



NF VALIDATION 102 (AFNOR Certification):
Protocol for validation of methods for the detection and quantification of veterinary drugs in
food products (revision N°1: June 1st, 2017)

RENEWAL STUDY

Validation report of evaluation of Delvotest® T kit for detection of a broad spectrum residues of antibiotics in raw cow milk

Version 1
OCTOBER 2021

Expert Laboratory:
ACTALIA Cecalait
Rue de Versailles
39800 Poligny
FRANCE
Phone +33 (0)3 84 73 63 20
Fax +33 (0)3 84 73 63 29

For:
DSM Food Specialties B. V
2600 MA Delft
The NETHERLANDS

Certification Body:

AFNOR

Validation protocol:

NF102 (AFNOR Certification): Protocol for validation of methods for the detection and quantification of veterinary drugs in food products (revision N°1: June 1st, 2017)

Kit to evaluate:

Delvotest® T

Principle of the method:

This method is a standard diffusion test for the qualitative detection of antibacterial substances as penicillins, tetracyclines, sulfonamides, cephalosporins, macrolides, aminoglycosides, lincomycin, chloramphenicol, trimethoprim, rifaximin and dapson in raw milk.

Scope:

Raw cow milk

Table of contents

| | | |
|----|---|-----|
| 1. | INTRODUCTION..... | 4 |
| | 1.1. First validation in 2013 (ANSES Fougères) | 4 |
| | 1.2. First renewal in 2016 (ANSES Fougères) | 4 |
| | 1.3. Second renewal in 2020-2021 (ACTALIA Cecalait) | 4 |
| 2. | DELVOTEST® T METHOD | 5 |
| | 2.1. Principle of the method | 5 |
| | 2.2. Protocol of the method | 5 |
| 3. | PRELIMINARY STUDY | 6 |
| | 3.1. Determination of the control time | 6 |
| | 3.2. Determination of detection capability (CCβ) | 7 |
| | 3.3. Test for false positive results | 10 |
| | 3.4. Applicability on individual cow milk | 10 |
| | 3.5. Robustness | 11 |
| | 3.6. Practicability | 16 |
| | 3.7. Bibliography | 17 |
| | 3.8. Conclusion of the preliminary study | 17 |
| | 3.9. User complaints | 18 |
| 4. | INTERLABORATORY STUDY..... | 20 |
| | 4.1. Interlaboratory study in 2013 (ANSES Fougères) | 20 |
| | 4.2. Interlaboratory study in 2021 (ACTALIA-Cecalait) | 21 |
| | 4.3. Calculation on both interlaboratory studies (2013 and 2021) | 27 |
| | 4.4. Conclusion of interlaboratory studies (2013 and 2021 studies) | 40 |
| 5. | GENERAL CONCLUSION..... | 41 |
| 6. | BIBLIOGRAPHIC REFERENCES | 43 |
| | Appendix 1: Details on antibiotics used in preliminary study | 44 |
| | Appendix 2: Results of robustness study (2020) | 46 |
| | Appendix 3: Results of preliminary and interlaboratory studies in 2013 (ANSES) | 112 |
| | Appendix 4: Details on antibiotics used in interlaboratory study (2021) | 114 |
| | Appendix 5: Raw data for homogeneity (2021) | 115 |
| | Appendix 6: Raw data for stability study | 116 |
| | Appendix 7: Results of interlaboratory study in 2021 (ACTALIA Cecalait) | 118 |

1. INTRODUCTION

The Delvotest® T method was certified by AFNOR in 2013 under the certification number DSM 28/02-02/12, then 2 renewal studies were performed in 2016 and 2020

1.1. First validation in 2013 (ANSES Fougères)

In the first validation, the scope was cow milk with an extension on ewe and goat milks. In addition, the presence of a preservative (azidiol) in milk has been studied. A comparative study has been performed with alternative method Delvotest® T and a reference method.

Results of Delvotest® T and of the reference method were similar. The method was applicable on ewe and goat milks. The presence of preservative (azidiol) did not impact the results. Conclusions of preliminary study were positive to the continuation of project and an interlaboratory study was performed.

1.2. First renewal in 2016 (ANSES Fougères)

A first renewal has been performed in 2016 without further study, as alternative method and AFNOR technical rules were not modified.

1.3. Second renewal in 2020-2021 (ACTALIA Cecalait)

The rules of AFNOR have been changed in 2017 according to NF102 (AFNOR Certification): 'Protocol for validation of methods for the detection and quantification of veterinary drugs in food products' (revision N°1: June 1st, 2017). The modifications in comparison with previous rules are:

- No comparison of the alternative method with a reference method ;
- Detection capability (CC β) study with minimum 20 repetitions per antibiotic and per level, and 20 antibiotics minimum for a broad spectrum test in milk ;
- Applicability with 10 repetitions minimum per antibiotic and per level;
- Analyses with at least 3 different batch numbers of kit (including a batch close to the manufacture date and a batch close to the expiry date) ;
- Influence of test protocol and composition of matrix are to test in robustness study.

As the 1st preliminary study was done only on 5 repetitions per antibiotic and per level, and age of reagents were not tested, all the validation study was performed again according to the new AFNOR Certification rules including:

- Determination of detection capability (CC β) with at least 3 batches ;
- Determination of false positive results ;
- Applicability ;
- Robustness ;
- Practicability.

A complement of interlaboratory study of 2013 was performed in 2021:

- To test again Tetracycline ;
- To align the interlaboratory study with the new version of AFNOR rules, with addition of 2 antibiotics.

2. DELVOTEST® T METHOD

2.1. Principle of the method

The Delvotest® T is a qualitative broad spectrum test for the detection of antibiotic residues in raw milk. The test is based on growth inhibition of *Geobacillus stearothermophilus*.

The product contains a solid agar medium seeded with standardised number of spores of *Geobacillus stearothermophilus* with required nutrients for growth. The medium is coloured by the pH indicator bromocresol purple.

Milk samples, which are free from antibiotic substances or contain them below specified level, were added to the test at the level of 0,1 mL and incubated at 64°C. It allows germination and growth of the bacteria. This will lead to a change in colour of the indicator to (partially) yellow.

When the milk sample contains antibiotic substances at or above the test sensitivity, growth is inhibited and the colour remains (predominantly) purple.

2.2. Protocol of the method

Test production and protocol have not changed since the first validation in 2012

Delvotest® T kit is in two formats according to the number of samples to use. Each format has different packaging. Once a month, the scanner is calibrated with a coloured card.

The steps are the following:

- Preheat the incubation device. The temperature of the dry incubator or water bath should be set at $64^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
- Select the required number of test material. Detach one or more ampoules, or break the plates in blocks depending on the number of milk sample to analyse. Take care that the aluminium foil from the remaining tests is not damaged. Remove the aluminium foil from the plate or perforate the foil of ampoules carefully.
- Add the milk sample. Milk samples should be representative of the milk to be tested and homogenized. Pipette 0,1 mL of sample in the test. For each sample use a new and clean pipette. Indicate clearly each test with for example a sample number.
- Incubate the test. When using test in plate, cover the plates using included adhesive foil. The ampoules have to be put into the incubator immediately after milk addition.

Incubate the test plates or ampoules in the preheated dry incubator or water bath. Incubate the test until the control time already determined.

- Read visually or with Delvo®Scan. The colour should be read from the 2/3 part of the ampoule or from underneath the test plate. Interpretation of results is presented in **Table 1**.

Table 1. Interpretation of results of Delvotest® T.

| COLOR OF MEDIUM | RESULTS |
|---------------------------|---|
| Purple (predominantly) | POSITIVE Milk sample contains antibiotics at or above the test sensitivity. |
| Yellow (partially) | NEGATIVE The milk analysed does not contain antibiotics or the antibiotic concentration is below the detection sensitivity of the test. |

3. PRELIMINARY STUDY

In the second renewal study in 2020, all preliminary study was performed again according to the new rules of AFNOR of 2017.

During this second renewal study, 2 formats of Delvotest® T were studied: ampoules and plates. In the same time, 2 types of readings were tested: visual and by Delvo®Scan.

All incubations of Delvotest® T in plates were done in a water bath at $64^{\circ}\text{C} \pm 2^{\circ}\text{C}$. For those in ampoules, DSM Delvotest® Incubators (Mini S block heater) were used at $64^{\circ}\text{C} \pm 2^{\circ}\text{C}$. All incubations were performed at ‘control time’ (see 3.1).

The Delvo®Scan software used was the version 5.08 (ampoules and plates) with an EPSON V600 scanner with a cut-off equal to 0.

All pipettings were done with a variable pipette (20 – 200 μL with an accuracy of $\pm 0,6 \mu\text{L}$).

3.1.Determination of the control time

The control time is the time when blank negative milk sample turns negative. It was determined for each batch according to DSM good practices by the analyze of 1 negative control provided by DSM, reads every 5 minutes from 2h45 to 3h15 incubation.

3.2. Determination of detection capability (CC β)

3.2.1. Materials and methods

The blank raw cow milk was commingled milk coming from at least 10 animals not treated with veterinary drugs within the last 8 weeks before milking. The maximum period for the cold storage (between 0°C and 6°C) of the fresh raw milk was 56 hours. Analyses of composition of milk were performed for each milk used (fat, protein, somatic cells, total count of microorganisms and pH).

The blank raw milk was tested before using by Delvotest® T and by another test (Bioeasy® β -lactam –tetracycline – Cefalexin – Ref YRM1008-40) in duplicate. Three blank raw milks of different origin were used.

Blank raw milk was spiked with different compounds (**Table 3**) belonging to different drug families. 42 molecules have been tested with two formats (plates and ampoules). As the quinolones (sum of enrofloxacin and ciprofloxacin) are not detected by Delvotest® T, they were not tested in this renewal study. Details on antibiotics used are reported in **Appendix 1**.

For each format, results were read by visually and by Delvo®Scan. All samples were codified previously and were analyzed in blind.

At least 3 different batch numbers have been tested (including a batch close to the manufacture date and a batch close to the expiry date).

Each compound was spiked separately. For each compound a minimum of 1 level around the test detection capability was tested.

- In the case where the CC β values were announced by DSM, in the first time the CC β announced was tested.
- In the case where the CC β values were not announced by DSM, in the first time the MRL was tested.

If the CC β still fails, the CC β level is increased according to NF 102 rules as follows:

- Range 1 – 10 ppb: increments of 1 ppb
- Range 11 – 20 ppb: increments of 2 ppb
- Range 21 – 50 ppb: increments of 5 ppb
- Range 51 – 250 ppb: increments of 10 ppb
- Range 251 – 500 ppb: increments of 25 ppb
- Range 500 – 1 000 ppb: increments of 100 ppb
- Range 1 000 – 5 000 ppb: increments of 500 ppb

The number of replicates tested at each level is based on closeness to the MRL according to AFNOR rules. The number of replicates is given in **Table 2**.

Table 2. Number of replicates to test according to the MRL

| Concentration tested | Number of replicates | Performance criterion Maximum number of negative results allowed |
|---|----------------------|---|
| > MRL | 20 | 1 |
| Close to the MLR (10% below to the MLR) | 60 | 3 |
| Between 50% and 90% of the MLR | 40 | 2 |
| ≤ 50% MRL | 20 | 1 |

Detection capability is defined as the lowest concentration tested giving at least 95% of positive results; it is the lowest concentration where at least 19 out of 20 tests, 38 out of 40 tests, or 57 out of 60 tests are positive, respectively.

Detection capability was determined with 3 batches for ampoules (20A09/31, 19L11/31 and 19L18/31) and 6 batches for plates (19K22/30, 19J18/30, 19J02/30, 20D31/20, 20D16/30 and 20D22/30). The tests were interpreted visually and by Delvo®Scan. All results (reader values) were collected in a data base.

3.2.2. Results

A summary of the detection capabilities obtained is given in **Table 3**.

3.2.3. Conclusion of CCB study

On 42 antibiotics tested:

- 25 compounds have a detection capability below or equal to MRL.
 - o The detection capability of gentamycin is equal to the MRL for both plates and ampoules.
 - o The detection capability of oxytetracycline is equal to the MRL for ampoules only;
 - o The detection capability of tetracycline is equal to the MRL for plates only.
- 16 compounds have a detection capability higher than regulatory limits (4-epioxytetracycline, chlortetracycline, 4-epitetracycline, 4-epichlortetracycline, sulfamethazine, tilmicosin, erythromycin A, spiramycin, streptomycin, dihydrostreptomycin, cefquinome, chloramphenicol, trimethoprim, lincomycin, clavulanic acid and dapsone);
- Doxycycline has no regulatory limite.

Quinolones (Enrofloxacin and ciprofloxacin) were not tested since Delvotest® T detection capability is far above MRL.

Table 3. Detection capabilities (CC β , in ppb) determined at control time by Delvotest® T kit in raw cow milk.

| Drug family | Compounds detected | MRL in milk (ppb) | Number of positive sample | | CC β (ppb) | =, < or > MRL | Number of positive sample | | CC β (ppb) | =, < or > MRL |
|-----------------|------------------------|-------------------|---------------------------|-------------------|------------------|---------------|---------------------------|-------------------|------------------|---------------|
| | | | Visual reading | Delvoscan reading | | | Visual reading | Delvoscan reading | | |
| | | | Ampoules | | | | | Plates | | |
| Penicillins | Amoxicillin | 4 | 20/20 | 20/20 | 2 | < | 20/20 | 20/20 | 2 | < |
| | Ampicillin | 4 | 20/20 | 20/20 | 2 | < | 20/20 | 20/20 | 2 | < |
| | Penicillin G | 4 | 40/40 | 39/40 | 3 | < | 20/20 | 20/20 | 1 | < |
| | Cloxacillin | 30 | 20/20 | 20/20 | 10 | < | 20/20 | 20/20 | 10 | < |
| | Oxacillin | 30 | 20/20 | 20/20 | 3 | < | 20/20 | 20/20 | 3 | < |
| | Nafcillin | 30 | 20/20 | 20/20 | 3 | < | 20/20 | 20/20 | 3 | < |
| Tetracyclines | Oxytetracycline | 100 | 60/60 | 60/60 | 100 | = | 40/40 | 39/40 | 80 | < |
| | 4-Epioxytetracycline | 100 | 20/20 | 20/20 | 600 | > | 20/20 | 20/20 | 800 | > |
| | Chlortetracycline | 100 | 20/20 | 20/20 | 150 | > | 20/20 | 20/20 | 150 | > |
| | 4-Epichlortetracycline | 100 | 20/20 | 20/20 | 600 | > | 20/20 | 20/20 | 600 | > |
| | Tetracycline | 100 | 40/40 | 40/40 | 80 | < | 60/60 | 60/60 | 100 | = |
| | 4-Epitetracycline | 100 | 20/20 | 20/20 | 800 | > | 20/20 | 20/20 | 1000 | > |
| | Doxycycline | * | 20/20 | 20/20 | 50 | * | 20/20 | 20/20 | 50 | * |
| Sulfonamides | Sulfamethazine | 100 | 20/20 | 20/20 | 125 | > | 20/20 | 20/20 | 125 | > |
| | Sulfathiazole | 100 | 20/20 | 20/20 | 30 | < | 20/20 | 20/20 | 30 | < |
| | Sulfadimethoxine | 100 | 20/20 | 20/20 | 40 | < | 20/20 | 20/20 | 40 | < |
| | Sulfadiazine | 100 | 40/40 | 40/40 | 55 | < | 20/20 | 20/20 | 50 | < |
| | Sulfadoxine | 100 | 40/40 | 40/40 | 80 | < | 40/40 | 40/40 | 80 | < |
| Macrolides | Tilmicosin | 50 | 20/20 | 20/20 | 60 | > | 20/20 | 20/20 | 100 | > |
| | Tylosin A | 50 | 40/40 | 40/40 | 35 | < | 40/40 | 40/40 | 35 | < |
| | Erythromycin A | 40 | 20/20 | 20/20 | 160 | > | 20/20 | 20/20 | 200 | > |
| | Spiramycin | 200 | 20/20 | 20/20 | 1500 | > | 20/20 | 20/20 | 2000 | > |
| Aminoglycosides | Neomycin B | 1500 | 20/20 | 20/20 | 140 | < | 20/20 | 20/20 | 140 | < |
| | Gentamycin | 100 | 59/60 | 59/60 | 100 | = | 58/60 | 58/60 | 100 | = |
| | Streptomycin | 200 | 20/20 | 20/20 | 700 | > | 20/20 | 20/20 | 1000 | > |
| | Dihydrostreptomycin | 200 | 20/20 | 20/20 | 700 | > | 20/20 | 20/20 | 800 | > |
| Cephalosporins | Cephapirin | 60 | 20/20 | 20/20 | 5 | < | 20/20 | 20/20 | 5 | < |
| | Desacetylcephapirin | 60 | 20/20 | 19/20 | 2 | < | 20/20 | 20/20 | 2 | < |
| | Ceftiofur | 100 | 20/20 | 20/20 | 20 | < | 20/20 | 20/20 | 20 | < |
| | Desfuroylceftiofur | 100 | 20/20 | 20/20 | 45 | < | 40/40 | 40/40 | 80 | < |
| | Cefoperazone | 50 | 20/20 | 20/20 | 20 | < | 20/20 | 20/20 | 20 | < |
| | Cefalexin | 100 | 20/20 | 20/20 | 30 | < | 20/20 | 20/20 | 30 | < |
| | Cefquinome | 20 | 20/20 | 20/20 | 50 | > | 20/20 | 20/20 | 60 | > |
| | Cefalonium | 20 | 20/20 | 20/20 | 5 | < | 20/20 | 20/20 | 5 | < |
| Cefazolin | 50 | 20/20 | 20/20 | 3 | < | 19/20 | 19/20 | 3 | < | |
| Others | Chloramphenicol | 0,3 ^a | 19/20 | 19/20 | 4000 | > | 20/20 | 20/20 | 3500 | > |
| | Trimethoprim | 50 | 20/20 | 20/20 | 110 | > | 20/20 | 19/20 | 120 | > |
| | Dapsone | 5 ^b | 20/20 | 20/20 | 10 | > | 20/20 | 20/20 | 10 | > |
| | Lincomycin | 150 | 20/20 | 20/20 | 275 | > | 20/20 | 20/20 | 220 | > |
| | Rifaximin | 60 | 40/40 | 40/40 | 40 | < | 40/40 | 40/40 | 40 | < |
| | Pirlimicin | 100 | 20/20 | 20/20 | 300 | < | 20/20 | 20/20 | 300 | < |
| | Clavulanic acid | 200 | 20/20 | 20/20 | 700 | > | 20/20 | 20/20 | 800 | > |

* No regulatory limit in milk

^a MRPL (Minimum Required Performance Limit)

^b MMPR (Minimum Method Performance Requirements)

3.3. Test for false positive results

3.3.1. Materials and methods

The blank raw milk was tested before using by Delvotest® T and other test (Bioeasy® β -lactam –tetracycline or Bioeasy® β -lactam – tetracycline – cefalexine) in duplicate.

93 samples of bulk tank commingled milks were tested by Delvotest® T kit with plates and 84 samples with ampoules. The false positive rate was determined at control time.

3.3.2. Results

On 93 samples analyzed with plates and 84 samples with ampoules, no false positive was detected in blank raw milk from different origin.

3.4. Applicability on individual cow milk

3.4.1. Materials and methods

10 blank milk from different origins were tested at control time.

At minimum 10 milk samples supplemented with antibiotics were tested. One or two representative compounds for each antibiotic family were spiked to its CC β level or above (maximum 20%) (**Table 4**). If one sample supplemented with antibiotic was negative, 10 additional samples were tested.

All samples were tested with two formats (ampoules and plates), incubated at control time and read visually and by Delvo®Scan. All samples were codified to be analysed in blind.

Table 4. List of compounds tested in applicability study

| Antibiotic family | Molecules | MRL in milk (ppb) | CC β validated for cow milk (ppb) | |
|-------------------|---------------------|-------------------|---|--------|
| | | | Ampoules | Plates |
| Penicillins | Amoxicillin | 4 | 2 | 2 |
| | Cloxacillin | 30 | 10 | 10 |
| Tetracyclines | Oxytetracycline | 100 | 100 | 80 |
| | Chlortetracycline | 100 | 150 | 150 |
| Sulfonamides | Sulfadimethoxine | 100 | 40 | 40 |
| | Sulfadiazine | 100 | 55 | 50 |
| Macrolides | Tylosin A | 50 | 35 | 35 |
| | Erythromycin A | 40 | 160 | 200 |
| Aminoglycosides | Dihydrostreptomycin | 200 | 700 | 800 |
| Cephalosporins | Cefalexin | 100 | 30 | 30 |
| Lincosamides | Lincomycin | 150 | 275 | 220 |

3.4.2. Results

Results of applicability on individual cow milk are presented in **Table 5**.

Table 5. Results of applicability on individual cow milk at control time

| Antibiotic family | Molecules tested | MRL in milk (ppb) | Cow milk | | | | Individual cow milk | | | |
|-------------------|----------------------|-------------------|------------------|---------------|------------------|---------------|---------------------------|---------------|---------------------------|---------------|
| | | | Ampoules | | Plates | | Ampoules | | Plates | |
| | | | CC β (ppb) | =, < or > MRL | CC β (ppb) | =, < or > MRL | Number of positive sample | Applicability | Number of positive sample | Applicability |
| Penicillins | Amoxicillin | 4 | 2 | < | 2 | < | 10/10 | YES | 10/10 | YES |
| | Cloxacillin | 30 | 10 | < | 10 | < | 10/10 | YES | 10/10 | YES |
| Tetracyclines | Oxytetracycline | 100 | 100 | > | 80 | < | 10/10 | YES | 10/10 | YES |
| | Chlortetracycline | 100 | 150 | > | 150 | > | 10/10 | YES | 10/10 | YES |
| Sulfonamides | Sulfadimethoxine | 100 | 40 | < | 40 | < | 10/10 | YES | 10/10 | YES |
| | Sulfadiazine | 100 | 55 | < | 50 | < | 10/10 | YES | 10/10 | YES |
| Macrolides | Tylosin A | 50 | 35 | < | 35 | < | 10/10 | YES | 10/10 | YES |
| | Erythromycin A | 40 | 160 | > | 200 | > | 10/10 | YES | 10/10 | YES |
| Aminoglycosides | Dihydro-streptomycin | 200 | 700 | > | 800 | > | 10/10 | YES | 10/10 | YES |
| Cephalosporins | Cefalexin | 100 | 30 | < | 30 | < | 10/10 | YES | 10/10 | YES |
| Lincosamides | Lincomycin | 150 | 275 | > | 220 | > | 19/20 | YES | 10/10 | YES |

3.4.3. Conclusion

No difference was observed in CC β between the results with commingled cow milk and with individual cow milk. Applicability of the method is verified with individual cow raw milk.

