  $-15^{\circ}\text{C}$  or colder in a non-frost-free freezer or in an insulated foam container in a frost-free freezer; use within 2 months \_\_\_\_\_

Lab Prep. Date: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

a. Thaw and use within 24 hours. Store at  $0.0-4.5^{\circ}\text{C}$  \_\_\_\_\_

d. Scintillation fluid expires six (6) months after opening \_\_\_\_\_

Date opened: \_\_\_\_\_ Lab Exp. Date: \_\_\_\_\_

**TECHNIQUE**

**6. Control point and Zero Control Average to be determined for each new lot of reagents** \_\_\_\_\_

a. Sulfonamide Assay Control Point (CP) and Negative Control Average \_\_\_\_\_

- |                                  |                                |
|----------------------------------|--------------------------------|
| 1. Run six 10 ppb Sulfamethazine | 2. Run three Negative Controls |
|----------------------------------|--------------------------------|

Sulfamethazine

Negative Control

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- 4. \_\_\_\_\_
- 5. \_\_\_\_\_
- 6. \_\_\_\_\_
- Av. \_\_\_\_\_
- +24% \_\_\_\_\_
- CP. \_\_\_\_\_

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- Av. \_\_\_\_\_

b. Chloramphenicol Assay Control Point (CP) and Negative Control Average \_\_\_\_\_

- |                                  |                                |
|----------------------------------|--------------------------------|
| 1. Run six 1 ppb chloramphenicol | 2. Run three Negative Controls |
|----------------------------------|--------------------------------|

Chloramphenicol

Negative Control

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- 4. \_\_\_\_\_
- 5. \_\_\_\_\_
- 6. \_\_\_\_\_
- Av. \_\_\_\_\_
- +25% \_\_\_\_\_
- CP. \_\_\_\_\_

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- Av. \_\_\_\_\_

c. Tetracycline Assay Control Point (CP) and Negative Control Average \_\_\_\_\_

- 1. Run six 30 ppb Oxytetracycline
- 2. Run three Negative Controls

Oxytetracycline

Negative Control

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- 4. \_\_\_\_\_
- 5. \_\_\_\_\_
- 6. \_\_\_\_\_
- Av. \_\_\_\_\_
- +23% \_\_\_\_\_
- CP. \_\_\_\_\_

- 1. \_\_\_\_\_
- 2. \_\_\_\_\_
- 3. \_\_\_\_\_
- Av. \_\_\_\_\_

**7. Acceptability of control point determinations** \_\_\_\_\_

- a. If any of the 6 control point determinations deviate from the average, redo that determination \_\_\_\_\_
  - 1. For Sulfonamide Assay cannot deviate by more than  $\pm 24\%$  \_\_\_\_\_
  - 2. For Tetracycline Assay cannot deviate by more than  $\pm 23\%$  \_\_\_\_\_
  - 3. For Chloramphenicol Assay cannot deviate by more than  $\pm 25\%$  \_\_\_\_\_
- b. If the re-determined value is within the allowed deviation recalculate the average and proceed with testing \_\_\_\_\_
- c. If the value is not within allowed deviation then another set of 6 standards must be run \_\_\_\_\_
- d. A common control point for multiple analysts may be used \_\_\_\_\_
  - 1. Control point determination performed by one analyst only \_\_\_\_\_
  - 2. Control point determination rotated and inclusive of all certified/approved analysts \_\_\_\_\_
  - 3. If daily performance check fails and is not resolved by using fresh controls, technique should be reviewed for consistency and corrective action taken as necessary \_\_\_\_\_

**8. Daily Performance and Operation Check (also see App. N GR item 10)** \_\_\_\_\_

- a. The Negative Control tests  $\pm 30\%$  (20% Chloramphenicol Assay) established for each new lot of kits \_\_\_\_\_
- b. The positive control tests less than or equal to the control point \_\_\_\_\_

- c. If these conditions are not met re-determine control point(s) \_\_\_\_\_
- 1. Conditions met; proceed with testing \_\_\_\_\_
- 2. Conditions not met; discontinue testing and seek technical assistance \_\_\_\_\_

**9. Test Procedures** \_\_\_\_\_

- a. Sulfonamide Assay \_\_\_\_\_
  - 1. Label test tubes, one for each test sample \_\_\_\_\_
  - 2. Add 1 white tablet to each tube \_\_\_\_\_
  - 3. Add 300  $\mu$ L water to each tube \_\_\_\_\_
  - 4. Breakup tablets in tubes by vortexing tubes 10 times in a rise and fall motion in 10 sec, white tablets must be completely broken apart or continue vortexing before proceeding \_\_\_\_\_
  - 5. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min (samples/controls must be in appropriate containers to allow the use of vortexing) \_\_\_\_\_
  - 6. Add 5 mL of mixed sample/control to corresponding tube \_\_\_\_\_
    - a. Using pipettor (item 3.j.2) with new tip for each sample/control, draw up 5 mL avoiding foam or bubbles \_\_\_\_\_
    - b. Remove tip from liquid \_\_\_\_\_
    - c. Expel test portion into appropriate tube \_\_\_\_\_
  - 7. The following steps must be completed within 40 sec (all sample tubes being assayed) \_\_\_\_\_
    - a. Add pink tablet to each tube \_\_\_\_\_
    - b. Vortex tubes 10 times in a rise and fall motion in 10 sec (pink tablets do not breakup) \_\_\_\_\_
  - 8. Incubate tubes for 3 min at  $85\pm 2^{\circ}\text{C}$  \_\_\_\_\_
  - 9. Remove tubes and centrifuge for 3 min; optionally for 5 min (use same time used to determine control point) \_\_\_\_\_
  - 10. After centrifugation, immediately pour off milk \_\_\_\_\_
  - 11. While still draining tubes, remove fat ring with 2 or more cotton swabs, continue until dry, do not touch pellet (do not go much below the fat ring) \_\_\_\_\_
  - 12. Add 300  $\mu$ L of water to tubes and break up pellets using vortex mixer \_\_\_\_\_

13. Pellets must be completely suspended before proceeding to next step \_\_\_\_\_
14. Add 3 mL of scintillation fluid to each tube, cap and vortex until uniformly mixed \_\_\_\_\_
15. Count tubes on scintillation counter for 1 min using [3H] channel \_\_\_\_\_
16. Record counts as counts per minute (CPM) \_\_\_\_\_

b. Chloramphenicol Assay \_\_\_\_\_

1. Label test tubes, one for each test sample \_\_\_\_\_
2. Add 1 white tablet to each tube \_\_\_\_\_
3. Add 300  $\mu$ L water to each tube \_\_\_\_\_
4. Breakup tablets in tubes by vortexing tubes 10 times in a rise and fall motion in 10 sec, white tablets must be completely broken apart or continue vortexing before proceeding \_\_\_\_\_
5. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting, use within 3 min (samples/controls must be in appropriate containers to allow the use of vortexing) \_\_\_\_\_
6. Add 1.0 mL of mixed sample/control to corresponding tube \_\_\_\_\_
  - a. Using pipettor (item 3.j.3) with new tip for each sample/control, draw up 1 mL avoiding foam and bubbles \_\_\_\_\_
  - b. Remove tip from liquid \_\_\_\_\_
  - c. Expel test portion into appropriate tube \_\_\_\_\_
7. The following steps must be completed within 40 sec (all assay tubes being assayed) \_\_\_\_\_
  - a. Add 1 green tablet to each tube \_\_\_\_\_
  - b. Vortex tubes as in 4 above \_\_\_\_\_
  - c. Add black tablet to each tube \_\_\_\_\_
  - d. Vortex tubes as in 4 above \_\_\_\_\_
8. Incubate tubes in an ice bath (50% ice, 50% water) at 0.0-4.5°C for 3 min \_\_\_\_\_
9. Remove tubes and centrifuge for 5 min \_\_\_\_\_
10. Using 300  $\mu$ L pipettor immediately add 300  $\mu$ L of centrifuged sample to a new labeled tube (remove by avoiding fat and without disturbing pellet) \_\_\_\_\_

11. Use fresh tip for each sample \_\_\_\_\_
12. Add 3 mL of scintillation fluid to each tube, cap and vortex until uniformly mixed \_\_\_\_\_
13. Count tubes on scintillation counter for 1 min using [3H] channel \_\_\_\_\_
14. Record counts as counts per minute (CPM) \_\_\_\_\_

c. Tetracycline Assay \_\_\_\_\_

1. Label test tubes, one for each test sample \_\_\_\_\_
2. Add 1 white tablet to each empty tube \_\_\_\_\_
3. Add 300  $\mu$ L water to each tube \_\_\_\_\_
4. Breakup tablets in tubes by vortexing tubes 10 times in a rise and fall motion in 10 sec, white tablets must be completely broken apart or continue vortexing before proceeding \_\_\_\_\_
5. Mix sample(s)/control(s) by shaking 25 times in 7 sec through 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min. Dilute 1 mL of sample with 9 mL of Zero Control, repeat mixing.  
**Controls are not diluted before testing** \_\_\_\_\_
6. Add 5.0 mL diluted milk sample or undiluted control to corresponding tube \_\_\_\_\_
  - a. Using pipettor (item 3,j,2) with new tip for each sample/control, draw up 5 mL avoiding foam or bubbles \_\_\_\_\_
  - b. Remove tip from liquid \_\_\_\_\_
  - c. Expel test portion into appropriate tube \_\_\_\_\_
7. The following steps must be completed within 40 sec (all sample tubes being assayed) \_\_\_\_\_
  - a. Add orange tablet to each tube \_\_\_\_\_
  - b. Vortex tubes 15 times in a rise and fall motion in 20 sec (orange tablets do not breakup) \_\_\_\_\_
8. Incubate tubes for 3 min at  $35\pm 2^{\circ}\text{C}$  \_\_\_\_\_
9. Remove tubes and centrifuge for 5 min \_\_\_\_\_
10. After centrifugation immediately pour off milk \_\_\_\_\_
11. While still draining tubes, remove fat ring with 2 or more cotton swabs, continue until dry, do not touch pellet (do not go much below the fat ring) \_\_\_\_\_

- 12. Add 300 µL of water to tubes and break up pellets using vortex mixer \_\_\_\_\_
- 13. Pellets must be completely suspended before proceeding to next step \_\_\_\_\_
- 14. Add 3 mL of scintillation fluid to a tube, cap and vortex until uniformly mixed. Count tubes on scintillation counter for 1 min using [3H] channel \_\_\_\_\_
- 15. Repeat step 14 with each tube to be analyzed. \_\_\_\_\_
- 16. Record counts as counts per minute (CPM) \_\_\_\_\_

**10. Interpretation** \_\_\_\_\_

- a. If the number of the measured activity in the analyzer is greater than the control point, then the sample is Negative (NF) \_\_\_\_\_
- b. If the number of the measured activity in the analyzer is less than or equal to the control point then the sample is Presumptive Positive \_\_\_\_\_

**11. Verification of Initial Positive Samples (see App. N GR item 11); Confirmation of Presumptive Positive Samples (see App. N GR item 12);and Producer Traceback (see App. N GR item 13)** \_\_\_\_\_

**12. Reporting (see App. N GR item 14)** \_\_\_\_\_

**13. Handling of Exempt Quantities of Radioactive Materials** \_\_\_\_\_

- a. No mouth pipetting \_\_\_\_\_
- b. No smoking, eating or use of cosmetics while reagents are being handled \_\_\_\_\_
- c. Nuclear Regulatory Commission (NRC) licensed facilities must meet requirements as they relate to the use of gloves, other protective measures, and handling of wastes \_\_\_\_\_
- d. Wash hands thoroughly after handling reagents \_\_\_\_\_
- e. Wipe up spills immediately and thoroughly \_\_\_\_\_
- f. Properly dispose of all contaminated waste \_\_\_\_\_

**APPENDIX N BULK MILK TANKER SCREENING TEST FORM**

**Neogen BetaStar® Plus (Raw Commingled Cow Milk) Beta-Lactam Test  
IMS# 9-N1**

**GENERAL REQUIREMENTS**

1. See Appendix N General Requirements (App. N GR) items 1-8 & 15 \_\_\_\_\_

**SAMPLES**

2. See App. N GR item 9 \_\_\_\_\_

**APPARATUS & REAGENTS**

3. **Equipment** \_\_\_\_\_

- a. Heater Block, 47.5±2.0°C \_\_\_\_\_

1. Check temperature daily (day of use); maintain records \_\_\_\_\_

2. Temperature measuring device must be supplied by manufacturer and meet all App. N GR item 3 requirements \_\_\_\_\_

3. Temperature measuring device for each incubator (App. N GR item 3) \_\_\_\_\_

- b. Accuscan® III Reader (software V 3.551 or higher) with Accuscan III program installed, with printout or download of data. Manual available \_\_\_\_\_

Serial Number: \_\_\_\_\_

1. Printer or computer link for hardcopy download \_\_\_\_\_

2. Maintain records \_\_\_\_\_

- c. Pipettor – 200 µL and disposable tips (see App. N GR item 7) \_\_\_\_\_

- d. Or single use 200 µL Poly-pipet with overflow bulb to accurately measure amount of sample, supplied by manufacturer (**screening only**) \_\_\_\_\_

- e. Timer \_\_\_\_\_

4. **Reagents** \_\_\_\_\_

- a. BetaStar Plus Test Kit

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

QC Date: \_\_\_\_\_ By: \_\_\_\_\_

1. Kit contains lyophilized receptor vials, dipsticks, and Poly-pipets (item 3.d) \_\_\_\_\_

a. Verify that first 5 characters of the lot code on the dipstick tube, dipstick and vial matches the first 5 characters of the Test Kit lot code \_\_\_\_\_

Kit Lot #: \_\_\_\_\_

2. Verify acceptable status of the moisture indicator card in the dipstick tube prior to use. If all of the dots on the indicator card have changed from blue to pink, do not use the kit \_\_\_\_\_

b. Positive Control \_\_\_\_\_

1. Lyophilized 5 ppb Penicillin G/100ppb Desfuroylceftiofur \_\_\_\_\_

Lot #: \_\_\_\_\_ Exp. Date: \_\_\_\_\_

c. Negative control \_\_\_\_\_

1. Previously tested negative raw milk (item 5.d) \_\_\_\_\_

**5. Reagent Stability (frozen standards are not allowed)** \_\_\_\_\_

a. Test Kit including reagents and dipsticks received refrigerated in insulated chest with cold pack block shipped overnight \_\_\_\_\_

b. Store reagents and dipsticks at 0.0-4.5°C, maintain no longer than manufacturer's expiration date \_\_\_\_\_

c. Positive Control – Manufacturer supplied, maintain no longer than manufacturer's expiration date \_\_\_\_\_

1. Reconstituted with 1 mL of raw commingled cow milk tested at 2.0 or higher ratio with BetaStar Plus Beta Lactam test; use within 48 hours and maintain at 0.0-4.5°C \_\_\_\_\_

Lab Prep Date: \_\_\_\_\_ Lab Exp Date: \_\_\_\_\_

2. Day of use, must produce 0.85 or lower ratio reading; maintain records \_\_\_\_\_

Test Value: \_\_\_\_\_

**Do not proceed if out of range** \_\_\_\_\_

- d. Negative Control – raw commingled cow milk tested at 2.0 or higher ratio reading with BetaStar Plus Beta-Lactam test. Maintain at 0.0-4.5°C and use within 72 hours

Sample ID: \_\_\_\_\_ Test Value: \_\_\_\_\_

Date Tested: \_\_\_\_\_

**Do not proceed if out of range**

## TECHNIQUE

### 6. Daily Performance and Operation Check

- a. See App. N GR item 10
- b. Accuscan III Reader Calibration
1. The reader calibration occurs automatically when the Accuscan III Program (V3.551 or higher) is initiated on the reader
    - a. The Accuscan III program must be re-initiated daily prior to use, when the unit is turned off, or when the unit goes off if the battery charge is too low
  2. If the reader calibration is unsuccessful, the reader will not operate. A warning message will prompt the user “Calibration unsuccessful. Contact Neogen.”
  3. Calibrator cartridges must be read daily. There are two calibrators, one as Positive and one as Negative. Each calibrator has a line for both beta-lactam and Ceftiofur
  4. Both lines on each of the calibration cartridges must read within the limits as indicated on the cartridge device

Positive Calibrator Readings:

Beta-lactam: \_\_\_\_\_

Ceftiofur: \_\_\_\_\_

Negative Calibrator Reading: \_\_\_\_\_

Beta-lactam: \_\_\_\_\_

Ceftiofur: \_\_\_\_\_

5. If Calibrators are out of range, contact Neogen before proceeding
6. Maintain records

**7. Test Procedure [Precautions – when handling BetaStar Plus test, make sure hands are clean and dry. This will protect against contamination of test reagents]**

- a. The test is designed for use under normal ambient environmental conditions (15-30°C). Remove the kit from the refrigerator. Remove the number of test vials required and the dipstick container. Immediately return the balance of the kit to the refrigerator. Allow the dipstick container to equilibrate to room temperature (15-30°C) for 10-15 min prior to opening to prevent condensation
- b. Remove the number of dipsticks required and return the dipstick container to the refrigerator
- c. A maximum of 4 tests can be run at one time. If more than one sample is being run, all milk samples should be prepared prior to inserting into the heater block and beginning the incubation period
- d. Any receptor vials or dipsticks removed from the kit that remain unused at the end of the testing day must be discarded
- e. Dipsticks that have been removed from the dipstick container must be kept clean and dry
- f. Label one vial and one dipstick for each test sample and each control
- g. Gently tap the vial on a hard surface in order to assure all solid material is in bottom of vial
- h. Mix milk sample(s)/control(s) 25 times in 7 sec with a 1 ft movement or vortex for 10 sec at maximum setting; use within 3 min (sample must be in appropriate containers to allow the use of vortexing)
- i. Carefully remove the cap and rubber stopper from the vial
- j. Pipette 200 µL milk sample into the vial
  - 1. **Certified laboratories must**, utilize a fixed volume pipettor
    - a. Attach a 200 µL pipette tip to the pipettor
    - b. Draw up sample, avoiding foam and bubbles
    - c. Deliver the sample into the vial by depressing the plunger
    - d. Replace the rubber stopper in the vial

2. Using new manufacturer-provided Poly-pipet (item 3.d) for each sample/control [**Screening only**]
  - a. Squeeze top bulb while holding vertically with bulb and overflow reservoir side pointing down, draw up test portion avoiding foam and bubbles. Sample should completely fill pipet shaft and overflow into the bottom half of the overflow reservoir
  - b. Remove tip from liquid
  - c. While holding the Poly-pipet vertically, expel test portion slowly into the vial. Excess portion should remain in reservoir
  - d. Replace the rubber stopper in the vial
- k. Mix the milk and the reagents thoroughly by inverting the vial twice and swirling in a circular motion until all solids are in solution
- l. Remove and discard stopper from vial and place the vial into the heater block and incubate at  $47.5 \pm 2.0^{\circ}\text{C}$  for 3 min, not to exceed 3 min and 30 sec
- m. At the completion of the 3 min incubation, place labeled dipstick into the vial in the heater block. The arrows on the dipstick must be oriented downward in the vial
- n. Incubate the dipstick in the vial for 2 min, not to exceed 2 min and 30 sec at  $47.5 \pm 2.0^{\circ}\text{C}$
- o. At the completion of the 2 min incubation, remove the dipstick from the vial and visually inspect the control line; an absent, partial or indistinct control line indicates an invalid test and the sample/control must be re-test.  
**CAUTION: Insert only valid test(s) into the Reader**
- p. Place the dipstick arrows first, into the holder, and insert the holder into the Accuscan III reader (software V3.551 or higher)
- q. Read dipsticks within 3 min of completion of the incubation

**8. Interpretation with Reader**

- a. If there is a negative reading (ratio > 1.0) on the reader, sample is Negative (NF)
- b. If there is a positive reading (ratio  $\leq$  1.0) on the reader, sample is an Initial Positive

**9. Verification of Initial Positive Tanker Samples (see App. N GR item 11); Confirmation of Presumptive Positive Tanker Samples (see App. N GR item 12); and Traceback of Producer(s) on a Confirmed Positive Tanker (see App. N GR item 13)**

**10. Reporting (see App. N GR item 14)**

\_\_\_\_\_

- a. Maintain reader tapes or computer print outs

\_\_\_\_\_

# QC FORMS

<u>BFSL#</u>	<u>NAME OF RECORD</u>	<u>SHEET REVISION DATE</u>
497	Monthly Analytical Balance Check Records	1/14
498	Monthly Electronic Balance Check Records	1/14
500	Daily Drug Screening Test Log	1/14
500a	Daily Drug Screening Test Log	1/14
501a	Temperature Records - Block Heater	1/14
501b	Temperature Records - Freezer	1/14
501c	Temperature Records - Refrigerator	1/14
503	Semi-Annual Pipettor Calibration	12/13
513	Test Kit Suitability Check for Drug Residue Testing	1/14
513A	Positive Control Suitability Check	7/11
513B	Negative Control Suitability Check	7/11
515	Thermometer Accuracy Check Log	12/13
515a	Thermometer Accuracy Check Log	12/13
528	Appendix N Training Log	1/14
528a	Appendix N Training Session(New Analyst)	1/14
534	SNAP Image Performance Check Set	7/09
535	Charm Rosa Reader Primary Calibration Strips	7/09

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

## MONTHLY ANALYTICAL ELECTRONIC BALANCE CHECK RECORDS

Year: \_\_\_\_\_

Make/ Model/Type: \_\_\_\_\_ Serial # or ID#: \_\_\_\_\_

Date(s) Serviced: \_\_\_\_\_

Date	Analysts ID# or Initials	Actual Scale Readings									Comments
		10 mg	50 mg	100 mg	200 mg	300 mg	500 mg	1 g	5 g	10g	

1. Electronic only, sensitive to 0.1g for general laboratory purposes and to 0.0001g for pipettor checks and antibiotics.
2. Checked monthly with Class S or S1, or equivalent ASTM 1, 2, or 3, weights. Certificate of authenticity or other verification, required.
3. Check weights that correspond to normal use of balance and maintain records. Record actual display reading of balance.
4. Checked at least annually by a qualified representative for good working order with proof of check in laboratory.