3.5. Robustness

3.5.1. Materials and methods

3.5.1.1. Study performed by ILVO (2012)

Both formats (ampoules and plates) were tested and read visually and by Delvo®Scan. The different parameters: somatic cells, fat content and protein content have been tested by ILVO lab in 2012 (Reybroeck, W. and Ooghe, S., 2012). The results were kept for this second renewal study.

3.5.1.2. Study performed by ACTALIA Cecalait (2020)

All incubations for the robustness study were performed at control time except for the variation in incubation time, where incubation time was fixed at 3h15.

Both formats (ampoules and plates) were tested and read visually and by Delvo®Scan.

One or two representative compounds for each antibiotic family were spiked to its CC β level or above (maximum 20%). Fresh raw cow milks were used to prepare positive pool samples. For each parameter tested, samples were tested with:

- At least 3 different blank raw milk samples
- At least 3 different raw milk samples spiked with antibiotics described in **Table 6**.

Table 6. List of compounds tested in robustness study

| Antibiotic families | Molecules | MRL in milk (ppb) | CC β validated for cow milk (ppb) | |
|---------------------|---------------------|-------------------|---|--------|
| | | | Ampoules | Plates |
| Penicillins | Amoxicillin | 4 | 2 | 2 |
| | Cloxacillin | 30 | 10 | 10 |
| Tetracyclines | Oxytetracycline | 100 | 100 | 80 |
| | Chlortetracycline | 100 | 150 | 150 |
| Sulfonamides | Sulfadimethoxine | 100 | 40 | 40 |
| | Sulfadiazine | 100 | 55 | 50 |
| Macrolides | Tylosin A | 50 | 35 | 35 |
| | Erythromycin A | 40 | 160 | 200 |
| Aminoglycosides | Dihydrostreptomycin | 200 | 700 | 800 |
| Cephalosporins | Cefalexin | 100 | 30 | 30 |
| Lincosamides | Lincomycin | 150 | 275 | 220 |

Reagents, sample volumes, temperatures and incubation times were tested:

- **Sample volume:** 110 μ L and 90 μ L *versus* 100 μ L
- **Incubation time:** 3h15 *versus* control time
- **Incubation temperature:** 66°C and 62°C *versus* 64°C
- **Delay in reading:** 15 min after incubation time with test at room temperature and 15 min after incubation time with test in cold water *versus* immediately after incubation

Different parameters were tested concerning composition of the milk. Parameters are following:

- **pH values:** 6 < pH < 6,3 and 7,1 < pH < 7,5 *versus* 6,6 < pH < 6,9
- **Total bacterial count:** TBC > 5.10⁵/mL *versus* TBC < 1.10⁵/mL
- **Frozen milk *versus* unfrozen milk:** frozen milk was thawed overnight at 4°C
- **Milk temperature:** cold milk (3 \pm 2°C) *versus* milk at 20 \pm 2°C.

Low pH was obtained by addition of lactic acid and high pH by addition of sodium hydroxide. Total bacterial counts were obtained after maturing of milk during 15 hours at 20°C.

3.5.2. Results

3.5.2.1. Study performed by ILVO (2012)

Both formats (ampoules and plates) were tested and read visually and by Delvo®Scan. This study showed that there was a high rate of false positive results for milk with a high somatic cells count and high fat content:

- 19,1% for a content of somatic cells between 5.10^5 and 10^6 /mL;
- 50% for a content of somatic cells $>10^6$ /mL.

For raw milk samples with a fat content $> 6\%$, rate of false positive was 14,9%.

There was no false positive for milk samples with a protein content $> 4\%$ and $< 3\%$ (**Table 7**).

Table 7. Conclusion of robustness study by ILVO (2012)

| | Somatic cells between 5.10^5 and 1.10^6 /mL | Somatic cells $> 10^6$ /mL | Fat content $> 6\%$ | Protein content $> 4\%$ | Protein content $< 3\%$ |
|----------------------------|---|----------------------------|---------------------|-------------------------|-------------------------|
| Number of raw milk samples | 71 | 69 | 47 | 10 | 13 |
| Rate of false positive | 19,1% | 50% | 14,9% | No false positive | No false positive |

3.5.2.2. Complement of ILVO's study performed by ACTALIA Cecalait (2020)

The robustness study by ILVO lab showed high rate of false positive results (negative sample which gives positive result) when milk composition was out of the scope (somatic cells $> 5.10^5$ /mL and fat content $> 6\%$). These parameters (somatic cells and fat content) were not tested in ACTALIA Cecalait but some data were collected when milk samplings were not in the AFNOR scope for milk composition.

On 144 samples of bulk tank commingled sampling:

- no milk had a fat content higher than 6%;
- only 3 milks had a level higher than 5.10^5 somatic cells/mL. These samples were not used in this study, but blank milks were tested and were all negative (**Table 8**).

Table 8. Results of blank milk with high somatic cells content

| Dates | Somatic cells /mL | Ampoules batches | Plates batches | AMPOULES | | PLATES | |
|------------|-------------------|------------------|----------------|----------------|-------------------|----------------|-------------------|
| | | | | Visual reading | Delvoscan reading | Visual reading | Delvoscan reading |
| 27/04/2020 | 532 000 | 20A09/31 | 19K22/30 | - | - 6,13 | - | - 8,34 |
| | | 19L11/31 | 19J18/30 | - | - 4,34 | - | - 10,49 |
| | | 19L18/31 | 19J02/30 | - | - 2,23 | - | - 5,66 |
| 25/05/2020 | 655 000 | 20A09/31 | 19K22/30 | - | - 6,06 | - | - 5,39 |
| | | 19L11/31 | 19J18/30 | - | - 4,6 | - | - 8,07 |
| | | 19L18/31 | 19J02/30 | - | - 1,69 | - | - 3,42 |
| 04/06/2020 | 1 233 000 | 20A09/31 | 19K22/30 | - | - 5,82 | - | - 7,00 |
| | | 19L11/31 | 19J18/30 | - | - 4,82 | - | - 8,31 |
| | | 19L18/31 | 19J02/30 | - | - 0,85 | - | - 4,32 |

3.5.2.3. Study performed by ACTALIA Cecalait (2020)

Other parameters were tested in ACTALIA Cecalait.

Results of robustness on raw cow milk are presented in **Table 9**. All the results for ampoules and plates are presented in **Appendix 2**.

Table 9. Results of robustness study for both formats (ampoules and plates) read visually and by Delvo®Scan.

| | Robustness test | False positive results | False negative results | Conclusion |
|-------------------------------|----------------------------|------------------------|------------------------|---------------------|
| Sample volume | 90 µL | NO | NO | Robust |
| | 110 µL | NO | NO | |
| Incubation time | 3h15 | NO | YES * | Not robust * |
| Incubation temperature | 62°C | NO | NO | Robust |
| | 66°C | NO | NO | |
| Delay in reading | 15 min at room temperature | NO | NO | Robust |
| | 15 min at 4°C | NO | NO | |
| pH | Low (6,0 < pH < 6,3) | NO | YES** | Not robust** |
| | High (7,1 < pH < 7,5) | NO | NO | Robust |
| Total bacterial count | > 5.10 ⁵ /mL | NO | NO | Robust |
| Frozen milk | Frozen milk | NO | NO | Robust |
| Milk temperature | 3 ± 2°C | NO | NO | Robust |

*: There are false negative for 2 antibiotics (dihydrostreptomycin and lincomycin) on plates format only read visually and by Delvo®Scan.

** : There are false negative for 4 antibiotics (tylosin A, erythromycin, dihydrostreptomycin and lincomycin) on both formats (ampoules and plates) read visually and by Delvo®Scan.

3.5.3. Conclusion of robustness

All robustness study was carried out at the control time of 2h55 for the 2 batches used (20D21/30 and 20D16/30). Only for robustness of incubation time, it was replaced by 3h15.

For all parameters tested, no false positive results were observed

For 6 parameters tested (sample volume, incubation temperature, delay in reading, total bacterial count, frozen milk and milk temperature) no false negative was observed.

False negative results were observed for the robustness parameter incubation time (incubation of 3h15 instead of control time of 2h55), on 2 antibiotics (dihydrostreptomycin and lincomycin) and only with plates format read visually and using Delvo®Scan. Nevertheless, the CCβ of these antibiotics are higher than the Maximum Residue Limit (MRL).

False negative results were also obtained with incubation at control time of milk samples with low pH ($6 < \text{pH} < 6,3$) on 4 antibiotics (tylosin A, erythromycin, dihydrostreptomycin and lincomycin) with both formats (plates and ampoules) read visually and using Delvo®Scan. Nevertheless, during the detection capability study the pH of the 144 samples tested was never lower than 6,5. We can notice also that the $\text{CC}\beta$ of 3 antibiotics of them (erythromycin, dihydrostreptomycin and lincomycin) were higher than the Maximum Residue Limit (MRL).

During the detection capability study, no difference was detected between different batches including reagent close to the manufactory or expiry dates. We can conclude that age of batches for plates and ampoules, has no impact on the data.

3.6 Practicability

The practicability of the alternative method was evaluated according to the 12 criteria defined in AFNOR rules (**Table 10**).

Table 10. Practicability's criteria of alternative method

| N° | Criteria | Communication of criteria | Results of expert lab | |
|----|---|----------------------------|---|---|
| | | | AMPOULES | PLATES |
| 1 | Reagent packaging | Kit wrapping or kit insert | Cardboard box with kit insert in 6 languages and colorcard for results interpretation | Cardboard box with kit insert in 6 languages and colorcard for results interpretation (in 5 and 20 packs) + adhesive tape |
| 2 | Reagent volume | Kit wrapping or kit insert | 1 or 4 packages of 25 ampoules | 5, 20 or 80 plates of 96 samples divisible in 6 x 16 samples per plate |
| 3 | Storage conditions (+ expiry date) | Kit wrapping or kit insert | Sense of storage, storage temperature (2-8°C), away from the light, protect from freezing, expiry date and batch number | |
| 4 | Use after one use | Kit wrapping or kit insert | To prevent from freezing and between 2-8°C | |
| 5 | Equipment or specific places | Kit insert | DSM incubator or water bath | |
| 6 | Reagent (ready to use or to reconstitute) | Kit wrapping or kit insert | Kit ready to use; Control to reconstitute for determination of control time | |
| 7 | Training time | Report | Half day including incubation time | |
| 8 | Real time of manipulation | Report | Sample inoculation is very short (few seconds) Incubation time for raw cow milk : 3h00 ± 15 min | |
| 9 | Delay to obtain results | Report | Between 3h00 and 3h30 from the beginning to reading | |
| 10 | Operator's qualification | Report | Laboratory agent | |
| 11 | Results traceability | Kit insert | Printing of result report, save csv and picture file with Delvo@Scan software | |
| 12 | Maintenance | Report | Monthly calibration of Delvo@Scan | |

3.7 Bibliography

2 publications were published.

- A study had compared 9 microbial inhibitor tests including Delvotest® T on milk samples from 200 different individual goats (Romero *et al.*, 2016).

Results were based on visual and instrumental reading. For Delvotest® T, specificity with instrumental reading was 99,5% and 98,5% with visual reading. This result shows that Delvotest® T is suitable for goat milk. This study shows that fat removal followed by heat treatment is the most appropriate milk treatment to reduce false positive results for almost all tests except for the Delvotest® T. Delvotest® T was not influenced by the milk pre-treatment.

- Another study has used Delvotest® T for a search of antibiotic residues in commercialized milk in Constantine region (North East Algeria) (Boultif *et al.*, 2016). Sampling was cow milk produced locally as well as imported milk powder. A total 180 samples were analysed (120 samples of local milk and 60 samples of imported milk).

Delvotest® T data showed that 40% of milk samples (25% positive and 15% doubtful samples) produced locally were contaminated with antibiotic residues. For milk powder, Delvotest® T revealed 5% (3% positive and 2% doubtful samples) of samples contaminated with antibiotic residues.

3.8 Conclusion of the preliminary study

The rules of AFNOR have been changed in 2017 according to NF102 (AFNOR Certification): 'Protocol for validation of methods for the detection and quantification of veterinary drugs in food products' (revision N°1: June 1st, 2017), so the preliminary study was performed again in the second renewal study in 2020 according to these new rules.

The Delvo®Scan software used was the version 5.08 (ampoules and plates) with an EPSON V600 scanner with a cut-off equal to 0.

All incubations of this renewal study were performed at control time except when the incubation time was tested in robustness (incubation at 3h15).

The results of the preliminary study on Delvotest® T with 2 formats (plates and ampoules) and 2 readings (visual and Delvo®Scan) were:

- No false positive detected with plates and ampoules;
- Detection capabilities determined at control time on 42 antibiotics;
- Several parameters tested in the robustness study: sample volume, incubation time, incubation temperature, delay in reading, pH, total bacterial count, frozen milk, milk temperature and age of batches.
 - No false positive results were observed

- False negative results were observed for 2 robustness parameters:
 - **Incubation time of 3h15** for dihydrostreptomycin and lincomycin, only on plates read visually and by Delvo®Scan.
 - **Low pH** for tylosin A, erythromycin, dihydrostreptomycin and lincomycin by ampoules and plates read visually and by Delvo®Scan.
- In detection capability study, no difference was detected between different batches for plates and ampoules.
- The robustness study performed by ILVO lab showed high rate of false positive results when milk composition was not in conformity (somatic cells > 5.10⁵ /mL and fat content > 6%), but in the samples of the second renewal study performed by ACTALIA Cecalait no false positive results was detected.

- Applicability on individual cow milk was verified with plates and ampoules, read visually and by Delvo®Scan.

- The complaints observed and the bibliography study did not lead to a protocol modification or exclusion of material.

- No modification of the method was observed since the first validation study.

3.9. User complaints

From 2016 to 2019, DSM recorded complaints concerning all the formats (ampoules and plates) on around 500 batches per year. In that period, DSM received 101 complaints, 88 complaints were considered by DSM as justified. The distributions of overall and justified complaints are presented in **Table 14**.

Concerning justified complaints, the most common found were performance and appearance of products (48 complaints of 88 complaints). Complaints are mainly related to the formats: 100 ampoules and 20 plates. These complaints did not lead to a protocol modification or exclusion of material. Details of complaints are presented in **Appendix 15**.

Table 14. Types of complaints for Delvotest® T from 2016 to 2019

| Overall complaints | | | | |
|-------------------------|------------|--------------------------------|--------------------------|-------------------------|
| Format | Overall | Logistic (packaging, delivery) | Performance / Appearance | Administrative / Others |
| 25 ampoules | 11 | 3 | 4 | 4 |
| 100 ampoules | 51 | 13 | 28 | 10 |
| 5 plates | 10 | 2 | 7 | 1 |
| 20 plates | 29 | 8 | 20 | 1 |
| Total complaints | 101 | 26 | 59 | 16 |

| Justified complaints | | | | |
|-----------------------------|----------------|---|-------------------------------------|------------------------------------|
| Format | Overall | Logistic (packaging, delivery) | Performance / Appearance | Administrative / Others |
| 25 ampoules | 9 | 3 | 3 | 3 |
| 100 ampoules | 46 | 12 | 24 | 10 |
| 5 plates | 8 | 2 | 5 | 1 |
| 20 plates | 25 | 8 | 16 | 1 |
| Total complaints | 88 | 25 | 48 | 15 |

4. INTERLABORATORY STUDY

A first interlaboratory study was conducted by the expert laboratory ANSES Fougères in 2013.

According to the new AFNOR rules of 2017, for a broad spectrum test 6 antibiotics must be tested in an interlaboratory study.

4.1. Interlaboratory study in 2013 (ANSES Fougères)

9 laboratories participated to this interlaboratory study. The choice of concentration was based on MRL and the results of phase 1 of preliminary study (sensitivity of reference method and alternative method).

Each material was prepared in double-blind, codified, such as 56 samples were sent to be analysed by each laboratory. In addition, 4 negative controls were provided to laboratory to determinate optimum incubation time: 1 cow milk, 1 ewe milk, 1 goat milk and 1 cow milk with azidiol. Milk samples were sent frozen. The **Table 12** presents the samples prepared for the interlaboratory study.

Table 12. Samples of the interlaboratory study in 2013

| Preservative | With azidiol | Without azidiol | | | | | |
|----------------------|--------------|-----------------|---------|---------|------------|--------------|-----------|
| Antibiotics | Penicillin G | Penicillin G | | | Cefquinome | Tetracycline | Tylosin A |
| MRL | 4 | 4 | 4 | 4 | 20 | 100 | 50 |
| Species | Cow | Cow | Ewe | Goat | Cow | | |
| Concentrations (ppb) | 'Blank' | 'Blank' | 'Blank' | 'Blank' | 'Blank' | 'Blank' | 'Blank' |
| | 1 | 1 | 1 | 1 | 20 | 40 | 20 |
| | 4 | 4 | 4 | 4 | 80 | 200 | 50 |
| | 6 | 6 | 6 | 6 | 300 | 300 | 300 |

The results of expert laboratory were:

- 1- 14 on 14 blank milks were negative (L0);
- 2- Below the supposed detection limit (L1), 12 on 14 samples were negative;
- 3- At the supposed detection limit (L2), all samples were positive;
- 4- Above the supposed detection limit (L3), all samples were positive.

All results of this study are presented in **Appendix 3**, and were satisfactory.

8 or 9 laboratories were retained for results interpretation. Overall for sensitivity study, results of the interlaboratory study are similar to results of the preliminary study.

With all these data, two formats of Delvotest® T have been validated:

- Ampoules with visual reading
- Plates with visual and Delvo®Scan reading

4.2. Interlaboratory study in 2021 (ACTALIA-Cecalait)

To answer to AFNOR requirements, 3 antibiotics left have to be tested. AFNOR Technical Board asked to test again **Tetracycline** (Tetracycline) because the detection capabilities changed compared to the last study. The 2 antibiotics left were selected for their antibiotic family and for a practicability reason, because they have the same CC β for the 2 formats of Delvotest® T (ampoule and plate): **Sulfadimethoxine** (Sulfonamide) and **Gentamycin** (Aminoglycoside).

Before using Delvotest® T (ampoules or plates) for the interlaboratory study, collaborative laboratories had to determine the control time of each batch of Delvotest® T.

All analyses were performed at control time. The Delvo®Scan software used was the version 5.08 (ampoules and plates) with an EPSON V600 scanner and the cut-off was equal to 0.

The detection capabilities of antibiotics tested in 2013 (penicillin G, cefquinome and tylosin A) were equivalent to these obtained in the renewal study in 2020, except for tetracycline (**Table 11**). Results of penicillin G, cefquinome and tylosin A of the first interlaboratory study in 2013

were kept and completed with 3 antibiotics during the new interlaboratory study in 2021 conducted by ACTALIA Cecalait: tetracycline (tetracycline), sulfadimethoxine (sulfonamide) and gentamycin (aminoglycoside).

Table 11. Detection capabilities determined in 2013 and 2020

| Antibiotic families | Antibiotics | LMR (ppb) | CC β (ppb) determined by ANSES in 2013 | | CC β (ppb) determined by ACTALIA in 2020 | |
|---------------------|--------------|-----------|--|--------|--|--------|
| | | | Ampoules | Plates | Ampoules | Plates |
| β -lactams | Penicillin G | 4 | 4 | 2 | 3 | 1 |
| | Cefquinome | 20 | 40 | 40 | 50 | 60 |
| Tetracycline | Tetracycline | 100 | 200 | 200 | 80 | 100 |
| Macrolide | Tylosin A | 50 | 50 | 50 | 35 | 35 |

4.2.1 Preparation of samples

Specifications of the blank raw milk:

- Be used within 36 hours after sampling;
- Be stored between 0 and 6°C;
- Contain at least the milk of 10 animals, without treatment during at least 8 weeks before sampling;
- Have a milk composition corresponding to AFNOR rules.

The commingled raw cow milk was tested to confirm the absence of antibiotic by:

- 3 BioEasy® tests performed in duplicate:
 - o BioEasy® β -lactam – Tetracycline – Cefalexine (reference: 763.000008.40);
 - o BioEasy® Sulphonamide (reference: 763.001024.10);
 - o BioEasy® Gentamycin (reference: 763.001007.05).
- 2 formats of Delvotest® T (ampoules and plates) in duplicate.

The raw cow milk was sampled the day of samples preparation and its composition was in conformity.

Antibiotic stock solutions were prepared the day of samples preparation. Details of antibiotics used are presented in **Appendix 4** (brand, reference and batch).

For each antibiotic, blank milk was spiked at 4 levels:

- L0: Antibiotic-free sample
- L1: Sample spiked at 50% of the CC β
- L2: Sample spiked at +20% of CC β
- L3: Sample spiked at +50% of CC β

The **Table 13** presents the list of antibiotics and their concentrations tested with Delvotest® T in ampoules and plates formats.

Table 13. List of antibiotics tested

| Antibiotic families | Antibiotics | MRL (ppb) | Delvotest® T format | CCβ (ppb) | Concentration levels tested (ppb) | | | |
|---------------------|------------------|-----------|---------------------|-----------|-----------------------------------|----|-----|-----|
| | | | | | L0 | L1 | L2 | L3 |
| Sulfonamide | Sulfadimethoxine | 100 | Ampoules and plates | 40 | 0 | 20 | 48 | 60 |
| Aminoglycoside | Gentamycin | 100 | Ampoules and plates | 100 | 0 | 50 | 120 | 150 |
| Tetracycline | Tetracycline | 100 | Ampoules | 80 | 0 | 40 | 96 | 120 |
| | | | Plates | 100 | 0 | 50 | 120 | 150 |

The collaborative laboratories also received 2 negative and 1 positive controls. The first negative control was provided by DSM Food Specialties in the same time as tests necessary for this study. It was used to determine the control time of each batch of Delvotest® T (ampoules and plates). The second negative control was milk without antibiotic. The positive control was a sample at L3 level of gentamycin. These 2 controls (1 negative and 1 positive) were used to validate the run.

Spiked samples were distributed under agitation in tubes codified in blind duplicates. Milk samples (samples and controls) were frozen at -80°C for 1 day and then stored at -20°C until the shipment.

To sum up, each laboratory received from ACTALIA Cecalait:

- 1 negative control and 1 positive control to analyze in twice, with 2 formats (ampoules and plates) and with 2 types of reading (visual and by Delvo®Scan).
- 32 samples to analyze in twice with 2 types of reading (visual and by Delvo®Scan)

All laboratories had to perform analyses the same day, 7 days after sending (20/04/2021).

4.2.2. verification of homogeneity and stability

Homogeneity and stability of milk samples were verified at control time with 2 formats of Delvotest® T (ampoules and plates) and interpreted from Delvo®Scan results (Z-values)

4.2.2.1. Homogeneity

Homogeneity was performed with 10 samples spiked at L3 level for each antibiotic in duplicate. These samples were selected randomly during the distribution. For each sample, visual and Delvo®Scan readings were performed. For each antibiotic, a mean, a standard deviation and a coefficient of variation were calculated from the Z-values (Delvo®Scan results).

All results were positives. Raw data are presented in **Appendix 5**. The calculations from Delvo®Scan results are presented in **Table 14**. The higher standard deviation is 0,56 and the

higher coefficient of variation is 10,5 %. From these results it can be concluded that the homogeneity of samples is verified.

Table 14. Control of homogeneity

| DELVOTEST® T - AMPOULES | | | |
|--------------------------|-------------------------|------------------------------|---------------------------|
| Antibiotics | Gentamycin (120 ppb) | Sulfadimethoxine (48 ppb) | Tetracycline (96 ppb) |
| Mean of Z-values | 6,21 | 3,47 | 4,53 |
| Standard deviation | 0,34 | 0,36 | 0,38 |
| Coefficient of variation | 5,42 % | 10,51 % | 8,30 % |
| DELVOTEST® T - PLATES | | | |
| Antibiotics | Gentamycin (120 ppb) | Sulfadimethoxine (48 ppb) | Tetracycline (120 ppb) |
| Mean of Z-values | 5,83 | 4,63 | 5,60 |
| Standard deviation | 0,56 | 0,45 | 0,47 |
| Coefficient of variation | 9,57 % | 9,75 % | 8,40 % |

4.2.2.2. Stability

Samples stability was performed with 3 samples spiked at L3 level for each antibiotic. These samples were analyzed in duplicate at 3 different times: after 24 hours in freezer (T1), the day of samples shipment (T2) and the day of samples analyses (T3). For each sample, visual and Delvo®Scan readings were performed. For each antibiotic, a mean of the Z-values was calculated

Raw data are presented in **Appendix 6**. The means of Z-values are presented in **Table 15** and **Figure 1**. All readings (visual and by Delvo®Scan) were positives. A low decrease of Z-values was noticed for all antibiotics for plates format at the third point of stability (day of sample analysis), but had no impact because all laboratories performed analyses the same day. It can be concluded that the stability of samples is satisfactory.

Table 15. Control of stability based on interpretation of Z-values (not quantitative data)

| DELVOTEST® T - AMPOULE | | | |
|------------------------|----------------------|---------------------------|------------------------|
| Mean of Z-values | Gentamycin (120 ppb) | Sulfadimethoxine (48 ppb) | Tetracycline (96 ppb) |
| T0 | 6,05 | 3,40 | 4,47 |
| T1 | 6,62 | 4,54 | 4,95 |
| T2 | 6,79 | 4,64 | 5,07 |
| DELVOTEST® T - PLATE | | | |
| Mean of Z-values | Gentamycin (120 ppb) | Sulfadimethoxine (48 ppb) | Tetracycline (120 ppb) |
| T0 | 6,56 | 5,62 | 6,14 |
| T1 | 6,72 | 6,02 | 6,66 |
| T2 | 4,60 | 4,08 | 3,87 |

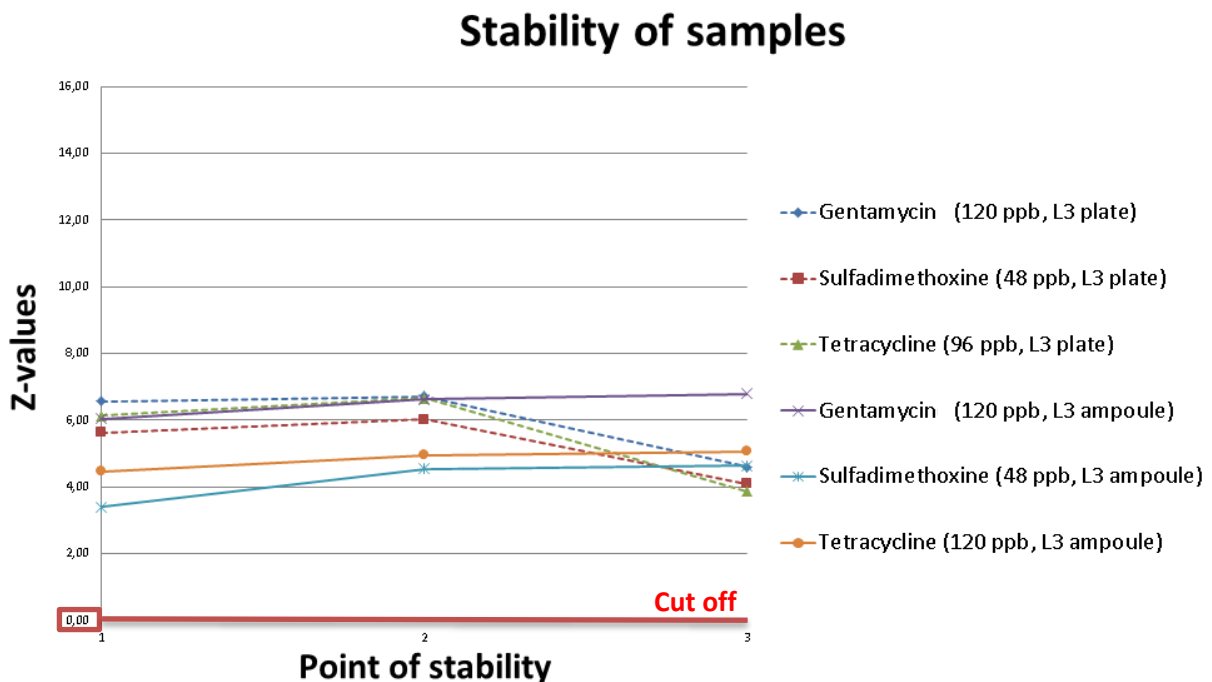


Figure 1. Stability of samples

4.2.3 Shipment

Frozen samples were sent to the collaborative laboratories on dry ice. Upon receipt, collaborative laboratories checked the temperature indicator (Giovatemp +2/3°C; reference P2-3; C.C.F Technologies) and checked if samples were still frozen.

ACTALIA Cecalait performed analysis on the same samples as collaborative laboratories, but results were not included in the interpretation of the interlaboratory study. To be in the same conditions of travel, samples were also kept on dry ice in a package at room temperature.

4.2.4. Exclusion of laboratories

In order to obtain at least 8 valid sets of results, 11 laboratories were in the list of collaborative laboratories.

On these 11 collaborative laboratories, few days before the interlaboratory study, the **laboratory 11** announced that it could not participate.

The collaborative **laboratory 10** did not receive its package, that was held at customs.

For other participants (including the expert laboratory), milk samples were still frozen at receipt without temperature alarm.

According to AFNOR rules, some results can be excluded of interpretation of the interlaboratory study. In this case, the reason of exclusion has to be explained in the report. Collaborative laboratories knew the conditions of exclusion in the protocol, which are:

- Discordance in results of controls (if positive control is negative or negative control is positive);
- Non respect of storage temperature during the shipment (defrosting of samples) and in the collaborative laboratory (positive cold storage before the protocol of defrosting);
- Non respect of the day analyses (20/04/2021).

In this study, 2 collaborative laboratories were excluded:

- The collaborative **laboratory 9** did not respect the protocol of the method. This laboratory did not determine the control time, as required in the instructions and during the training, and used an incubation time of 3h15. This over incubation leads to a decrease of Z-values (around -15 instead of -7/ -6 for blank milk) and by extension a loss of sensitivity;
- The collaborative **laboratory 5** had problem with its software for ampoules the day of samples analyses, despite the fact that the software was correctly installed and was already used before to determine the control time. For results with ampoules format, only data from visual reading were interpretable.

To overcome the high number of missing data, the collaborative **laboratory 7** kindly accepted to participate with a second collaborator, on a different set of samples and in the same conditions as other collaborative laboratories.

All laboratories performed analyses the same day.

Therefore, 9 sets of results were interpreted in this report (including 1 set without results read by Delvo®Scan on ampoules format).

All information about the shipment, reception of samples and reasons of exclusion are presented in the **Table 16**.

Table 16. Conditions of reception and exclusion of collaborative laboratories

| Collaborative laboratories | Labs identification | Alarm of temperature indicator | Receipt dates | Reasons of exclusion |
|----------------------------|---------------------|--------------------------------|---------------|---|
| 1 | 1 | No | 14/04/2021 | No |
| 2 | 2 | No | 14/04/2021 | No |
| 3 | 3 | No | 14/04/2021 | No |
| 4 | 4 | No | 14/04/2021 | No |
| 5 | 5 | No | 14/04/2021 | No |
| 6 | 6 | No | 14/04/2021 | No |
| 7 Collaborator A | 7 | No | 13/04/2021 | No |
| 7 Collaborator B | 9 | No | 19/04/2021 | No |
| 8 | 8 | No | 15/04/2021 | Software problem, no results with ampoules |
| 9 | / | No | 14/04/2021 | No determination of the control time |
| 10 | / | / | / | Package held at customs |
| 11 | / | / | / | Withdrawal of participation before sending |

For each antibiotic, the results with visual and by Delvo®Scan readings are presented in **Appendix 7** for ampoules and plates formats. Raw data of the expert laboratory are included in these tables and are consistent to expected results, but were not taken into account for the statistic evaluation (specificity, selectivity, repeatability and reproducibility)

4.3. Calculation on both interlaboratory studies (2013 and 2021)

4.3.1. Specificity, percentage of positive results and sensitivity

Based on these data, several parameters were calculated:

- **Specificity** (SP, %) of the method, with the following equation:

$$SP (\%) = (1 - (P_0/N_0)) \times 100$$

where P_0 is the number of positive results at level L0

N_0 is the total of results at level L0

- **Percentage of positive results** at level L1 (PR₁, %), with the following equation:

$$PR_1 (\%) = P_1/N_1 \times 100$$

where P₁ is the number of positive results at level L1

N₁ is the total of results at level L1

- **Sensitivity** (SE, %) for each level L2 and L3, with the following equation:

$$SE_{L2} (\%) = (P_2/N_2) \times 100$$

where P₂ is the number of positive results at level L2

N₂ is the total of results at level L2

$$SE_{L3} (\%) = (P_3/N_3) \times 100$$

where P₃ is the number of positive results at level L3

N₃ is the total of results at level L3

- **Global sensitivity** (GSE, %) for the levels L2 + L3, with the following equation:

$$GSE (\%) = (P/N) \times 100$$

where P is the number of positive results at the levels L2 and L3

N is the total of results at the levels L2 and L3

Interpretation of global results from both interlaboratory studies of 2013 and 2021 (Tables 17 and 18):

-Ampoules format:

- Specificity:

The specificity was very satisfactory, 100 % obtained for each type of reading.

- Sensitivity:

At level L1, positive results were between 39 and 47 %.

The sensitivity of levels L2, L3 and L2+L3 was very satisfactory, 100 % obtained for each type of reading.

- Plates format:

- Specificity

The specificity was satisfactory (100 % for visual reading and 97 % for Delvo®Scan reading).

- Sensitivity

At level L1, positive results were between 25 and 44 %.

The sensitivity at levels L2 and L3 were close to 100 % (L2 > 96% and L3 > 98 %). Therefore, the global sensitivity was also satisfactory (98 % for visual reading and 97 % for Delvo®Scan reading).

Table 17. Specificity, percentage of positive results, sensitivity and global sensitivity for both interlaboratory studies (2013 and 2021) with Delvotest® T in ampoules format.

| Delvotest® T formats | AMPOULES | | | | | | | | |
|---|----------|---------|---------|---------|---------|---------|------------|---------|---------|
| | Visual | | | | | | Delvo®Scan | | |
| Readings | P | C | Ty | G | S | T | G | S | T |
| Antibiotics | | | | | | | | | |
| Number of positive results (L0) / Total of results (L0) | 0 / 32 | 0 / 32 | 0 / 32 | 0 / 36 | 0 / 36 | 0 / 36 | 0 / 32 | 0 / 32 | 0 / 32 |
| Specificity per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 |
| Specificity L0 (%) | 100,0 | | | | | | 100,0 | | |
| Number of positive results (L1) / Total of results (L1) | 2 / 32 | 2 / 32 | 15 / 32 | 32 / 36 | 28 / 36 | 15 / 36 | 20 / 32 | 13 / 32 | 5 / 32 |
| Positive results L1 per antibiotic (%) | 6,3 | 6,3 | 46,9 | 88,9 | 77,8 | 41,7 | 62,5 | 40,6 | 15,6 |
| Positive results L1 (%) | 46,1 | | | | | | 39,6 | | |
| Number of positive results (L2) / Total of results (L2) | 32 / 32 | 32 / 32 | 32 / 32 | 36 / 36 | 36 / 36 | 36 / 36 | 32 / 32 | 32 / 32 | 32 / 32 |
| Positive results L2 per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 |
| Sensitivity L2 (%) | 100,0 | | | | | | 100,0 | | |
| Number of positive results (L3) / Total of results (L3) | 32 / 32 | 32 / 32 | 32 / 32 | 36 / 36 | 36 / 36 | 36 / 36 | 32 / 32 | 32 / 32 | 32 / 32 |
| Positive results L3 per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 |
| Sensitivity L3 (%) | 100,0 | | | | | | 100,0 | | |
| Number of positive results (L2+L3) / Total of results (L2+L3) | 64 / 64 | 64 / 64 | 64 / 64 | 72 / 72 | 72 / 72 | 72 / 72 | 64 / 64 | 64 / 64 | 64 / 64 |
| Positive results L2+L3 per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 |
| Global sensitivity L2+L3 (%) | 100,0 | | | | | | 100,0 | | |

P = Penicillin G; C = Cefquinome; Ty = Tylosin A; G = Gentamycin; S = Sulfadimethoxine; T = Tetracycline.

Table 18. Specificity, percentage of positive results, sensitivity and global sensitivity for both interlaboratory studies (2013 and 2021) with Delvotest® T in plates format.

| Delvotest® T formats | PLATES | | | | | | | | | | | |
|---|-------------|---------|---------|---------|---------|---------|-------------|---------|---------|---------|---------|---------|
| | Visual | | | | | | Delvo®Scan | | | | | |
| Readings | P | C | Ty | G | S | T | P | C | Ty | G | S | T |
| Antibiotics | | | | | | | | | | | | |
| Number of positive results (L0) / Total of results (L0) | 0 / 36 | 0 / 36 | 0 / 36 | 0 / 36 | 0 / 36 | 0 / 34 | 2 / 36 | 1 / 36 | 1 / 36 | 0 / 36 | 0 / 36 | 0 / 36 |
| Specificity per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 94,4 | 97,2 | 97,2 | 100,0 | 100,0 | 100,0 |
| Specificity L0 (%) | 100,0 | | | | | | 97,8 | | | | | |
| Number of positive results (L1) / Total of results (L1) | 6 / 36 | 11 / 36 | 24 / 36 | 12 / 36 | 28 / 36 | 13 / 36 | 4 / 36 | 8 / 36 | 16 / 36 | 5 / 36 | 16 / 36 | 6 / 36 |
| Positive results L1 per antibiotic (%) | 16,7 | 30,6 | 66,7 | 33,3 | 77,8 | 36,1 | 11,1 | 22,2 | 44,4 | 13,9 | 44,4 | 16,7 |
| Positive results L1 (%) | 43,5 | | | | | | 25,5 | | | | | |
| Number of positive results (L2) / Total of results (L2) | 32 / 36 | 36 / 36 | 34 / 36 | 36 / 36 | 36 / 36 | 36 / 36 | 34 / 36 | 33 / 36 | 35 / 36 | 36 / 36 | 35 / 36 | 36 / 36 |
| Positive results L2 per antibiotic (%) | 88,9 | 100,0 | 94,4 | 100,0 | 100,0 | 100,0 | 94,4 | 91,7 | 97,2 | 100,0 | 97,2 | 100,0 |
| Sensitivity L2 (%) | 97,2 | | | | | | 96,8 | | | | | |
| Number of positive results (L3) / Total of results (L3) | 36 / 36 | 36 / 36 | 36 / 36 | 36 / 36 | 34 / 36 | 36 / 36 | 35 / 36 | 34 / 36 | 35 / 36 | 36 / 36 | 36 / 36 | 36 / 36 |
| Positive results L3 per antibiotic (%) | 100,0 | 100,0 | 100,0 | 100,0 | 94,4 | 100,0 | 97,2 | 94,4 | 97,2 | 100,0 | 100,0 | 100,0 |
| Sensitivity L3 (%) | 99,1 | | | | | | 98,1 | | | | | |
| Number of positive results (L2+L3) / Total of results (L2+L3) | 68 / 72 | 72 / 72 | 70 / 72 | 72 / 72 | 70 / 72 | 72 / 72 | 69 / 72 | 67 / 72 | 70 / 72 | 72 / 72 | 71 / 72 | 72 / 72 |
| Positive results L2+L3 per antibiotic (%) | 94,4 | 100,0 | 97,2 | 100,0 | 97,2 | 100,0 | 95,8 | 93,1 | 97,2 | 100,0 | 98,6 | 100,0 |
| Global sensitivity L2+L3 (%) | 98,1 | | | | | | 97,5 | | | | | |

P = Penicillin G; C = Cefquinome; Ty = Tylosin A; G = Gentamycin; S = Sulfadimethoxine; T = Tetracycline.

4.3.2. Repeatability

Two types of repeatability (%) were calculated for each laboratory:

- The comparison between duplicates of the same sample;
- The comparison between two identical samples.

Tables 19 to 22 present the repeatability of samples used during the interlaboratory study performed by ANSES in 2013 and **Tables 23 to 26** by ACTALIA Cecalait in 2021.

Repeatability is between 90 % and 100 % for each sample (blank or spiked milks), each format, each type of reading and each type of repeatability.

Only repeatability between two identical samples were around 80% for samples spiked with:

- Sulfadimethoxine with Delvo®Scan reading in plate format;
- Cefquinome with visual reading in plate format.

Table 19. Repeatability of blank milk (ANSES, 2013).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | |
|--------------------------|--|--------------|-------------|---|--------------|-------------|
| | AMPOULES | PLATES | | AMPOULES | PLATES | |
| | Visual | Visual | Delvo®Scan | Visual | Visual | Delvo®Scan |
| AA | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AB | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AD | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AE | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AF | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AG | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AH | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 | 8/8 |
| AI | Excluded (1 analysis vs 2) | 8/8 | 8/8 | Excluded (1 analysis vs 2) | 8/8 | 8/8 |
| AK | 8/8 | 8/8 | 2/8 | 8/8 | 8/8 | 2/8 |
| Total | 64/64 | 72/72 | 66/72 | 64/64 | 72/72 | 66/72 |
| Repeatability (%) | 100,0 | 100,0 | 91,7 | 100,0 | 100,0 | 91,7 |

Table 20. Repeatability of penicillin G (ANSES, 2013).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | |
|--------------------------|--|-------------|-------------|---|-------------|-------------|
| | AMPOULES | PLATES | | AMPOULES | PLATES | |
| | Visual | Visual | Delvo®Scan | Visual | Visual | Delvo®Scan |
| AA | 6/6 | 6/6 | 5/6 | 6/6 | 6/6 | 5/6 |
| AB | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AD | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AE | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AF | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AG | 6/6 | 6/6 | 6/6 | 4/6 | 6/6 | 6/6 |
| AH | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 4/6 |
| AI | Excluded (1 analysis vs 2) | 6/6 | 6/6 | Excluded (1 analysis vs 2) | 4/6 | 4/6 |
| AK | 6/6 | 2/6 | 6/6 | 6/6 | 4/6 | 6/6 |
| Total | 48/48 | 50/54 | 53/54 | 46/48 | 50/54 | 49/54 |
| Repeatability (%) | 100,0 | 92,6 | 98,1 | 95,8 | 92,6 | 90,7 |

Table 21. Repeatability of cefquinome (ANSES, 2013).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | |
|--------------------------|---|-------------|-------------|--|-------------|-------------|
| | AMPOULES | PLATES | | AMPOULES | PLATES | |
| | Visual | Visual | Delvo®Scan | Visual | Visual | Delvo®Scan |
| AA | 6/6 | 6/6 | 5/6 | 6/6 | 4/6 | 5/6 |
| AB | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AD | 6/6 | 6/6 | 6/6 | 6/6 | 4/6 | 6/6 |
| AE | 6/6 | 5/6 | 6/6 | 6/6 | 5/6 | 6/6 |
| AF | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AG | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AH | 6/6 | 5/6 | 6/6 | 4/6 | 4/6 | 6/6 |
| AI | Excluded (1 analysis vs 2) | 6/6 | 6/6 | Excluded (1 analysis vs 2) | 6/6 | 6/6 |
| AK | 6/6 | 4/6 | 6/6 | 6/6 | 4/6 | 6/6 |
| Total | 48/48 | 50/54 | 53/54 | 46/48 | 45/54 | 53/54 |
| Repeatability (%) | 100,0 | 92,6 | 98,1 | 95,8 | 83,3 | 98,1 |

Table 22. Repeatability of tylosin A (ANSES, 2013).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | |
|--------------------------|---|-------------|--------------|--|-------------|-------------|
| | AMPOULES | PLATES | | AMPOULES | PLATES | |
| | Visual | Visual | Delvo®Scan | Visual | Visual | Delvo®Scan |
| AA | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AB | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AD | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 4/6 |
| AE | 5/6 | 6/6 | 6/6 | 5/6 | 6/6 | 6/6 |
| AF | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AG | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 | 6/6 |
| AH | 6/6 | 6/6 | 6/6 | 4/6 | 4/6 | 6/6 |
| AI | Excluded (1 analysis vs 2) | 6/6 | 6/6 | Excluded (1 analysis vs 2) | 6/6 | 6/6 |
| AK | 6/6 | 4/6 | 6/6 | 6/6 | 4/6 | 4/6 |
| Total | 47/48 | 52/54 | 54/54 | 45/48 | 50/54 | 50/54 |
| Repeatability (%) | 97,9 | 96,3 | 100,0 | 93,8 | 92,6 | 92,6 |

Table 23. Repeatability of blank milk (ACTALIA Cecalait, 2021).