Facility/Laboratory Name: Utter's Dairy

**MONTHLY ANALYTICAL ELECTRONIC BALANCE CHECK RECORDS**

Year: 2013, 2014

Make/ Model/Type: Mettler Toledo Serial # or ID#: 1002894

Date(s) Serviced: 8/15/12,8/18/13

Date	Analysts ID# or Initials	Actual Scale Readings									Comments
		10 mg	50 mg	100 mg	200 mg	300 mg	500 mg	1 g	5 g	10g	
11/25/13	J. Michaels, #02	0.0099g	0.0501g	0.1001g	0.2003g	0.2997g	0.4997g	1.0000g	5.0001g	10.0001	√ Cleaned area & balance
12/15/13	A. Thomas	0.0100g	0.0495g	0.1002g	0.1995g	0.2996g	0.4996g	0.9995g	4.9998g	10.0000	√ Cleaned area & balance
1/23/14	J. Michaels, #02	0.0099g	0.0501g	0.1000g	0.2001g	0.2999g	0.5000g	1.0001g	4.9997g	9.9998	√ Cleaned area & balance

1. Electronic only, sensitive to 0.1g for general laboratory purposes and to 0.0001g for pipettor checks and antibiotics.
2. Checked monthly with Class S or S1, or equivalent ASTM 1, 2, or 3, weights. Certificate of authenticity or other verification, required.
3. Check weights that correspond to normal use of balance and maintain records. Record actual display reading of balance.
4. Checked at least annually by a qualified representative for good working order with proof of check in laboratory.



COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: Northpoint Laboratory

**MONTHLY MILK/MEDIA ELECTRONIC BALANCE CHECK RECORDS**

Year: 2013, 2014

Make/ Mode/ Type: Ohaus Scout Serial # or ID#: 21198

Date(s) Serviced: 4/12/12, 4/19/13

Date	Analysts ID# or Initials	Actual Scale Readings							Comments
		1 gm	5 gm	10 gm	25 gm	50 gm	100 gm	150 gm	
11/20/13	J. Smith	1.02g	5.00g	9.99g	25.01g	50.01g	99.98g	150.02g	√ cleaned balance
12/15/13	A. Jones	1.01g	5.02g	9.99g	25.00g	50.02g	100.01g	149.99g	√ cleaned balance
1/18/14	A. Jones	1.02g	5.01g	10.00g	25.01g	50.01g	99.99g	150.03g	√ cleaned balance

1. Electronic only, sensitive to 0.1g for general laboratory purposes and to 0.0001g for pipettor checks and antibiotics.
2. Checked monthly with Class S or S1, or equivalent ASTM 1, 2, or 3, weights. Certificate of authenticity or other verification, required.
3. Check weights that correspond to normal use of balance and maintain records. Record actual display reading of balance.
4. Checked at least annually by a qualified representative for good working order with proof of check in laboratory.

SCREENING TEST USED \_\_\_\_\_

**DAILY DRUG SCREENING TEST LOG**

FACILITY/LABORATORY NAME: \_\_\_\_\_

FDA ID# \_\_\_\_\_

YEAR \_\_\_\_\_

ADDRESS: \_\_\_\_\_

SAMPLE COLLECTED			TANKER TEMP. °F	OWNER OF MILK/ FIPS #	COMPLETE TANKER LICENSE PLATE NUMBER	BILL OF LADING #	POUNDS	LAB. TEMP. CONTROL °C	TIME START TESTING	TIME READ RESULT	RESULT (NUMERICAL VALUE)	INTERP. (POS/NF)	ANALYST ID#
DATE (mm/dd)	TIME	SAMPLER ID											

**A VALID POSITIVE AND NEGATIVE CONTROL MUST BE RUN EACH DAY SCREENING TEST IS PERFORMED WITH RESULTS RECORDED.**

COMMERCIAL POSITIVE CONTROL		RECONSTITUTED POSITIVE CONTROL		PRE-TESTED NEGATIVE CONTROL		TEST KIT INFORMATION		READER PERFORMANCE CHECKS	
MFG		LOT#		ID (i.e. SILO #)		LOT#		IDEXX	ROSA
LOT #		DATE PREP'D		DATE PREP'D		EXPIRATION DATE		DEVICE 1:	LOW:
DATE RECEIVED		TIME PREP'D		TIME PREP'D:				DEVICE 2:	HIGH:
DATE OPENED		FROZEN DATE		FROZEN DATE		<b>LEVEL CHECK</b> (Charm SL or SL3 only)		Analysts ID #	
EXPIRATION DATE		THAW DATE		THAW DATE		Satisfactory?			
COMMENTS:		EXPIRATION DATE		EXPIRATION DATE		Yes	No		
		NUMERICAL RESULT		NUMERICAL RESULT					

SCREENING TEST USED Charm SL

**DAILY DRUG SCREENING TEST LOG**

FACILITY/LABORATORY NAME: Utter's Dairy

FDA ID# 42-689

YEAR 2014

ADDRESS: \_\_\_\_\_

SAMPLE COLLECTED			TANKER TEMP. °F	OWNER OF MILK/ FIPS #	COMPLETE TANKER LICENSE PLATE NUMBER	BILL OF LADING #	POUNDS	LAB. TEMP. CONTROL °C	TIME START TESTING	TIME READ RESULT	RESULT (NUMERICAL VALUE)	INTERP. (POS/NF)	ANALYST ID#
DATE (mm/dd)	TIME	SAMPLER ID											
1/27	09:05	JR	39.0°F	42-405	PYK8005	37092	45,289 lbs	3.9°C	09:22	09:30	-2698	Not Found	JK
1/27	10:15	MJ	38.0°F	Hilltop 42-341	XFT8736	38112	35,599 lbs	3.3°C	10:20	10:28	-1831	Not Found	JM
1/27	10:45	MJ	37.5°F	42-405	XFK5592	38561	37, 268 lbs	3.2C	10:51	10:59	-1587	Not Found	JK

A VALID POSITIVE AND NEGATIVE CONTROL MUST BE RUN EACH DAY SCREENING TEST IS PERFORMED WITH RESULTS RECORDED.

COMMERCIAL POSITIVE CONTROL		RECONSTITUTED POSITIVE CONTROL		PRE-TESTED NEGATIVE CONTROL		TEST KIT INFORMATION		READER PERFORMANCE CHECKS	
MFG	Charm Sciences	LOT#	18A	ID (i.e. SILO #)	37090	LOT#	127	IDEXX	ROSA
LOT #	18A	DATE PREP'D	1/26/14	DATE PREP'D	1/26/14	EXPIRATION DATE	4/2014	DEVICE 1: NA	LOW:-0987
DATE RECEIVED	12/29/13	TIME PREP'D	09:00AM	TIME PREP'D:	9:55AM			DEVICE 2: NA	HIGH:+1156
DATE OPENED	1/6/14	FROZEN DATE	1/26/14	FROZEN DATE	NA	LEVEL CHECK (Charm SL or SL3 only)			Analysts ID # <b>JR</b>
EXPIRATION DATE	5/2014	THAW DATE	1/27/14	THAW DATE	NA	Satisfactory?			
COMMENTS:		EXPIRATION DATE	1/28/14	EXPIRATION DATE	1/29/14	Yes	No		
		NUMERICAL RESULT	+2598	NUMERICAL RESULT	-1697	X			

SCREENING TEST USED IDEXX New Snap

**DAILY DRUG SCREENING TEST LOG**

FACILITY/LABORATORY NAME: Utter's Dairy

FDA ID# 42-689

YEAR 2014

ADDRESS: 4242 Wide lane, Hometown PA 19856

SAMPLE COLLECTED			TANKER TEMP. °F	OWNER OF MILK/ FIPS #	COMPLETE TANKER LICENSE PLATE NUMBER	BILL OF LADING #	POUNDS	LAB. TEMP. CONTROL °C	TIME START TESTING	TIME READ RESULT	RESULT (NUMERICAL VALUE)	INTERP. (POS/NF)	ANALYST ID#
DATE (mm/dd)	TIME	SAMPLER ID											
1/27	09:05	JR	39.0°F	42-405	PYK8005	37092	45,289 lbs	3.9°C	09:22	09:30	0.67	Not Found	JK
1/27	10:15	MJ	38.0°F	Hilltop 42-341	XFT8736	38112	35,599 lbs	3.3°C	10:20	10:28	0.71	Not Found	JM
1/27	10:45	MJ	37.5°F	42-405	XFK5592	38561	37, 268 lbs	3.2C	10:51	10:59	0.59	Not Found	JK

A VALID POSITIVE AND NEGATIVE CONTROL MUST BE RUN EACH DAY SCREENING TEST IS PERFORMED WITH RESULTS RECORDED.

COMMERCIAL POSITIVE CONTROL		RECONSTITUTED POSITIVE CONTROL		PRE-TESTED NEGATIVE CONTROL		TEST KIT INFORMATION		READER PERFORMANCE CHECKS	
MFG	IDEXX	LOT#	EH598	ID (i.e. SILO #)	37090	LOT#	DA188	IDEXX	ROSA
LOT #	EH598	DATE PREP'D	1/27/14	DATE PREP'D	1/26/14	EXPIRATION DATE	5/14/14	DEVICE 1: 0.73	LOW: NA
DATE RECEIVED	12/25/13	TIME PREP'D	09:00AM	TIME PREP'D:	9:55AM			DEVICE 2: 1.56	HIGH: NA
DATE OPENED	1/2/14	FROZEN DATE	NA	FROZEN DATE	1/26/14	LEVEL CHECK (Charm SL or SL3 only)			
EXPIRATION DATE	4/1/14	THAW DATE	NA	THAW DATE	1/27/14				
COMMENTS:	EXPIRATION DATE	1/28/14	EXPIRATION DATE	1/28/14	Yes	No			
	NUMERICAL RESULT	5.01	NUMERICAL RESULT	0.87	NA				

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

**DAILY DRUG SCREENING TEST LOG**

SCREENING TEST USED \_\_\_\_\_

YEAR \_\_\_\_\_

FACILITY/LABORATORY NAME: \_\_\_\_\_

FDA ID# \_\_\_\_\_

ADDRESS: \_\_\_\_\_

SAMPLE COLLECTED			TANKER TEMP. (°F)	OWNER OF MILK/ FIPS #	COMPLETE TANKER LICENSE PLATE NUMBER	BILL OF LADING #	POUNDS	LAB. TEMP. CONTROL (°C) 0.0-4.5C	TIME START TESTING	TIME READ RESULTS	RESULTS (NUMERICAL VALUE)	INTERP. (POS/NF)	NAME/ID#	COMMENTS
DATE (mm/dd)	TIME	Sampler ID												

A POSITIVE AND NEGITIVE CONTROL MUST BE RUN EACH DAY THAT SCREENING TEST IS PERFORMED WITH RESULTS MAINTAINED.

COMMERCIAL POSITIVE CONTROL		RECONSTITUTED POSITIVE CONTROL		PRE-TESTED NEGATIVE CONTROL		TEST KIT INFORMATION		READER PERFORMANCE CHECKS	
MFG.		LOT #		ID (i.e. SILO #):		LOT#:		ROSA	SERIAL #:
LOT #		DATE PREP'D		DATE PREP'D:		EXPIRATION DATE:		LOW STRIP RESULT	
DATE RECEIVED:		TIME PREP'D:		TIME PREP'D:			HIGH STRIP RESULT		
DATE OPENED:		FROZEN DATE		FROZEN DATE			LOW RANGE		
LOT EXPIRES ON:		THAW DATE		THAW DATE			HIGH RANGE		
		EXPIRES:		EXPIRES:					
		NUMERICAL RESULT:		NUMERICAL RESULT:					
HEATER BLOCK TEMPERATURE		FRIDGE TEMPERATURE		FREEZER TEMPERATURE		LEVEL CHECK		IDEXX	
		0.0-4.5C		< -15.0C		(Charm ROSA only)		DEVICE 1 RESULT	
HEATER BLOCK SN#		FRIDGE SN#		FREEZER SN#		SATISFACTORY?		DEVICE 2 RESULT	
AM	PM	AM	PM	AM	PM	YES / NO		DEVICE 1 RANGE	
°C	°C	°C	°C	°C	°C				
°C	°C	°C	°C	°C	°C	ANALYST ID#		DEVICE 2 RANGE	
INITIALS	INITIALS	INITIALS	INITIALS	INITIALS	INITIALS				

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

**DAILY DRUG SCREENING TEST LOG**

SCREENING TEST USED IDEXX New Snap

YEAR 2014

FACILITY/LABORATORY NAME: Utter's Dairy

FDA ID# 42-689

ADDRESS: 4242 Wide Lane, Hometown PA 19856

SAMPLE COLLECTED			TANKER TEMP. (°F)	OWNER OF MILK/ FIPS #	COMPLETE TANKER LICENSE PLATE NUMBER	BILL OF LADING #	POUNDS	LAB. TEMP. CONTROL (°C) 0.0-4.5C	TIME START TESTING	TIME READ RESULTS	RESULTS (NUMERICAL VALUE)	INTERP. (POS/NF)	NAME/ID#	COMMENTS
DATE (mm/dd)	TIME	Sampler ID												
1/27	9:05	JR	39.0°F	42-405	PYK8005	37092	45,289 lbs	3.9°C	9:22	9:30	0.67	Not Found	JK	
1/27	10:15	MJ	38.0°F	Hilltop 42-341	XFT8736	38112	35,599 lbs	3.3°C	10:20	10:28	0.71	Not Found	JM	
1/27	10:45	MJ	37.5°F	42-405	XFK5592	38561	37, 268 lbs	3.2C	10:51	10:59	0.59	Not Found	JK	

A POSITIVE AND NEGITIVE CONTROL MUST BE RUN EACH DAY THAT SCREENING TEST IS PERFORMED WITH RESULTS MAINTAINED.

COMMERCIAL POSITIVE CONTROL			RECONSTITUTED POSITIVE CONTROL		PRE-TESTED NEGATIVE CONTROL		TEST KIT INFORMATION		READER PERFORMANCE CHECKS	
MFG.	IDEXX		LOT #	EH598	ID (i.e. SILO #):	37090	LOT#:	DA188	ROSA	SERIAL #:
LOT #	EH598		DATE PREP'D	1/27/2014	DATE PREP'D:	1/26/2014	EXPIRATION DATE:	5/14/2014	LOW STRIP RESULT	
DATE RECEIVED:	12/25/2013		TIME PREP'D:	09:00AM	TIME PREP'D:	9:55AM			HIGH STRIP RESULT	
DATE OPENED:	1/2/2014		FROZEN DATE	NA	FROZEN DATE	1/26/2014			LOW RANGE	
LOT EXPIRES ON:	4/1/2014		THAW DATE	NA	THAW DATE	1/27/2014			HIGH RANGE	
			EXPIRES:	1/28/2014	EXPIRES:	1/28/2014				
			NUMERICAL RESULT:	5.01	NUMERICAL RESULT:	0.87				
HEATER BLOCK TEMPERATURE	40-50C		FRIDGE TEMPERATURE	0.0-4.5C	FREEZER TEMPERATURE	< -15.0C	LEVEL CHECK	IDEXX		
HEATER BLOCK SN#	12578		FRIDGE SN#	S1985E58	FREEZER SN#		(Charm ROSA only)	DEVICE 1 RESULT	0.73	
AM	PM		AM	PM	AM	PM	SATISFACTORY?	DEVICE 2 RESULT	1.56	
47.8°C	46.5°C		1.2°C	1.5°C	-15.5°C	-16.1°C	YES / NO	DEVICE 1 RANGE	0.58-0.88	
°C	°C		2.5°C	2.6°C	°C	°C	ANALYST ID#	DEVICE 2 RANGE	1.25-1.85	
INITIALS	JR	INITIALS	MJ	INITIALS	JR	INITIALS	MJ			



**TEMPERATURE RECORDS (BLOCK HEATER)**

Facility/Laboratory Name \_\_\_\_\_

Heater Block Make/ Model \_\_\_\_\_ Unit ID# \_\_\_\_\_

Testing Procedure Used \_\_\_\_\_ Temp. Range of Use: \_\_\_\_\_

MONTH/YEAR \_\_\_\_\_

Day	Temperature AM	Analysts Initials/ID#	Temperature PM	Analysts Initials/ID#
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				
31				

1. Temperature are checked each day of use prior to use.
2. All temperature readings need to be made to the nearest 0.1°C.

**TEMPERATURE RECORDS (BLOCK HEATER)**

Facility/Laboratory Name Utter's Dairy

Heater Block Make/ Model LAB-LINE MULTI-BLOK HEATER/2052 Unit ID# Block#1

Testing Procedure Used IDEXX NEW SNAP Temp. Range of Use: 45±5°C

MONTH/YEAR January 2014

Day	Temperature AM	Analysts Initials/ID#	Temperature PM	Analysts Initials/ID#
1				
2	45.9°C	JK		
3				
4	45.0°C	AT		
5				
6	45.8°C	AT		
7				
8				
9	45.3°C	JM		
10				
11	45.1°C	JK		
12				
13	44.9°C	JK		
14				
15				
16	44.8°C	AT		
17				
18	45.3°C	AT		
19				
20	45.0°C	AT		
21				
22				
23	45.1°C	JK		
24				
25	45.0°C	JM		
26				
27	44.8°C	JK		
28				
29				
30	44.9°C	AT		
31				

1. Temperatures are checked each day of use prior to use.
2. All temperature readings need to be made to the nearest 0.1°C.

**TEMPERATURE RECORDS (BLOCK HEATER)**

Facility/Laboratory Name Utter's Dairy

Heater Block Make/ Model ROSA Incubator Unit ID# RR0795

Testing Procedure Used Charm SL Temp. Range of Use: 56±1°C

MONTH/YEAR January 2014

Day	Temperature AM	Analysts Initials/ID#	Temperature PM	Analysts Initials/ID#
1				
2	56.1°C	JK	56.0°C	AS
3				
4	56.1°C	AT		
5				
6	56.1°C	AT		
7				
8				
9	56.1°C	JM	55.9°C	AS
10				
11	56.1°C	JK		
12				
13	56.9°C	JK		
14				
15				
16	56.1°C	AT	56.6°C	AS
17				
18	56.3°C	AT		
19				
20	56.0°C	AT		
21				
22				
23	56.1°C	JK	55.9°C	AS
24				
25	56.0°C	JM		
26				
27	55.8°C	JK		
28				
29				
30	55.9°C	AT	55.7°C	JT
31				

1. Temperatures are checked each day of use prior to use.
2. All temperature readings need to be made to the nearest 0.1°C.

### TEMPERATURE RECORDS (FREEZER)

Facility/Laboratory Name \_\_\_\_\_

Make/ Model \_\_\_\_\_ Unit ID# or Serial No. \_\_\_\_\_

MONTH/YEAR \_\_\_\_\_

Temp. Range: < -15°C

Day	Temperature AM		Analysts Initials/ID#	Temperature PM		Analysts Initials/ID#
	(top)	(bottom)		(top)	(bottom)	
Thermometer location (shelf)						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						

1. Temperatures are to be checked daily for screening-only locations; temperatures are checked twice daily (AM and PM) for CIS and Milk Industry locations.
2. All temperature readings need to be made to the nearest 0.1°C.

### TEMPERATURE RECORDS (FREEZER)

Facility/Laboratory Name Utter's Dairy

Make/ Model Revco Unit ID# or Serial No. REL5004E

MONTH/YEAR January 2014

Temp. Range: < -15°C

Day Thermometer location (shelf)	Temperature AM		Analysts Initials/ID#	Temperature PM		Analysts Initials/ID#
	(top)	(bottom)		(top)	(bottom)	
1	-15.5	-16.3	JK	-18.0	-22.0	JM
2	-17.5	-21.1	JK	-16.5	-21.5	JK
3	-17.5	-22.0	AT	-18.5	-19.5	JK
4	-18.0	-22.0	JM	-18.0	-19.0	AT
5	-16.5	-21.5	AT	-20.6	-22.0	JM
6	-18.5	-19.5	JK	-19.5	-20.2	AT
7	-18.0	-19.0	AT	-16.5	-18.5	JK
8	-20.6	-22.0	AT	-15.5	-16.3	AT
9	-19.5	-20.2	JK	-17.5	-21.1	AT
10	-16.5	-18.5	JM	-17.5	-22.0	JK
11	-15.5	-16.3	JK	-18.5	-19.5	JK
12	-17.5	-21.1	JK	-18.0	-19.0	JK
13	-17.5	-22.0	AT	-20.6	-22.0	JK
14	-18.0	-22.0	JM	-19.5	-20.2	AT
15	-16.5	-21.5	AT	-16.5	-18.5	JM
16	-18.5	-19.5	JK	-15.5	-16.3	AT
17	-18.0	-19.0	AT	-15.5	-16.3	JK
18	-20.6	22.0	AT	-17.5	-21.1	AT
19	-19.5	-20.2	JK	-17.5	-22.0	AT
20	-16.5	-18.5	JM	-18.0	-22.0	JK
21	-15.5	-16.3	JK	-16.5	-21.5	JM
22	-15.5	-16.3	JK	-16.5	-21.5	JK
23	-17.5	-21.1	JK	-18.5	-19.5	JK
24	-17.5	-22.0	AT	-18.0	-19.0	AT
25	-18.0	-22.0	JM	-20.6	-22.0	JM
26	-16.5	-21.5	AT	-19.5	-20.2	AT
27	-18.5	-19.5	JK	-16.5	-18.5	JK
28	-18.0	-19.0	AT	-15.5	-16.3	AT
29	-20.6	-22.0	AT	-15.5	-16.3	AT
30	-19.5	-20.2	JK	-17.5	-21.1	JK
31	-16.5	-18.5	JM	-17.5	-22.0	JM

1. Temperatures are to be checked daily for screening-only locations; temperatures are checked twice daily (AM and PM) for CIS and Milk Industry locations.
2. All temperature readings need to be made to the nearest 0.1°C.