| Labs | Number of analysis for same sample / Number of total sample) x 100 (%) | | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | | |
|--------------------------|---|------------------|--------------|--------------|---|------------------|--------------|--------------|
| | AMPOULES | | PLATES | | AMPOULES | | PLATES | |
| | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan |
| ACTALIA | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 1 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 2 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 3 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 4 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 5 | 6/6 | Software problem | 6/6 | 6/6 | 3/3 | Software problem | 3/3 | 3/3 |
| 6 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 7 | 6/6 | 6/6 | 5/5 | 5/5 | 3/3 | 3/3 | 2/2 | 2/2 |
| 8 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 9 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| Total | 54/54 | 48/48 | 53/53 | 53/53 | 27/27 | 24/24 | 26/26 | 26/26 |
| Repeatability (%) | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 |

Table 24. Repeatability of gentamycin (ACTALIA Cecalait, 2021).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | | |
|--------------------------|--|------------------|--------------|-------------|---|------------------|--------------|-------------|
| | AMPOULES | | PLATES | | AMPOULES | | PLATES | |
| | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan |
| ACTALIA | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 1 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 2 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 3 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 2/3 | 3/3 | 3/3 |
| 4 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 5 | 6/6 | Software problem | 6/6 | 6/6 | 3/3 | Software problem | 3/3 | 3/3 |
| 6 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 7 | 6/6 | 4/6 | 6/6 | 6/6 | 3/3 | 2/3 | 3/3 | 3/3 |
| 8 | 6/6 | 6/6 | 6/6 | 5/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 9 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| Total | 54/54 | 46/48 | 54/54 | 47/48 | 27/27 | 22/24 | 27/27 | 26/27 |
| Repeatability (%) | 100,0 | 95,8 | 100,0 | 97,9 | 100,0 | 91,7 | 100,0 | 96,3 |

Table 25. Repeatability of sulfadimethoxine (ACTALIA Cecalait, 2021).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | | |
|--------------------------|--|------------------|--------------|-------------|---|------------------|-------------|-------------|
| | AMPOULES | | PLATES | | AMPOULES | | PLATES | |
| | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan |
| ACTALIA | 6/6 | 6/6 | 6/6 | 5/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 1 | 6/6 | 6/6 | 6/6 | 5/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 2 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 2/3 | 3/3 |
| 3 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 4 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 5 | 6/6 | Software problem | 6/6 | 6/6 | 3/3 | Software problem | 3/3 | 3/3 |
| 6 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 7 | 6/6 | 5/6 | 6/6 | 5/6 | 3/3 | 2/3 | 3/3 | 2/3 |
| 8 | 6/6 | 6/6 | 6/6 | 4/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 9 | 6/6 | 6/6 | 6/6 | 5/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| Total | 54/54 | 47/48 | 54/54 | 49/54 | 27/27 | 23/24 | 26/27 | 22/27 |
| Repeatability (%) | 100,0 | 97,9 | 100,0 | 90,7 | 100,0 | 95,8 | 96,3 | 81,5 |

Table 26. Repeatability of tetracycline (ACTALIA Cecalait, 2021).

| Labs | (Number of analysis for same sample / Number of total sample) x 100 (%) | | | | (Number of analysis for identical sample / Number of total sample) x 100 (%) | | | |
|--------------------------|--|------------------|-------------|-------------|---|------------------|-------------|-------------|
| | AMPOULES | | PLATES | | AMPOULES | | PLATES | |
| | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan | Visual | Delvo@Scan |
| ACTALIA | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 1 | 6/6 | 5/6 | 6/6 | 6/6 | 3/3 | 2/3 | 3/3 | 3/3 |
| 2 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 3 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 4 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 2/3 | 3/3 | 3/3 |
| 5 | 6/6 | Software problem | 6/6 | 5/6 | 3/3 | Software problem | 3/3 | 2/3 |
| 6 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| 7 | 5/6 | 6/6 | 5/6 | 6/6 | 2/3 | 3/3 | 2/3 | 3/3 |
| 8 | 6/6 | 6/6 | 6/6 | 4/6 | 3/3 | 3/3 | 3/3 | 2/3 |
| 9 | 6/6 | 6/6 | 6/6 | 6/6 | 3/3 | 3/3 | 3/3 | 3/3 |
| Total | 53/54 | 47/48 | 53/54 | 51/54 | 26/27 | 22/24 | 26/27 | 25/27 |
| Repeatability (%) | 98,1 | 97,9 | 98,1 | 94,4 | 96,3 | 91,7 | 96,3 | 92,6 |

4.3.3. Reproducibility

For both interlaboratory studies performed by ANSES and by ACTALIA Cecalait, the reproducibility was calculated for each level of antibiotic (L0, L1, L2 and L3).

The interlaboratory reproducibility (%) is the ratio of the number of identical and excepted results (negative for blank samples and positive when antibiotic was added) on the total number of results. For L1 level, the most frequent results were taken into account, because positive and negative results were obtained at this level under the detection capability of the method.

Tables 27 to 30 present the reproducibility for each format (ampoules and plates) and for each type of reading (visual and by Delvo®Scan) for the 6 antibiotics.

The reproducibility for blank milk was equal to 100 %, except for 3 antibiotics analyzed in 2013 with Delvo®Scan reading in plates format (reproducibility higher than 94 %).

L1 level showed a reproducibility between 71 and 77 %. These results can be explained by a level under the detection capabilities of the method.

All L2 and L3 levels had reproducibility equal to 100 % for ampoules format with both readings. With plates format (visual and Delvo®Scan readings), the reproducibility was close to 100% (L2 > 96 %; L3 > 98 %).

The global reproducibility (L1+L2+L3) is between 89 and 92 %. This decrease is due to the low reproducibility of L1 level.

Table 27. Reproducibility for Delvotest® T in ampoules format with visual reading.

| Sample type (blank milk or antibiotic) | Levels | Concentrations (µg/kg) | Number of identical results | Total of results | Reproducibility (%) |
|--|-----------------------|---------------------------|--------------------------------------|---------------------|------------------------|
| Blank milk | L0 (penicillin G) | - | 32 | 32 | 100,0 |
| | L0 (cefquinome) | - | 32 | 32 | 100,0 |
| | L0 (tylosin A) | - | 32 | 32 | 100,0 |
| | L0 (gentamycin) | - | 36 | 36 | 100,0 |
| | L0 (sulfadimethoxine) | - | 36 | 36 | 100,0 |
| | L0 (tetracycline) | - | 36 | 36 | 100,0 |
| Total (L0) : | | | 204 | 204 | 100,0 |
| Penicillin G | L1 | 1 | 30* | 32 | 93,8 |
| | L2 | 4 | 32 | 32 | 100,0 |
| | L3 | 6 | 32 | 32 | 100,0 |
| Cefquinome | L1 | 20 | 30* | 32 | 93,8 |
| | L2 | 80 | 32 | 32 | 100,0 |
| | L3 | 300 | 32 | 32 | 100,0 |
| Tylosin A | L1 | 20 | 17* | 32 | 53,1 |
| | L2 | 50 | 32 | 32 | 100,0 |
| | L3 | 300 | 32 | 32 | 100,0 |
| Gentamycin | L1 | 50 | 32 | 36 | 88,9 |
| | L2 | 120 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Sulfadimethoxine | L1 | 20 | 28 | 36 | 77,8 |
| | L2 | 48 | 36 | 36 | 100,0 |
| | L3 | 60 | 36 | 36 | 100,0 |
| Tetracycline | L1 | 40 | 21* | 36 | 58,3 |
| | L2 | 96 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Total (L1) : | | | 158 | 204 | 77,5 |
| Total (L2) : | | | 204 | 204 | 100,0 |
| Total (L3) : | | | 204 | 204 | 100,0 |
| Total (L1+L2+L3) : | | | 566 | 612 | 92,5 |

*: Negative results were taken into account.

Table 28. Reproducibility for Delvotest® T in ampoules format with Delvo®Scan reading.

| Sample type (blank milk or antibiotic) | Levels | Concentrations (µg/kg) | Number of identical results | Total of results | Reproducibility (%) |
|--|-----------------------|---------------------------|-----------------------------------|---------------------|------------------------|
| Blank milk | L0 (gentamycin) | - | 32 | 32 | 100,0 |
| | L0 (sulfadimethoxine) | - | 32 | 32 | 100,0 |
| | L0 (tetracycline) | - | 32 | 32 | 100,0 |
| Total (L0) : | | | 96 | 96 | 100,0 |
| Gentamycin | L1 | 50 | 20 | 32 | 62,5 |
| | L2 | 120 | 32 | 32 | 100,0 |
| | L3 | 150 | 32 | 32 | 100,0 |
| Sulfadimethoxine | L1 | 20 | 23 | 32 | 71,9 |
| | L2 | 48 | 32 | 32 | 100,0 |
| | L3 | 60 | 32 | 32 | 100,0 |
| Tetracycline | L1 | 40 | 27* | 32 | 84,4 |
| | L2 | 96 | 32 | 32 | 100,0 |
| | L3 | 150 | 32 | 32 | 100,0 |
| Total (L1) : | | | 70 | 96 | 72,9 |
| Total (L2) : | | | 96 | 96 | 100,0 |
| Total (L3) : | | | 96 | 96 | 100,0 |
| Total (L1+L2+L3) : | | | 262 | 288 | 91,0 |

*: Negative results were taken into account.

Table 29. Reproducibility for Delvotest® T in plates format with visual reading.

| Sample type (blank milk or antibiotic) | Levels | Concentrations (µg/kg) | Number of identical results | Total of results | Reproducibility (%) |
|--|-----------------------|---------------------------|--------------------------------------|---------------------|------------------------|
| Blank milk | L0 (penicillin G) | - | 36 | 36 | 100,0 |
| | L0 (cefquinome) | - | 36 | 36 | 100,0 |
| | L0 (tylosin A) | - | 36 | 36 | 100,0 |
| | L0 (gentamycin) | - | 36 | 36 | 100,0 |
| | L0 (sulfadimethoxine) | - | 36 | 36 | 100,0 |
| | L0 (tetracycline) | - | 34 | 34 | 100,0 |
| Total (L0) : | | | 214 | 214 | 100,0 |
| Penicillin G | L1 | 1 | 30* | 36 | 83,3 |
| | L2 | 4 | 32 | 36 | 88,9 |
| | L3 | 6 | 36 | 36 | 100,0 |
| Cefquinome | L1 | 20 | 25* | 36 | 69,4 |
| | L2 | 80 | 36 | 36 | 100,0 |
| | L3 | 300 | 36 | 36 | 100,0 |
| Tylosin A | L1 | 20 | 24 | 36 | 66,7 |
| | L2 | 50 | 34 | 36 | 94,4 |
| | L3 | 300 | 36 | 36 | 100,0 |
| Gentamycin | L1 | 50 | 24* | 36 | 66,7 |
| | L2 | 120 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Sulfadimethoxine | L1 | 20 | 28 | 36 | 77,8 |
| | L2 | 48 | 36 | 36 | 100,0 |
| | L3 | 60 | 34 | 36 | 94,4 |
| Tetracycline | L1 | 40 | 23* | 36 | 63,9 |
| | L2 | 96 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Total (L1) : | | | 154 | 216 | 71,3 |
| Total (L2) : | | | 210 | 216 | 97,2 |
| Total (L3) : | | | 214 | 216 | 99,1 |
| Total (L1+L2+L3) : | | | 578 | 648 | 89,2 |

*: Negative results were taken into account.

Table 30. Reproducibility for Delvotest® T in plates format with Delvo®Scan reading.

| Sample type (blank milk or antibiotic) | Levels | Concentration (µg/kg) | Number of identical results | Total of results | Reproducibility (%) |
|--|-----------------------|--------------------------|-----------------------------------|---------------------|------------------------|
| Blank milk | L0 (penicillin G) | - | 34 | 36 | 94,4 |
| | L0 (cefquinome) | - | 35 | 36 | 97,2 |
| | L0 (tylosin A) | - | 35 | 36 | 97,2 |
| | L0 (gentamycin) | - | 36 | 36 | 100,0 |
| | L0 (sulfadimethoxine) | - | 36 | 36 | 100,0 |
| | L0 (tetracycline) | - | 34 | 34 | 100,0 |
| Total (L0) : | | | 210 | 214 | 98,1 |
| Penicillin G | L1 | 1 | 32* | 36 | 88,9 |
| | L2 | 4 | 34 | 36 | 94,4 |
| | L3 | 6 | 35 | 36 | 97,2 |
| Cefquinome | L1 | 20 | 28* | 36 | 77,8 |
| | L2 | 80 | 33 | 36 | 91,7 |
| | L3 | 300 | 34 | 36 | 94,4 |
| Tylosin A | L1 | 20 | 20* | 36 | 55,6 |
| | L2 | 50 | 35 | 36 | 97,2 |
| | L3 | 300 | 35 | 36 | 97,2 |
| Gentamycin | L1 | 50 | 31* | 36 | 86,1 |
| | L2 | 120 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Sulfadimethoxine | L1 | 20 | 20* | 36 | 55,6 |
| | L2 | 48 | 35 | 36 | 97,2 |
| | L3 | 60 | 36 | 36 | 100,0 |
| Tetracycline | L1 | 40 | 30* | 36 | 83,3 |
| | L2 | 96 | 36 | 36 | 100,0 |
| | L3 | 150 | 36 | 36 | 100,0 |
| Total (L1) : | | | 161 | 216 | 74,5 |
| Total (L2) : | | | 208 | 216 | 96,8 |
| Total (L3) : | | | 212 | 216 | 98,1 |
| Total (L1+L2+L3) : | | | 581 | 648 | 89,8 |

*: Negative results were taken into account.

4.4 Conclusion of interlaboratory studies (2013 and 2021 studies)

The renewal interlaboratory study was conducted on raw cow's milk.

Specificity, sensitivity and global sensitivity were very satisfactory (100% or close to 100%)
Percentage of positive results at L1 level (lower than detection capability) were between 25 and 46%.

Repeatability was between 80 % and 100 %.

The reproducibility for blank milk was equal or close to 100 %.

The global reproducibility (L1+L2+L3) was between 89 and 93 %, due to the low reproducibility of L1 level.

In general, all results were satisfactory, but we can observe that:

- better results were obtained with Delvotest® T in ampoules than in plates format.
- the results were slightly better with visual reading than with Delvo®Scan.

5. GENERAL CONCLUSION

The Delvo®Scan software used was the version 5.08 (ampoules and plates) with an EPSON V600 scanner and cut-off equal to 0.

All incubations were performed at control time except when the incubation time was tested in robustness (incubation of 3h15).

The rules of AFNOR have been changed in 2017, so the preliminary study was performed again in the second renewal study in 2020 according to these new rules.

The results of the preliminary study on Delvotest® T with 2 formats (plates and ampoules) and with 2 readings (visual and Delvo®Scan) are summarized in **Table 31**, and were satisfactory.

During this second renewal study conducted by ACTALIA Cecalait, an interlaboratory study was carried out in 2021 in addition to the one already performed by ANSES expert laboratory in 2013. Global results of these interlaboratory studies are presented in the **Table 32**, and were satisfactory.

Table 31. Summary of preliminary study performed in 2020 at control time by visually and by Delvo®Scan readings in raw cow milk.

| CHARACTERISTICS OF PERFORMANCE | | CONCLUSION | | MRL in milk (ppb) |
|--------------------------------|----------------------------------|------------------------------------|-------------------------|-------------------|
| | | AMPOULES | PLATES | |
| False positive (%) | | 0 | 0 | |
| Detection capability | CCβ Amoxicillin (ppb) | 2 | 2 | 4 |
| | CCβ Ampicillin (ppb) | 2 | 2 | 4 |
| | CCβ Penicillin G (ppb) | 3 | 1 | 4 |
| | CCβ Cloxacillin (ppb) | 10 | 10 | 30 |
| | CCβ Oxacillin (ppb) | 3 | 3 | 30 |
| | CCβ Nafcillin (ppb) | 3 | 3 | 30 |
| | CCβ Oxytetracycline (ppb) | 100 | 80 | 100 |
| | CCβ 4-epioxytetracycline (ppb) | 600 | 800 | 100 |
| | CCβ Chlortetracycline (ppb) | 150 | 150 | 100 |
| | CCβ 4-epichlortetracycline (ppb) | 600 | 600 | 100 |
| | CCβ Tetracycline (ppb) | 80 | 100 | 100 |
| | CCβ 4-epitetracycline (ppb) | 800 | 1 000 | 100 |
| | CCβ Doxycycline (ppb) | 50 | 50 | ^a |
| | CCβ Sulfamethazine (ppb) | 125 | 125 | 100 |
| | CCβ Sulfathiazole (ppb) | 30 | 30 | 100 |
| | CCβ Sulfadimethoxine (ppb) | 40 | 40 | 100 |
| | CCβ Sulfadiazine (ppb) | 55 | 50 | 100 |
| | CCβ Sulfadoxine (ppb) | 80 | 80 | 100 |
| | CCβ Tilmicosin (ppb) | 60 | 100 | 50 |
| | CCβ Tylosin A (ppb) | 35 | 35 | 50 |
| | CCβ Erythromycin A(ppb) | 160 | 200 | 40 |
| | CCβ Spiramycin (ppb) | 1 500 | 2 000 | 200 |
| | CCβ Neomycin B (ppb) | 140 | 140 | 1500 |
| | CCβ Gentamycin (ppb) | 100 | 100 | 100 |
| | CCβ Streptomycin (ppb) | 700 | 1 000 | 200 |
| | CCβ Dihydrostreptomycin(ppb) | 700 | 800 | 200 |
| | CCβ Cephapirin (ppb) | 5 | 5 | 60 |
| | CCβ Desacetylcephapirin (ppb) | 2 | 2 | 60 |
| | CCβ Ceftiofur (ppb) | 20 | 20 | 100 |
| | CCβ Desfuroylceftiofur (ppb) | 45 | 80 | 100 |
| | CCβ Cefoperazone (ppb) | 20 | 20 | 50 |
| | CCβ Cefalexin (ppb) | 30 | 30 | 100 |
| | CCβ Cefquinome (ppb) | 50 | 60 | 20 |
| | CCβ Cefalonium (ppb) | 5 | 5 | 20 |
| | CCβ Cefazolin (ppb) | 3 | 3 | 50 |
| | CCβ Chloramphénicol (ppb) | 4 000 | 3 500 | 0,3 ^b |
| | CCβ Trimethoprim (ppb) | 110 | 120 | 50 |
| | CCβ Dapsone (ppb) | 10 | 10 | 5 ^c |
| | CCβ Lincomycin (ppb) | 275 | 220 | 150 |
| | CCβ Rifaximin (ppb) | 40 | 40 | 60 |
| CCβ Pirlimicin (ppb) | 300 | 300 | 100 | |
| CCβ Clavulanic acid (ppb) | 700 | 800 | 200 | |
| Applicability | Individual cow milk | Suitable | | |
| Robustness | Sample volume | Robust | | |
| | Incubation time | Robust | Not robust ^d | |
| | Incubation temperature | Robust | | |
| | Delay of reading | Robust | | |
| | pH | Not robust for low pH ^e | | |
| | Total Bact Count | Robust | | |
| | Frozen milk | Robust | | |
| | Milk temperature | Robust | | |
| Age of batches | Robust | | | |

^a No MRL in milk

^b MRPL (Minimum Required Performance Limit)

^c MMPR (Minimum Method Performance Requirements)

^d There are false negative results for 2 antibiotics (dihydrostreptomycin and lincomycin) on plates only.

^e There are false negative results for 4 antibiotics (tylosin A, erythromycin, dihydrostreptomycin and lincomycin).

Table 32. Results of interlaboratory studies (2013 and 2021) on Delvotest® T.

| Delvotest® T format | | AMPOULES | | | | PLATES | | | |
|----------------------------|-----------------------|----------|-------|------------|-------|--------|-------|------------|------|
| Type of reading | | Visual | | Delvo®Scan | | Visual | | Delvo®Scan | |
| Specificity (L0) | | 100,0 | | 100,0 | | 100,0 | | 97,8 | |
| % positive results (L1) | | 46,1 | | 39,6 | | 43,5 | | 25,5 | |
| Global sensitivity (L2+L3) | | 100,0 | | 100,0 | | 98,1 | | 97,5 | |
| Repeatability | Type of repeatability | (1) | (2) | (1) | (2) | (1) | (2) | (1) | (2) |
| | Blank | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 100,0 | 95,2 | 93,9 |
| | Gentamycin | 100,0 | 100,0 | 95,8 | 91,7 | 100,0 | 100,0 | 97,9 | 96,3 |
| | Sulfadimethoxine | 100,0 | 100,0 | 97,9 | 95,8 | 90,7 | 96,3 | 100,0 | 81,5 |
| | Tetracycline | 98,1 | 96,3 | 97,9 | 91,7 | 98,1 | 96,3 | 94,4 | 92,6 |
| | Penicillin G | 100,0 | 95,8 | | | 92,6 | 92,6 | 98,1 | 90,7 |
| | Cefquinome | 100,0 | 95,8 | | | 92,6 | 83,3 | 98,1 | 98,1 |
| | Tylosin A | 97,9 | 93,8 | | | 96,3 | 92,6 | 100,0 | 92,6 |
| Reproducibility (L0) | | 100,0 | | 100,0 | | 100,0 | | 98,1 | |
| Reproducibility (L1+L2+L3) | | 92,5 | | 91,0 | | 89,2 | | 89,8 | |

(1): Repeatability of same samples

(2): Repeatability of identical sample

6. BIBLIOGRAPHIC REFERENCES

- Reybroeck, W. and Ooghe, S., 2012. ILVO-T&V ‘Validation report of the Delvotest® T’.
- Boultif, L. Bassem, B. Cherif, A.M. Djahida, D. Ismahane, R. 2016. Comparative analysis of antibiotic residues in local and imported milk by microbiological inhibition test in Constantine region (north east Algeria). Der Pharma Chemica : 8(16):105-111.
- Romero, T. Van Weyenberg, S. Molina, M.P. Reybroeck, W. 2016. Detection of antibiotics in goat’s milk: comparison of different commercial microbial inhibitor tests developed for the testing of cows’ milk. International Dairy Journal: 62:39-42.

Poligny, the 20th of July,

Pauline Bouveret

Project Manager – Validation of methods

Patricia ROLLIER

Head of microbiology

Appendix 1: Details on antibiotics used in preliminary study

| Drug family | Compounds detected | Supplier | Reference | Batch number |
|-----------------|------------------------|----------------------------------|-------------------|----------------------------------|
| Penicillins | Amoxicillin | Sigma-Aldrich | A8523-1G A8523 | 0000085664 108M4891V |
| | Ampicillin | Sigma-Aldrich | 31591 | BCBZ5016 |
| | Penicillin G | Sigma-Aldrich | P3032 | 059M4827V |
| | Cloxacillin | Sigma-Aldrich | 46140 | BCBW1061 |
| | Oxacillin | Sigma-Aldrich | 46589 | BCBT8512 |
| | Nafcillin | Sigma-Aldrich | 32071 | BCCC5791 |
| Tetracyclines | Oxytetracycline | Sigma-Aldrich | 46598 | BCCC5114 BCBZ6310 |
| | 4-Epioxytetracycline | Acros organics | 25771 | A0415682 |
| | Chlortetracycline | Sigma-Aldrich | C4881 | 069M4122V |
| | 4-Epichlortetracycline | Acros organics | 26823 | A0423144 |
| | Tetracycline | Sigma-Aldrich | T3383 31741 | 049M4834V BCBX5586 |
| | 4-Epitetracycline | Sigma-Aldrich | 37918 | BCBZ7193 BCCB7706 BCCD0437 |
| | Doxycycline | Sigma-Aldrich | D3447 | 109M4082V |
| Sulfonamides | Sulfamethazine | Sigma-Aldrich | S6256 | 048M4017V |
| | Sulfathiazole | Sigma-Aldrich | 46902 | BCBW1884 |
| | Sulfadimethoxine | Sigma-Aldrich | S7007 | 059M4032V |
| | Sulfadiazine | Sigma-Aldrich | S8626 | 069M4751V |
| | Sulfadoxine | Sigma-Aldrich | 31736 | BCBV7742 |
| Macrolides | Tilmicosin | Sigma-Aldrich | 33864 | BCCB4507 |
| | Tylosin A | Sigma-Aldrich | 33847 | BCCD1311 BCBX4839 BCCB6064 |
| | Erythromycin A | Sigma-Aldrich | E5389 | WXBD0760V WXBC7091V |
| | Spiramycin | Sigma-Aldrich | S9132 | MKCG3562 |
| Aminoglycosides | Neomycin B | Sigma-Aldrich | N6386 | 036K0078 |
| | Gentamycin | Sigma-Aldrich | 46305 | BCCB6394 |
| | Streptomycin | Sigma-Aldrich | 46738 S6501 | SZBF194XV SLBP6412V |
| | Dihydrostreptomycin | Sigma-Aldrich | D7253 | 117M4820V |
| Cephalosporins | Cephapirin | Sigma-Aldrich | 43989 | BCCC5208 BCBW7147 |
| | Desacetylcephapirin | Toronto Research Chemicals | D288970 | 16-AKS-79-4 |
| | Ceftiofur | Sigma-Aldrich | 32422 | BCCB8697 |

| | | | | |
|--------|--------------------|----------------------------|---------|------------|
| | Desfuroylceftiofur | Toronto Research Chemicals | D289980 | 5-WBZ-57-5 |
| | Cefoperazone | Sigma-Aldrich | C4292 | 118M4841V |
| | Cefalexin | Sigma-Aldrich | 33989 | BCBW7031 |
| | Cefquinome | Sigma-Aldrich | 32472 | BCBW2550 |
| | Cefalonium | Sigma-Aldrich | 32904 | BCBV1595 |
| | Cefazolin | Sigma-Aldrich | C5020 | 019M4852V |
| Others | Chloramphenicol | Sigma-Aldrich | C0378 | SLCD7425 |
| | Trimethoprim | Sigma-Aldrich | T7883 | 019M4019V |
| | Dapsone | Sigma-Aldrich | A74807 | STBJ1870 |
| | Lincomycin | Sigma-Aldrich | 31727 | BCBW4661 |
| | Rifaximin | Sigma-Aldrich | 33999 | BCBT5109 |
| | Pirlimicin | Cayman Chemical | 20138 | 0574568 |
| | Clavulanic acid | Sigma-Aldrich | 33454 | STBJ0056 |

Appendix 2: Results of robustness study (2020)

1. Results of robustness study for ampoules

1.1. Influence of protocol

1.1.1. Sample volume

1.1.1.1. Penicillins

1.1.1.1.1. Amoxicillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Amox2 - 90 µL | 6 | 90µL - A3 | + | 5,14 | + |
| Milk 1 Amox2 - 110 µL | 6 | 110µL - A1 | + | 7,36 | + |
| Milk 2 Amox2 - 90 µL | 6 | 90µL - A8 | + | 4,32 | + |
| Milk 2 Amox2 - 110 µL | 6 | 110µL - A8 | + | 6,53 | + |
| Milk 3 Amox2 - 90 µL | 6 | 90µL - A9 | + | 3,83 | + |
| Milk 3 Amox2 - 110 µL | 6 | 110µL - A4 | + | 7,13 | + |

1.1.1.1.2. Cloxacillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Cloxa10 - 90 µL | 6 | 90µL - A12 | + | 7 | + |
| Milk 1 Cloxa10 - 110 µL | 6 | 110µL - A6 | + | 5,88 | + |
| Milk 2 Cloxa10 - 90 µL | 6 | 90µL - A15 | + | 5,76 | + |
| Milk 2 Cloxa10 - 110 µL | 6 | 110µL - A13 | + | 6,86 | + |
| Milk 3 Cloxa10 - 90 µL | 6 | 90µL - A18 | + | 6,12 | + |
| Milk 3 Cloxa10 - 110 µL | 6 | 110µL - A11 | + | 6,65 | + |

1.1.1.2. Tetracyclines

1.1.1.2.1. Oxytetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 90 µL | 6 | 90µL - A10 | + | 4,95 | + |
| Milk 1 Oxytetra110 - 110 µL | 6 | 110µL - A20 | + | 5,27 | + |
| Milk 2 Oxytetra110 - 90 µL | 6 | 90µL - A4 | + | 4,48 | + |
| Milk 2 Oxytetra110 - 110 µL | 6 | 110µL - A12 | + | 5,05 | + |
| Milk 3 Oxytetra110 - 90 µL | 6 | 90µL - A6 | + | 5,27 | + |
| Milk 3 Oxytetra110 - 110 µL | 6 | 110µL - A5 | + | 6,51 | + |

1.1.1.2.2. Chlortetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 90 µL | 6 | 90µL - A1 | + | 5,82 | + |
| Milk 1 Chlortetra150 - 110 µL | 6 | 110µL - A7 | + | 6,37 | + |
| Milk 2 Chlortetra150 - 90 µL | 6 | 90µL - A2 | + | 5,65 | + |
| Milk 2 Chlortetra150 - 110 µL | 6 | 110µL - A15 | + | 6,07 | + |
| Milk 3 Chlortetra150 - 90 µL | 6 | 90µL - A5 | + | 5,58 | + |
| Milk 3 Chlortetra150 - 110 µL | 6 | 110µL - A17 | + | 6,15 | + |

1.1.1.3. Sulfonamides

1.1.1.3.1. Sulfadimethoxine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 90 µL | 6 | 90µL - A7 | + | 5,78 | + |
| Milk 1 Sulfadimet40 - 110 µL | 6 | 110µL - A18 | + | 6,5 | + |
| Milk 2 Sulfadimet40 - 90 µL | 6 | 90µL - A14 | + | 5,73 | + |
| Milk 2 Sulfadimet40 - 110 µL | 6 | 110µL - A24 | + | 5,96 | + |
| Milk 3 Sulfadimet40 - 90 µL | 6 | 90µL - A11 | + | 5,81 | + |
| Milk 3 Sulfadimet40 - 110 µL | 6 | 110µL - A25 | + | 5,73 | + |

1.1.1.3.2. Sulfadiazine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 90 µL | 6 | 90µL - A1 | + | 5,02 | + |
| Milk 1 Sulfadiaz55 - 110 µL | 6 | 110µL - B4 | + | 5,61 | + |
| Milk 2 Sulfadiaz55 - 90 µL | 6 | 90µL - A5 | + | 5,08 | + |
| Milk 2 Sulfadiaz55 - 110 µL | 6 | 110µL - B11 | + | 5,01 | + |
| Milk 3 Sulfadiaz55 - 90 µL | 6 | 90µL - A10 | + | 5,02 | + |
| Milk 3 Sulfadiaz55 - 110 µL | 6 | 110µL - B13 | + | 4,73 | + |

Date : 20/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 5 | 90µL - A1 | - | -6,78 | - |
| Milk 1 Neg - 110 µL | 5 | 110µL - B2 | - | -7,27 | - |
| Milk 2 Neg - 90 µL | 5 | 90µL - A4 | - | -5,28 | - |
| Milk 2 Neg - 110 µL | 5 | 110µL - B5 | - | -6,63 | - |
| Milk 3 Neg - 90 µL | 5 | 90µL - A5 | - | -7,06 | - |
| Milk 3 Neg - 110 µL | 5 | 110µL - B3 | - | -7,24 | - |

Date : 20/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 90 µL | 5 | 90µL - A2 | + | 5,22 | + |
| Milk 1 Sulfadiaz50 - 110 µL | 5 | 110µL - B4 | + | 4,3 | + |
| Milk 2 Sulfadiaz50 - 90 µL | 5 | 90µL - A3 | + | 4,72 | + |
| Milk 2 Sulfadiaz50 - 110 µL | 5 | 110µL - B6 | + | 4,56 | + |
| Milk 3 Sulfadiaz50 - 90 µL | 5 | 90µL - A6 | + | 4,19 | + |
| Milk 3 Sulfadiaz50 - 110 µL | 5 | 110µL - B1 | + | 4,05 | + |

1.1.1.4. Macrolides

1.1.1.4.1. Tylosin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Tylo35 - 90 µL | 6 | 90µL - A15 | + | 4,22 | + |
| Milk 1 Tylo35 - 110 µL | 6 | 110µL - B5 | + | 4,5 | + |
| Milk 2 Tylo35 - 90 µL | 6 | 90µL - A7 | + | 3,33 | + |
| Milk 2 Tylo35 - 110 µL | 6 | 110µL - B2 | + | 4,62 | + |
| Milk 3 Tylo35 - 90 µL | 6 | 90µL - A2 | + | 3,83 | + |
| Milk 3 Tylo35 - 110 µL | 6 | 110µL - B6 | + | 4,69 | + |

1.1.1.4.2. Erythromycin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Erythromicine 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Erythro160 - 90 µL | 6 | 90µL - A3 | + | 4,85 | + |
| Milk 1 Erythro160 - 110 µL | 6 | 110µL - B3 | + | 5,5 | + |
| Milk 2 Erythro160 - 90 µL | 6 | 90µL - A12 | + | 3,9 | + |
| Milk 2 Erythro160 - 110 µL | 6 | 110µL - B8 | + | 4,55 | + |
| Milk 3 Erythro160 - 90 µL | 6 | 90µL - A16 | + | 4,85 | + |
| Milk 3 Erythro160 - 110 µL | 6 | 110µL - B19 | + | 5,03 | + |

1.1.1.5. Aminoglycosides : dihydrostreptomycin

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - 90 µL | 6 | 90µL - A13 | + | 4,73 | + |
| Milk 1 Dihydrostrepto700 - 110 µL | 6 | 110µL - B14 | + | 5,68 | + |
| Milk 2 Dihydrostrepto700 - 90 µL | 6 | 90µL - A25 | + | 3,9 | + |
| Milk 2 Dihydrostrepto700 - 110 µL | 6 | 110µL - B15 | + | 5,04 | + |
| Milk 3 Dihydrostrepto700 - 90 µL | 6 | 90µL - A20 | + | 4,16 | + |
| Milk 3 Dihydrostrepto700 - 110 µL | 6 | 110µL - B24 | + | 4,99 | + |

1.1.1.6. Cephalosporins : céfalexine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Cefal30 - 90 µL | 6 | 90µL - A21 | + | 5,67 | + |
| Milk 1 Cefal30 - 110 µL | 6 | 110µL - B21 | + | 5,6 | + |
| Milk 2 Cefal30 - 90 µL | 6 | 90µL - A27 | + | 5,21 | + |
| Milk 2 Cefal30 - 110 µL | 6 | 110µL - B26 | + | 5,61 | + |
| Milk 3 Cefal30 - 90 µL | 6 | 90µL - A24 | + | 5,54 | + |
| Milk 3 Cefal30 - 110 µL | 6 | 110µL - B25 | + | 6,29 | + |

1.1.1.7. Lincosamides : lincomycine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Linco275 - 90 µL | 6 | 90µL - A16 | + | 2 | + |
| Milk 1 Linco275 - 110 µL | 6 | 110µL - A23 | + | 3,98 | + |
| Milk 2 Linco275 - 90 µL | 6 | 90µL - A24 | + | 2,15 | + |
| Milk 2 Linco275 - 110 µL | 6 | 110µL - A27 | + | 2,97 | + |
| Milk 3 Linco275 - 90 µL | 6 | 90µL - A21 | + | 3,37 | + |
| Milk 3 Linco275 - 110 µL | 6 | 110µL - A19 | + | 3,7 | + |

1.1.2. Incubation time

1.1.2.1. Penicillins

1.1.2.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Amoxicilline 2 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Amox2 - 3h15 | 6 | 3h15 - A2 | + | 5,32 | + |
| Milk 2 Amox2 - 3h15 | 6 | 3h15 - A8 | + | 3,69 | + |
| Milk 3 Amox2 - 3h15 | 6 | 3h15 - A6 | + | 4,19 | + |

1.1.2.1.2. Cloxacillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Cloxacilline 30 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - 3h15 | 6 | 3h15 - A7 | + | 6,43 | + |
| Milk 2 Cloxa10 - 3h15 | 6 | 3h15 - A4 | + | 5,25 | + |
| Milk 3 Cloxa10 - 3h15 | 6 | 3h15 - A9 | + | 5,15 | + |

1.1.2.2. Tetracyclines

1.1.2.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Oxytetracycline 110 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 3h15 | 6 | 3h15 - A11 | + | 0,52 | + |
| Milk 2 Oxytetra110 - 3h15 | 6 | 3h15 - A10 | + | 0,38 | + |
| Milk 3 Oxytetra110 - 3h15 | 6 | 3h15 - A12 | + | 0,72 | + |

1.1.2.2.2. Chlortetracycline

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3H15 - A1 | - | -14,35 | - |
| Milk 2 Neg - 3h15 | 4 | 3H15 - A10 | - | -12,77 | - |
| Milk 3 Neg - 3h15 | 4 | 3H15 - A8 | - | -15,13 | - |

Date : 19/08/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 3h15 | 4 | 3H15 - A5 | + | 1,17 | + |
| Milk 2 Chlortetra150 - 3h15 | 4 | 3H15 - A16 | + | 2,64 | + |
| Milk 3 Chlortetra150 - 3h15 | 4 | 3H15 - A20 | + | 3,86 | + |

1.1.2.3. Sulfonamides

1.1.2.3.1. Sulfadimethoxine

Date : 24/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 5 | 3h15 - A1 | - | -16,25 | - |
| Milk 2 Neg - 3h15 | 5 | 3h15 - A6 | - | -14,37 | - |
| Milk 3 Neg - 3h15 | 5 | 3h15 - A9 | - | -14,88 | - |

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 3h15 | 5 | 3h15 - A7 | + | 3,21 | + |
| Milk 2 Sulfadimet40 - 3h15 | 5 | 3h15 - A15 | + | 2,86 | + |
| Milk 3 Sulfadimet40 - 3h15 | 5 | 3h15 - A10 | + | 2,25 | + |

1.1.2.3.2. Sulfadiazine

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 3h15 | 4 | 3h15 - A3 | + | 0,95 | + |
| Milk 2 Sulfadiaz55 - 3h15 | 4 | 3h15 - A8 | + | 0,9 | + |
| Milk 3 Sulfadiaz55 - 3h15 | 4 | 3h15 - A14 | + | 2,29 | + |

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 5 | 3h15 - D7 | - | -13,15 | - |
| Milk 2 Neg - 3h15 | 5 | 3h15 - D8 | - | -13,75 | - |
| Milk 3 Neg - 3h15 | 5 | 3h15 - D9 | - | -15,72 | - |

Date : 01/09/2020

Sulfadiazine 60 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|---------|----------------|-------------------|---|
| Milk 1 Sulfadiaz60 - 3h15 | 5 | 3h15-D1 | + | 5,71 | + |
| Milk 2 Sulfadiaz60 - 3h15 | 5 | 3h15-D2 | + | 5,36 | + |
| Milk 3 Sulfadiaz60 - 3h15 | 5 | 3h15-D3 | + | 5,75 | + |

1.1.2.4. Macrolides

1.1.2.4.1. Tylosin A

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Tylosine 42 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Tylo42 - 3h15 | 4 | 3h15 - A12 | + | 3,66 | + |
| Milk 2 Tylo42 - 3h15 | 4 | 3h15 - A14 | + | 2,15 | + |
| Milk 3 Tylo42 - 3h15 | 4 | 3h15 - A15 | + | 2,19 | + |

1.1.2.4.2. Erythromycin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Erythromycine 192 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Erythro192 - 3h15 | 4 | 3h15 - E5 | + | 1,84 | + |
| Milk 2 Erythro192 - 3h15 | 4 | 3h15 - E11 | + | 2,04 | + |
| Milk 3 Erythro192 - 3h15 | 4 | 3h15 - E10 | + | 2,41 | + |

1.1.2.5. Aminoglycosides : dihydrostreptomycin

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Dihydrostreptomycine 840 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E14 | + | 2,85 | + |
| Milk 2 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E17 | + | 3,18 | + |
| Milk 3 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E20 | + | 3,38 | + |

1.1.2.6. Cephalosporins : céfalexine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Cefalexine 36 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Cefal36 - 3h15 | 4 | 3h15 - A4 | + | 4,02 | + |
| Milk 2 Cefal36 - 3h15 | 4 | 3h15 - A11 | + | 5,08 | + |
| Milk 3 Cefal36 - 3h15 | 4 | 3h15 - A13 | + | 4,66 | + |

1.1.2.7. Lincosamides : lincomycine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Lincomycine 330 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Linco330 - 3h15 | 4 | 3h15 - A3 | + | 1,74 | + |
| Milk 2 Linco330 - 3h15 | 4 | 3h15 - A7 | + | 0,36 | + |
| Milk 3 Linco330 - 3h15 | 4 | 3h15 - A10 | + | 0,61 | + |

1.1.3. Incubation temperature

1.1.3.1. Penicillins

1.1.3.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 6 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 6 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 6 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 6 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 6 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 6 | 66°C - A8 | - | -7,02 | - |

Date :20/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Amox2 - 62°C | 6 | 62°C - A2 | + | 7,02 | + |
| Milk 1 Amox2 - 66°C | 6 | 66°C - A5 | + | 5,67 | + |
| Milk 2 Amox2 - 62°C | 6 | 62°C - A1 | + | 6,1 | + |
| Milk 2 Amox2 - 66°C | 6 | 66°C - A9 | + | 5,44 | + |
| Milk 3 Amox2 - 62°C | 6 | 62°C - A14 | + | 5,82 | + |
| Milk 3 Amox2 - 66°C | 6 | 66°C - A3 | + | 5,31 | + |

1.1.3.1.2. Cloxacillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 6 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 6 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 6 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 6 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 6 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 6 | 66°C - A8 | - | -7,02 | - |

Date :20/07/2020

Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Cloxa10 - 62°C | 6 | 62°C - A3 | + | 7,78 | + |
| Milk 1 Cloxa10 - 66°C | 6 | 66°C - A12 | + | 6,33 | + |
| Milk 2 Cloxa10 - 62°C | 6 | 62°C - A11 | + | 7,09 | + |
| Milk 2 Cloxa10 - 66°C | 6 | 66°C - A15 | + | 3,53 | + |
| Milk 3 Cloxa10 - 62°C | 6 | 62°C - A13 | + | 7,31 | + |
| Milk 3 Cloxa10 - 66°C | 6 | 66°C - A14 | + | 4,27 | + |

1.1.3.2. Tetracyclines

1.1.3.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 6 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 6 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 6 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 6 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 6 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 6 | 66°C - A8 | - | -7,02 | - |

Date :20/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 62°C | 6 | 62°C - A12 | + | 6,15 | + |
| Milk 1 Oxytetra110 - 66°C | 6 | 66°C - A7 | + | 4,75 | + |
| Milk 2 Oxytetra110 - 62°C | 6 | 62°C - A15 | + | 4,9 | + |
| Milk 2 Oxytetra110 - 66°C | 6 | 66°C - A13 | + | 3,77 | + |
| Milk 3 Oxytetra110 - 62°C | 6 | 62°C - A10 | + | 5,07 | + |
| Milk 3 Oxytetra110 - 66°C | 6 | 66°C - A11 | + | 4,22 | + |

1.1.3.2.2. Chlortetracycline

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -4,66 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -5,95 | - |
| Milk 2 Neg - 62°C | 3 | 62°C - A1 | - | -3,54 | - |
| Milk 2 Neg - 66°C | 3 | 66°C - A7 | - | -7,38 | - |
| Milk 3 Neg - 62°C | 3 | 62°C - A4 | - | -5,63 | - |
| Milk 3 Neg - 66°C | 3 | 66°C - A5 | - | -7,55 | - |

Date : 22/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 62°C | 5 | 62°C - B9 | + | 5,99 | + |
| Milk 1 Chlortetra150 - 66°C | 5 | 66°C - C7 | + | 5,24 | + |
| Milk 2 Chlortetra150 - 62°C | 3 | 62°C - A6 | + | 6,47 | + |
| Milk 2 Chlortetra150 - 66°C | 3 | 66°C - A2 | + | 4,61 | + |
| Milk 3 Chlortetra150 - 62°C | 3 | 62°C - A8 | + | 6 | + |
| Milk 3 Chlortetra150 - 66°C | 3 | 66°C - A8 | + | 5,15 | + |

1.1.3.3. Sulfonamides

1.1.3.3.1. Sulfadimethoxine

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -4,66 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -5,95 | - |
| Milk 2 Neg - 62°C | 3 | 62°C - A1 | - | -3,54 | - |
| Milk 2 Neg - 66°C | 3 | 66°C - A7 | - | -7,38 | - |
| Milk 3 Neg - 62°C | 3 | 62°C - A4 | - | -5,63 | - |
| Milk 3 Neg - 66°C | 3 | 66°C - A5 | - | -7,55 | - |

Date : 22/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 62°C | 5 | 62°C - B2 | + | 5,72 | + |
| Milk 1 Sulfadimet40 - 66°C | 5 | 66°C - C1 | + | 4,62 | + |
| Milk 2 Sulfadimet40 - 62°C | 3 | 62°C - A9 | + | 6,01 | + |
| Milk 2 Sulfadimet40 - 66°C | 3 | 66°C - A9 | + | 4,75 | + |
| Milk 3 Sulfadimet40 - 62°C | 3 | 62°C - A5 | + | 6,24 | + |
| Milk 3 Sulfadimet40 - 66°C | 3 | 66°C - A1 | + | 5,03 | + |