**TEMPERATURE RECORDS (REFRIGERATOR)**

Facility/Laboratory Name \_\_\_\_\_

Make/ Model \_\_\_\_\_ Unit ID# \_\_\_\_\_

MONTH/YEAR \_\_\_\_\_

**Temp. Range: 0.0°C to 4.5°C**

Day	Temperature AM		Analysts Initials/ID#	Temperature PM		Analysts Initials/ID#
	(top)	(bottom)		(top)	(bottom)	
Thermometer location (shelf)						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						

1. Temperatures are to be checked daily for screening-only locations; temperatures are checked twice daily (AM and PM) for CIS and Milk Industry locations.
2. All temperature readings need to be made to the nearest 0.1°C.

**TEMPERATURE RECORDS (REFRIGERATOR)**

Facility/Laboratory Name Utter's Dairy

Make/ Model Revco Unit ID# or Serial No. REL5004E

MONTH/YEAR January 2014

Temp. Range: 0.0°C to 4.5°C

Day	Temperature AM		Analysts Initials/ID#	Temperature PM		Analysts Initials/ID#
	(top)	(bottom)		(top)	(bottom)	
Thermometer location (shelf)						
1	2.5	3.0	JK	2.5	3.0	JK
2	2.5	3.1	JK	2.6	3.1	JK
3	2.7	3.0	AT	2.5	3.0	AT
4	2.5	3.0	JM	2.6	3.1	JM
5	2.6	3.1	AT	2.5	3.0	AT
6	2.5	3.0	JK	2.7	3.1	JK
7	2.6	3.1	AT	2.5	3.0	AT
8	2.5	3.0	AT	2.5	3.0	AT
9	2.7	3.1	JK	2.5	3.1	JK
10	2.5	3.0	JM	2.7	3.0	JM
11	2.5	3.0	JK	2.7	3.0	JK
12	2.5	3.1	JK	2.5	3.0	JK
13	2.7	3.0	AT	2.6	3.1	JK
14	2.5	3.0	JM	2.5	3.0	AT
15	2.6	3.1	AT	2.6	3.1	JM
16	2.5	3.0	JK	2.5	3.0	AT
17	2.6	3.1	AT	2.7	3.1	JK
18	2.5	3.0	AT	2.5	3.0	AT
19	2.7	3.1	JK	2.5	3.0	AT
20	2.5	3.0	JM	2.6	3.1	JK
21	2.5	3.0	JK	2.5	3.0	JM
22	2.6	3.1	JK	2.5	3.0	JK
23	2.5	3.0	JK	2.5	3.1	JK
24	2.6	3.1	AT	2.7	3.0	AT
25	2.5	3.0	JM	2.5	3.0	JM
26	2.7	3.1	AT	2.6	3.1	AT
27	2.5	3.0	JK	2.5	3.0	JK
28	2.5	3.0	AT	2.6	3.1	AT
29	2.5	3.1	AT	2.5	3.0	AT
30	2.7	3.0	JK	2.7	3.1	JK
31	2.5	3.0	JM	2.5	3.0	JM

1. Temperatures are to be checked daily for screening-only locations; temperatures are checked twice daily (AM and PM) for CIS and Milk Industry locations.
2. All temperature readings need to be made to the nearest 0.1°C.

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

### SEMI-ANNUAL PIPETTOR ACCURACY CHECK

Test Kit for Use: \_\_\_\_\_

Calibration Location:  On-site  Other Name: \_\_\_\_\_

Date:		Date:		Date:		Date:	
Pipettor ID:		Pipettor ID:		Pipettor ID:		Pipettor ID:	
Analyst:		Analyst:		Analyst:		Analyst:	
Balance used (SN#):		Balance used (SN#):		Balance used (SN#):		Balance used (SN#):	
Reading #	Weight(gm) <sup>4</sup>						
1		1		1		1	
2		2		2		2	
3		3		3		3	
4		4		4		4	
5		5		5		5	
6		6		6		6	
7		7		7		7	
8		8		8		8	
9		9		9		9	
10		10		10		10	
Average		Average		Average		Average	

1. Check accuracy with ten (10) consecutive weighings once every 6 months.
2. Use with an analytical balance that reads to four decimal points.
3. Pipet and dispense as used during normal test procedure.
4. If pipettor specified volume is  $\geq 1.0$  mL, measurements may be by volume using class A graduated cylinder.
5. Average of all 10 weighing must be  $\pm 5\%$  of pipettor specified delivery volume.
6. If accuracy check fails ( $>5\%$ ), pipettor is to be taken out of service.
7. Individual pipettors must be etched or imprinted with identification and tagged with the average volume and date of accuracy check.

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: Utter's Dairy

### SEMI-ANNUAL PIPETTOR ACCURACY CHECK

Test Kit for Use: CHARM SL , Finnpiquette 300µlCalibration Location:  On-site  Other Name: \_\_\_\_\_

Date: <u>1/15/13</u>		Date: <u>7/25/13</u>		Date: <u>1/20/14</u>		Date:	
Pipettor ID: <u>J44426</u>		Pipettor ID: <u>J44426</u>		Pipettor ID: <u>J44426</u>		Pipettor ID:	
Analyst: <u>A Thomas</u>		Analyst: <u>A Thomas</u>		Analyst: <u>J. Michaels_</u>		Analyst:	
Balance used (SN#): <u>10226978</u>		Balance used (SN#): <u>10226978</u>		Balance used (SN#): <u>10229978</u>		Balance used (SN#):	
Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>
1	<u>.2922</u>	1	<u>.3205</u>	1	<u>.3278</u>	1	
2	<u>.2988</u>	2	<u>.3111</u>	2	<u>.3021</u>	2	
3	<u>.2921</u>	3	<u>.3125</u>	3	<u>.3098</u>	3	
4	<u>.2890</u>	4	<u>.3075</u>	4	<u>.3542</u>	4	
5	<u>.2980</u>	5	<u>.3061</u>	5	<u>.3134</u>	5	
6	<u>.3054</u>	6	<u>.3041</u>	6	<u>.3062</u>	6	
7	<u>.3214</u>	7	<u>.3126</u>	7	<u>.3076</u>	7	
8	<u>.3232</u>	8	<u>.3116</u>	8	<u>.3057</u>	8	
9	<u>.2998</u>	9	<u>.3128</u>	9	<u>.3322</u>	9	
10	<u>.2960</u>	10	<u>.3180</u>	10	<u>.3032</u>	10	
	<u>3.017</u>		<u>3.1168</u>		<u>3.1622</u>		
Average	<u>.3017</u>	Average	<u>.3117</u>	Average	<u>.3162</u>	Average	

1. Check accuracy with ten (10) consecutive weighings once every 6 months.
2. Use with an analytical balance that reads to four decimal points.
3. Pipet and dispense as used during normal test procedure.
4. If pipettor specified volume is  $\geq 1.0$  mL, measurements may be by volume using class A graduated cylinder.
5. Average of all 10 weighing must be  $\pm 5\%$  of pipettor specified delivery volume.
6. If accuracy check fails ( $>5\%$ ), pipettor is to be taken out of service.
7. Individual pipettors must be etched or imprinted with identification and tagged with the average volume and date of accuracy check.

Facility/Laboratory Name: Utter's Dairy

**SEMI-ANNUAL PIPETTOR ACCURACY CHECK**

Test Kit for Use: IDEXX New Snap, Eppendorf 450µl

Calibration Location:  On-site  Other Name: \_\_\_\_\_

Date: <u>1/15/13</u>		Date: <u>7/25/13</u>		Date: <u>1/20/14</u>		Date:	
Pipettor ID: <u>9588463</u>		Pipettor ID: <u>9588463</u>		Pipettor ID: <u>9588463</u>		Pipettor ID:	
Analyst: <u>A Thomas</u>		Analyst: <u>A Thomas</u>		Analyst: <u>J. Michaels</u>		Analyst:	
Balance used (SN#): <u>10226978</u>		Balance used (SN#): <u>10226978</u>		Balance used (SN#): <u>10229978</u>		Balance used (SN#):	
Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>	Reading #	Weight(gm) <sup>4</sup>
1	.4773	1	.4596	1	.4719	1	
2	.4611	2	.4593	2	.4601	2	
3	.4645	3	.4570	3	.4592	3	
4	.4712	4	.4596	4	.4594	4	
5	.4646	5	.4616	5	.4722	5	
6	.4628	6	.4575	6	.4621	6	
7	.4497	7	.4493	7	.4730	7	
8	.4708	8	.4524	8	.4709	8	
9	.4672	9	.4509	9	.4690	9	
10	.4578	10	.4593	10	.4652	10	
	4.647		4.567		4.663		
Average	.4647	Average	.4567	Average	.4663	Average	

EXAMPLE

1. Check accuracy with ten (10) consecutive weighings once every 6 months.
2. Use with an analytical balance that reads to four decimal points.
3. Pipet and dispense as used during normal test procedure.
4. If pipettor specified volume is ≥ 1.0 mL, measurements may be by volume using class A graduated cylinder.
5. Average of all 10 weighing must be ±5% of pipettor specified delivery volume.
6. If accuracy check fails (>5%), pipettor is to be taken out of service.
7. Individual pipettors must be etched or imprinted with identification and tagged with the average volume and date of accuracy check.



COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: Utter's Dairy

**TEST KIT SUITABILITY CHECK FOR DRUG RESIDUE TESTING**

Test Method Used: Charm SL

Date Received	Lot Number	Expiration Date	Date Tested <sup>1</sup>	Date Start Using <sup>2</sup>	Control Results/Interpretation				Analyst ID or Initials
					POSITIVE CONTROL		NEGATIVE CONTROL		
					Result	Suitability Check Date <sup>3</sup>	Result	Suitability Check Date <sup>3</sup>	
12/15/13	127	4/2014	12/16/13	12/20/13	+1954 POS	12/16/13	-1149 NF	12/15/13	JM
1/8/14	128	6/2014	1/10/14	1/11/14	+2257 POS	1/10/14	-2087 NF	1/8/14	JM

1. 'Date tested' is the date the first positive and negative control is run to check the suitability of the new lot.
2. 'Date Start Using' is the date this lot of kits is put into use, testing controls & trucks.
3. Suitability check date the POS/NEG control that was used to check the new kit was first tested and found suitable.

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

Facility/Laboratory Name: Utter's Dairy

**TEST KIT SUITABILITY CHECK FOR DRUG RESIDUE TESTING**

Test Method Used: IDEXX New Snap

Date Received	Lot Number	Expiration Date	Date Tested <sup>1</sup>	Date Start Using <sup>2</sup>	Control Results/Interpretation				Analyst ID or Initials
					POSITIVE CONTROL		NEGATIVE CONTROL		
					Result	Suitability Check Date <sup>3</sup>	Result	Suitability Check Date <sup>3</sup>	
12/15/13	MT995	6 FEB 14	12/16/13	12/20/13	5.69 POS	12/16/13	0.76 NF	12/15/13	JM
1/8/14	KD143	30 APR 14	1/10/14	1/11/14	3.30 POS	1/10/14	0.67 NF	1/8/14	JM

1. 'Date tested' is the date the first positive and negative control is run to check the suitability of the new lot.
2. 'Date Start Using' is the date this lot of kits is put into use, testing controls & trucks.
3. Suitability check date the POS/NEG control that was used to check the new kit was first tested and found suitable.



COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: Utter's Dairy

**Positive Control Suitability Test**

**Positive Control Information**

**Test Kit Information**

Manufacturer	Lot #	Mfg. Expiration Date	Date Prepared	Expiration Date	Date Tested	Start Test Time	Read Results Time	Test Results Positive Control	Analyst ID# or Initials		Test Kit Used	Manufacturer	Lot #	Mfg. Expiration Date
Charm Sciences	18D	2/2014	12/16/13	12/17/13	12/16/13	08:35	08:45	+1954/POS	JM		Charm SL	Charm Sciences	127	4/2014
Charm Sciences	18D	2/2014	12/23/13	12/24/13	12/23/13	09:30	09:40	+2155/POS	AT		Charm SL	Charm Sciences	127	4/2014
Charm Sciences	18F	5/2014	12/30/13	12/31/13	12/30/13	08:20	08:30	+1874/POS	AT		Charm SL	Charm Sciences	127	4/2014
Charm Sciences	18F	5/2014	1/10/14	1/11/14	1/10/14	09:15	09:25	+2358/POS	JM		Charm SL	Charm Sciences	128	6/2014

EXAMPLE

COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

**Positive Control Suitability Test**

**Positive Control Information**

**Test Kit Information**

Manufacturer	Lot #	Mfg. Expiration Date	Date Prepared	Expiration Date	Date Tested	Start Test Time	Read Results Time	Test Results Positive Control	Analyst ID# or Initials		Test Kit Used	Manufacturer	Lot #	Mfg. Expiration Date
IDEXX	EK669	2/16/14	12/16/13	12/17/13	12/16/13	08:35	08:45	5.69/Pos	JM		SNAP	IDEXX	MT995	2/6/14
IDEXX	EK669	2/16/14	12/23/13	12/24/13	12/23/13	09:30	09:40	4.17/POS	AT		SNAP	IDEXX	MT995	2/6/14
IDEXX	EK669	2/16/14	12/30/13	12/31/13	12/30/13	08:20	08:30	6.68/POS	AT		SNAP	IDEXX	MT995	2/6/14
IDEXX	EK669	2/16/14	1/10/14	1/11/14	1/10/14	09:15	09:25	3.30/POS	JM		SNAP	IDEXX	KD143	4/30/14

EXAMPLE





COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

**Negative Control Suitability Test**

**Negative Control Information**

**Test Kit Information**

Source (bulk tank, silo, tanker, etc)Date	ID # ( tanker license #, silo#, etc)	Date Prepared	Expiration Date	Date Tested	Start Test Time	Read Results Time	Test Results Negative Control	Analyst ID# or Initials		Test Kit Used	Manufacturer	Lot #	Mfg. Expiration Date
Tanker	1235587	12/16/13	2/16/14	12/16/13	08:35	08:45	0.69/NF	JM		SNAP	IDEXX	MT995	2/6/14
Tanker	1236489	12/23/13	2/23/14	12/23/13	09:30	09:40	0.77/ NF	AT		SNAP	IDEXX	MT995	2/6/14
Tanker	1236954	12/30/13	2/30/14	12/30/13	08:20	08:30	0.68/ NF	AT		SNAP	IDEXX	MT995	2/6/14
Tanker	1237259	1/10/14	3/10/14	1/10/14	09:15	09:25	0.80/ NF	JM		SNAP	IDEXX	KD143	4/30/14

EXAMPLE



Facility/Laboratory Name: \_\_\_\_\_

Year \_\_\_\_\_

**THERMOMETER ACCURACY CHECK LOG**

Date NIST Tested	NIST	Serial /ID Number	Range	Graduation Interval	Calibration points	Ice point result	Correction Factor <sup>7</sup> °C	Analyst
1/6/14	NIST 1	F95-389	-1 to 101C	0.2	0,32,45,64,85	0.0C	0.0	JM
1/8/14	NIST 2	3697	-50 to 10C	0.2	-30, -15, 0	0.0C	0.0	JM
Date Tested	Test thermometer Location of use	Serial Number	Lab ID	Temp range of use °C	Temp of Test Thermometer °C	Temp and ID of NIST Reference Thermometer °C	Correction Factor °C	Analyst
1/6/14	Sampling	J3398	TC1	0.0-4.5	0.2	0.0 NIST 1	-0.2	JM
1/6/14	Sample receiving	J6689	TC2	0.0-4.5	0.0	0.0 NIST 1	0.0	JM
1/6/14	Fridge, top shelf	Ertco 14479	F1	0.0-4.5	0.6	0.0 NIST 1	-0.6	JM
1/6/14	Fridge, bottom shelf	Ertco 1245	F2	0.0-4.5	-0.2	0.0 NIST 1	+0.2	JM
1/7/14	Incubator, top shelf	Ertco 6695	I1	31-33	31.5	32.1 NIST 1	+0.6	JM
1/7/14	Incubator, bottom shelf	Ertco 1176	I2	31-33	31.9	32.1 NIST 1	+0.2	JM
1/7/14	Charm SL heater block	Ertco 5572	HB1	55-57	56.2	56.0 NIST 1	-0.2	JM
1/8/14	Freezer	Fisher F669	FZ1	<-15.0	-18.2	-18.6 NIST 2	-0.4	JM

- To be done before initial use and at least annually thereafter.
- National Institute of Standards and Testing (NIST) Certified thermometer, or equivalent, with a certificate of calibration.
- Range of test thermometers appropriate for designated use.
- Accuracy of test thermometers checked against certified thermometer.
- Accurate to ± 1.0°C when checked at temperature(s) of use.
- Results recorded and thermometers tagged with the following information: Identification, date of check, temperature of check, correction factor(s) and analyst ID.
- If NIST has a correction other than 0.0°C, use form BFSLS 515a.



Facility/Laboratory Name: \_\_\_\_\_ Year \_\_\_\_\_

**THERMOMETER ACCURACY CHECK LOG**

Date NIST Tested	NIST	Serial /ID Number	Range	Graduation Interval	Calibration points	Ice point result	Correction Factor °C	Analyst ID	
1/6/14	NIST 1	F95-389	-1 to 101C	0.2	0,32,45,64,85	0.0C	0.0	JM	
1/8/14	NIST 2	3697	-50 to 10C	0.2	-30, -15, 0	0.3C	-0.3	JM	
Date Tested	Test Thermometer Location of use	Serial Number	Lab ID	Temp range of use °C	Temp of Test Thermometer °C	Read Temp and ID of NIST Reference Thermometer °C	Adjusted NIST Reading °C	Correction Factor of Test Thermometer °C	Analyst ID
1/6/14	Sampling	J3398	TC1	0.0-4.5	0.2	0.0 NIST 1	0.0	-0.2	JM
1/6/14	Sample receiving	J6689	TC2	0.0-4.5	0.0	0.0 NIST 1	0.0	0.0	JM
1/6/14	Fridge, top shelf	Ertco 14479	F1	0.0-4.5	0.6	0.0 NIST 1	0.0	-0.6	JM
1/6/14	Fridge, bottom shelf	Ertco 1245	F2	0.0-4.5	-0.2	0.0 NIST 1	0.0	+0.2	JM
1/7/14	Incubator, top shelf	Ertco 6695	I1	31-33	31.5	32.1 NIST 1	32.1	+0.6	JM
1/7/14	Incubator, bottom shelf	Ertco 1176	I2	31-33	31.9	32.1 NIST 1	32.1	+0.2	JM
1/7/14	Charm SL heater block	Ertco 5572	HB1	55-57	56.2	56.0 NIST 1	56.0	-0.2	JM
1/8/14	Freezer	Fisher F669	FZ1	<-15.0	-18.2	-18.6 NIST 2	-18.9	-0.7	JM

- To be done before initial use and at least annually thereafter.
- National Institute of Standards and Testing (NIST) Certified thermometer, or equivalent, with a certificate of calibration.
- Range of test thermometers appropriate for designated use.
- Accuracy of test thermometers checked against certified thermometer.
- Accurate to ± 1.0°C when checked at temperature(s) of use.
- Results recorded and thermometers tagged with the following information: Identification, date of check, temperature of check, correction factor(s) and analyst ID.



Facility/Laboratory Name: Utter's Dairy

### Annual Appendix N Training Log

Name	PDA Analyst #	Position (CIS or IS or IA)	Date of Initial Training <sup>1</sup>	Date of On-site Review by IS <sup>2</sup>	Date of On-site review by State LEO <sup>3</sup>	Annual Split Sample Participation Date	Results from Split Samples (Pass/Fail)
Alyssa Thomas	03	IA	4/15/12	3/1/13	NA	3/12/13	PASS
Jeff Michaels	02	IA	10/19/13	3/1/13	NA	3/12/13	PASS
Jason Kirk	01	CIS	NA	NA	6/15/13	3/12/13	PASS
Alice Stone	04	CIS	3/3/12	NA	6/15/13	3/12/13	PASS

EXAMPLE

- Notes:
1. Date of the initial training for Industry Analyst (IA) to gain approval for testing.
  2. Date of annual in-house training and observation of the IA by the Supervisor.
  3. Date of audit with state LEO. Audit participation is optional for IA's and mandatory for all Industry Supervisors.
  4. All IA's and Supervisory must have a successful participation in the annual split samples to maintain approval/certification.

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

## APPENDIX N TRAINING SESSION APPROVAL REQUEST FOR NEW ANALYST

The following individuals have participated in training at: (Facility) \_\_\_\_\_ in (Town) \_\_\_\_\_ PA, concerning the Appendix N Testing Program for Drug Residues for (test) \_\_\_\_\_.

This training was held on \_\_\_\_\_, 20\_\_\_\_ by \_\_\_\_\_ Information and materials presented dealt with the review of the (current) Pasteurized Milk Ordinance (PMO)- Appendix N. Drug Residue Testing and Farm Surveillance, Industry Analyst, Industry Supervisor and Certified Industry Supervisor responsibilities. FDA 2400 forms and product inserts, along with quality control records, were used to evaluate approved methods for testing for animal drug residues. Each analyst properly demonstrated testing procedure of approved Appendix N method used at this facility.

The undersigned have been trained in the Appendix N requirements. They understand the responsibilities associated with this testing procedure.

DETERMINED BY FACILITY TRAINER			DETERMINED BY LABORATORY EVALUATION OFFICER		
Name of Participant (print)	SIGNATURE of Participant	Date Trained	Classification	Status	PDA #

Classification: IA= Ind. Analyst, IS = Ind. Supervisor, CIS = Certified Ind. Supervisor    Status: F<sup>A</sup>-Fully Approved, C<sup>A</sup> = Conditionally Approved, P<sup>A</sup> = Provisionally Approved

\_\_\_\_\_  
Facility Supervisor Signature

\_\_\_\_\_  
Date

\_\_\_\_\_  
State Laboratory Evaluation Officer Signature

\_\_\_\_\_  
Date Approved

COMMONWEALTH OF PENNSYLVANIA  
DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
LABORATORY DIVISION

**APPENDIX N TRAINING SESSION**  
**APPROVAL REQUEST FOR NEW ANALYST**

The following individuals have participated in training at: (Facility) Utter's Dairy in (Town) Hometown PA, concerning the Appendix N Testing Program for Drug Residues for (test) Charm SL.