1.1.3.3.2. Sulfadiazine

Date : 24/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -4,66 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -5,95 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - B4 | - | -3,86 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - C3 | - | -6,11 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - B6 | - | -4,34 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - C4 | - | -6,39 | - |

Date : 24/08/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 62°C | 5 | 62°C - B3 | + | 5,37 | + |
| Milk 1 Sulfadiaz55 - 66°C | 5 | 66°C - C6 | + | 4,3 | + |
| Milk 2 Sulfadiaz55 - 62°C | 5 | 62°C - B5 | + | 5,03 | + |
| Milk 2 Sulfadiaz55 - 66°C | 5 | 66°C - C8 | + | 4,05 | + |
| Milk 3 Sulfadiaz55 - 62°C | 5 | 62°C - B7 | + | 5,24 | + |
| Milk 3 Sulfadiaz55 - 66°C | 5 | 66°C - C11 | + | 3,9 | + |

Date : 24/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 62°C | 5 | 62°C - B8 | + | 4,7 | + |
| Milk 1 Sulfadiaz50 - 66°C | 5 | 66°C - C9 | + | 3,4 | + |
| Milk 2 Sulfadiaz50 - 62°C | 5 | 62°C - B11 | + | 5,33 | + |
| Milk 2 Sulfadiaz50 - 66°C | 5 | 66°C - C5 | + | 4,08 | + |
| Milk 3 Sulfadiaz50 - 62°C | 5 | 62°C - B10 | + | 4,81 | + |
| Milk 3 Sulfadiaz50 - 66°C | 5 | 66°C - C10 | + | 3,61 | + |

1.1.3.4. Macrolides

1.1.3.4.1. Tylosin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 4 | 62°C - A2 | - | -4,33 | - |
| Milk 1 Neg - 66°C | 4 | 66°C - B1 | - | -6,89 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - A6 | - | -4,52 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - B10 | - | -6,31 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - A3 | - | -4,79 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - B7 | - | -7,06 | - |

Date : 25/08/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - 62°C | 4 | 62°C - A1 | + | 3,39 | + |
| Milk 1 Tylo35 - 66°C | 4 | 66°C - B2 | + | 4,49 | + |
| Milk 2 Tylo35 - 62°C | 4 | 62°C - A5 | + | 3,81 | + |
| Milk 2 Tylo35 - 66°C | 4 | 66°C - B3 | + | 4,68 | + |
| Milk 3 Tylo35 - 62°C | 4 | 62°C - A4 | + | 4,3 | + |
| Milk 3 Tylo35 - 66°C | 4 | 66°C - B5 | + | 6,05 | + |

1.1.3.4.2. Erythromycin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 4 | 62°C - A2 | - | -4,33 | - |
| Milk 1 Neg - 66°C | 4 | 66°C - B1 | - | -6,89 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - A6 | - | -4,52 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - B10 | - | -6,31 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - A3 | - | -4,79 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - B7 | - | -7,06 | - |

Date : 25/08/2020

Erythromycine 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Erythro160 - 62°C | 4 | 62°C - A8 | + | 4,91 | + |
| Milk 1 Erythro160 - 66°C | 4 | 66°C - B12 | + | 4,25 | + |
| Milk 2 Erythro160 - 62°C | 4 | 62°C - A10 | + | 5,5 | + |
| Milk 2 Erythro160 - 66°C | 4 | 66°C - B6 | + | 5,12 | + |
| Milk 3 Erythro160 - 62°C | 4 | 62°C - A12 | + | 5,71 | + |
| Milk 3 Erythro160 - 66°C | 4 | 66°C - B8 | + | 4,72 | + |

1.1.3.5. Aminoglycosides : dihydrostreptomycin

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 4 | 62°C - A2 | - | -4,33 | - |
| Milk 1 Neg - 66°C | 4 | 66°C - B1 | - | -6,89 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - A6 | - | -4,52 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - B10 | - | -6,31 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - A3 | - | -4,79 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - B7 | - | -7,06 | - |

Date : 25/08/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - 62°C | 4 | 62°C - A15 | + | 2,43 | + |
| Milk 1 Dihydrostrepto700 - 66°C | 4 | 66°C - B14 | + | 5,4 | + |
| Milk 2 Dihydrostrepto700 - 62°C | 4 | 62°C - A21 | + | 2,52 | + |
| Milk 2 Dihydrostrepto700 - 66°C | 4 | 66°C - B19 | + | 5,35 | + |
| Milk 3 Dihydrostrepto700 - 62°C | 4 | 62°C - A18 | + | 1,86 | + |
| Milk 3 Dihydrostrepto700 - 66°C | 4 | 66°C - B15 | + | 5 | + |

1.1.3.6. Cephalosporins : céfalexine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 4 | 62°C - B1 | - | -2,07 | - |
| Milk 1 Neg - 66°C | 4 | 66°C - C4 | - | -4,19 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - B2 | - | -3,96 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - C1 | - | -6,3 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - B3 | - | -5,81 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - C2 | - | -7,01 | - |

Date : 26/08/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cefal30 - 62°C | 4 | 62°C - B4 | + | 6,89 | + |
| Milk 1 Cefal30 - 66°C | 4 | 66°C - C3 | + | 0,11 | + |
| Milk 2 Cefal30 - 62°C | 4 | 62°C - B8 | + | 6,57 | + |
| Milk 2 Cefal30 - 66°C | 4 | 66°C - C5 | + | 0,67 | + |
| Milk 3 Cefal30 - 62°C | 4 | 62°C - B6 | + | 6,4 | + |
| Milk 3 Cefal30 - 66°C | 4 | 66°C - C9 | + | 0,67 | + |

1.1.3.7. Lincosamides : lincomycine

Date : 12/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - D8 | - | -4,09 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - E1 | - | -4,78 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - D1 | - | -4,01 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - E6 | - | -5,48 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - D3 | - | -2,36 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - E9 | - | -5,69 | - |

Date : 12/08/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco275 - 62°C | 5 | 62°C - D4 | + | 3,29 | + |
| Milk 1 Linco275 - 66°C | 5 | 66°C - E7 | + | 3,53 | + |
| Milk 2 Linco275 - 62°C | 5 | 62°C - D7 | + | 4,25 | + |
| Milk 2 Linco275 - 66°C | 5 | 66°C - E2 | + | 3,74 | + |
| Milk 3 Linco275 - 62°C | 5 | 62°C - D9 | + | 4,13 | + |
| Milk 3 Linco275 - 66°C | 5 | 66°C - E8 | + | 3,78 | + |

1.1.4. Delay of reading

1.1.4.1. Penicillins

1.1.4.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 3 | 4°C - A9 | - | -9,21 | - |
| Milk 1 Neg - TA | 3 | TA - A1 | - | -10,56 | - |
| Milk 2 Neg - 4°C | 3 | 4°C - A11 | - | -8,68 | - |
| Milk 2 Neg - TA | 3 | TA - A4 | - | -9,1 | - |
| Milk 3 Neg - 4°C | 3 | 4°C - A14 | - | -10,6 | - |
| Milk 3 Neg - TA | 3 | TA - A9 | - | -10,38 | - |

Date : 20/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Amox2 - 4°C | 3 | 4°C - A7 | + | 4,92 | + |
| Milk 1 Amox2 - TA | 3 | TA - A2 | + | 4,67 | + |
| Milk 2 Amox2 - 4°C | 3 | 4°C - A1 | + | 0,58 | + |
| Milk 2 Amox2 - TA | 3 | TA - A6 | + | 4,9 | + |
| Milk 3 Amox2 - 4°C | 3 | 4°C - A12 | + | 5,34 | + |
| Milk 3 Amox2 - TA | 3 | TA - A3 | + | 4,04 | + |

1.1.4.1.2. Cloxacillin

Date : 20/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - C1 | - | -8,87 | - |
| Milk 1 Neg - TA | 5 | TA - D2 | - | -7,19 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - C6 | - | -6,78 | - |
| Milk 2 Neg - TA | 5 | TA - D1 | - | -6,75 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - C5 | - | -9,4 | - |
| Milk 3 Neg - TA | 5 | TA - D4 | - | -7,7 | - |

Date : 20/08/2020

Cloxacilline 12 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa12 - 4°C | 5 | 4°C - C11 | + | 6,8 | + |
| Milk 1 Cloxa12 - TA | 5 | TA - D11 | + | 6,49 | + |
| Milk 2 Cloxa12 - 4°C | 5 | 4°C - C12 | + | 6,75 | + |
| Milk 2 Cloxa12 - TA | 5 | TA - D13 | + | 7,07 | + |
| Milk 3 Cloxa12 - 4°C | 5 | 4°C - C13 | + | 6,72 | + |
| Milk 3 Cloxa12 - TA | 5 | TA - D12 | + | 6,87 | + |

1.1.4.2. Tetracyclines

1.1.4.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 3 | 4°C - A9 | - | -9,21 | - |
| Milk 1 Neg - TA | 3 | TA - A1 | - | -10,56 | - |
| Milk 2 Neg - 4°C | 3 | 4°C - A11 | - | -8,68 | - |
| Milk 2 Neg - TA | 3 | TA - A4 | - | -9,1 | - |
| Milk 3 Neg - 4°C | 3 | 4°C - A14 | - | -10,6 | - |
| Milk 3 Neg - TA | 3 | TA - A9 | - | -10,38 | - |

Date : 20/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 4°C | 3 | 4°C - A4 | + | 3,48 | + |
| Milk 1 Oxytetra110 - TA | 3 | TA - A14 | + | 4,02 | + |
| Milk 2 Oxytetra110 - 4°C | 3 | 4°C - A5 | + | 3,55 | + |
| Milk 2 Oxytetra110 - TA | 3 | TA - A15 | + | 3,19 | + |
| Milk 3 Oxytetra110 - 4°C | 3 | 4°C - A6 | + | 3,27 | + |
| Milk 3 Oxytetra110 - TA | 3 | TA - A13 | + | 3,2 | + |

1.1.4.2.2. Chlortetracycline

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - D2 | - | -7,71 | - |
| Milk 1 Neg - TA | 4 | TA - C1 | - | -8,68 | - |
| Milk 2 Neg - 4°C | 3 | 4°C - A6 | - | -7,76 | - |
| Milk 2 Neg - TA | 3 | TA - A7 | - | -8,81 | - |
| Milk 3 Neg - 4°C | 3 | 4°C - A2 | - | -7,82 | - |
| Milk 3 Neg - TA | 3 | TA - A3 | - | -8,53 | - |

Date : 22/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 4°C | 4 | 4°C - B12 | + | 3,83 | + |
| Milk 1 Chlortetra150 - TA | 4 | TA - C13 | + | 3,31 | + |
| Milk 2 Chlortetra150 - 4°C | 3 | 4°C - A4 | + | 5,96 | + |
| Milk 2 Chlortetra150 - TA | 3 | TA - A4 | + | 5,14 | + |
| Milk 3 Chlortetra150 - 4°C | 3 | 4°C - A7 | + | 5,25 | + |
| Milk 3 Chlortetra150 - TA | 3 | TA - A5 | + | 5,01 | + |

1.1.4.3. Sulfonamides

1.1.4.3.1. Sulfadimethoxine

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - C1 | - | -8,87 | - |
| Milk 1 Neg - TA | 5 | TA - D2 | - | -7,19 | - |
| Milk 2 Neg - 4°C | 3 | 4°C - A6 | - | -7,76 | - |
| Milk 2 Neg - TA | 3 | TA - A7 | - | -8,81 | - |
| Milk 3 Neg - 4°C | 3 | 4°C - A2 | - | -7,82 | - |
| Milk 3 Neg - TA | 3 | TA - A3 | - | -8,53 | - |

Date : 22/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 4°C | 5 | 4°C - C9 | + | 5,19 | + |
| Milk 1 Sulfadimet40 - TA | 5 | TA - D10 | + | 5,85 | + |
| Milk 2 Sulfadimet40 - 4°C | 3 | 4°C - A3 | + | 6,04 | + |
| Milk 2 Sulfadimet40 - TA | 3 | TA - A8 | + | 5,39 | + |
| Milk 3 Sulfadimet40 - 4°C | 3 | 4°C - A5 | + | 5,08 | + |
| Milk 3 Sulfadimet40 - TA | 3 | TA - A2 | + | 4,96 | + |

1.1.4.3.2. Sulfadiazine

Date : 20/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - C1 | - | -8,87 | - |
| Milk 1 Neg - TA | 5 | TA - D2 | - | -7,19 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - C6 | - | -6,78 | - |
| Milk 2 Neg - TA | 5 | TA - D1 | - | -6,75 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - C5 | - | -9,4 | - |
| Milk 3 Neg - TA | 5 | TA - D4 | - | -7,7 | - |

Date : 20/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 4°C | 5 | 4°C - C3 | + | 3,64 | + |
| Milk 1 Sulfadiaz50 - TA | 5 | TA - D9 | + | 4,16 | + |
| Milk 2 Sulfadiaz50 - 4°C | 5 | 4°C - C8 | + | 4,05 | + |
| Milk 2 Sulfadiaz50 - TA | 5 | TA - D8 | + | 4,43 | + |
| Milk 3 Sulfadiaz50 - 4°C | 5 | 4°C - C4 | + | 3,63 | + |
| Milk 3 Sulfadiaz50 - TA | 5 | TA - D6 | + | 4,1 | + |

Date : 20/08/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 4°C | 5 | 4°C - C2 | + | 4,03 | + |
| Milk 1 Sulfadiaz55 - TA | 5 | TA - D5 | + | 4,66 | + |
| Milk 2 Sulfadiaz55 - 4°C | 5 | 4°C - C7 | + | 4,17 | + |
| Milk 2 Sulfadiaz55 - TA | 5 | TA - D3 | + | 4,43 | + |
| Milk 3 Sulfadiaz55 - 4°C | 5 | 4°C - C10 | + | 4,18 | + |
| Milk 3 Sulfadiaz55 - TA | 5 | TA - D7 | + | 3,98 | + |

1.1.4.4. Macrolides

1.1.4.4.1. Tylosin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - D2 | - | -7,71 | - |
| Milk 1 Neg - TA | 4 | TA - C1 | - | -8,68 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - D3 | - | -7,54 | - |
| Milk 2 Neg - TA | 4 | TA - C3 | - | -7,35 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - D1 | - | -8,48 | - |
| Milk 3 Neg - TA | 4 | TA - C2 | - | -7,76 | - |

Date : 25/08/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - 4°C | 4 | 4°C - D7 | + | 2,82 | + |
| Milk 1 Tylo35 - TA | 4 | TA - C7 | + | 2,13 | + |
| Milk 2 Tylo35 - 4°C | 4 | 4°C - D4 | + | 3,29 | + |
| Milk 2 Tylo35 - TA | 4 | TA - C10 | + | 2,48 | + |
| Milk 3 Tylo35 - 4°C | 4 | 4°C - D10 | + | 3,64 | + |
| Milk 3 Tylo35 - TA | 4 | TA - C8 | + | 4,18 | + |

1.1.4.4. Erythromycin A

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - B1 | - | -10,66 | - |
| Milk 1 Neg - TA | 4 | TA - C4 | - | -10,5 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - B5 | - | -9,29 | - |
| Milk 2 Neg - TA | 4 | TA - C5 | - | -9,6 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - B6 | - | -11 | - |
| Milk 3 Neg - TA | 4 | TA - C6 | - | -10,86 | - |

Date : 19/08/2020

Erythromicine 160 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Erythro160 - 4°C | 4 | 4°C - B14 | + | 4,02 | + |
| Milk 1 Erythro160 - TA | 4 | TA - C14 | + | 4,55 | + |
| Milk 2 Erythro160 - 4°C | 4 | 4°C - B18 | + | 3,81 | + |
| Milk 2 Erythro160 - TA | 4 | TA - C19 | + | 3,92 | + |
| Milk 3 Erythro160 - 4°C | 4 | 4°C - B19 | + | 3,26 | + |
| Milk 3 Erythro160 - TA | 4 | TA - C15 | + | 3,59 | + |

1.1.4.5. Aminoglycosides : dihydrostreptomycin

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - B1 | - | -10,66 | - |
| Milk 1 Neg - TA | 4 | TA - C4 | - | -10,5 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - B5 | - | -9,29 | - |
| Milk 2 Neg - TA | 4 | TA - C5 | - | -9,6 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - B6 | - | -11 | - |
| Milk 3 Neg - TA | 4 | TA - C6 | - | -10,86 | - |

Date : 19/08/2020

Dihydrostreptomycine 700 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - 4°C | 4 | 4°C - B4 | + | 1,48 | + |
| Milk 1 Dihydrostrepto700 - TA | 4 | TA - C11 | + | 2,27 | + |
| Milk 2 Dihydrostrepto700 - 4°C | 4 | 4°C - B10 | + | 3,66 | + |
| Milk 2 Dihydrostrepto700 - TA | 4 | TA - C8 | + | 2,51 | + |
| Milk 3 Dihydrostrepto700 - 4°C | 4 | 4°C - B16 | + | 3,34 | + |
| Milk 3 Dihydrostrepto700 - TA | 4 | TA - C9 | + | 3,35 | + |

1.1.4.6. Cephalosporins : céfalexine

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - B1 | - | -10,66 | - |
| Milk 1 Neg - TA | 4 | TA - C4 | - | -10,5 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - B5 | - | -9,29 | - |
| Milk 2 Neg - TA | 4 | TA - C5 | - | -9,6 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - B6 | - | -11 | - |
| Milk 3 Neg - TA | 4 | TA - C6 | - | -10,86 | - |

Date : 19/08/2020

Cefalexine 30 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Cefal30 - 4°C | 4 | 4°C - B3 | + | 1,72 | + |
| Milk 1 Cefal30 - TA | 4 | TA - C2 | + | 0,86 | + |
| Milk 2 Cefal30 - 4°C | 4 | 4°C - B7 | + | 4,69 | + |
| Milk 2 Cefal30 - TA | 4 | TA - C3 | + | 4,01 | + |
| Milk 3 Cefal30 - 4°C | 4 | 4°C - B8 | + | 3,19 | + |
| Milk 3 Cefal30 - TA | 4 | TA - C1 | + | 1,7 | + |

1.1.4.7. Lincosamides : lincomycine

Date : 12/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - B1 | - | -8,26 | - |
| Milk 1 Neg - TA | 5 | TA - C2 | - | -8,06 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - B2 | - | -8,44 | - |
| Milk 2 Neg - TA | 5 | TA - C5 | - | -9,34 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - B3 | - | -5,77 | - |
| Milk 3 Neg - TA | 5 | TA - C6 | - | -8,55 | - |

Date : 12/08/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Linco275 - 4°C | 5 | 4°C - B7 | + | 1,13 | + |
| Milk 1 Linco275 - TA | 5 | TA - C3 | + | 1,16 | + |
| Milk 2 Linco275 - 4°C | 5 | 4°C - B8 | + | 1,53 | + |
| Milk 2 Linco275 - TA | 5 | TA - C4 | + | 1,07 | + |
| Milk 3 Linco275 - 4°C | 5 | 4°C - B9 | + | 1,77 | + |
| Milk 3 Linco275 - TA | 5 | TA - C9 | + | 1,43 | + |

1.2. Matrix quality

1.2.1. pH

1.2.1.1. Penicillins

1.2.1.1.1. Amoxicillin

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -13,09 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,99 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -11,42 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020

Amoxicilline 2 ppb and 2,4 ppb

Validated +20% weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Amox2,4 - weak pH | 4 | pH-D22 | + | 2,69 | + |
| Milk 1 Amox2 - high pH | 5 | pH fort - B4 | + | 8,05 | + |
| Milk 2 Amox2,4 - weak pH | 4 | pH-D23 | + | 3,29 | + |
| Milk 2 Amox2 - high pH | 5 | pH fort - B25 | + | 8,6 | + |
| Milk 3 Amox2,4 - weak pH | 4 | pH-D24 | + | 3,47 | + |
| Milk 3 Amox2 - high pH | 5 | pH fort - B26 | + | 3,76 | + |

1.2.1.1.2. Cloxacillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -10,6 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -5,16 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,43 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,79 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,72 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -7,79 | - |

Date : 02/09/2020

Cloxaciline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|----------------|----------------|-------------------|---|
| Milk 1 Cloxa10 - weak pH | 5 | pH faible - A7 | + | 3,21 | + |
| Milk 1 Cloxa10 - high pH | 5 | pH fort - B23 | + | 8,83 | + |
| Milk 2 Cloxa10 - weak pH | 5 | pH faible - A4 | + | 3,87 | + |
| Milk 2 Cloxa10 - high pH | 5 | pH fort - B3 | + | 8,85 | + |
| Milk 3 Cloxa10 - weak pH | 5 | pH faible - A9 | + | 3,39 | + |
| Milk 3 Cloxa10 - high pH | 5 | pH fort - B21 | + | 9,13 | + |

1.2.1.2. Tetracyclines

1.2.1.2.1. Oxytetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -10,6 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -5,16 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,43 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,79 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,72 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -7,79 | - |

Date : 02/09/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - weak pH | 5 | pH faible - A11 | + | 3,25 | + |
| Milk 1 Oxytetra110 - high pH | 5 | pH fort - B22 | + | 5,39 | + |
| Milk 2 Oxytetra110 - weak pH | 5 | pH faible - A3 | + | 2,22 | + |
| Milk 2 Oxytetra110 - high pH | 5 | pH fort - B24 | + | 4,9 | + |
| Milk 3 Oxytetra110 - weak pH | 5 | pH faible - A10 | + | 2,97 | + |
| Milk 3 Oxytetra110 - high pH | 5 | pH fort - B2 | + | 4,93 | + |

1.2.1.2.2. Chlortetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -10,6 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -5,16 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,43 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,79 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,72 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -7,79 | - |

Date : 02/09/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - weak pH | 5 | pH faible - A16 | + | 4,14 | + |
| Milk 1 Chlortetra150 - high pH | 5 | pH fort - B18 | + | 5,91 | + |
| Milk 2 Chlortetra150 - weak pH | 5 | pH faible - A23 | + | 3,74 | + |
| Milk 2 Chlortetra150 - high pH | 5 | pH fort - B6 | + | 5,95 | + |
| Milk 3 Chlortetra150 - weak pH | 5 | pH faible - A21 | + | 3,99 | + |
| Milk 3 Chlortetra150 - high pH | 5 | pH fort - B20 | + | 5,76 | + |

1.2.1.3. Sulfonamides

1.2.1.3.1. Sulfadimethoxine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -10,6 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -5,16 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,43 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,79 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,72 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -7,79 | - |

Date : 02/09/2020

Sulfadimethoxine 40 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|-------------------------------|-------|-----------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Sulfadimet40 - weak pH | 5 | pH faible - A18 | + | 3,3 | + |
| Milk 1 Sulfadimet40 - high pH | 5 | pH fort - B10 | + | 7,8 | + |
| Milk 2 Sulfadimet40 - weak pH | 5 | pH faible - A24 | + | 4,9 | + |
| Milk 2 Sulfadimet40 - high pH | 5 | pH fort - B16 | + | 7,53 | + |
| Milk 3 Sulfadimet40 - weak pH | 5 | pH faible - A27 | + | 2,62 | + |
| Milk 3 Sulfadimet40 - high pH | 5 | pH fort - B9 | + | 7,54 | + |

1.2.1.3.2. Sulfadiazine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH faible - A1 | - | -11,21 | - |
| Milk 1 Neg - high pH | 4 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH faible - A5 | - | -10,37 | - |
| Milk 2 Neg - high pH | 4 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH faible - A2 | - | -10,66 | - |
| Milk 3 Neg - high pH | 4 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020

Sulfadiazine 50 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|------------------------------|-------|----------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Sulfadiaz50 - weak pH | 4 | pH faible - A4 | + | 1,02 | + |
| Milk 1 Sulfadiaz50 - high pH | 4 | pH fort - B5 | + | 7,28 | + |
| Milk 2 Sulfadiaz50 - weak pH | 4 | pH faible - A6 | + | 0,93 | + |
| Milk 2 Sulfadiaz50 - high pH | 4 | pH fort - B4 | + | 6,75 | + |
| Milk 3 Sulfadiaz50 - weak pH | 4 | pH faible - A9 | + | 0,38 | + |
| Milk 3 Sulfadiaz50 - high pH | 4 | pH fort - B6 | + | 6,51 | + |

Date : 01/09/2020

Sulfadiazine 55 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|------------------------------|-------|----------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Sulfadiaz55 - weak pH | 4 | pH faible - A7 | + | 1,5 | + |
| Milk 1 Sulfadiaz55 - high pH | 4 | pH fort - B3 | + | 6,38 | + |
| Milk 2 Sulfadiaz55 - weak pH | 4 | pH faible - A3 | + | 1,63 | + |
| Milk 2 Sulfadiaz55 - high pH | 4 | pH fort - B7 | + | 6,83 | + |
| Milk 3 Sulfadiaz55 - weak pH | 4 | pH faible - A8 | + | 1,6 | + |
| Milk 3 Sulfadiaz55 - high pH | 4 | pH fort - B1 | + | 6,55 | + |

1.2.1.4. Macrolides

1.2.1.4.1. Tylosin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -13,09 | - |
| Milk 1 Neg - high pH | 4 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,99 | - |
| Milk 2 Neg - high pH | 4 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -11,42 | - |
| Milk 3 Neg - high pH | 4 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020

Tylosine 35 ppb and 42 ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Tylo42 - weak pH | 4 | pH-D11 | - | -3,3 | - |
| Milk 1 Tylo35 - high pH | 4 | pH fort - B8 | + | 6,15 | + |
| Milk 2 Tylo42 - weak pH | 4 | pH-D5 | - | -2,25 | - |
| Milk 2 Tylo35 - high pH | 4 | pH fort - B10 | + | 7,29 | + |
| Milk 3 Tylo42 - weak pH | 4 | pH-D9 | - | -2,34 | - |
| Milk 3 Tylo35 - high pH | 4 | pH fort - B9 | + | 6,84 | + |

1.2.1.4.2. Erythromycin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -13,09 | - |
| Milk 1 Neg - high pH | 4 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,99 | - |
| Milk 2 Neg - high pH | 4 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -11,42 | - |
| Milk 3 Neg - high pH | 4 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020

Erythromycine 160 ppb and 192 ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Erythro160 - high pH | 4 | pH fort - B12 | + | 6,82 | + |
| Milk 1 Erythro192 - weak pH | 4 | pH-D13 | - | -2,21 | - |
| Milk 2 Erythro160 - high pH | 4 | pH fort - B11 | + | 6,66 | + |
| Milk 2 Erythro192 - weak pH | 4 | pH-D4 | - | -2,22 | - |
| Milk 3 Erythro160 - high pH | 4 | pH fort - B14 | + | 7,21 | + |
| Milk 3 Erythro192 - weak pH | 4 | pH-D12 | - | -1,6 | - |

1.2.1.5. Aminoglycosides : dihydrostreptomycin

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -13,09 | - |
| Milk 1 Neg - high pH | 4 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,99 | - |
| Milk 2 Neg - high pH | 4 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -11,42 | - |
| Milk 3 Neg - high pH | 4 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020
Dihydrostreptomycine 700 ppb and 840 ppb

Not Validated weak pH
Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto840 - weak pH | 4 | pH-D18 | - | -2,43 | - |
| Milk 1 Dihydrostrepto700 - high pH | 4 | pH fort - B21 | + | 6,62 | + |
| Milk 2 Dihydrostrepto840 - weak pH | 4 | pH-D14 | - | -2,9 | - |
| Milk 2 Dihydrostrepto700 - high pH | 4 | pH fort - B20 | + | 7,12 | + |
| Milk 3 Dihydrostrepto840 - weak pH | 4 | pH-D19 | - | -0,83 | - |
| Milk 3 Dihydrostrepto700 - high pH | 4 | pH fort - B24 | + | 6,76 | + |

1.2.1.6. Cephalosporins : céfalexine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH faible - A1 | - | -11,21 | - |
| Milk 1 Neg - high pH | 4 | pH fort - B2 | - | -5,78 | - |
| Milk 2 Neg - weak pH | 4 | pH faible - A5 | - | -10,37 | - |
| Milk 2 Neg - high pH | 4 | pH fort - B13 | - | -4,45 | - |
| Milk 3 Neg - weak pH | 4 | pH faible - A2 | - | -10,66 | - |
| Milk 3 Neg - high pH | 4 | pH fort - B18 | - | -6,09 | - |

Date : 01/09/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Cefal30 - weak pH | 4 | pH faible - A25 | + | 2,04 | + |
| Milk 1 Cefal30 - high pH | 4 | pH fort - B26 | + | 7,86 | + |
| Milk 2 Cefal30 - weak pH | 4 | pH faible - A26 | + | 1,64 | + |
| Milk 2 Cefal30 - high pH | 4 | pH fort - B25 | + | 5,35 | + |
| Milk 3 Cefal30 - weak pH | 4 | pH faible - A27 | + | 0,31 | + |
| Milk 3 Cefal30 - high pH | 4 | pH fort - B27 | + | 8,95 | + |

1.2.1.7. Lincosamides : lincomycine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -10,6 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -5,16 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,43 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,79 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,72 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -7,79 | - |

Date : 02/09/2020

Lincomycine 275 ppb and 330 ppb

Not Validated weak pH
Validated strong pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Linco330 - weak pH | 5 | pH faible - A17 | - | -4,33 | - |
| Milk 1 Linco275 - high pH | 5 | pH fort - B17 | + | 6,49 | + |
| Milk 2 Linco330 - weak pH | 5 | pH faible - A20 | - | -3,82 | - |
| Milk 2 Linco275 - high pH | 5 | pH fort - B12 | + | 6,75 | + |
| Milk 3 Linco330 - weak pH | 5 | pH faible - A25 | - | -4,56 | - |
| Milk 3 Linco275 - high pH | 5 | pH fort - B14 | + | 7,03 | + |

1.2.2. Total Bacteria Count

1.2.2.1. Penicillins

1.2.2.1.1. Amoxicillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Amoxicilline 2 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Amox2 - high TBC | 5 | Mat - C2 | + | 4,43 | + |
| Milk 2 Amox2 - high TBC | 5 | Mat - C8 | + | 5,03 | + |
| Milk 3 Amox2 - high TBC | 5 | Mat - C7 | + | 4,31 | + |

1.2.2.1.2. Cloxacillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Cloxacilline 30 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - high TBC | 5 | Mat - C14 | + | 8,07 | + |
| Milk 2 Cloxa10 - high TBC | 5 | Mat - C6 | + | 9,27 | + |
| Milk 3 Cloxa10 - high TBC | 5 | Mat - C12 | + | 8,58 | + |

1.2.2.2. Tetracyclines

1.2.2.2.1. Oxytetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Oxytetracycline 110 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - high TBC | 5 | Mat - C17 | + | 3,74 | + |
| Milk 2 Oxytetra110 - high TBC | 5 | Mat - C11 | + | 4,05 | + |
| Milk 3 Oxytetra110 - high TBC | 5 | Mat - C15 | + | 3,95 | + |

1.2.2.2.2. Chlortetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - high TBC | 5 | Mat - C20 | + | 5 | + |
| Milk 2 Chlortetra150 - high TBC | 5 | Mat - C5 | + | 5,88 | + |
| Milk 3 Chlortetra150 - high TBC | 5 | Mat - C24 | + | 5,57 | + |

1.2.2.3. Sulfonamides

1.2.2.3.1. Sulfadimethoxine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - high TBC | 5 | Mat - C25 | + | 5,72 | + |
| Milk 2 Sulfadimet40 - high TBC | 5 | Mat - C18 | + | 5,49 | + |
| Milk 3 Sulfadimet40 - high TBC | 5 | Mat - C22 | + | 5,79 | + |

1.2.2.3.2. Sulfadiazine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 4 | Mat - C14 | - | -7,95 | - |
| Milk 2 Neg - high TBC | 4 | Mat - C1 | - | -8,11 | - |
| Milk 3 Neg - high TBC | 4 | Mat - C11 | - | -7,95 | - |

Date : 01/09/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - high TBC | 4 | Mat - C4 | + | 5,47 | + |
| Milk 2 Sulfadiaz50 - high TBC | 4 | Mat - C8 | + | 4,94 | + |
| Milk 3 Sulfadiaz50 - high TBC | 4 | Mat - C3 | + | 5,68 | + |

Date : 01/09/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - high TBC | 4 | Mat - C5 | + | 6,03 | + |
| Milk 2 Sulfadiaz55 - high TBC | 4 | Mat - C10 | + | 5,1 | + |
| Milk 3 Sulfadiaz55 - high TBC | 4 | Mat - C2 | + | 5,19 | + |

1.2.2.4. Macrolides

1.2.2.4.1. Tylosin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 4 | Mat - C14 | - | -7,95 | - |
| Milk 2 Neg - high TBC | 4 | Mat - C1 | - | -8,11 | - |
| Milk 3 Neg - high TBC | 4 | Mat - C11 | - | -7,95 | - |

Date : 01/09/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - high TBC | 4 | Mat - C12 | + | 4,42 | + |
| Milk 2 Tylo35 - high TBC | 4 | Mat - C13 | + | 3,66 | + |
| Milk 3 Tylo35 - high TBC | 4 | Mat - C7 | + | 5,09 | + |

1.2.2.4.2. Erythromycin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 4 | Mat - C14 | - | -7,95 | - |
| Milk 2 Neg - high TBC | 4 | Mat - C1 | - | -8,11 | - |
| Milk 3 Neg - high TBC | 4 | Mat - C11 | - | -7,95 | - |

Date : 01/09/2020

Erythromycine A (3 éch) 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Erythro160 - high TBC | 4 | Mat - C17 | + | 4,88 | + |
| Milk 2 Erythro160 - high TBC | 4 | Mat - C6 | + | 5,58 | + |
| Milk 3 Erythro160 - high TBC | 4 | Mat - C15 | + | 4,97 | + |

1.2.2.5. Aminoglycosides : dihydrostreptomycin

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 4 | Mat - C14 | - | -7,95 | - |
| Milk 2 Neg - high TBC | 4 | Mat - C1 | - | -8,11 | - |
| Milk 3 Neg - high TBC | 4 | Mat - C11 | - | -7,95 | - |

Date : 01/09/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - high TBC | 4 | Mat - C23 | + | 4,66 | + |
| Milk 2 Dihydrostrepto700 - high TBC | 4 | Mat - C18 | + | 4,87 | + |
| Milk 3 Dihydrostrepto700 - high TBC | 4 | Mat - C26 | + | 5,27 | + |

1.2.2.6. Cephalosporins : céfalexine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 4 | Mat - C14 | - | -7,95 | - |
| Milk 2 Neg - high TBC | 4 | Mat - C1 | - | -8,11 | - |
| Milk 3 Neg - high TBC | 4 | Mat - C11 | - | -7,95 | - |

Date : 01/09/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cefal30 - high TBC | 4 | Mat - C25 | + | 4,1 | + |
| Milk 2 Cefal30 - high TBC | 4 | Mat - C27 | + | 4,95 | + |
| Milk 3 Cefal30 - high TBC | 4 | Mat - C24 | + | 3,51 | + |

1.2.2.7. Lincosamides : lincomycine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -8,08 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -8,69 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -8,64 | - |

Date : 02/09/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco275 - high TBC | 5 | Mat - C19 | + | 2,55 | + |
| Milk 2 Linco275 - high TBC | 5 | Mat - C26 | + | 3,76 | + |
| Milk 3 Linco275 - high TBC | 5 | Mat - C27 | + | 3,32 | + |

1.2.3. Frozen milk

1.2.3.1. Penicillins

1.2.3.1.1. Amoxicillin

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------|----------------|-------------------|---|
| Milk 1 Amox2 - frozen | 3 | 1-A1 | + | 4,87 | + |
| Milk 2 Amox2 - frozen | 3 | 2-B4 | + | 4,76 | + |
| Milk 3 Amox2 - frozen | 3 | 3-C9 | + | 4,67 | + |

1.2.3.1.2. Cloxacillin

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-D3 | - | -4,73 | - |
| Milk 2 Neg - frozen | 3 | 2-E5 | - | -5,31 | - |
| Milk 3 Neg - frozen | 3 | 3-F2 | - | -7 | - |

Date : 14/08/2020

Cloxacilline 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Cloxa10 - frozen | 3 | 1-D2 | + | 6,47 | + |
| Milk 2 Cloxa10 - frozen | 3 | 2-E9 | + | 6,52 | + |
| Milk 3 Cloxa10 - frozen | 3 | 3-F9 | + | 5,77 | + |

1.2.3.2. Tetracyclines

1.2.3.2.1. Oxytetracycline

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - frozen | 3 | 1-A5 | + | 4,2 | + |
| Milk 2 Oxytetra110 - frozen | 3 | 2-B6 | + | 4,28 | + |
| Milk 3 Oxytetra110 - frozen | 3 | 3-C7 | + | 4,4 | + |

1.2.3.2.2. Chlortetracycline

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-D3 | - | -4,73 | - |
| Milk 2 Neg - frozen | 3 | 2-E5 | - | -5,31 | - |
| Milk 3 Neg - frozen | 3 | 3-F2 | - | -7 | - |

Date : 14/08/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - frozen | 3 | 1-D9 | + | 5,92 | + |
| Milk 2 Chlortetra150 - frozen | 3 | 2-E8 | + | 5,46 | + |
| Milk 3 Chlortetra150 - frozen | 3 | 3-F8 | + | 5,82 | + |

1.2.3.3. Sulfonamides

1.2.3.3.1. Sulfadimethoxine

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - frozen | 3 | 1-A3 | + | 5,67 | + |
| Milk 2 Sulfadimet40 - frozen | 3 | 2-B5 | + | 5,68 | + |
| Milk 3 Sulfadimet40 - frozen | 3 | 3-C3 | + | 5,63 | + |

1.2.3.3.2. Sulfadiazine

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-D3 | - | -4,73 | - |
| Milk 2 Neg - frozen | 3 | 2-E5 | - | -5,31 | - |
| Milk 3 Neg - frozen | 3 | 3-F2 | - | -7 | - |

Date : 14/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - frozen | 3 | 1-D5 | + | 4,62 | + |
| Milk 2 Sulfadiaz50 - frozen | 3 | 2-E3 | + | 4,5 | + |
| Milk 3 Sulfadiaz50 - frozen | 3 | 3-F4 | + | 4,29 | + |

Date : 14/08/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - frozen | 3 | 1-D4 | + | 4,5 | + |
| Milk 2 Sulfadiaz55 - frozen | 3 | 2-E2 | + | 4,75 | + |
| Milk 3 Sulfadiaz55 - frozen | 3 | 3-F3 | + | 4,52 | + |

1.2.3.4. Macrolides

1.2.3.4.1. Tylosin A

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Tylo35 - frozen | 3 | 1-A9 | + | 4,14 | + |
| Milk 2 Tylo35 - frozen | 3 | 2-B7 | + | 2,67 | + |
| Milk 3 Tylo35 - frozen | 3 | 3-C1 | + | 4,27 | + |

1.2.3.4.2. Erythromycin A

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-D3 | - | -4,73 | - |
| Milk 2 Neg - frozen | 3 | 2-E5 | - | -5,31 | - |
| Milk 3 Neg - frozen | 3 | 3-F2 | - | -7 | - |

Date : 14/08/2020

Erythromycine A (3 éch) 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Erythro160 - frozen | 3 | 1-D8 | + | 4,94 | + |
| Milk 2 Erythro160 - frozen | 3 | 2-E7 | + | 4,81 | + |
| Milk 3 Erythro160 - frozen | 3 | 3-F1 | + | 4,84 | + |

1.2.3.5. Aminoglycosides : dihydrostreptomycin

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - frozen | 3 | 1-A8 | + | 5,84 | + |
| Milk 2 Dihydrostrepto700 - frozen | 3 | 2-B9 | + | 5,37 | + |
| Milk 3 Dihydrostrepto700 - frozen | 3 | 3-C6 | + | 5,33 | + |

1.2.3.6. Cephalosporins : céfalexine

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-A4 | - | -7,32 | - |
| Milk 2 Neg - frozen | 3 | 2-B2 | - | -5,95 | - |
| Milk 3 Neg - frozen | 3 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Cefal30 - frozen | 3 | 1-A7 | + | 3,89 | + |
| Milk 2 Cefal30 - frozen | 3 | 2-B3 | + | 4,13 | + |
| Milk 3 Cefal30 - frozen | 3 | 3-C2 | + | 2,55 | + |

1.2.3.7. Lincosamides : lincomycine

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 3 | 1-D3 | - | -4,73 | - |
| Milk 2 Neg - frozen | 3 | 2-E5 | - | -5,31 | - |
| Milk 3 Neg - frozen | 3 | 3-F2 | - | -7 | - |

Date : 14/08/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Linco275 - frozen | 3 | 1-D6 | + | 3,89 | + |
| Milk 2 Linco275 - frozen | 3 | 2-E6 | + | 3,19 | + |
| Milk 3 Linco275 - frozen | 3 | 3-F5 | + | 3,24 | + |

1.2.4. Milk temperature

1.2.4.1. Penicillins

1.2.4.1.1. Amoxicillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,88 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -7,53 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -3,46 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -3,9 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -4,25 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,05 | - |

Date : 13/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Amox2 - room temperature | 6 | TA - C1 | + | 3,34 | + |
| Milk 1 Amox2 - cold | 6 | 4°C - D6 | + | 5,71 | + |
| Milk 2 Amox2 - room temperature | 6 | TA - C8 | + | 4,31 | + |
| Milk 2 Amox2 - cold | 6 | 4°C - D4 | + | 4,67 | + |
| Milk 3 Amox2 - room temperature | 6 | TA - C13 | + | 4,98 | + |
| Milk 3 Amox2 - cold | 6 | 4°C - D7 | + | 4,4 | + |

1.2.4.1.2. Cloxacillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,88 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -7,53 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -3,46 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -3,9 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -4,25 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,05 | - |

Date : 13/07/2020

Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - room temperature | 6 | TA - C12 | + | 6,48 | + |
| Milk 1 Cloxa10 - cold | 6 | 4°C - D16 | + | 6,11 | + |
| Milk 2 Cloxa10 - room temperature | 6 | TA - C5 | + | 6,24 | + |
| Milk 2 Cloxa10 - cold | 6 | 4°C - D2 | + | 5,49 | + |
| Milk 3 Cloxa10 - room temperature | 6 | TA - C3 | + | 5,56 | + |
| Milk 3 Cloxa10 - cold | 6 | 4°C - D9 | + | 5,1 | + |

1.2.4.2. Tetracyclines

1.2.4.2.1. Oxytetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,88 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -7,53 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -3,46 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -3,9 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -4,25 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,05 | - |

Date : 13/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - room temperature | 6 | TA - C6 | + | 3,74 | + |
| Milk 1 Oxytetra110 - cold | 6 | 4°C - D11 | + | 4,9 | + |
| Milk 2 Oxytetra110 - room temperature | 6 | TA - C23 | + | 4,39 | + |
| Milk 2 Oxytetra110 - cold | 6 | 4°C - D13 | + | 4,79 | + |
| Milk 3 Oxytetra110 - room temperature | 6 | TA - C24 | + | 4,41 | + |
| Milk 3 Oxytetra110 - cold | 6 | 4°C - D5 | + | 5,34 | + |

1.2.4.2.2. Chlortetracycline

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,14 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -8,14 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -6,63 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -4,85 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,08 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -7,33 | - |

Date : 15/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - room temperature | 6 | TA - C27 | + | 5,45 | + |
| Milk 1 Chlortetra150 - cold | 6 | 4°C - D27 | + | 5,54 | + |
| Milk 2 Chlortetra150 - room temperature | 6 | TA - C24 | + | 5,95 | + |
| Milk 2 Chlortetra150 - cold | 6 | 4°C - D26 | + | 4,96 | + |
| Milk 3 Chlortetra150 - room temperature | 6 | TA - C26 | + | 5,92 | + |
| Milk 3 Chlortetra150 - cold | 6 | 4°C - D21 | + | 5,44 | + |

1.2.4.3. Sulfonamides

1.2.4.3.1. Sulfadimethoxine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,88 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -7,53 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -3,46 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -3,9 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -4,25 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,05 | - |

Date : 13/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - room temperature | 6 | TA - C2 | + | 5,56 | + |
| Milk 1 Sulfadimet40 - cold | 6 | 4°C - D12 | + | 6,1 | + |
| Milk 2 Sulfadimet40 - room temperature | 6 | TA - C4 | + | 5,41 | + |
| Milk 2 Sulfadimet40 - cold | 6 | 4°C - D17 | + | 5,85 | + |
| Milk 3 Sulfadimet40 - room temperature | 6 | TA - C9 | + | 5,46 | + |
| Milk 3 Sulfadimet40 - cold | 6 | 4°C - D22 | + | 5,3 | + |