This training was held on October 19, 20 12 by Jason Kirk, CIS. Information and materials presented dealt with the review of the (current) Pasteurized Milk Ordinance (PMO)- Appendix N. Drug Residue Testing and Farm Surveillance, Industry Analyst, Industry Supervisor and Certified Industry Supervisor responsibilities. FDA 2400 forms and product inserts, along with quality control records, were used to evaluate approved methods for testing for animal drug residues. Each analyst properly demonstrated testing procedure of approved Appendix N method used at this facility.

The undersigned have been trained in the Appendix N requirements. They understand the responsibilities associated with this testing procedure.

DETERMINED BY FACILITY TRAINER			DETERMINED BY LABORATORY EVALUATION OFFICER		
Name of Participant (print)	SIGNATURE of Participant	Date Trained	Classification	Status	PDA #
Jeff Michaels		10/19/12	IS	Ca	03

Classification: IA= Ind. Analyst, IS = Ind. Supervisor, CIS = Certified Ind. Supervisor Status: F<sup>A</sup>-Fully Approved, C<sup>A</sup> = Conditionally Approved, P<sup>A</sup> = Provisionally Approved

\_\_\_\_\_  
Facility Supervisor Signature

\_\_\_\_\_  
Date

\_\_\_\_\_  
State Laboratory Evaluation Officer Signature

\_\_\_\_\_  
Date Approved

COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

**SNAPSHOT PERFORMANCE CHECK SET**

YEAR: \_\_\_\_\_ MONTH: \_\_\_\_\_

SERIAL # OF PERFORMANCE CHECK SET: \_\_\_\_\_

DAY	DEVICE 1:C/S _____	DEVICE 2:C/S _____	ANALYST ID# OR INITIALS
	-.15 _____ +.15 _____	-.30 _____ +.30 _____	
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			

1. Performance Check Set needs to be done day of use along with a positive and negative control.

COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: WIDE CREEK FARMS

**SNAPSHOT PERFORMANCE CHECK SET**

YEAR: 2013

MONTH: December

SERIAL # OF PERFORMANCE CHECK SET: SNAP001347

DAY	DEVICE 1:C/S = <u>0.73</u>	DEVICE 2:C/S = <u>1.55</u>	ANALYST ID# OR INITIALS
	<u>-.15 = .58</u> <u>+.15 = .88</u>	<u>-.30 = 1.25</u> <u>+.30 = 1.85</u>	
1	.77	1.58	AT, #03
2	.76	1.58	JK, #01
3	.77	1.58	AT, #03
4	.77	1.59	AT, #03
5	.76	1.58	JM, #02
6	.77	1.58	AT, #03
7	.77	1.59	JK, #01
8	.77	1.58	JK, #01
9	.77	1.58	JK, #01
10	.77	1.58	JM, #02
11	.76	1.58	AT, #03
12	.77	1.58	JK, #01
13	.77	1.58	AT, #03
14	.77	1.58	AT, #03
15	.77	1.58	JM, #02
16	.77	1.58	AT, #03
17	.77	1.58	JK, #01
18	.77	1.58	JK, #01
19	.77	1.58	JK, #01
20	.77	1.58	JM, #02
21	.77	1.58	AT, #03
22	.77	1.58	AT, #03
23	.77	1.59	JK, #01
24	.77	1.58	AT, #03
25	.79	1.58	AT, #03
26	.77	1.58	JM, #02
27	.77	1.58	AT, #03
28	.77	1.58	JK, #01
29	.77	1.58	JK, #01
30	.77	1.58	JK, #01
31	.77	1.58	JM, #02

**EXAMPLE**

1. Performance Check Set needs to be done day of use along with a positive and negative control.

COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: \_\_\_\_\_

**CHARM ROSA READER (ROSA Reader, ROSA Pearl Reader or Charm Sciences equivalent)**

**PRIMARY CALIBRATION STRIPS**

YEAR \_\_\_\_\_ MONTH \_\_\_\_\_

SERIAL # OF PRIMARY CALIBRATION STRIPS \_\_\_\_\_

DAY	<u>LOW RANGE:</u>		<u>HIGH RANGE:</u>		ANALYST ID# OR INITIALS
	-20% _____	+20% _____	-20% _____	+20% _____	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					

1. Primary Calibration Strips need to be done day of use along with a positive and negative control.
2. Primary Calibration Strips match ROSA serial number. Calibration strips are specific to an individual reader. Do not interchange strips between different readers.

COMMONWEALTH OF PENNSYLVANIA  
 DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
 LABORATORY DIVISION

Facility/Laboratory Name: WIDE CREEK FARMS

**CHARM ROSA READER (ROSA Reader, ROSA Pearl Reader or Charm Sciences equivalent)**

**PRIMARY CALIBRATION STRIPS**

YEAR 2012

MONTH October

SERIAL #(S) OF PRIMARY CALIBRATION STRIPS RR0795 (CHARM SL)

DAY	LOW RANGE:		HIGH RANGE:		ANALYST ID# OR INITIALS
	-20%	+20%	-20%	+20%	
1	<u>-1063</u>	<u>-1594</u>	<u>+1297</u>	<u>+1945</u>	<u>JK</u>
2	<u>-1246</u>		<u>+1391</u>		
3	<u>-1445</u>		<u>+1685</u>		<u>JK</u>
4					
5	<u>-1330</u>		<u>+1403</u>		<u>AT</u>
6					
7	<u>-1455</u>		<u>+1537</u>		<u>JM</u>
8	<u>-1501</u>		<u>+1667</u>		<u>JM</u>
9					
10					
11	<u>-1422</u>		<u>+1333</u>		<u>JK</u>
12					
13	<u>-1456</u>		<u>+1785</u>		<u>JK</u>
14					
15	<u>-1099</u>		<u>+1469</u>		<u>JK</u>
16					
17					
18	<u>-1363</u>		<u>+1537</u>		<u>AT</u>
19					
20	<u>-1489</u>		<u>+1372</u>		<u>JM</u>
21					
22					
23	<u>-1125</u>		<u>+1403</u>		<u>AT</u>
24	<u>-1199</u>		<u>+1743</u>		<u>JM</u>
25					
26					
27	<u>-1099</u>		<u>+1899</u>		<u>AT</u>
28					
29	<u>-1159</u>		<u>+1589</u>		<u>AT</u>
30					
31	<u>-1426</u>		<u>+1900</u>		<u>JK</u>

**EXAMPLE**

1. Primary Calibration Strips need to be done day of use along with a positive and negative control.
2. Primary Calibration Strips match ROSA serial number. Calibration strips are specific to an individual reader. Do not interchange strips between different readers.

# APPENDIX N

## MEMOS



**pennsylvania**  
DEPARTMENT OF AGRICULTURE

BUREAU OF FOOD SAFETY & LABORATORY SERVICES

**Date:** February 4, 2014  
**Subject:** New FDA 2400 Form Changes  
**To:** All Pennsylvania Approved Dairy Laboratories/Facilities  
**From:** Michael F. Hydock, Chief  
Laboratory Division

**IMPLEMENTATION DATE: Immediately**

Enclosed is one (1) original copy of the revised FDA-2400 form series for the following procedures that may apply to your laboratory/facility:

1. FORM FDA/NCIMS 2400a-4 Petrifilm™ Aerobic & Coliform Count Rev. 1/14
2. FORM FDA/NCIMS 2400d Direct Microscopic Somatic Cell Count Rev. 1/14
3. FORM FDA/NCIMS 2400h-2 Bentley Somacount™ Rev. 1-14
4. FORM FDA/NCIMS 2400h-3 Foss 5000-FC Rev. 1/14
5. FORM FDA/NCIMS 2400h-4 SomaScope™ MKII/Smart Rev 1/14
6. FORM FDA/NCIMS 2400h-5 Foss Minor Rev. 1/14
7. FORM FDA/NCIMS 2400n-4 Charm® II Beta-Lactam Assays Rev. 1/14
8. FORM FDA/NCIMS 2400n-5 Charm® II Combined Non-Beta-Lactam Assays rev. 1/14

These forms have been reviewed and approved by the LPET in conjunction with the NCIMS Laboratory Committee.

In the case of these forms there are NO procedure changes. The changes in these forms are mainly the result of Conference actions (IMS#, footer FDA/NCIMS 2400x Form and 4.5C). In addition there are some editorial and format changes/fixes.

All laboratory surveys conducted after today shall be conducted using these new forms.

If you have any questions please contact me at 717-772-3236 or email [mhydock@pa.gov](mailto:mhydock@pa.gov)

cc. L. Johnson  
cc. J. Martin



**pennsylvania**  
DEPARTMENT OF AGRICULTURE

**BUREAU OF FOOD SAFETY & LABORATORY SERVICES**

Date: January 27, 2014

Subject: Laboratory Quality Control Forms Revised 12/13 and 1/14

To: Pennsylvania Approved Laboratories and Screening Facilities

From: Michael F. Hydock, Chief  
BFSLS – Laboratory Division

**EFFECTIVE IMMEDIATELY**

**Enclosed is a packet of the Laboratory Quality Control Forms Revised 12/13 or 1/14.**

The forms are as follows:

1. BFSLS 495 Incubator Temperature Log (Revision 12-13)
2. BFSLS 501a Temperature Record Block Heater (Revision 1-14)
3. BFSLS 501b Temperature Record Freezer (Revision 1-14)
4. BFSLS 501c Temperature Record Refrigerator (Revision 1-14)
5. BFSLS 510 Refrigerator Temperature Log (Revision 12-13)
6. BFSLS 515 Thermometer Accuracy check (Revision 12-13)
7. BFSLS 515a Thermometer Accuracy check (Revision 12-13 -New Form)
8. BFSLS 532 Freezer Temperature Log (Revision 12-13)
9. BFSLS 532a Freezer Temperature Log (Revision 12-13)
10. BFSLS 497 Monthly Analytical Balance Check Records (Revision 1-14)
11. BFSLS 498 Monthly Milk-Media Electronic Balance Check Records (Revision 1-14)
12. BFSLS 500 Drug Screening Test Log (Revision 1-14)
13. BFSLS 500a Daily Screening Log (Revision 1-14)
14. BFSLS 503 Semi-annual Pipettor Calibration (Revision 12-13)
15. BFSLS 513 Test Kit Suitability Check (Revision 1-14)
16. BFSLS 528 Appendix N Training Log (Revision 1-14)
17. BFSLS 528a Appendix N Training Sheet New Analyst (Revision 1-14)
18. BFSLS 480 Microbiological Suitable Water (MS) (Revision 12-13)
19. BFSLS 485 Daily pH Meter Maintenance (Revision 12-13)
20. BFSLS 485b Daily pH Meter Calibration Log (Revision 12-13)
21. BFSLS 486 Stock Phosphate Buffer(Purchased) (Revision 12-13)
22. BFSLS 486b Stock Phosphate Buffer(Lab Prepared) (Revision 12-13)
23. BFSLS 490 Quarterly Percent Weight Loss Records-Agar Method (Revision 12-13)
24. BFSLS 490a Quarterly Percent Weight Loss Records-Petrifilm Method (Revision 12-13)
25. BFSLS 492 Dilution Blank Checks (Revision 12-13)
26. BFSLS 492a Pre-Dispensed Rinse Solution Checks for Gallon Containers (Revision 12-13)
27. BFSLS 492b Pre-Dispensed Rinse Solution Checks for Half Pint, Pint, & Quart Containers (Revision 12-13)
28. BFSLS 492c Pre-Dispensed Rinse Solution Checks for Half Gallon Containers (Revision 12-13)
29. BFSLS 493 Autoclave Sterilization Record (Revision 12-13)
30. BFSLS 493a Autoclave Sterilization Record - Waste Only (Revision 12-13)

- 31. BFSLS 494 Hot Air Oven Sterilization Record (Revision 12-13)
- 32. BFSLS 503b Volume Check Continuous Pipetting Outfit (Revision 1-14)

If your laboratory/facility is using an older revision of the above forms, they are to be replaced with the current revision and used immediately for recording laboratory/facility required records. The forms and reports must be maintained and available for review during a laboratory/facility evaluation. If you desire to make any change in the format of these forms, a request outlining the change(s) must be submitted to the Laboratory Division for review and acceptance prior to initiating the change(s). A written response will be sent to the laboratory/ facility from BFSLS-Laboratory Division granting or not granting any change(s) in the format as acceptable for use as an official record form.

If you have any questions, call me at (717) 787-4315.

Thank you for your cooperation.

cc. J. Rakowski



**pennsylvania**  
DEPARTMENT OF AGRICULTURE

**BUREAU OF FOOD SAFETY & LABORATORY SERVICES**

**Date:** October 25, 2013  
**Subject:** New FDA 2400 Form Changes - Effective Immediately  
**To:** All Pennsylvania Approved Dairy Laboratories/Facilities  
**From:** Michael F. Hydock, Chief  
Laboratory Division

**IMPLEMENTATION DATE: NOVEMBER 1, 2013**

Enclosed is one (1) original copy of the revised FDA-2400 form series for the following procedure that applies to your laboratory/facility:

1. FORM FDA/NCIMS 2400n Appendix N - General Requirements rev. 10/13
2. FORM FDA/NCIMS 2400n-1 Charm® SL/SL3 rev. 10/13

These forms has been reviewed and approved by the LPET in conjunction with the NCIMS Laboratory Committee.

General Conference changes: Add IMS# to the top of each method, change 4.4C to 4.5C where specified and recognize the efforts of the NCIMS in the revision of these forms in the footer of each form, FDA/NCIMS 2400 form.

1. Appendix N General Requirements (FORM FDA/NCIMS 2400n): Clarification of balance calibration checks (item 6.c), clarification on use of pipettors (item 7.c), specifies need to record date and time of tanker testing (item 9.4), clarifies interpretation of positive and negative controls prior to reading duplicate test results for presumptive (item 11.b.1), BMT confirmation (item 12.b.1) and producer traceback (item 13.e.1) testing, and fixes producer traceback procedure so that there is an initial screen of producers on a load prior to running the samples in duplicate with controls (item 13.a-g).
2. Charm SL/SL3 BL Test (FORM FDA/NCIMS 2400n-1): Adds EZ reader wording including allowance to use EZ reader to incubate and read (item 7) and removes SL6 (no longer an approved method).

All laboratory surveys conducted **after November 1, 2013** shall be conducted using these new forms.

If you have any questions please contact me at 717-772-3236 or email [mhydock@pa.gov](mailto:mhydock@pa.gov)

cc. L. Johnson  
cc. J. Martin



**pennsylvania**  
DEPARTMENT OF AGRICULTURE

**BUREAU OF FOOD SAFETY & LABORATORY SERVICES**

**Date:** October 25, 2013  
**Subject:** New FDA 2400 Form Changes - Effective Immediately  
**To:** All Pennsylvania Approved Dairy Laboratories/Facilities  
**From:** Michael F. Hydock, Chief  
Laboratory Division

**IMPLEMENTATION DATE: NOVEMBER 1, 2013**

Enclosed is one (1) original copy of the revised FDA-2400 form series for the following procedure that applies to your laboratory/facility:

1. FORM FDA/NCIMS 2400n Appendix N - General Requirements rev. 10/13
2. FORM FDA/NCIMS 2400n-2 IDEXX New Snap® Beta-Lactam Test rev. 10/13

These forms has been reviewed and approved by the LPET in conjunction with the NCIMS Laboratory Committee.

General Conference changes: Add IMS# to the top of each method, change 4.4C to 4.5C where specified and recognize the efforts of the NCIMS in the revision of these forms in the footer of each form, FDA/NCIMS 2400 form.

1. Appendix N General Requirements (FORM FDA/NCIMS 2400n): Clarification of balance calibration checks (item 6.c), clarification on use of pipettors (item 7.c), specifies need to record date and time of tanker testing (item 9.4), clarifies interpretation of positive and negative controls prior to reading duplicate test results for presumptive (item 11.b.1), BMT confirmation (item 12.b.1) and producer traceback (item 13.e.1) testing, and fixes producer traceback procedure so that there is an initial screen of producers on a load prior to running the samples in duplicate with controls (item 13.a-g)
2. IDEXX New Snap BL Test (FORM FDA/NCIMS 2400n-2): Adds approval for testing raw camel and raw goat milk, and allows test kits to be shipped and received without refrigeration as long as shipping is less than 72 hour (item 3.f). Reminder: IDEXX has announced that they are no longer able to service the old SNAPshot reader as of October 31, 2013. The old readers may still be used as long as they continue to work as specified in the 2400 form.

All laboratory surveys conducted **after November 1, 2013** shall be conducted using these new forms.

If you have any questions please contact me at 717-772-3236 or email [mhydock@pa.gov](mailto:mhydock@pa.gov)

cc. L. Johnson  
cc. J. Martin



# pennsylvania

## DEPARTMENT OF AGRICULTURE

### BUREAU OF FOOD SAFETY & LABORATORY SERVICES

Date: June 15<sup>th</sup>, 2012

Subject: Appendix N Positive Drug Residue Dumping Procedure for Multi-Compartment Tankers

To: Milk Receiving Locations conducting Appendix N Drug Residue Testing  
Milk Sanitarian Supervisors  
Milk Sanitarians

From: Dr. Lydia Johnson | Director  
Bureau of Food Safety & Laboratory Services  
Pennsylvania Department of Agriculture  
2301 North Cameron Street | Harrisburg, PA 17110  
Phone: 717.787.4315 | Fax: 717.787.1873

In response to the recent questions raised concerning dumping of milk from multi-compartment tankers, the Pennsylvania Department of Agriculture (PDA) is adopting the following procedure effective June 1<sup>st</sup>, 2012. This policy rescinds any prior PDA policies or memorandums concerning dumping of Appendix N positive testing milk on multi-compartment tankers. This policy is intended to align PDA's policy with the interpretation of FDA as stated in the answer to question number 72 c) contained in M-I-12-9.

- If all compartments test negative, all compartments may be received.
- If all compartments test presumptive positive and are confirmed positive, all compartments must be dumped.
- When one compartment tests presumptive positive and the other compartment(s) test negative, the milk from the negative compartment(s) shall not be unloaded until the confirmatory tests and producer trace back tests are completed and a positive producer is confirmed.
- If milk from the bulk milk tank of the confirmed positive producer has been split between compartments, that milk is considered adulterated and must also be dumped.
- If a producer has multiple bulk milk tanks, only the compartments with milk from individual tank samples confirmed positive needs to be dumped.

Your cooperation is appreciated. If you have any questions or concerns, please contact me.

5100 Paint Branch Parkway  
College Park, MD 20740-3835

M-I-96-10 (Revision #8)

March 22, 2012

TO: All Regional Food and Drug Directors  
Attn: Regional Milk Specialists

FROM: Dairy and Egg Branch (HFS-316)

SUBJECT: Drug Residue Test Methods For Confirmation Of Presumptive Positive Results And Initial Producer Trace Back

This coded memorandum replaces and rescinds the previous revision of this coded memorandum (M-I-96-10 (Revision #7), Issued January 4, 2010). Revision #7 will be identified in the next Index of Memoranda of Information (M-I) as "INACTIVE".

This revision is based on the phasing out and addition of several Tests as reflected in M-a-85 (Revision #14), Issued March 22, 2012. It modifies the Tables to reflect the acceptance of the following Tests:

- Neogen Corporation BetaStar® Plus Beta Lactam Test;
- Charm Sciences, Inc. Charm® 3 SL3 Beta Lactam Test; and
- Charm Sciences, Inc. Charm® FLUSLBL Flunixin and Beta Lactam Test.

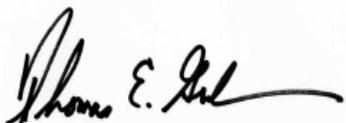
The following Tests have been removed as they are no longer available:

- Neogen Corporation BetaStar® US Beta Lactam Test; and
- Charm® SL3 Beta Lactam Test.

The following Tables have been developed by FDA's Center for Veterinary Medicine (CVM) to demonstrate those screening Tests that have been accepted under the requirements of Appendix N of the *Grade "A" Pasteurized Milk Ordinance* (PMO) to determine whether presumptive positive milk tank truck results are screening positive (load confirmation). These Tables may also be used to determine an appropriate initial producer trace back test to identify the positive producer(s) who contributed the milk containing drug(s) on a positive milk tank truck load. These Tables do not include any additional requirements for testing and are a reference for which Tests are appropriate to test samples from a producer(s), which has contributed milk containing drug(s) to a positive milk tank truck.

Copies of this memorandum are enclosed for distribution to Regional Milk Specialists, State Milk Regulatory Agencies, State Laboratory Evaluation Officers and State Milk Sanitation Rating Officers in your region. This memorandum should be widely distributed to representatives of the dairy industry, State Veterinarians, State Veterinary and Pharmacy Boards, Veterinarian Professional Organizations and other interested parties and will also be available on the FDA Web Site at <http://www.fda.gov> at a later date.

If you would like an electronic version of this document prior to it being available on the FDA Web Site, please e-mail your request to [Robert.Hennes@fda.hhs.gov](mailto:Robert.Hennes@fda.hhs.gov).



Thomas Graham, PhD, Team Leader  
Laboratory Proficiency & Evaluation Team



Robert F. Hennes, RS, MPH  
CAPT U.S. Public Health Service  
Dairy and Egg Branch

<b>PRESUMPTIVE POSITIVE TEST</b>	<b>SCREENING TEST POSITIVE (CONFIRMATION TEST) OPTIONS</b>
BetaStar® Plus Beta Lactam Test	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® II Tablet Beta Lactam Test (Competitive Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test
Charm® <i>B. stearothermophilus</i> Tablet Disk Assay  Not a Beta lactam Specific Test. Refer to Footnote <sup>1</sup> .	BetaStar® Plus Beta Lactam Test Charm® <i>B. stearothermophilus</i> Tablet Disk Assay Charm® II Tablet Beta Lactam Test (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® SL Beta Lactam Test Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test Charm® FLUSLBL Flunixin and Beta Lactam Test Delvotest P 5 Pack (Reader and Visual) Delvotest P/Delvotest P Mini Delvotest SP/Delvotest SP Mini New Snap® Beta Lactam Test
Charm® II Tablet Beta Lactam Test (Competitive Assay)	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® SL Beta Lactam Test Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test Charm® FLUSLBL Flunixin and Beta Lactam Test New Snap® Beta Lactam Test
Charm® II Tablet Beta Lactam Test (Sequential Assay)	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® SL Beta Lactam Test Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test Charm® FLUSLBL Flunixin and Beta Lactam Test New Snap® Beta Lactam Test

<b>PRESUMPTIVE POSITIVE TEST</b>	<b>SCREENING TEST POSITIVE (CONFIRMATION TEST) OPTIONS</b>
Charm® II Tablet Beta Lactam Test (Quantitative Assay)	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® II Tablet Beta Lactam Test (Competitive Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test
Charm® SL Beta Lactam Test	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® SL Beta Lactam Test Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test Charm® FLUSLBL Flunixin and Beta Lactam Test New Snap® Beta Lactam Test
Charm® SL6 Beta Lactam Test	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® II Tablet Beta Lactam Test (Competitive Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test
Charm® 3 SL3 Beta Lactam Test	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® II Tablet Beta Lactam Test (Competitive Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) + Charm® II Test for Cloxacillin in Milk (Competitive Assay) Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test
Charm® FLUSLBL Flunixin and Beta Lactam Test	Charm® FLUSLBL Flunixin and Beta Lactam Test
Charm® II Chloramphenicol Test	Charm® II Chloramphenicol Test
Charm® II Sulfa Drug Test (Competitive Assay)	Charm® II Sulfa Drug Test (Competitive Assay)
Charm® II Tetracycline Drug Test (Competitive Assay)	Charm® II Tetracycline Drug Test (Competitive Assay)

<b>PRESUMPTIVE POSITIVE TEST</b>	<b>SCREENING TEST POSITIVE (CONFIRMATION TEST) OPTIONS</b>
<p>Delvotest P 5 Pack (Reader and Visual)</p> <p>Not a Beta lactam Specific Test. Refer to Footnote<sup>1</sup>.</p>	<p>BetaStar® Plus Beta Lactam Test            Charm® <i>B. stearothermophilus</i> Tablet Disk Assay            Charm® II Tablet Beta Lactam Test (Competitive Assay)            Charm® II Tablet Beta Lactam Test (Sequential Assay)            Charm® II Tablet Beta Lactam Test (Quantitative Assay)            Charm® SL Beta Lactam Test            Charm® SL6 Beta Lactam Test            Charm® 3 SL3 Beta Lactam Test            Charm® FLUSLBL Flunixin and Beta Lactam Test            Delvotest P 5 Pack (Reader and Visual)            Delvotest P/Delvotest P Mini            Delvotest SP/Delvotest SP Mini            New Snap® Beta Lactam Test</p>
<p>Delvotest P/Delvotest P Mini</p> <p>Not a Beta lactam Specific Test. Refer to Footnote<sup>1</sup>.</p>	<p>BetaStar® Plus Beta Lactam Test            Charm® <i>B. stearothermophilus</i> Tablet Disk Assay            Charm® II Tablet Beta Lactam Test (Competitive Assay)            Charm® II Tablet Beta Lactam Test (Sequential Assay)            Charm® II Tablet Beta Lactam Test (Quantitative Assay)            Charm® SL Beta Lactam Test            Charm® SL6 Beta Lactam Test            Charm® 3 SL3 Beta Lactam Test            Charm® FLUSLBL Flunixin and Beta Lactam Test            Delvotest P 5 Pack (Reader and Visual)            Delvotest P/Delvotest P Mini            Delvotest SP/Delvotest SP Mini            New Snap® Beta Lactam Test</p>
<p>Delvotest SP/Delvotest SP Mini</p> <p>Not a Beta lactam Specific Test. Refer to Footnote<sup>1</sup>.</p>	<p>BetaStar® Plus Beta Lactam Test            Charm® <i>B. stearothermophilus</i> Tablet Disk Assay            Charm® II Tablet Beta Lactam Test (Competitive Assay)            Charm® II Tablet Beta Lactam Test (Sequential Assay)            Charm® II Tablet Beta Lactam Test (Quantitative Assay)            Charm® SL Beta Lactam Test            Charm® SL6 Beta Lactam Test            Charm® 3 SL3 Beta Lactam Test            Charm® FLUSLBL Flunixin and Beta Lactam Test            Delvotest P 5 Pack (Reader and Visual)            Delvotest P/Delvotest P Mini            Delvotest SP/Delvotest SP Mini            New Snap® Beta Lactam Test</p>

<b>PRESUMPTIVE POSITIVE TEST</b>	<b>SCREENING TEST POSITIVE (CONFIRMATION TEST) OPTIONS</b>
New Snap® Beta Lactam Test	BetaStar® Plus Beta Lactam Test Charm® II Tablet Beta Lactam Test (Competitive Assay) Charm® II Tablet Beta Lactam Test (Sequential Assay) Charm® II Tablet Beta Lactam Test (Quantitative Assay) Charm® SL Beta Lactam Test Charm® SL6 Beta Lactam Test Charm® 3 SL3 Beta Lactam Test Charm® FLUSLBL Flunixin and Beta Lactam Test New Snap® Beta Lactam Test

<sup>1</sup>These Tests are not specific for Beta lactams. While they are not validated to NCIMS standards for any drugs other than Beta lactams, a non-Beta lactam drug residue can cause a positive test. A negative or not found (NF) test result using one of the Beta lactam specific Tests to follow-up or confirm a positive result with this test kit will require re-testing the sample using the Charm® *B. stearothersophilus* Tablet Disk Assay, Delvotest P 5 Pack, Delvotest P/Delvotest P Mini or Delvotest SP/Delvotest SP Mini. Any of the listed confirmation test options, other than the four (4) identified in the Tables, are Beta lactam specific Tests.

5100 Paint Branch Parkway  
College Park, MD 20740-3835

M-a-85 (Revision #14)

March 22, 2012

TO: All Regional Food and Drug Directors  
Attn: Regional Milk Specialists

FROM: Dairy and Egg Branch (HFS-316)

SUBJECT: Beta Lactam And Other Test Methods For Use Under Appendix N And  
Section 6 Of The *Grade "A" Pasteurized Milk Ordinance* (PMO)

This coded memorandum replaces and rescinds the previous revision of this coded memorandum (M-a-85 (Revision #13), Issued January 4, 2010).

This revision addresses the following changes:

- The acceptance of the Neogen Corporation BetaStar® Plus Beta Lactam Test and concurrent removal of the Neogen BetaStar® US Beta Lactam Test (M-I-11-3, Issued June 3, 2011);
- The acceptance of the Charm Sciences, Inc. Charm® 3 SL3 Beta Lactam Test and the removal of the Charm Sciences, Inc. Charm® SL3 Beta Lactam Test as Charm Sciences is no longer manufacturing the test kit and all lots have reached their expiration date (M-I-10-8, Issued November 4, 2010); and
- The acceptance of the Charm Sciences, Inc. Charm® FLUSLBL Flunixin and Beta Lactam Test (M-I-12-3, Issued February 3, 2102).

The individual Test Tables presented in this revision provide data points that were derived from testing at least thirty (30) samples at each concentration for each drug detected.

The attached information is summarized from the evaluation of data submitted by test sponsors. Information related to the protocol used in this evaluation is available from Dr. David White, FDA's Center for Veterinary Medicine (CVM), (301) 210-4760. Additional information regarding the performance of these Tests may be available from the test kit manufacturers.

Label claims for these new approved Tests were evaluated for use on raw, commingled bovine milk samples. The evaluation protocol did not measure the performance of these Tests in the assay of drug residues in other milk matrices, i.e., pasteurized milk or milk

taken from individual cows, although claims for such use are made by some of the manufacturers of these Tests.

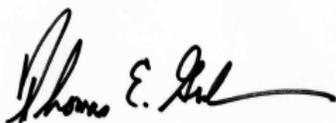
**NOTE:** FDA recognizes that six (6) Beta lactams are widely used in treating disease in lactating dairy cattle and are the most likely to cause a residue in milk if misused. These are penicillin, ceftiofur, cloxacillin, cephalosporin, amoxicillin, and ampicillin. While it is preferred that monitoring for Beta lactams include all of these drugs, at this time, the Agency is recommending that methods be utilized that have been shown to detect at least four (4) of the six (6) Beta lactams identified above.

Testing for drug residue(s) in compliance with the provisions of Sections 6 and 7 of the PMO may be accomplished by the use of any accepted Appendix N Test for raw milk or an accepted Section 6 Test for raw and pasteurized milk.

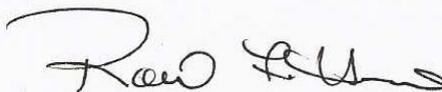
The NCIMS Executive Board has agreed that future updates to M-a-85 that add, delete or revise these Tests will not require a public comment period or follow the protocol established in the Procedures document for the issuance of M-a's.

Copies of this coded memorandum are enclosed for distribution to Regional Milk Specialists, State Milk Regulatory Agencies, State Laboratory Evaluation Officers and State Milk Sanitation Rating Officers in your region. This memorandum should be widely distributed to representatives of the dairy industry, State Veterinarians, State Veterinary and Pharmacy Boards, Veterinarian Professional Organizations and other interested parties and also will be available on the FDA Web site at [http:// www.fda.gov](http://www.fda.gov) at a later date.

If you would like an electronic version of this document prior to it being available on the FDA Web Site, please e-mail your request to [Robert.Hennes@fda.hhs.gov](mailto:Robert.Hennes@fda.hhs.gov).



Thomas Graham, PhD, Team Leader  
Laboratory Proficiency & Evaluation Team



Robert F. Hennes, RS, MPH  
CAPT U.S. Public Health Service  
Dairy and Egg Branch

**ATTACHMENT TO M-a-85 (REVISION #14)**

**MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS<sup>1</sup>**  
**Beta lactams**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>CLOXACILLIN</b>	<b>PENICILLIN</b>
TOLERANCE OR SAFE LEVEL	10 ppb	10 ppb	100 ppb <sup>2</sup>	20 ppb	10 ppb	5 ppb
<b>SCREENING TEST</b>						
BETASTAR <sup>®</sup> PLUS BETA LACTAM TEST	5.5	5.2	80	19.0	8.2	4.7
CHARM <sup>®</sup> <i>B. stearothermophilus</i> TABLET DISK ASSAY <sup>4, 5, 6</sup>	7.5	6.7	ND <sup>3</sup>	11.7	50 <sup>7</sup>	3.8
CHARM <sup>®</sup> II TABLET BETA LACTAM TEST (COMPETITIVE ASSAY) <sup>4</sup>	7.5	5.7	47	4.2	70 <sup>7</sup>	3.0
CHARM <sup>®</sup> II TABLET BETA LACTAM TEST (SEQUENTIAL ASSAY) <sup>6</sup>	8.1	6.6	58	4.1	50 <sup>7</sup>	3.4
CHARM <sup>®</sup> II TABLET BETA LACTAM TEST (QUANTITATIVE ASSAY) <sup>8</sup>	8.1	6.6	58	4.1	8.5	3.4
CHARM <sup>®</sup> II TEST FOR CLOXACILLIN IN MILK (COMPETITIVE ASSAY) <sup>4, 9</sup>	ND <sup>3</sup>	ND <sup>3</sup>	ND <sup>3</sup>	ND <sup>3</sup>	8.5	ND <sup>3</sup>
CHARM <sup>®</sup> SL BETA LACTAM TEST <sup>10, 11, 12</sup>	5.6	8.5	77	13.7	50 <sup>7</sup>	3.6
CHARM <sup>®</sup> SL6 BETA LACTAM TEST	7.1	9.6	72	18.7	8.3	4.2
CHARM <sup>®</sup> 3 SL3 BETA LACTAM TEST	8.4	8.0	79	20.0	8.6	3.8
CHARM <sup>®</sup> FLUSLBL FLUNIXIN AND BETA LACTAM TEST <sup>13</sup>	5.9	6.8	63	13.4	NA <sup>14</sup>	2.0
DELVOTEST P 5 PACK (READER) <sup>4, 15</sup>	4.6	4.0	ND <sup>3</sup>	8.2	NA <sup>14</sup>	2.1
DELVOTEST P 5 PACK (VISUAL) <sup>4, 5, 16</sup>	4.6	4.0	ND <sup>3</sup>	8.2	NA <sup>14</sup>	2.1
DELVOTEST P/DELVOTEST P MINI <sup>5, 6, 11</sup>	7.7	5.1	NA <sup>14</sup>	7.0	30 <sup>7</sup>	3.1
DELVOTEST SP/DELVOTEST SP MINI <sup>11</sup>	6.0	7.9	NA <sup>14</sup>	7.7	33 <sup>7</sup>	2.7
NEW SNAP <sup>®</sup> BETA LACTAM TEST KIT <sup>17</sup>	7.3	5.8	12	11.7	50 <sup>7</sup>	3.0

Continued on next page

**CONTINUED: MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS<sup>1</sup>**

**Beta lactams**

**FOOTNOTES:**

1. Parts per billion (ppb), which can be detected 90% of the time with 95% confidence. Additional drug level response data are provided for each Test in the following Tables and should be considered when selecting drug residue monitoring tests. The 90/95% concentrations (ppb) were determined by fitting a statistical model to the dose response data designed to estimate this value. The lower, one-sided 95% confidence limit was used. This data was either collected at an independent laboratory or the test samples were prepared at an independent laboratory.
2. The ceftiofur tolerance is based on measuring the sum of ceftiofur and desfuroylceftiofur related metabolites in milk as desfuroylceftiofur. The screening test detection concentrations for ceftiofur were evaluated using milk containing ceftiofur and desfuroylceftiofur related metabolites from treated animals. Due to the approval of "Spectramast", an intramammary ceftiofur product, the safe level of 50 ppb as parent ceftiofur is no longer used.
3. ND indicates "Not Detected" at or below tolerance.
4. This Test is acceptable for use to detect Beta lactam residues when used with bovine pasteurized whole and skim milk.
5. Refer to M-I-01-4, Issued July 2, 2001, for certification requirements to use this visual test.
6. This Test is acceptable for testing raw, commingled goat milk.
7. 90/95% concentrations were not determined for sensitivities significantly above the tolerance/safe level.
8. Test sensitivity when presumptive positive milk samples are verified in accordance with label directions using the Charm® II Tablet Beta Lactam Test (Sequential Assay) and the Charm® II Test for Cloxacillin in Milk (Competitive Assay).
9. For Appendix N bulk milk tanker screening, this Test must be used in combination with other approved screening methods in order to detect at least four (4) of the six (6) targeted Beta lactam drugs.
10. The Charm® SL Beta Lactam Test is acceptable for testing raw, commingled goat milk (M-I-03-3, Issued 2/25/2003).
11. The Charm® SL Beta Lactam Test, Delvotest P/Delvotest P Mini and Delvotest SP/Delvotest SP Mini are acceptable for testing raw, commingled water buffalo milk (M-I-09-6, Issued October 16, 2009).
12. The Charm® SL Beta Lactam Test is acceptable for testing raw, commingled sheep milk (M-I-09-7, Issued 11/3/2009).
13. The Charm® FLUSLBL Flunixin and Beta Lactam Test is a multi-class Test. The information listed here is only for the performance of the test kit in detecting Beta lactam drug residues. For information on flunixin, refer to MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS NSAIDs on page 15.
14. NA indicates "Data Not Available".
15. The DelvoScan Reader option for the Delvotest 5 P Pack has not been validated in fat-free chocolate, whole chocolate, half & half, heavy cream and pasteurized goat milk.
16. The Delvotest 5 P Pack (VISUAL) is acceptable to detect ampicillin, amoxicillin, cephalosporin and penicillin residues in bovine fat-free chocolate, whole chocolate, half & half, heavy cream and pasteurized goat milk.
17. The visual reading option is not available with the New Snap® Beta Lactam Test.

**BETASTAR® PLUS BETA LACTAM TEST  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEFTIOFUR	CEPHAPIRIN	CLOXACILLIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>10</b>	<b>5</b>
DRUG CONCENTRATION (ppb)						
1						0
2	0	0			0	0
3						0
4	0	0		0	0	0
5	27	10				100
6	100	100			0	
8	100	100		0	83	
10	100	100			100	
12				0		
15				63		
20			0	100		
40			0			
60			20			
80			90			
100			100			

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuoylcefotiofur related metabolites

**CHARM® *B. stearothermophilus* TABLET DISK ASSAY  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEPHAPIRIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)				
1				0
2	0	0	0	0
3				0
4	10	3	0	55
5				100
6	30	67		
8	90	100	0	
10	100	100		
14			100	
20			100	

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

**CHARM® II TABLET BETA LACTAM TEST (COMPETITIVE ASSAY)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)					
1					10
2	3	3		30	67
3					97
4	10	43		100	100
5			0		100
6	83	97			
8	100	100		100	
10	100	100	20		
14				100	
20			43	100	
40			100		
60			97		
80			100		
100			100		

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuoylcefthiofur related metabolites

**CHARM® II TABLET BETA LACTAM TEST (SEQUENTIAL ASSAY)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)					
1					0
2	0	0		3	10
3					80
4	20	10		100	100
5			0		100
6	23	83			
8	93	97		100	
10	100	100	0		
14				100	
20			3	100	
40			67		
60			97		
80			100		
100			100		

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuroylceftiofur related metabolites

**CHARM® II TABLET BETA LACTAM TEST (QUANTITATIVE ASSAY)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEFTIOFUR	CEPHAPIRIN	CLOXACILLIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>10</b>	<b>5</b>
DRUG CONCENTRATION (ppb)						
1						0
2	0	0		3	0	10
3						80
4	20	10		100	3	100
5			0			100
6	23	83			17	
8	93	97		100	87	
10	100	100	0		100	
14				100		
20			3	100		
40			67			
60			97			
80			100			
100			100			

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuroylcefthiofur related metabolites

**CHARM® II TEST FOR CLOXACILLIN IN MILK (COMPETITIVE ASSAY)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	CLOXACILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>
DRUG CONCENTRATION (ppb)	
2	0
4	3
6	17
8	87
10	100

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

**CHARM® SL BETA LACTAM TEST  
DRUG CONCENTRATION RESPONSE <sup>1,2</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)					
1					0
2	3	3			13
3					73
4	70	13		0	100
5			0		100
6	100	83			
8	100	100		50	
10	100	97 <sup>4</sup>	0		
12				97	
16				100	
20			0	100	
40			0		
60			23		
80			100		
100			100		

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuoylceftiofur related metabolites

<sup>4</sup>All statistical models used to calculate 90/95 allow for a single negative result at tolerance

**CHARM® SL6 BETA LACTAM TEST  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>CLOXACILLIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>10</b>	<b>5</b>
DRUG CONCENTRATION (ppb)						
1						7
2	0	0			0	23
3						93
4	7	3		0	40	100
5						100
6	57	17			83	
8	97	77		7	97	
10	100	100			100	
12				53		
16				100		
20			3	100		
40			37			
60			90			
80			97			
100			100			

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuoylcefiofur related metabolites

**CHARM® 3 SL3 BETA LACTAM TEST  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>CLOXACILLIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>10</b>	<b>5</b>
DRUG CONCENTRATION (ppb)						
1						0
2	0	0			0	0
3						13
4	0	0		0	0	97
5						100
6	3	23			13	
8	83	97		0	93	
10	100	100			100	
12				3		
16				83		
20			0	100		
40			0			
60			50			
80			100			
100			100			

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuoylceftiofur related metabolites

**CHARM® FLUSLBL FLUNIXIN AND BETA LACTAM TEST<sup>1</sup>  
DRUG CONCENTRATION RESPONSE<sup>2,3</sup>**

<b>DRUG</b>	<b>AMOXICILLIN</b>	<b>AMPICILLIN</b>	<b>CEFTIOFUR</b>	<b>CEPHAPIRIN</b>	<b>PENICILLIN</b>
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>4</sup></b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)					
1					0
2	0	0			20
3					97
4	13	10		0	100
5					100
6	90	43			
8	97	97		3	
10	100	100			
12				67	
16				97	
20			0	100	
40			37		
60			97		
80			100		
100			100		

<sup>1</sup>Beta-lactam data only. See separate listing under NSAIDs for flunixin drug concentration response on page 15.

<sup>2</sup> Percent positive

<sup>3</sup> Based on 30 samples at each concentration

<sup>4</sup> Total parent and desfuoylceftiofur related metabolites

**DELVOTEST P 5 PACK (VISUAL AND READER)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEPHAPIRIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)				
1				3
2	10	7	3	60
3				100
4	100	97	100	100
5				100
6	100	100		
8	100	100	100	
10	100	100		
14			100	
20			100	

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

**DELVOTEST P/DELVOTEST P MINI  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEPHAPIRIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)				
1				0
2	33	3	0	0
3				100
4	47	70	7	100
5				100
6	93	100		
8	97	100	100	
10	100	97 <sup>3</sup>		
14			100	
20			100	

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>All statistical models used to calculate 90/95 allow for a single negative result at tolerance

**DELVOTEST SP/DELVOTEST SP MINI  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEPHAPIRIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)				
1				7
2	0	0	3	47
3				100
4	80	10	17	100
5		33		100
6	100			
8	100	100	100	
10	97 <sup>3</sup>	100		
14			100	
20			100	

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>All statistical models used to calculate 90/95 allow for a negative result at tolerance

**NEW SNAP® BETA LACTAM TEST KIT  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	AMOXICILLIN	AMPICILLIN	CEFTIOFUR	CEPHAPIRIN	PENICILLIN
TOLERANCE/SAFE LEVEL (ppb)	<b>10</b>	<b>10</b>	<b>100<sup>3</sup></b>	<b>20</b>	<b>5</b>
DRUG CONCENTRATION (ppb)					
1					7
2	0	0		0	37
3					93
4	20	37		0	100
5			7		100
6	70	100			
8	100	100		0	
10	100	100	90		
12				100	
20			100	100	
40			100		
60			100		
80			100		
100			100		

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>Total parent and desfuroylceftiofur related metabolites

**MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS<sup>1</sup>  
NON-STEROIDAL ANTI-INFLAMMATORY DRUGS (NSAIDs)**

<b>DRUG</b>	<b>FLUNIXIN<sup>2</sup></b>
TOLERANCE/SAFE LEVEL (ppb)	2 ppb
<b>SCREENING TEST</b>	
CHARM® FLUSLBL FLUNIXIN AND BETA LACTAM TEST	1.9

<sup>1</sup>Parts per billion (ppb), which can be detected 90% of the time with 95% confidence. Additional drug level response data are provided for each Test in the following Table. The 90/95% concentrations (ppb) were determined by fitting a statistical model to the dose response data designed to estimate this value. The lower, one-sided 95% confidence limit was used. This data was either collected at an independent laboratory or the test samples were prepared at an independent laboratory.

<sup>2</sup>As 5-hydroxyflunixin, the major metabolic form of flunixin and the chemical marker of flunixin in milk.

**CHARM® FLUSLBL FLUNIXIN AND BETA LACTAM TEST<sup>1</sup>  
DRUG CONCENTRATION RESPONSE<sup>2,3</sup>**

<b>DRUG</b>	<b>FLUNIXIN<sup>4</sup></b>
TOLERANCE/SAFE LEVEL (ppb)	<b>2</b>
<b>DRUG CONCENTRATION (ppb)</b>	
0.4	30
0.8	70
1.0	
1.2	97
1.6	97
2.0	100

<sup>1</sup>Flunixin data only. See separate listing under Beta lactams for Beta lactam drug concentration response on page 12.

<sup>2</sup>Percent positive

<sup>3</sup>Based on 30 samples at each concentration

<sup>4</sup>As 5-hydroxyflunixin, the major metabolic form of flunixin and the chemical marker of flunixin in milk.

## MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS<sup>1</sup> SULFONAMIDES

DRUG	SULFADIMETHOXINE	SULFAMETHAZINE	SULFATHIAZOLE	SULFADIAZINE
TOLERANCE/SAFE LEVEL (ppb)	10 ppb	10 ppb	10 ppb	10 ppb
<b>SCREENING TEST</b>				
CHARM® II SULFA DRUG TEST (COMPETITIVE ASSAY)	4.0	9.4	7.3	4.9

<sup>1</sup>Parts per billion (ppb), which can be detected 90% of the time with 95% confidence. Additional drug level response data are provided for each test in the following Table. The 90/95% concentrations (ppb) were determined by fitting a statistical model to the dose response data designed to estimate this value. The lower, one-sided 95% confidence limit was used. This data was either collected at an independent laboratory or the test samples were prepared at an independent laboratory.

### CHARM® II SULFA DRUG TEST (COMPETITIVE ASSAY) DRUG CONCENTRATION RESPONSE<sup>1,2</sup>

DRUG	SULFADIMETHOXINE	SULFAMETHAZINE	SULFATHIAZOLE	SULFADIAZINE
TOLERANCE/SAFE LEVEL (ppb)	10	10	10	10
DRUG CONCENTRATION (ppb)				
2	97	7	0	40
4	100	80	57	100
6	100	97	100	100
8	100	100	100	100
10	100	100	100	97 <sup>3</sup>

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>All statistical models used to calculate 90/95 allow for a single negative result at tolerance

**MILK DRUG RESIDUE SCREENING TEST DETECTION CONCENTRATIONS<sup>1</sup>  
TETRACYCLINES**

DRUG	CHLORTETRACYCLINE	OXYTETRACYCLINE	TETRACYCLINE
TOLERANCE/SAFE LEVEL (ppb)	<b>300 ppb (Chlortetracycline + Tetracycline + Oxytetracycline)</b>		
DRUG CONCENTRATION (ppb)			
CHARM® II TETRACYCLINE DRUG TEST (COMPETITIVE ASSAY)	257	119	67

<sup>1</sup>Parts per billion (ppb), which can be detected 90% of the time with 95% confidence. Additional drug level response data are provided for each test in the following Table. The 90/95% concentrations (ppb) were determined by fitting a statistical model to the dose response data designed to estimate this value. The lower, one-sided 95% confidence limit was used. This data was either collected at an independent laboratory or the test samples were prepared at an independent laboratory.

**CHARM® II TETRACYCLINE DRUG TEST (COMPETITIVE ASSAY)  
DRUG CONCENTRATION RESPONSE<sup>1,2</sup>**

DRUG	Chlortetracycline	Oxytetracycline	Tetracycline
TOLERANCE/SAFE LEVEL (ppb)	<b>300 ppb (Chlortetracycline + Tetracycline + Oxytetracycline)</b>		
DRUG CONCENTRATION (ppb)			
20			0
30			7
40			37
60		13	93
70		37	
90	17		
100		87	
120	20		
150		100	
160	77		
230	93		
300	97 <sup>3</sup>	100	100

<sup>1</sup>Percent positive

<sup>2</sup>Based on 30 samples at each concentration

<sup>3</sup>All standard statistical models used to calculate 90/95 allow for a single negative result at tolerance



Pennsylvania Department of  
**AGRICULTURE**  
Bureau of Food Safety and Laboratory Services  
Laboratory Division

Date: September 19, 2005  
Subject: Change in Address/Location and Personnel.  
To: All Pennsylvania Approved Laboratories and Appendix N Testing Facilities.  
From: Michael F. Hydock, Chief  
Laboratory Division

**EFFECTIVE OCTOBER 1, 2005**

To Maintain certification, all Pennsylvania Approved Laboratories and Appendix N Testing Facilities **must notify the Laboratory Division in writing with in 5 days, of any changes made in the address/location and/or personnel.**

**Policy Purpose:**

- To maintain the correct information for on-site survey certification and Laboratory/Facility status changes for each Pennsylvania Approved Dairy Laboratory/Facility.
- To maintain a current list of certified laboratory analysts based on periodic review of the laboratory/facility personnel's status.
- To determine if a **major change in personnel** will likely affect the facility's Quality Assurance Program. If a major change in quality assurance records/training does occur, an INTERIM on-site evaluation by a certified Laboratory Evaluation Officer, may be justified to determine compliance.

Failure to comply with this policy will result in **decertification of the laboratory/facility and/or analysts** conducting testing of dairy products for regulatory compliance.

Responses should be mailed to the following address:

Commonwealth of Pennsylvania  
Department of Agriculture  
Bureau of Food Safety and Laboratory Services  
2301 North Cameron Street  
Harrisburg, Pa. 17110-9408  
Attention: Michael F. Hydock, Chief, Laboratory Division

If you have any questions call me at (717) 787-4315 Ext. 207

Cc: B. McLean  
J. Dell

# APPENDIX N REPORTING FORMS

<u>BFSLS#</u>	<u>NAME OF RECORD SHEET</u>	<u>REVISION DATE</u>
431	Notice of Milk Action Report	2/00
472	Emergency Laboratory Report - Drug Residue/Phosphatase	5/09
476	Bulk Milk Pick-Up Tanker Information	8/01
477	Appendix N Bulk Milk Tanker Positive Drug Residue Test Report	1/14
502	Producer Trace-Back for Positive Confirmed Loads Test Report	1/14

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
DIVISION OF MILK SANITATION

NOTICE OF MILK PRODUCER ACTION REPORT

Pennsylvania Department of Agriculture, Region \_\_\_\_\_  
Milk Sanitarian: \_\_\_\_\_  
Region Address: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**In accordance with Chapter 59.31. Milk Sanitation and Standards, you are hereby advised of the following producer action:**

Producer No. \_\_\_\_\_  
Herd No. \_\_\_\_\_  
Producer Name \_\_\_\_\_  
Address: \_\_\_\_\_  
\_\_\_\_\_

**Action Taken:**

Initial Instatement\* Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
Previous Handler \_\_\_\_\_  
  
Suspension\* Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
Reason \_\_\_\_\_  
Reinstatement Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
  
Handler Initiated Termination\*\* Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
  
Producer Initiated Termination\*\* Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
  
Reason \_\_\_\_\_

**\*Attach a copy of the Dairy Farm Sanitation Report.**

**\*\*Attach a copy of the Dairy Farm Sanitation Report and include a copy of the producer record. Mail or deliver to the appropriate Region Office within twenty-four (24) hours of this action.**

Permit Holder \_\_\_\_\_ FIPS# 42 - \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Signature \_\_\_\_\_ Date \_\_\_\_/\_\_\_\_/\_\_\_\_  
Approved Inspector or Authorized Agent



**PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
DIVISION OF MILK SANITATION  
2301 NORTH CAMERON STREET, PA 17110-9408  
FAX 717-787-1873**

**EMERGENCY LABORATORY REPORT**  
(Drug Residue / Phosphatase)

Please use this form to report **all positive official monthly test results**, which require an immediate report to the Department of Agriculture, and then send this report to the address shown above. Phone notification of positive drug residues during off hours, holidays, and weekends may be made to **717-787-4315** (via voicemail system). If this positive test result involves a producer who also is a raw or pasteurized jugger, please include this information with your phone call report.

**Positive phosphatase results or drug residue results in finished products will now require phone call notification to the Harrisburg office at 717-787-4315.**

**In accordance the Section 59.309, Milk Sanitation Standards, you are hereby advised of the following positive result:**

<u>Drug Residue</u>	<input type="checkbox"/>	Test Kit Used _____	Lot # _____
<u>Phosphatase</u>	<input type="checkbox"/>	Fluorophos _____	Charm _____
<u>Pathogens</u>	<input type="checkbox"/>	Confirmed Type _____	

**REPORTING INFORMATION**

Producer Name / Address \_\_\_\_\_  
 or \_\_\_\_\_  
 Finished Product ID Code \_\_\_\_\_

Producer No. & Herd No. / Sell By Code \_\_\_\_\_

Date Sampled \_\_\_\_\_ Temperature Control \_\_\_\_\_  
 Date and Time of Analysis \_\_\_\_\_ Temperature Control \_\_\_\_\_

**RESULTS FOUND**

Initial Result (Values / Interpretation) \_\_\_\_\_  
 Confirmatory Test(s) Used \_\_\_\_\_  
 Confirmation Results (Values / Interpretation) \_\_\_\_\_

**RECORD KEEPING INFORMATION**

Date/Time PDA was notified by phone \_\_\_\_\_ Date Report was mailed \_\_\_\_\_

Approved Inspector \_\_\_\_\_

Permit Holder \_\_\_\_\_ BTU No. \_\_\_\_\_

LABORATORY \_\_\_\_\_

SIGNATURE \_\_\_\_\_  
 Laboratory Director

**This report must be mailed (Received in Harrisburg) within 48 hours from initial notification**

**PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY AND LABORATORY SERVICES  
DIVISION OF MILK SANITATION  
2301 NORTH CAMERON STREET, PA 17110-9408  
FAX 717-787-1873**

**EMERGENCY LABORATORY REPORT**  
(Drug Residue / Phosphatase)

Please use this form to report **all positive official monthly test results**, which require an immediate report to the Department of Agriculture, and then send this report to the address shown above. Phone notification of positive drug residues during off hours, holidays, and weekends may be made to **717-787-4315** (via voicemail system). If this positive test result involves a producer who also is a raw or pasteurized jugger, please include this information with your phone call report.

**Positive phosphatase results or drug residue results in finished products will now require phone call notification to the Harrisburg office at 717-787-4315.**

**In accordance the Section 59.309, Milk Sanitation Standards, you are hereby advised of the following positive result:**

Drug Residue  Test Kit Used Delvo 5-pak Lot # 07I23/I  
Phosphatase  Fluorophos \_\_\_\_\_ Charm \_\_\_\_\_  
Pathogens  Confirmed Type \_\_\_\_\_

**REPORTING INFORMATION**

Producer Name / Address Bob Nokandu  
 or RR 1 Box 168  
 Finished Product ID Code Ronks, PA

Producer No. & Herd No. / Sell By Code 47-10002693  
 Date Sampled 11/8/13 Temperature Control 2.9C  
 Date and Time of Analysis 11/8/13 12:30PM Temperature Control 3.1C

**RESULTS FOUND**

Initial Result (Values / Interpretation) Purple - Pos  
 Confirmatory Test(s) Used Delvo 5-pak  
 Confirmation Results (Values / Interpretation) Purple-Pos

**RECORD KEEPING INFORMATION**

Date/Time PDA was notified by phone 11/8/13 2:00PM Date Report was mailed 11/8/13  
 Approved Inspector Z. Kennedy  
 Permit Holder Utter's Dairy BTU No. 42-995  
 LABORATORY Quality Laboratory  
 SIGNATURE Michael Peters

Laboratory Director

**This report must be mailed (Received in Harrisburg) within 48 hours from initial notification**

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY & LABORATORY SERVICES  
DIVISION OF MILK SANITATION  
2301 N. CAMERON STREET  
HARRISBURG, PA 17110-9408

In accordance with the provisions of The Pennsylvania Drug Residue Testing Program, I am submitting the following information regarding positive drug residue tests involving a producer under my supervision.

Bulk Milk Pick-up Tanker Information

Tanker License Plate Number: \_\_\_\_\_ Date Report Mailed: \_\_\_\_\_

Presumptive Test Used /Date      Screen Test Used /Date      Producer Trace Back Test /Date

Presumptive Test Location      Screen Test Location      Producer Trace Back Location

Presumptive Test Result (Duplicate)      Screen Test Result (Duplicate)      Producer Trace Back Result (Triplicate)

Disposition of \_\_\_\_\_

Adulterated Tanker: \_\_\_\_\_

Date and Location: \_\_\_\_\_

\*\*\*\*\*

Violative Producer Information

PA Producer Name and Number: \_\_\_\_\_

Herd Number: \_\_\_\_\_

Address: \_\_\_\_\_

Out-of-State Producer ID No.: \_\_\_\_\_

Cause of Adulterated Bulk Tank: \_\_\_\_\_

Drug Used: \_\_\_\_\_

**THIS REPORT MUST BE MAILED WITHIN 72 HOURS OF INITIAL PRESUMPTIVE POSITIVE TEST RESULT.**

PERMIT HOLDER: \_\_\_\_\_

Name FIPS No.

Street

City State Zip

Signature: \_\_\_\_\_

Approved Inspector or Authorized Agent

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
BUREAU OF FOOD SAFETY & LABORATORY SERVICES  
DIVISION OF MILK SANITATION  
2301 N. CAMERON STREET  
HARRISBURG, PA 17110-9408

In accordance with the provisions of The Pennsylvania Drug Residue Testing Program, I am submitting the following information regarding positive drug residue tests involving a producer under my supervision.

Bulk Milk Pick-up Tanker Information

Tanker License Plate Number: YR-0935 Date Report Mailed: 1-7-08  
IDEXX SNAP      /1-5-08 Charm SL      /1-5-08 Charm SL      /1-5-08  
Presumptive Test Used /Date Screen Test Used /Date Producer Trace Back Test /Date  
Mountainside Dairy      Utters Dairy      Utters Dairy       
Presumptive Test Location Screen Test Location Producer Trace Back Location  
2.31, 2.43      +2351, +2153      +2147, +2044, +2189       
Presumptive Test Result Screen Test Result Producer Trace Back Result  
(Duplicate) (Duplicate) (Triplicate)  
Disposition of      Dumped at Lee Oswald Manure pit       
Adulterated Tanker:       
Date and Location: 1-6-08 Rockville PA

\*\*\*\*\*

Violative Producer Information

PA Producer Name and Number: 19832 Joe Somebody  
Herd Number: 10-26-H54  
Address: RD 1 Box 94  
Spring Creek, PA 19823  
Out-of-State Producer ID No.:       
Cause of Adulterated Bulk Tank: Milked treated cow  
      
      
Drug Used: Tomorrow

**THIS REPORT MUST BE MAILED WITHIN 72 HOURS OF INITIAL PRESUMPTIVE POSITIVE TEST RESULT.**

PERMIT HOLDER: Utters Dairy 42-999  
    Name FIPS No.  
RD 3 Box 147  
    Street  
Rockville PA 19745  
    City State Zip  
Signature: A. DeMann  
      
Approved Inspector or Authorized Agent

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY & LABORATORY SERVICES  
 LABORATORY DIVISION  
 2301 N. CAMERON STREET  
 HARRISBURG, PA 17110-9408  
 Office (717) 787-4315 Fax (717) 787-1873

**APPENDIX N BULK MILK TANKER *POSITIVE* DRUG RESIDUE TEST REPORT**

<b>Receiving Location</b> _____	<b>Collection of Sample</b> Date ___/___/___ Time ___:___ am/pm Temp. . ___°F	<b>Owner of Milk</b> _____  FIPS # _____	<b>Route #</b> _____  <b>Load #</b> _____
<b>Milk Hauler</b> _____	<b>Rejection Information</b> Positive compartment: Single _____ Front _____ Rear _____	<b>Weight of Load</b> _____	<b>Tanker License Plate # / State</b> _____

**INITIAL TEST RESULT**

<b>Date /Time</b> ___/___/___  ___:___ am/pm	<b>Test Method Used</b> _____	<b>Test Kit Lot #</b> _____  <b>Expiration Date</b> _____	<b>Initial Result</b> (number / interpretation) FRONT _____ / _____  REAR _____ / _____	<b>Analyst I.D./</b> <b>Initials</b> _____
---	----------------------------------	---	---	--

**PRESUMPTIVE TEST RESULT\*\***

<b>Temperature</b> _____°C	<b>Test Method Used</b> _____	<b>Test Kit Lot #</b> _____  <b>Expiration Date</b> _____	<b>Presumptive Result</b> <b>DUPLICATE</b> (number / interpretation) _____/_____ _____/_____	<b>Analyst I.D./</b> <b>Initials</b> _____
-------------------------------	----------------------------------	---	--	--

<b>Printout:</b> (enclosed)  Yes <input type="checkbox"/>  No <input type="checkbox"/>	<b>Control Results</b>  Positive _____  Negative _____	<b>Charm II Control Point Results</b> Control Point _____ Date Established _____  Positive _____ Negative _____ (Average) + _____ -- _____	<b>Department Notification:</b> Phone ___ Fax ___ Email ___ Date ___/___/___ Time ___:___ am/pm Reported By: _____ Who contacted _____
---	--	---	---

<b>Disposition of Load</b> (secure <u>initial</u> test sample, secure tanker, attach weight slip) Seal numbers: _____ Sent to: _____  Dumped / Diverted Where? _____  Analyst _____ Supervisor _____ Date _____	<b>Received</b> <input type="checkbox"/>  <b>Condemned</b> <input type="checkbox"/>  <b>Rejected</b> <input type="checkbox"/>
--	---

**Comments:**  
\_\_\_\_\_  
\_\_\_\_\_

**SCREENING TEST (CONFIRMATION) RESULTS**

<b>Date / Time</b> <b>Tested</b> ___/___/___  ___:___ am/pm	<b>Test Method Used</b> _____	<b>Test Kit Lot #</b> _____  <b>Expiration Date</b> _____	<b>Confirmation Results</b> <b>DUPLICATE</b> (number / interpretation) _____/_____  _____/_____	<b>Analyst</b> <b>I.D./Initials</b> _____
---	----------------------------------	---	--	---

<b>Confirmatory</b> <b>Location</b> _____ _____	<b>Control Results</b>  Positive _____  Negative _____	<b>Charm II Control Point Results</b> Control Point _____ Date Established _____  Positive _____ Negative _____ (Average) + _____ -- _____	<b>Department Notification:</b> Phone ___ Fax ___ Email ___ Date ___/___/___ Time ___:___ am/pm Reported By: _____ Who contacted _____
--	--	---	---

<b>Disposition of Load</b> (secure <u>initial</u> test sample, secure tanker, attach weight slip) Seal numbers: _____ Sent to: _____  Dumped / Diverted Where? _____	<b>Received</b> <input type="checkbox"/>  <b>Condemned</b> <input type="checkbox"/>
---	---

**CERTIFIED ANALYST/SUPERVISOR** \_\_\_\_\_ **DATE** \_\_\_\_\_

**\*\*SCREENING FACILITIES - A COPY OF THIS REPORT MUST ACCOMPANY THE TRUCK AND PRODUCER SAMPLES TO THE CONFIRMATION LOCATION, BE KEPT ON FILE AT THE SCREENING LOCATION, AND ALSO BE SENT TO THE PENNSYLVANIA DEPARTMENT OF AGRICULTURE WITHIN 72 HOURS OF INITIAL TESTING.**

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY & LABORATORY SERVICES  
 LABORATORY DIVISION  
 2301 N. CAMERON STREET  
 HARRISBURG, PA 17110-9408  
 Office (717) 787-4315 Fax (717) 787-1873

**APPENDIX N BULK MILK TANKER POSITIVE DRUG RESIDUE TEST REPORT**

Receiving Location <u>Brown Cow Dairy</u>	Collection of Sample Date <u>2</u> / <u>4</u> / <u>14</u> Time <u>9</u> : <u>45</u> <u>am</u> /pm Temp. <u>38</u> °F	Owner of Milk <u>Utter's Dairy</u> FIPS # <u>42-995</u>	Route # <u>18</u> Load # <u>168123</u>
Milk Hauler <u>My-T-Trucks</u>	Rejection Information Positive compartment: Single _____ Front <u>X</u> Rear _____	Weight of Load <u>52,269</u>	Tanker License Plate # / State <u>PT-3698F</u>

**INITIAL TEST RESULT**

Date / Time <u>2</u> / <u>4</u> / <u>14</u> <u>9</u> : <u>55</u> <u>am</u> /pm	Test Method Used <u>IDEXX Snap</u>	Test Kit Lot # <u>KD159</u> Expiration Date <u>4/2/14</u>	Initial Result (number / interpretation) FRONT <u>6.58</u> / <u>POS</u> REAR <u>0.75</u> / <u>NF</u>	Analyst I.D./ Initials <u>JT</u>
--	---------------------------------------	--	---	-------------------------------------

**PRESUMPTIVE TEST RESULT\*\***

Temperature <u>3.2</u> °C	Test Method Used <u>IDEXX Snap</u>	Test Kit Lot # <u>KD159</u> Expiration Date <u>4/2/14</u>	Presumptive Result <b>DUPLICATE</b> (number / interpretation) <u>5.95</u> / <u>POS</u> <u>6.12</u> / <u>POS</u>	Analyst I.D./ Initials <u>JT</u>
------------------------------	---------------------------------------	--	---	-------------------------------------

Printout: (enclosed) Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>	Control Results Positive <u>3.59</u> Negative <u>0.72</u>	Charm II Control Point Results Control Point _____ Date Established _____ Positive _____ Negative _____ (Average) + _____ -- _____	Department Notification: Phone _____ Fax _____ Email <u>X</u> Date <u>2</u> / <u>4</u> / <u>14</u> Time <u>10</u> : <u>30</u> <u>am</u> /pm Reported By: <u>JT</u> Who contacted <u>M. Hydock</u>
---	---	--	--

Disposition of Load (secure initial test sample, secure tanker, attach weight slip) Seal numbers: <u>0134, 1121, 1139</u> Sent to: <u>Utter's Dairy for confirmation</u> Dumped / Diverted Where? _____ Analyst <u>J. Thompson</u> Supervisor <u>F. James</u> Date <u>2/4/14</u>	Received <input type="checkbox"/> Condemned <input type="checkbox"/> Rejected <input checked="" type="checkbox"/>
---	---

Comments:  
\_\_\_\_\_  
\_\_\_\_\_

**SCREENING TEST (CONFIRMATION) RESULTS**

Date / Time Tested <u>2</u> / <u>4</u> / _____ <u>1</u> : <u>45</u> <u>am</u> /pm Temp. Control _____ °C	Test Method Used <u>Charm SL</u>	Test Kit Lot # <u>109</u> Expiration Date <u>5/2014</u>	Confirmation Results <b>DUPLICATE</b> (number / interpretation) <u>+2689</u> / <u>POS</u> <u>+2548</u> / <u>POS</u>	Analyst I.D./Initials <u>S. M</u>
---	-------------------------------------	--	---	--------------------------------------

Confirmatory Location <u>Utter's Dairy</u>	Control Results Positive <u>+1659</u> Negative <u>-1452</u>	Charm II Control Point Results Control Point _____ Date Established _____ Positive _____ Negative _____ (Average) + _____ -- _____	Department Notification: Phone _____ Fax <u>X</u> Email _____ Date <u>2</u> / <u>4</u> / <u>14</u> Time <u>3</u> : <u>00</u> <u>am</u> /pm Reported By: <u>J. W</u> Who contacted <u>M. Hydock</u>
---	---	--	---

Disposition of Load (secure initial test sample, secure tanker, attach weight slip) Seal numbers: <u>899,1574</u> Sent to: <u>A. Stoltzfus manure pit</u> Dumped / Diverted Where? <u>Ronks, PA</u>	Received <input type="checkbox"/> Condemned <input checked="" type="checkbox"/>
---	--

CERTIFIED ANALYST/SUPERVISOR Sam Marshal / James Williams DATE 2/4/14

\*\*SCREENING FACILITIES - A COPY OF THIS REPORT MUST ACCOMPANY THE TRUCK AND PRODUCER SAMPLES TO THE CONFIRMATION LOCATION, BE KEPT ON FILE AT THE SCREENING LOCATION, AND ALSO BE SENT TO THE PENNSYLVANIA DEPARTMENT OF AGRICULTURE WITHIN 72 HOURS OF INITIAL TESTING.

**PENNSYLVANIA DEPARTMENT OF AGRICULTURE**  
**BUREAU OF FOOD SAFETY & LABORATORY SERVICES**  
**LABORATORY DIVISION**  
**2301 N. CAMERON STREET**  
**HARRISBURG, PA 17110-9408**  
**Office (717) 787-4315      Fax (717) 787-1873**

**PRODUCER TRACE-BACK FOR POSITIVE CONFIRMED LOADS**  
**(DRUG RESIDUE) TEST REPORT**

<b>Confirmatory Location</b>  _____	<b>Collection of Sample</b> Date ___/___/___ Time ___:___am/pm Temp. _____°F	<b>Owner of Milk</b>  _____  FIPS # _____	<b>Route #</b> _____ <b>Load #</b> _____
<b>Laboratory ID #</b>  _____  <b>Printout (enclosed):</b> Yes <input type="checkbox"/> No <input type="checkbox"/>	<b>Test Method(s) Used</b>  _____  _____	<b>Test Kit Lot #</b>  _____  <b>Expiration Date</b>  _____	<b>Department Notification:</b>  Phone __ Fax __ Email __ Date ___/___/___ Time ___:___am/pm Reported By: _____ Who contacted _____

**Comments:**

Samples Received:    Date: \_\_\_/\_\_\_/\_\_\_    Time: \_\_\_:\_\_\_am/pm    Temp. : \_\_\_\_\_°C.    Analyst Initials \_\_\_\_\_

Samples Tested:      Date: \_\_\_/\_\_\_/\_\_\_    Time: \_\_\_:\_\_\_am/pm    Temp. : \_\_\_\_\_°C.    Analyst Initials \_\_\_\_\_

**PRODUCER TRACE-BACK INFORMATION TEST RESULTS**

Sample #	FIPS #	Producer #	Result (#)	Interpretation (POS or NF)	Control Results
					<b>Positive Control</b> _____
					<b>Negative Control</b> _____
					<b><u>Charm II Control Point Results</u></b>
					Control Point _____
					Date Established _____
					Positive _____ Negative _____
					(Average) + _____ -- _____
					<b>Producer Confirmation</b>
					<b>Positive Producer(s)</b>
					<b><u>DUPLICATE RESULTS</u></b> (number / interpretation)
					_____ / _____
					_____ / _____
					<b>Positive Control</b> _____
					<b>Negative Control</b> _____

**CERTIFIED ANALYST / SUPERVISOR** \_\_\_\_\_ **DATE** \_\_\_\_\_

**\*\*A COPY OF BFSLS-477 MUST ACCOMPANY THIS REPORT AND BE SENT WITHIN 48 HOURS OF TRACE-BACK RESULTS. A COPY MUST BE KEPT ON FILE AT THE CONFIRMATORY LOCATION.**

PENNSYLVANIA DEPARTMENT OF AGRICULTURE  
 BUREAU OF FOOD SAFETY & LABORATORY SERVICES  
 LABORATORY DIVISION  
 2301 N. CAMERON STREET  
 HARRISBURG, PA 17110-9408  
 Office (717) 787-4315 Fax (717) 787-1873

**PRODUCER TRACE-BACK FOR POSITIVE CONFIRMED LOADS**  
**(DRUG RESIDUE) TEST REPORT**

<b>Confirmatory Location</b> <u>Utter's Dairy</u>		<b>Collection of Sample</b> Date <u>2 / 4 / 14</u> Time <u>9 : 45</u> am/pm Temp. <u>2.6</u> °C		<b>Owner of Milk</b> <u>Utter's Dairy</u> FIPS # <u>42-995</u>		<b>Route #</b> <u>18</u> <b>Load #</b> <u>168123</u>	
<b>Laboratory ID #</b> <u>42-399</u>		<b>Test Method(s) Used</b> <u>Charm SL</u>		<b>Test Kit Lot #</b> <u>109</u>		<b>Department Notification:</b> Phone ___ Fax <input checked="" type="checkbox"/> Email ___ Date <u>2 / 4 / 14</u> Time <u>3 : 00</u> am/pm Reported By: <u>J. W</u> Who contacted <u>M. Hydock</u>	
<b>Printout (enclosed):</b> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>				<b>Expiration Date</b> <u>5/2014</u>			

**Comments:**

Samples Received: Date: 2 / 4 / 14 Time: 1 : 30 am/pm Temp. : 2.5 °C. Analyst Initials SM  
 Samples Tested: Date: 2 / 4 / 14 Time: 2 : 00 am/pm Temp. : 2.3 °C. Analyst Initials SM

**PRODUCER TRACE-BACK INFORMATION TEST RESULTS**

Sample #	FIPS #	Producer #	Result (#)	Interpretation (POS or NF)	Control Results
1	42-995	26995	-1459	NF	Positive Control <u>+1699</u>
2	42-995	26845	-1589	NF	Negative Control <u>-1544</u>
3	42-995	26541	+4239	POS	
4	42-995	26854	-1259	NF	
5	42-995	56771	-2095	NF	<b>Charm II Control Point Results</b> Control Point _____ Date Established _____ Positive _____ Negative _____ (Average) + _____ -- _____
					<b>Producer Confirmation</b>
					<b>Positive Producer(s)</b>
					<b>DUPLICATE RESULTS</b> (number / interpretation) <u>+4369</u> / <u>POS</u> <u>+4254</u> / <u>POS</u>
					Positive Control <u>+1854</u>
					Negative Control <u>-1584</u>

CERTIFIED ANALYST / SUPERVISOR Sam Marshal / James Williams DATE 2/4/14

\*\*A COPY OF BFSL-477 MUST ACCOMPANY THIS REPORT AND BE SENT WITHIN 48 HOURS OF TRACE-BACK RESULTS. A COPY MUST BE KEPT ON FILE AT THE CONFIRMATORY LOCATION.

**GRADE “A” PASTEURIZED**

**MILK ORDINANCE (PMO)**

**– APPENDIX N**

## APPENDIX N. DRUG RESIDUE TESTING AND FARM SURVEILLANCE

### I. INDUSTRY RESPONSIBILITIES

#### MONITORING AND SURVEILLANCE:

Industry shall screen all bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers, regardless of final use, for Beta lactam drug residues. Additionally, other drug residues shall be screened for by employing a random sampling program on bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers when the Commissioner of the FDA determines that a potential problem exists as cited in Section 6 of this *Ordinance*. The random bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers sampling program shall represent and include, during any consecutive six (6) months, at least four (4) samples collected in at least four (4) separate months, except when three (3) months show a month containing two (2) sampling dates separated by at least twenty (20) days. Samples collected under this random sampling program shall be analyzed as specified by FDA. (Refer to Section 6 of this *Ordinance*.)

The bulk milk pickup tanker shall be sampled after the last producer has been picked up and before any additional commingling. These bulk milk pickup tanker samples may be collected using an approved aseptic sampler. The sample shall be representative. Bulk milk pickup tanker testing shall be completed prior to processing the milk. Bulk milk pickup tanker samples confirmed positive for drug residues shall be retained as determined necessary by the Regulatory Agency.

All raw milk supplies that have not been transported in bulk milk pickup tankers shall be sampled prior to processing the milk. The sample(s) shall be representative of each farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. Testing of all raw milk supplies that have not been transported in bulk milk pickup tankers shall be completed prior to processing the milk.

**NOTE:** On-farm producer/processors that plan to store or ship their raw sheep milk frozen, shall sample their raw sheep milk prior to freezing. The sample shall be obtained by a bulk milk hauler/sampler permitted by the Regulatory Agency where the dairy farm is located. The raw sheep milk sample shall then be tested in a certified laboratory or screening facility. If this is the on-farm producer/processor's only raw sheep milk supply, this testing would suffice for the required Appendix N testing for all raw milk supplies that have not been transported in bulk milk pickup tankers, which are required to be completed prior to processing the milk. In the case of sheep milk dairy farms, the raw milk sample may be frozen in accordance with a sample protocol approved by the Regulatory Agency in which the dairy farm is located as specified in Appendix B and transported to a certified laboratory for testing. The test results, or raw milk samples, shall clearly distinguish the lot number of the frozen raw sheep milk and accompany the frozen raw sheep milk to the plant.

All presumptive positive test results for drug residues from analysis conducted on commingled raw milk tanks, bulk milk pickup tankers and/or all raw milk supplies that have not been

transported in bulk milk pickup tankers, farm raw milk tanks/silos (only milk offered for sale) or finished milk or milk product samples shall be reported to the Regulatory Agency in which the testing was conducted. Bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers samples confirmed positive for drug residues shall be retained or disposed of as determined by the Regulatory Agency.

Industry plant samplers shall be evaluated according to the requirements specified in Section 6. THE EXAMINATION OF MILK AND MILK PRODUCTS and at the frequency addressed in Section 5. INSPECTION OF DAIRY FARMS AND MILK PLANTS of this *Ordinance*.

### **REPORTING AND FARM TRACE BACK:**

When a bulk milk pickup tanker and/or a raw milk supply that has not been transported in bulk milk pickup tankers is found to be presumptive positive for drug residues, the Regulatory Agency in which the testing was conducted, shall be immediately notified of the results and the ultimate disposition of the raw milk.

The producer samples from the bulk milk pickup tanker, found to be positive for drug residues, shall be individually tested to determine the farm of origin. The samples shall be tested as directed by the Regulatory Agency.

When a farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc., is (are) used for a milk plant's raw milk supply(ies) that has (have) not been transported in bulk milk pickup tankers, is (are) found to be positive (confirmed) for drug residues, the farm of origin of the drug residue has consequently already been determined and further testing is not required to determine the farm of origin.

Further pickups or use of the violative individual producer's milk shall be immediately discontinued, until such time, that subsequent tests are no longer positive for drug residues.

### **RECORD REQUIREMENTS:**

Results of all testing may be recorded in any format acceptable to the Regulatory Agency that includes at least the following information:

1. Identity of the person doing the test;
2. Identity of the bulk milk pickup tanker or farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. used for the storage of all raw milk supplies that have not been transported in bulk milk pickup tankers being tested\*;
3. Date/time the test was performed (Time, Day, Month and Year);
4. Identity of the test performed/lot #/any and all controls (+/-);
5. Results of the test;
6. Follow-up testing if the initial test was positive/any and all controls (+/-);
7. Site where test was performed, and
8. Prior test documentation shall be provided for a presumptive positive load.

\*Include the BTU number(s) of the dairy farms present on the bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers with the above information.

Records of all sample results shall be maintained for a minimum of six (6) months by the industry at the location where the tests were run, and/or another location as directed by the Regulatory Agency.

## **II. REGULATORY AGENCY RESPONSIBILITIES**

Upon receipt of notification from industry of a bulk milk pickup tanker and/or a raw milk supply that has not been transported in bulk milk pickup tankers, which contains milk from another Regulatory Agency's jurisdiction, is found to be presumptive positive for drug residues it is the responsibility of the receiving Regulatory Agency to notify the Regulatory Agency(ies) from which the milk originated.

### **MONITORING AND SURVEILLANCE:**

Regulatory Agencies shall monitor industry surveillance activities during either routine or unannounced, on-site quarterly inspections to collect samples from bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers and to review industry records of their sampling program. Samples should be collected and analyzed from at least ten percent (10%) of the bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers scheduled to arrive on the day of the inspection. The method used shall be appropriate for the drug being analyzed and shall be capable of detecting the same drugs at the same concentrations as the method being used by industry. Alternately, the Regulatory Agency or Laboratory Evaluation Officer (LEO) may take known samples with them on the audit visit and observe the industry analyst test the samples. Receiving locations that choose to certify all receiving analysts, certified under the provisions of the NCIMS Laboratory Certification Program, are exempt from the sample collection requirements of this Section. Receiving locations where all approved receiving Industry Analysts and Industry Supervisors successfully participate in a biennial on-site evaluation and annual spilt sample comparisons by LEOs are also exempt from the sample collection requirements of this Section.

A review shall include, but not be limited to, the following:

1. Is the program an appropriate routine monitoring program for the detection of drug residues?
2. Is the program utilizing appropriate test methods?
3. Is each producer's milk represented in a testing program for drug residues and tested at the frequency prescribed in Section I. INDUSTRY RESPONSIBILITIES of this Appendix for drug residues?
4. Is the program assuring timely notification to the appropriate Regulatory Agency of positive results, the ultimate disposition of the bulk milk pickup tanker and/or a raw milk supply that has not been transported in bulk milk pickup tankers and of the trace back to the farm of origin?
5. Is the dairy farm pickup and/or use of the violative individual producer's milk suspended until subsequent testing establishes the milk is no longer positive for drug residues?

To satisfy these requirements:

- a. There should be an agreement between the Regulatory Agency and industry that specifies how this notification is to take place. This notification shall be “timely” for example by telephone or fax, and supported in writing.
- b. The ultimate disposition should either be prearranged in an agreement between the Regulatory Agency and the industry, or physically supervised by the Regulatory Agency. The milk should be disposed of in accordance with provisions of M-I-06-5 or an FDA and Regulatory Agency reviewed and accepted Beta lactam milk diversion protocol for use as animal feed.
- c. All screening test positive (confirmed) loads shall be broken down (producer trace back) using the same or an equivalent test method (M-I-96-10, latest revision). Confirmation tests (load and producer trace back/permit action) shall be performed by an Official Laboratory, Officially Designated Laboratory or Certified Industry Supervisor. Positive producers shall be handled in accordance with this Appendix.
- d. When a farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. is (are) used for a milk plant’s raw milk supply(ies) that has (have) not been transported in bulk milk pickup tankers, is (are) found to be positive (confirmed) for drug residues, the farm of origin of the drug residue has consequently already been determined and further testing is not required to determine the farm of origin. Confirmation tests shall be performed by an Official Laboratory, Officially Designated Laboratory or Certified Industry Supervisor. Positive producers shall be handled in accordance with this Appendix.
- e. The suspension and discontinuance of farm bulk milk tank pick up and/or the use of raw milk supplies that have not been transported in bulk milk pickup tankers is the responsibility of the industry; under the direction and supervision of the Regulatory Agency. At the discretion of the Regulatory Agency, records should be maintained by industry and/or the Regulatory Agency that:
  - 1) Establish the identity of the producer for raw milk supplies that have not been transported in bulk milk pickup tankers that tested positive or the producer and the identity of the load that tested positive; and
  - 2) Establish that milk is not picked up or used from the drug residue positive producer until the Regulatory Agency has fulfilled their obligations under Section II. ENFORCEMENT of this Appendix and has cleared the milk for pick up and/or use.

Sufficient records should be reviewed to assure that all bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers are sampled before additional commingling at the milk receiving facility and the results were made available to the appropriate BTU(s).

The Regulatory Agency shall also perform routine sampling and testing for drug residues determined to be necessary as outlined in Section 6 of this *Ordinance*.

#### **ENFORCEMENT:**

If testing reveals milk positive for drug residues, the milk shall be disposed of in a manner that removes it from the human or animal food chain, except where acceptably reconditioned under

FDA Compliance Policy Guide (CPG 7126.20). The Regulatory Agency shall determine the producer(s) responsible for the violation.

**Suspension:** Any time milk is found to test as a confirmed positive for a drug residue, the Regulatory Agency shall immediately suspend the producer's Grade "A" permit or equally effective measures shall be taken to prevent the sale of milk containing drug residues.

**Penalties:** Future pickups and/or use of the violative individual producer's milk are prohibited until subsequent testing reveals the milk is free of drug residue. The penalty shall be for the value of all milk on the contaminated load and/or raw milk supply that has not been transported in bulk milk pickup tankers plus any costs associated with the disposition of the contaminated load or raw milk supply that has not been transported in bulk milk pickup tankers. The Regulatory Agency may accept certification from the violative producer's milk marketing cooperative or purchaser of milk as satisfying the penalty requirements.

**Reinstatement:** The Grade "A" producer's permit may be reinstated, or other action taken, to allow the sale of milk for human food, when a representative sample taken from the producer's milk, prior to commingling with any other milk, is no longer positive for drug residue.

**Follow-Up:** Whenever a drug residue test is positive, an investigation shall be made to determine the cause. The farm inspection is completed by the Regulatory Agency or its agent to determine the cause of the residue and actions taken to prevent future violations including:

1. On-farm changes in procedures necessary to prevent future occurrences as recommended by the Regulatory Agency.
2. Discussion and education on the Drug Residue Avoidance Control measures outlined in Appendix C. of this *Ordinance*.

**Permit Revocation:** After a third violation in a twelve (12) month period, the Regulatory Agency shall initiate administrative procedures pursuant to the revocation of the producer's Grade "A" permit under the authority of Section 3. Permits of this *Ordinance*, due to repeated violations.

### **REGULATORY AGENCY RECORDS:**

In regards to the industry reporting a positive tanker and/or a raw milk supply that has not been transported in bulk milk pickup tankers result, the Regulatory Agency's records shall indicate the following:

1. What were the Regulatory Agency's directions?
2. When was the Regulatory Agency notified? By whom?
3. What was the identity of the load or farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. when used for a milk plant's raw milk supply(ies) that has (have) not been transported in bulk milk pickup tankers?
4. What screening and/or confirmatory test(s) were used and who were the analyst(s)?
5. What was the disposition of the adulterated milk?
6. Which producer(s) was responsible?
7. Record of negative test results prior to subsequent milk pickup from the violative producer(s).

### **III. TESTING PROGRAM FOR DRUG RESIDUES ESTABLISHED DEFINITIONS:**

For purposes of this Appendix the following definitions are to be used:

1. **Presumptive Positive:** A presumptive positive test is a positive result from an initial testing of a bulk milk pickup tanker and/or raw milk supply that has not been transported in bulk milk pickup tankers using an M-a-85 (latest revision) approved test, which has been promptly repeated in duplicate with positive and negative controls that give the proper results using the same test, on the same sample, with one (1) or both of these duplicate retests giving a positive result.
2. **Screening Test Positive (Load or Raw Milk Supply that has Not been Transported in Bulk Milk Pickup Tankers Confirmation):** A screening test positive result is obtained when the presumptive positive sample is tested in duplicate, using the same or equivalent (M-I-96-10, latest revision) test as that used for the presumptive positive, with a positive and negative control that give the proper results, and either or both of the duplicates are positive. A screening test positive (load or farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. when used for a milk plant's raw milk supply(ies) that has (have) not been transported in bulk milk pickup tankers confirmation) is to be performed by an Official Laboratory, Officially Designated Laboratory or Certified Industry Supervisor using the same or an equivalent test (M-I-96-10, latest revision).
3. **Producer Trace Back/Permit Action:** A producer trace back/permit action test is performed after a screening test positive load is identified by an Official Laboratory, Officially Designated Laboratory or Certified Industry Supervisor using the same or an equivalent (M-I-96-10, latest revision) test as was used to obtain the screening test positive (load confirmation). A confirmed producer test positive result is obtained in the same manner as a confirmation (screening test positive) for a load. After an initial positive result (producer presumptive positive) is obtained on a producer sample, that sample is then tested in duplicate using the same test as was used to obtain the producer presumptive positive result. This testing is performed with a positive and negative control and if either or both of the duplicates are positive and the controls give the proper results, the producer sample is confirmed as positive.

**NOTE:** When a farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. is used for a milk plant's raw milk supply(ies) that has not been transported in bulk milk pickup tankers, is found to be positive (confirmed) for drug residues, the farm of origin for the drug residue has consequently already been determined and further testing is not required to determine the farm of origin.

4. **Individual Producer Load:** An individual producer bulk milk pickup tanker is a bulk milk pickup tanker, or a compartment(s) of a bulk milk pickup tanker, that contains milk from only one (1) dairy farm.
5. **Individual On-Farm Producer/Processor's Raw Milk Supply:** An individual on-farm producer/processor's raw milk supply may be transported in bulk milk pickup tankers; and/or their raw milk supply may be stored in a farm bulk milk tank(s)/silo(s) on the dairy farm that directly feeds the batch (vat) pasteurizer(s) or constant-level tank of a HTST pasteurization system or piped from the a farm bulk milk tank(s)/silo(s) to a raw milk tank(s) and/or silo(s) in the milk plant that feeds the batch (vat) pasteurizer(s) or constant-level tank of a HTST pasteurization system; and/or other raw milk storage containers.

6. **Industry Analyst:** A person under the supervision of a Certified Industry Supervisor or Industry Supervisor who is assigned to conduct screening of bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers for Appendix N. drug residue requirements.

7. **Industry Supervisor/Certified Industry Supervisor:** An individual trained by a LEO who is responsible for the supervision and training of Industry Analysts who test milk tank trucks and/or all raw milk supplies that have not been transported in bulk milk pickup tankers for Appendix N. drug residue requirements.

8. **Certified Industry Supervisor:** An Industry Supervisor who is evaluated and listed by a LEO as certified to conduct drug residue screening tests at industry drug residue screening sites for *Grade "A" PMO*, Appendix N. regulatory actions (confirmation of bulk milk pickup tankers, farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), or other raw milk storage container(s), etc. when used for a milk plant's raw milk supply(ies) that has (have) not been transported in bulk milk pickup tankers, producer trace back and/or permit actions).

## **CERTIFIED INDUSTRY SUPERVISORS; EVALUATION AND RECORDS:**

Reference: *EML*

1. **Certified Industry Supervisors/Industry Supervisors/Industry Analysts:** Regulatory Agencies may choose to allow Industry Supervisors to be certified. Under this program, these Certified Industry Supervisors may officially confirm presumptive positive bulk milk pickup tanker loads and/or all raw milk supplies that have not been transported in bulk milk pickup tankers, and confirm producer milk for regulatory purposes (producer trace back/permit action). In the implementation of Appendix N. of this *Ordinance*, the LEO shall use the appropriate Appendix N. FDA/NCIMS 2400 Form when evaluating Official Laboratories, Officially Designated Laboratories or Certified Industry Supervisors, Industry Supervisors and Industry Analysts.

The Certified Industry Supervisor/Industry Supervisor shall report to the LEO the results of all competency evaluations performed on Industry Analysts. The names of all Certified Industry Supervisors, Industry Supervisors and Industry Analysts, as well as their training and evaluation status, shall be maintained by the LEO and updated as replacement, additions and/or removals occur. The LEO shall verify (document) that each Certified Industry Supervisor and/or Industry Supervisor has established a program that ensures the proficiency of the Industry Analysts they supervise. The LEO shall also verify that each Industry Supervisor and Industry Analyst has demonstrated proficiency in performing drug residue analysis at least biennially. Verification may include an analysis of split samples and/or an on-site performance evaluation or another proficiency determination that the LEO and the FDA Laboratory Proficiency Evaluation Team (LPET) agree is appropriate.

Failure by the Industry Supervisor or Industry Analyst to demonstrate adequate proficiency to the LEO shall lead to their removal from the LEO list of Industry Supervisors and/or Industry Analysts. Reinstatement of their testing status shall only be possible by completing retraining and/or successfully analyzing split samples and/or passing an on-site evaluation or otherwise demonstrating proficiency to the LEO. (Refer to the *EML*, which describes the certification requirements for Certified Industry Supervisors and the training requirements for Industry Supervisors and Industry Analysts.)

2. **Sampling and Testing of Bulk Milk Pickup Tankers:** The bulk milk pickup tanker shall be sampled after the last producer has been picked up and before any additional commingling. The sample shall be representative. The sample analysis shall be completed before the milk is processed.

3. **Sampling and Testing of Raw Milk Supplies that have Not been Transported in Bulk Milk Pickup Tankers:** All raw milk supplies that have not been transported in bulk milk pickup tankers shall be sampled prior to processing the milk. The sample(s) shall be representative of each farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo(s), or other raw milk storage container(s) supply. Testing of all raw milk supplies that have not been transported in bulk milk pickup tankers shall be completed prior to processing the milk.

4. **Bulk Milk Pickup Tanker Unloaded Prior to Negative Test Result:** If the bulk milk pickup tanker is unloaded and commingled prior to obtaining a negative test result and the screening test is presumptive positive, the Regulatory Agency shall be immediately notified. If the bulk milk tanker sample is confirmed positive, then the commingled milk is adulterated and unacceptable for human consumption regardless of any subsequent test results from the commingled milk. The milk shall be disposed of under the supervision of the Regulatory Agency.

5. **Raw Milk Supplies that have Not been Transported in Bulk Milk Pickup Tankers Processed Prior to Negative Results:** If the raw milk supply that has not been transported in bulk milk pickup tankers is processed prior to obtaining a negative test result and the screening test is presumptive positive, the Regulatory Agency shall be immediately notified. If the sample of the raw milk supply that has not been transported in bulk milk pickup tankers is confirmed positive, then the processed milk is adulterated and unacceptable for human consumption regardless of any subsequent test results from the raw milk supply and/or pasteurized milk or milk products. The processed milk shall be disposed of under the supervision of the Regulatory Agency.

#### **BULK MILK PICKUP TANKER AND/OR ALL RAW MILK SUPPLIES THAT HAVE NOT BEEN TRANSPORTED IN BULK MILK PICKUP TANKERS SCREENING TEST:**

1. **Performance Tests/Controls:** Each lot of test kits purchased shall be tested by positive (+) and negative (-) controls, as defined in the SCREENING TESTS NECESSARY TO IMPLEMENT THE PROVISIONS OF APPENDIX N. FOR BULK MILK PICKUP TANKERS AND/OR ALL RAW MILK SUPPLIES THAT HAVE NOT BEEN TRANSPORTED IN RAW BULK MILK PICKUP TANKERS of this Section, in each screening facility prior to its initial use and each testing day thereafter. Records of all positive (+) and negative (-) control performance tests shall be maintained.

2. **Initial Drug Testing Procedures:** The following procedures apply to testing bulk milk pickup tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers for drug residues following the provisions of Appendix N. Industry analysts may screen tankers and/or all raw milk supplies that have not been transported in bulk milk pickup tankers and receive or reject milk. Milk plants, receiving stations, transfer stations and other screening locations may choose to participate in the Industry Supervisor Certification Program.

a. **Industry Presumptive Positive Options:** There are two (2) industry options for the milk represented by a presumptive positive sample:

1) The Regulatory Agency involved (origin and receipt) shall be notified. The appropriate Regulatory Agency shall take control of the presumptive positive load and/or raw milk supply that has not been transported in bulk milk pickup tankers. A written copy of the presumptive positive test results shall follow the initial Regulatory Agency notification. Testing for confirmation of that presumptive positive load and/or raw milk supply that has not been transported in bulk milk pickup tankers shall be in an Official Laboratory, Officially Designated Laboratory or by a Certified Industry Supervisor at a location acceptable to the Regulatory Agency. Documentation of prior testing shall be provided to the analyst performing the load and/or raw milk supply that has not been transported in bulk milk pickup tankers confirmation. The presumptive positive load and/or raw milk supply that has not been transported in bulk milk pickup tankers may be re-sampled, at the direction of the Regulatory Agency, prior to analysis with the same or equivalent test (M-I-96-10, latest revision), as was used to obtain the presumptive positive result. This analysis shall be done in duplicate with positive (+) and negative (-) controls. If either or both of the duplicate samples are positive and the positive (+) and negative (-) controls give the correct reactions, the sample is deemed a Screening Test Positive (Confirmed Load and/or Raw Milk Supply that has Not been Transported in Bulk Milk Pickup Tankers). A written copy of the test results shall be provided to the Regulatory Agency. The milk, which that sample represents, is no longer available for sale or processing into human food.

2) The owner of the presumptive positive milk may reject the load and/or raw milk supply that has not been transported in bulk milk pickup tankers without further testing. At that time the milk represented by the presumptive positive test is not available for sale or processing into human food. The milk cannot be re-screened. The Regulatory Agency involved (origin and receipt) shall be notified. Under this option, producer trace backs shall be conducted for the reject load.

**NOTE:** When a farm bulk milk tank(s)/silos(s), milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. is used for a milk plant's raw milk supply(ies) that has not been transported in bulk milk pickup tankers, is found to be positive (confirmed) for drug residues, the farm of origin for the drug residue has consequently already been determined and further testing is not required to determine the farm of origin.

### 3. **Re-Sampling:**

a. **Presumptive Results:** Occasionally, an error in sampling or a suspicious test result is discovered after a presumptive result is initially obtained. When this happens, the Regulatory Agency may allow the industry to re-sample the bulk milk pickup tanker and/or raw milk supply that has not been transported in bulk milk pickup tankers. The reasons that made the re-sampling necessary shall be clearly documented in testing records and reported to the Regulatory Agency. This written record shall be provided to the Regulatory Agency and shall be maintained with the record of the testing for that load and/or raw milk supply that has not been transported in bulk milk pickup tankers.

b. **Screening Test Results:** Re-sampling or additional analysis of screening test results should be discouraged. However, the Regulatory Agency may direct re-sampling and/or analysis, when it has determined that procedures for sampling and/or analysis did

not adhere to accepted NCIMS practices (*SMEDP*, FDA/NCIMS 2400 Forms, Appendix N. and the applicable FDA interpretative or informational memoranda). This decision by the Regulatory

Agency shall be based on objective evidence. A Regulatory Agency allowing re-sampling shall plan a timely follow-up to identify the problem and initiate corrective action to ensure the problem that led to the need for re-sampling is not repeated. If re-sampling and/or analysis is necessary, it shall include a review of the samplers, analysts, and/or laboratories to identify the problem(s) and initiate corrective action to ensure the problem(s) is not repeated. The reasons that made the re-sampling or analysis necessary shall be clearly documented in testing records maintained by the Regulatory Agency, and shall be maintained with the record of the testing for that load and/or raw milk supply that has not been transported in bulk milk pickup tankers.

4. **Producer Trace Back:** All screening test positive (confirmed) loads shall be broken down (producer trace back) using the same or an equivalent test method (M-I-96-10, latest revision). Confirmation tests (load and producer trace back/permit action) shall be performed in an Official Laboratory, Officially Designated Laboratory or by a Certified Industry Supervisor. Positive producers shall be handled in accordance with this Appendix.

**NOTE:** When a farm bulk milk tank(s)/silos, milk plant raw milk tank(s) and/or silo(s), other raw milk storage container(s), etc. is used for a milk plant's raw milk supply(ies) that has not been transported in bulk milk pickup tankers, is found to be positive (confirmed) for drug residues, the farm of origin for the drug residue has consequently already been determined and further testing is not required to determine the farm of origin.

Assuring Representative Samples From Individual-Producer Loads And Multiple-Farm Tank Loads From An Individual Producer: Representative samples shall be secured from each farm storage tank(s)/silo(s) of milk prior to loading onto a bulk milk pickup tanker and/or other raw milk supply transportation method at the dairy farm. The representative sample(s) shall travel with the bulk milk pickup tanker and/or other raw milk supply transportation method to a designated location acceptable to the Regulatory Agency.

**Record Requirements:** Results of all testing may be recorded in any format acceptable to the Regulatory Agency that includes at least the following information:

1. Identity of the person doing the test;
2. Identity of the bulk milk pickup tanker or farm bulk milk tank(s)/silo(s), milk plant raw milk tank(s) and/or silo, or other raw milk storage container(s), etc. used for the storage of raw milk supplies that have not been transported in bulk milk pickup tankers being tested\* ;
3. Date/time the test was performed (Time, Day, Month and Year);
4. Identity of the test performed/lot #/any and all controls (+/-);
5. Results of the test, if the analysis results are positive the record shall show:
  - a. The identity of each producer contributing to the positive load;
  - b. Who at the Regulatory Agency was notified;
  - c. When did this notification take place; and
  - d. How was this notification accomplished.
6. Follow-up testing if initial test was positive/any and all controls (+/-);

7. Site where test was performed; and
8. Prior test documentation shall be provided for a presumptive positive load.

\*Include the BTU number(s) of the dairy farms present on the bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers with the above information.

## **SCREENING TESTS NECESSARY TO IMPLEMENT THE PROVISIONS OF APPENDIX N. FOR BULK MILK PICKUP TANKERS AND/OR ALL RAW MILK SUPPLIES THAT HAVE NOT BEEN TRANSPORTED IN BULK MILK PICKUP TANKERS:**

1. **Performance Tests/Controls (+/-):**
  - a. Each lot of kits purchased is tested by positive (+) and negative (-) controls.
  - b. Each screening facility runs a positive (+) and negative (-) control performance test each testing day.
  - c. All NCIMS Approved Bulk Milk Pickup Tanker and/or All Raw Milk Supplies that have Not been Transported in Bulk Milk Pickup Tankers Screening Tests Include the Following Format: All presumptive positive test results shall be repeated in duplicate as soon as possible at the direction of the Regulatory Agency on the same sample with single positive (+) and negative (-) controls by a certified analyst (Official Laboratory, Officially Designated Laboratory or Certified Industry Supervisor) using the same or equivalent test (M-I-96-10, latest revision). If the duplicate tests are negative, with appropriate (+/-) control results, the bulk milk pickup tanker and/or all raw milk supplies that have not been transported in raw milk bulk milk pickup tankers is reported as negative. If one (1) or both duplicate test(s) is positive (+), the test result is reported to the Regulatory Agency in which the testing was conducted, as a screening test positive (confirmed).
  - d. All positive (+) controls used for drug residue testing kits are labeled to indicate a specific drug and concentration level for that drug.
    - (1)For tests that have been validated and only detect Penicillin, Ampicillin, Amoxicillin and Cephapirin, the positive (+) control is Pen G @  $5 \pm 0.5$  ppb.
    - (2)For test kits validated for the detection of Cloxacillin, the positive (+) control may be Cloxacillin @  $10 \pm 1$  ppb.
    - (3)For test kits validated for one (1) drug residue only, the positive (+) control is  $\pm 10\%$  of the safe level/tolerance of the drug residue detected.
2. **Work Area:**
  - a. Temperature within specifications of the test kit manufacturer's labeling.
  - b. Adequate lighting for conducting the test kit procedure.
3. **Test Kit Thermometers:**
  - a. Thermometer traceable to a NIST Certified Thermometer.
  - b. Graduation interval not greater than  $1^{\circ}\text{C}$ .
  - c. Dial thermometers are not used to determine the temperatures of samples, reagents, refrigerators, or incubators in milk laboratories.
4. **Refrigeration:**
  - a. Test kit reagent storage temperature specified by manufacturer.

5. **Balance (Electronic):**
  - a. 0.01 g for preparation of positive (+) controls.
  - b. Balance with appropriate sensitivity for calibration of pipetting devices within a tolerance of  $\pm 5\%$ . These devices may be calibrated at another location acceptable to the LEO.
6. **Screening Test Sampling Requirements:**
  - a. Temperature of milk in the bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers determined and recorded.
  - b. Representative bulk milk pickup tanker and/or all raw milk supplies that have not been transported in bulk milk pickup tankers sample for drug residue testing collected.
  - c. Samples tested within seventy-two (72) hours of collection.
7. **Screening Test Volumetric Measuring Devices:**
  - a. Single use devices provided by kit manufacturers are acceptable for Appendix N. screening analysts.
  - b. NCIMS Certified Laboratories require calibrated pipetting/dispensing devices. These devices may be calibrated at another location acceptable to the LEO.
  - c. Measuring devices with tips bearing calibration lines provided by test kit manufacturers are acceptable for Appendix N. screening.

#### **IV. ESTABLISHED TOLERANCES AND/OR SAFE LEVELS OF DRUG RESIDUES**

"Safe levels" are used by FDA as guides for prosecutorial discretion. They do not legalize residues found in milk that are below the safe level. In short, FDA uses the "safe levels" as prosecutorial guidelines and in full consistency with *CNI v. Young* stating, in direct and unequivocal language, that the "safe levels" are not binding. They do not dictate any result; they do not limit FDA's discretion in any way; and they do not protect milk producers, or milk from court enforcement action.

"Safe levels" are not and cannot be transformed into tolerances that are established for animal drugs under Section 512 (b) of the *FFD&CA* as amended. "Safe levels" do not:

1. Bind the courts, the public, including milk producers, or FDA, including individual FDA employees; and
2. Do not have the "force of law" of tolerances, or of binding rules.

Notification, changes or additions of "safe levels" shall be transmitted via Memoranda of Information (M-I's).

#### **V. APPROVED METHODS**

Regulatory Agencies and industry shall use tests from the most recent revision of M-a-85 for analysis of bulk milk pickup tankers and/or all raw milk supplies that have not been transported in raw milk bulk milk pickup tankers for Beta lactam residues, following the testing procedures specified in Section III of this Appendix. AOAC First Action and AOAC Final Action methods are accepted in accordance with Section 6 of this *Ordinance*. Drug residue detection methods shall be evaluated at the safe level or tolerance. Regulatory action based on each test kit method may be delayed until the evaluation is completed and the method is found to be acceptable to FDA and complies with the provisions of Section 6 of this *Ordinance*.

One (1) year after test(s) have been evaluated by FDA and accepted by the NCIMS for a particular drug or drug family, other unevaluated tests are not acceptable for screening milk. The acceptance of evaluated tests does not mandate any additional screening by industry with the evaluated method.

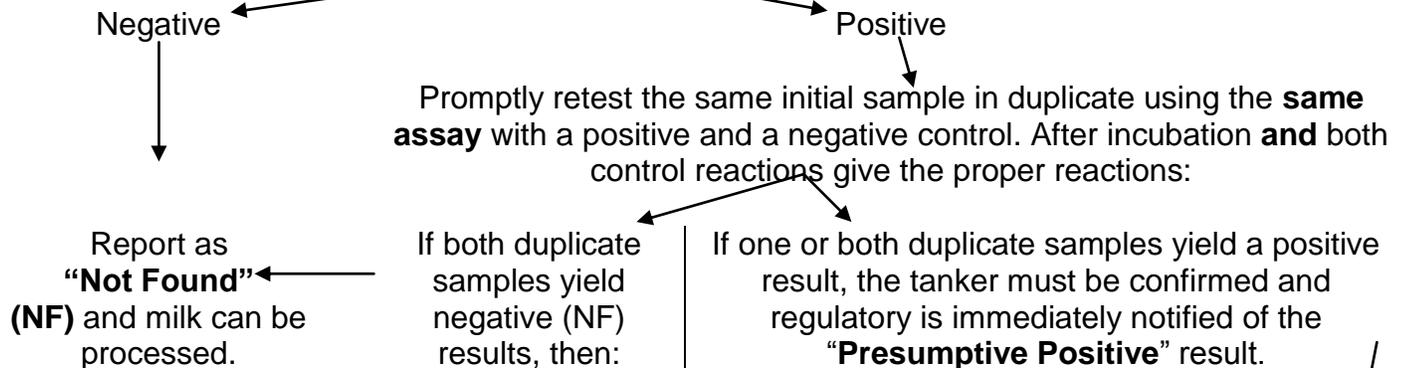
## Flowchart For Antibiotic Residue Analysis

Incoming tankers are screened for antibiotic residues either by an approved industry analyst(s) or by a certified analyst.

Daily monitoring performed on assays with readers – monitoring must be valid before proceeding.

**POSITIVE AND NEGATIVE CONTROL REACTIONS ARE VALID.**

Sample Reaction after initial analysis:



**Only certified analysts can continue the assay beyond this point.**

**Using the same initial positive sample\*:**

Optionally, rerun the reader performance controls where applicable.

**Promptly run a positive control, a negative control and same initial positive sample in duplicate on the same or equivalent assay.**

**Both controls give proper results before proceeding.**

NOTE: Controls and samples can be incubated at the same time, but control reactions must be determined before reading the sample results.

If both duplicate samples yield a negative (NF) result, then the tanker can be processed.

If either one or both duplicate samples yield a positive result, the tanker load is a **"Screening Test Positive (Confirmed Load)."**

The tanker represented by the sample **cannot** be processed, retested or offered for sale.

Producer trace back must be conducted.

Using the same or equivalent assay, test the producers that made up the **"Screening Test Positive"** tanker.

Positive producer samples are confirmed using the same procedure as for the tanker sample.

\*The Presumptive Positive Load can be re-sampled at the direction of the state Regulatory Agency prior to load confirmation. Records must indicate the reason for re-sampling the tanker and the regulator contacted.