1.2.4.3.2. Sulfadiazine

Date : 20/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 5 | Milk TA - E1 | - | -7,2 | - |
| Milk 1 Neg - cold | 5 | Milk 4°C - F1 | - | -6,78 | - |
| Milk 2 Neg - room temperature | 5 | Milk TA - E4 | - | -6,45 | - |
| Milk 2 Neg - cold | 5 | Milk 4°C - F2 | - | -6,65 | - |
| Milk 3 Neg - room temperature | 5 | Milk TA - E6 | - | -7,89 | - |
| Milk 3 Neg - cold | 5 | Milk 4°C - F3 | - | -7,32 | - |

Date : 20/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - room temperature | 5 | Milk TA - E2 | + | 3,93 | + |
| Milk 1 Sulfadiaz50 - cold | 5 | Milk 4°C - F4 | + | 4,29 | + |
| Milk 2 Sulfadiaz50 - room temperature | 5 | Milk TA - E5 | + | 4,47 | + |
| Milk 2 Sulfadiaz50 - cold | 5 | Milk 4°C - F5 | + | 4,9 | + |
| Milk 3 Sulfadiaz50 - room temperature | 5 | Milk TA - E3 | + | 4,11 | + |
| Milk 3 Sulfadiaz50 - cold | 5 | Milk 4°C - F6 | + | 4,39 | + |

1.2.4.4. Macrolides

1.2.4.4.1. Tylosin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,14 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -8,14 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -6,63 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -4,85 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,08 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -7,33 | - |

Date : 15/07/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - room temperature | 6 | TA - C13 | + | 3,72 | + |
| Milk 1 Tylo35 - cold | 6 | 4°C - D5 | + | 4,74 | + |
| Milk 2 Tylo35 - room temperature | 6 | TA - C12 | + | 3,67 | + |
| Milk 2 Tylo35 - cold | 6 | 4°C - D14 | + | 4,27 | + |
| Milk 3 Tylo35 - room temperature | 6 | TA - C4 | + | 4,38 | + |
| Milk 3 Tylo35 - cold | 6 | 4°C - D11 | + | 4,69 | + |

1.2.4.4.2. Erythromycin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,14 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -8,14 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -6,63 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -4,85 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,08 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -7,33 | - |

Date : 15/07/2020

Erythromycine 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Erythro160 - room temperature | 6 | TA - C11 | + | 4,86 | + |
| Milk 1 Erythro160 - cold | 6 | 4°C - D17 | + | 5,75 | + |
| Milk 2 Erythro160 - room temperature | 6 | TA - C20 | + | 4,1 | + |
| Milk 2 Erythro160 - cold | 6 | 4°C - D23 | + | 4,65 | + |
| Milk 3 Erythro160 - room temperature | 6 | TA - C16 | + | 4,43 | + |
| Milk 3 Erythro160 - cold | 6 | 4°C - D16 | + | 5,15 | + |

1.2.4.5. Aminoglycosides : dihydrostreptomycin

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,14 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -8,14 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -6,63 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -4,85 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,08 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -7,33 | - |

Date : 15/07/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---|-------|-----------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - room temperature | 6 | TA - C17 | + | 5,02 | + |
| Milk 1 Dihydrostrepto700 - cold | 6 | 4°C - D22 | + | 4,69 | + |
| Milk 2 Dihydrostrepto700 - room temperature | 6 | TA - C21 | + | 3,18 | + |
| Milk 2 Dihydrostrepto700 - cold | 6 | 4°C - D15 | + | 4,23 | + |
| Milk 3 Dihydrostrepto700 - room temperature | 6 | TA - C19 | + | 4,57 | + |
| Milk 3 Dihydrostrepto700 - cold | 6 | 4°C - D13 | + | 4,9 | + |

1.2.4.6. Cephalosporins : céfalexine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,14 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -8,14 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -6,63 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -4,85 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,08 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -7,33 | - |

Date : 15/07/2020

Cefalexine 30 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cefal30 - room temperature | 6 | TA - C23 | + | 5,39 | + |
| Milk 1 Cefal30 - cold | 6 | 4°C - D24 | + | 5,35 | + |
| Milk 2 Cefal30 - room temperature | 6 | TA - C25 | + | 5,57 | + |
| Milk 2 Cefal30 - cold | 6 | 4°C - D19 | + | 4,06 | + |
| Milk 3 Cefal30 - room temperature | 6 | TA - C22 | + | 5,66 | + |
| Milk 3 Cefal30 - cold | 6 | 4°C - D25 | + | 4,26 | + |

1.2.4.7. Lincosamides : lincomycine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,88 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -7,53 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -3,46 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -3,9 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -4,25 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,05 | - |

Date : 13/07/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco275 - room temperature | 6 | TA - C10 | + | 3,55 | + |
| Milk 1 Linco275 - cold | 6 | 4°C - D24 | + | 3,42 | + |
| Milk 2 Linco275 - room temperature | 6 | TA - C20 | + | 3,31 | + |
| Milk 2 Linco275 - cold | 6 | 4°C - D21 | + | 3,38 | + |
| Milk 3 Linco275 - room temperature | 6 | TA - C18 | + | 4,3 | + |
| Milk 3 Linco275 - cold | 6 | 4°C - D19 | + | 3,9 | + |

2. Results of robustness study for plates

2.1. Influence of protocol

2.1.1. Sample volume

2.1.1.1. Penicillins

2.1.1.1.1. Amoxicillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Amox2 - 90 µL | 6 | 90µL - A3 | + | 5,14 | + |
| Milk 1 Amox2 - 110 µL | 6 | 110µL - A1 | + | 7,36 | + |
| Milk 2 Amox2 - 90 µL | 6 | 90µL - A8 | + | 4,32 | + |
| Milk 2 Amox2 - 110 µL | 6 | 110µL - A8 | + | 6,53 | + |
| Milk 3 Amox2 - 90 µL | 6 | 90µL - A9 | + | 3,83 | + |
| Milk 3 Amox2 - 110 µL | 6 | 110µL - A4 | + | 7,13 | + |

2.1.1.1.2. Cloxacillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Cloxa10 - 90 µL | 6 | 90µL - A12 | + | 7 | + |
| Milk 1 Cloxa10 - 110 µL | 6 | 110µL - A6 | + | 5,88 | + |
| Milk 2 Cloxa10 - 90 µL | 6 | 90µL - A15 | + | 5,76 | + |
| Milk 2 Cloxa10 - 110 µL | 6 | 110µL - A13 | + | 6,86 | + |
| Milk 3 Cloxa10 - 90 µL | 6 | 90µL - A18 | + | 6,12 | + |
| Milk 3 Cloxa10 - 110 µL | 6 | 110µL - A11 | + | 6,65 | + |

2.1.1.2. Tetracyclines

2.1.1.2.1. Oxytetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 90 µL | 6 | 90µL - A10 | + | 4,95 | + |
| Milk 1 Oxytetra110 - 110 µL | 6 | 110µL - A20 | + | 5,27 | + |
| Milk 2 Oxytetra110 - 90 µL | 6 | 90µL - A4 | + | 4,48 | + |
| Milk 2 Oxytetra110 - 110 µL | 6 | 110µL - A12 | + | 5,05 | + |
| Milk 3 Oxytetra110 - 90 µL | 6 | 90µL - A6 | + | 5,27 | + |
| Milk 3 Oxytetra110 - 110 µL | 6 | 110µL - A5 | + | 6,51 | + |

2.1.1.2.2. Chlortetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 90 µL | 6 | 90µL - A1 | + | 5,82 | + |
| Milk 1 Chlortetra150 - 110 µL | 6 | 110µL - A7 | + | 6,37 | + |
| Milk 2 Chlortetra150 - 90 µL | 6 | 90µL - A2 | + | 5,65 | + |
| Milk 2 Chlortetra150 - 110 µL | 6 | 110µL - A15 | + | 6,07 | + |
| Milk 3 Chlortetra150 - 90 µL | 6 | 90µL - A5 | + | 5,58 | + |
| Milk 3 Chlortetra150 - 110 µL | 6 | 110µL - A17 | + | 6,15 | + |

2.1.1.3. Sulfonamides

2.1.1.3.1. Sulfadimethoxine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 90 µL | 6 | 90µL - A7 | + | 5,78 | + |
| Milk 1 Sulfadimet40 - 110 µL | 6 | 110µL - A18 | + | 6,5 | + |
| Milk 2 Sulfadimet40 - 90 µL | 6 | 90µL - A14 | + | 5,73 | + |
| Milk 2 Sulfadimet40 - 110 µL | 6 | 110µL - A24 | + | 5,96 | + |
| Milk 3 Sulfadimet40 - 90 µL | 6 | 90µL - A11 | + | 5,81 | + |
| Milk 3 Sulfadimet40 - 110 µL | 6 | 110µL - A25 | + | 5,73 | + |

2.1.1.3.2. Sulfadiazine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 90 µL | 6 | 90µL - A2 | + | 5,22 | + |
| Milk 1 Sulfadiaz50 - 110 µL | 6 | 110µL - B4 | + | 4,3 | + |
| Milk 2 Sulfadiaz50 - 90 µL | 6 | 90µL - A3 | + | 4,72 | + |
| Milk 2 Sulfadiaz50 - 110 µL | 6 | 110µL - B6 | + | 4,56 | + |
| Milk 3 Sulfadiaz50 - 90 µL | 6 | 90µL - A6 | + | 4,19 | + |
| Milk 3 Sulfadiaz50 - 110 µL | 6 | 110µL - B1 | + | 4,05 | + |

Date : 15/07/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 90 µL | 6 | 90µL - A1 | + | 5,02 | + |
| Milk 1 Sulfadiaz55 - 110 µL | 6 | 110µL - B4 | + | 5,61 | + |
| Milk 2 Sulfadiaz55 - 90 µL | 6 | 90µL - A5 | + | 5,08 | + |
| Milk 2 Sulfadiaz55 - 110 µL | 6 | 110µL - B11 | + | 5,01 | + |
| Milk 3 Sulfadiaz55 - 90 µL | 6 | 90µL - A10 | + | 5,02 | + |
| Milk 3 Sulfadiaz55 - 110 µL | 6 | 110µL - B13 | + | 4,73 | + |

2.1.1.4. Macrolides

2.1.1.4.1. Tylosin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Tylo35 - 90 µL | 6 | 90µL - A15 | + | 4,22 | + |
| Milk 1 Tylo35 - 110 µL | 6 | 110µL - B5 | + | 4,5 | + |
| Milk 2 Tylo35 - 90 µL | 6 | 90µL - A7 | + | 3,33 | + |
| Milk 2 Tylo35 - 110 µL | 6 | 110µL - B2 | + | 4,62 | + |
| Milk 3 Tylo35 - 90 µL | 6 | 90µL - A2 | + | 3,83 | + |
| Milk 3 Tylo35 - 110 µL | 6 | 110µL - B6 | + | 4,69 | + |

2.1.1.4.2. Erythromycin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Erythromicine 160 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Erythro160 - 90 µL | 6 | 90µL - A3 | + | 4,85 | + |
| Milk 1 Erythro160 - 110 µL | 6 | 110µL - B3 | + | 5,5 | + |
| Milk 2 Erythro160 - 90 µL | 6 | 90µL - A12 | + | 3,9 | + |
| Milk 2 Erythro160 - 110 µL | 6 | 110µL - B8 | + | 4,55 | + |
| Milk 3 Erythro160 - 90 µL | 6 | 90µL - A16 | + | 4,85 | + |
| Milk 3 Erythro160 - 110 µL | 6 | 110µL - B19 | + | 5,03 | + |

2.1.1.5. Aminoglycosides : dihydrostreptomycin

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Dihydrostreptomycine 700 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto700 - 90 µL | 6 | 90µL - A13 | + | 4,73 | + |
| Milk 1 Dihydrostrepto700 - 110 µL | 6 | 110µL - B14 | + | 5,68 | + |
| Milk 2 Dihydrostrepto700 - 90 µL | 6 | 90µL - A25 | + | 3,9 | + |
| Milk 2 Dihydrostrepto700 - 110 µL | 6 | 110µL - B15 | + | 5,04 | + |
| Milk 3 Dihydrostrepto700 - 90 µL | 6 | 90µL - A20 | + | 4,16 | + |
| Milk 3 Dihydrostrepto700 - 110 µL | 6 | 110µL - B24 | + | 4,99 | + |

2.1.1.6. Cephalosporins : céfalexine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A18 | - | -5,58 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - B22 | - | -4,92 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A26 | - | -4,74 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - B18 | - | -5,97 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,57 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - B27 | - | -4,75 | - |

Date : 15/07/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Cefal30 - 90 µL | 6 | 90µL - A21 | + | 5,67 | + |
| Milk 1 Cefal30 - 110 µL | 6 | 110µL - B21 | + | 5,6 | + |
| Milk 2 Cefal30 - 90 µL | 6 | 90µL - A27 | + | 5,21 | + |
| Milk 2 Cefal30 - 110 µL | 6 | 110µL - B26 | + | 5,61 | + |
| Milk 3 Cefal30 - 90 µL | 6 | 90µL - A24 | + | 5,54 | + |
| Milk 3 Cefal30 - 110 µL | 6 | 110µL - B25 | + | 6,29 | + |

2.1.1.7. Lincosamides : lincomycine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 90 µL | 6 | 90µL - A17 | - | -5,22 | - |
| Milk 1 Neg - 110 µL | 6 | 110µL - A2 | - | -2,4 | - |
| Milk 2 Neg - 90 µL | 6 | 90µL - A20 | - | -2,96 | - |
| Milk 2 Neg - 110 µL | 6 | 110µL - A3 | - | -0,41 | - |
| Milk 3 Neg - 90 µL | 6 | 90µL - A23 | - | -3,63 | - |
| Milk 3 Neg - 110 µL | 6 | 110µL - A9 | - | -2,28 | - |

Date : 13/07/2020

Lincomycine 275 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-------------|----------------|-------------------|---|
| Milk 1 Linco275 - 90 µL | 6 | 90µL - A16 | + | 2 | + |
| Milk 1 Linco275 - 110 µL | 6 | 110µL - A23 | + | 3,98 | + |
| Milk 2 Linco275 - 90 µL | 6 | 90µL - A24 | + | 2,15 | + |
| Milk 2 Linco275 - 110 µL | 6 | 110µL - A27 | + | 2,97 | + |
| Milk 3 Linco275 - 90 µL | 6 | 90µL - A21 | + | 3,37 | + |
| Milk 3 Linco275 - 110 µL | 6 | 110µL - A19 | + | 3,7 | + |

2.1.2. Incubation time

2.1.2.1. Penicillins

2.1.2.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Amox2 - 3h15 | 6 | 3h15 - A2 | + | 5,32 | + |
| Milk 2 Amox2 - 3h15 | 6 | 3h15 - A8 | + | 3,69 | + |
| Milk 3 Amox2 - 3h15 | 6 | 3h15 - A6 | + | 4,19 | + |

2.1.2.1.2. Cloxacillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Cloxacilline 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - 3h15 | 6 | 3h15 - A7 | + | 6,43 | + |
| Milk 2 Cloxa10 - 3h15 | 6 | 3h15 - A4 | + | 5,25 | + |
| Milk 3 Cloxa10 - 3h15 | 6 | 3h15 - A9 | + | 5,15 | + |

2.1.2.2. Tetracyclines

2.1.2.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 6 | 3h15 - A3 | - | -12,74 | - |
| Milk 2 Neg - 3h15 | 6 | 3h15 - A1 | - | -13,07 | - |
| Milk 3 Neg - 3h15 | 6 | 3h15 - A5 | - | -14,56 | - |

Date : 20/07/2020

Oxytetracycline 110 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Oxytetra110 - 3h15 | 6 | 3h15 - A11 | + | 0,52 | + |
| Milk 2 Oxytetra110 - 3h15 | 6 | 3h15 - A10 | + | 0,38 | + |
| Milk 3 Oxytetra110 - 3h15 | 6 | 3h15 - A12 | + | 0,72 | + |

2.1.2.2.2. Chlortetracycline

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3H15 - A1 | - | -14,35 | - |
| Milk 2 Neg - 3h15 | 4 | 3H15 - A10 | - | -12,77 | - |
| Milk 3 Neg - 3h15 | 4 | 3H15 - A8 | - | -15,13 | - |

Date : 19/08/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 3h15 | 4 | 3H15 - A5 | + | 1,17 | + |
| Milk 2 Chlortetra150 - 3h15 | 4 | 3H15 - A16 | + | 2,64 | + |
| Milk 3 Chlortetra150 - 3h15 | 4 | 3H15 - A20 | + | 3,86 | + |

2.1.2.3. Sulfonamides

2.1.2.3.1. Sulfadimethoxine

Date : 24/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 5 | 3h15 - A1 | - | -16,25 | - |
| Milk 2 Neg - 3h15 | 5 | 3h15 - A6 | - | -14,37 | - |
| Milk 3 Neg - 3h15 | 5 | 3h15 - A9 | - | -14,88 | - |

Date : 24/08/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 3h15 | 5 | 3h15 - A7 | + | 3,21 | + |
| Milk 2 Sulfadimet40 - 3h15 | 5 | 3h15 - A15 | + | 2,86 | + |
| Milk 3 Sulfadimet40 - 3h15 | 5 | 3h15 - A10 | + | 2,25 | + |

2.1.2.3.2. Sulfadiazine

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Sulfadiazine 55 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz55 - 3h15 | 4 | 3h15 - A3 | + | 0,95 | + |
| Milk 2 Sulfadiaz55 - 3h15 | 4 | 3h15 - A8 | + | 0,9 | + |
| Milk 3 Sulfadiaz55 - 3h15 | 4 | 3h15 - A14 | + | 2,29 | + |

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 5 | 3h15 - D7 | - | -13,15 | - |
| Milk 2 Neg - 3h15 | 5 | 3h15 - D8 | - | -13,75 | - |
| Milk 3 Neg - 3h15 | 5 | 3h15 - D9 | - | -15,72 | - |

Date : 01/09/2020

Sulfadiazine 60 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|---------|----------------|-------------------|---|
| Milk 1 Sulfadiaz60 - 3h15 | 5 | 3h15-D1 | + | 5,71 | + |
| Milk 2 Sulfadiaz60 - 3h15 | 5 | 3h15-D2 | + | 5,36 | + |
| Milk 3 Sulfadiaz60 - 3h15 | 5 | 3h15-D3 | + | 5,75 | + |

2.1.2.4. Macrolides

2.1.2.4.1. Tylosin A

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Tylosine 42 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Tylo42 - 3h15 | 4 | 3h15 - A12 | + | 3,66 | + |
| Milk 2 Tylo42 - 3h15 | 4 | 3h15 - A14 | + | 2,15 | + |
| Milk 3 Tylo42 - 3h15 | 4 | 3h15 - A15 | + | 2,19 | + |

2.1.2.4.2. Erythromycin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Erythromycine 192 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Erythro192 - 3h15 | 4 | 3h15 - E5 | + | 1,84 | + |
| Milk 2 Erythro192 - 3h15 | 4 | 3h15 - E11 | + | 2,04 | + |
| Milk 3 Erythro192 - 3h15 | 4 | 3h15 - E10 | + | 2,41 | + |

2.1.2.5. Aminoglycosides : dihydrostreptomycin

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - E6 | - | -14,65 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - E1 | - | -15,17 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - E3 | - | -15,22 | - |

Date : 25/08/2020

Dihydrostreptomycine 840 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E14 | + | 2,85 | + |
| Milk 2 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E17 | + | 3,18 | + |
| Milk 3 Dihydrostrepto840 - 3h15 | 4 | 3h15 - E20 | + | 3,38 | + |

2.1.2.6. Cephalosporins : céfalexine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Cefalexine 36 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Cefal36 - 3h15 | 4 | 3h15 - A4 | + | 4,02 | + |
| Milk 2 Cefal36 - 3h15 | 4 | 3h15 - A11 | + | 5,08 | + |
| Milk 3 Cefal36 - 3h15 | 4 | 3h15 - A13 | + | 4,66 | + |

2.1.2.7. Lincosamides : lincomycine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 3h15 | 4 | 3h15 - A1 | - | -11,99 | - |
| Milk 2 Neg - 3h15 | 4 | 3h15 - A6 | - | -13,02 | - |
| Milk 3 Neg - 3h15 | 4 | 3h15 - A9 | - | -15,35 | - |

Date : 26/08/2020

Lincomycine 330 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Linco330 - 3h15 | 4 | 3h15 - A3 | + | 1,74 | + |
| Milk 2 Linco330 - 3h15 | 4 | 3h15 - A7 | + | 0,36 | + |
| Milk 3 Linco330 - 3h15 | 4 | 3h15 - A10 | + | 0,61 | + |

2.1.3. Incubation temperature

2.1.3.1. Penicillins

2.1.3.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - A8 | - | -7,02 | - |

Date : 20/07/2020

Amoxicilline 2 ppb

| | | Validated | | | |
|--------------------|-------|------------|----------------|-------------------|---|
| Samples | Batch | Code | Visual reading | Delvoscan reading | |
| Milk 1 Amox2 - 62° | 5 | 62°C - A2 | + | 8,94 | + |
| Milk 1 Amox2 - 66° | 5 | 66°C - A5 | + | 7,82 | + |
| Milk 2 Amox2 - 62° | 5 | 62°C - A1 | + | 8,41 | + |
| Milk 2 Amox2 - 66° | 5 | 66°C - A9 | + | 6,98 | + |
| Milk 3 Amox2 - 62° | 5 | 62°C - A14 | + | 8,08 | + |
| Milk 3 Amox2 - 66° | 5 | 66°C - A3 | + | 6,74 | + |

2.1.3.1.2. Cloxacillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - A8 | - | -7,02 | - |

Date : 20/07/2020

Cloxacilline 10 ppb

| | | Validated | | | |
|----------------------|-------|------------|----------------|-------------------|---|
| Samples | Batch | Code | Visual reading | Delvoscan reading | |
| Milk 1 Cloxa10 - 62° | 5 | 62°C - A3 | + | 7,93 | + |
| Milk 1 Cloxa10 - 66° | 5 | 66°C - A12 | + | 4,96 | + |
| Milk 2 Cloxa10 - 62° | 5 | 62°C - A11 | + | 7,68 | + |
| Milk 2 Cloxa10 - 66° | 5 | 66°C - A15 | + | 4,01 | + |
| Milk 3 Cloxa10 - 62° | 5 | 62°C - A13 | + | 7,58 | + |
| Milk 3 Cloxa10 - 66° | 5 | 66°C - A14 | + | 4,29 | + |

2.1.3.2. Tetracyclines

2.1.3.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A8 | - | -1,98 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - A1 | - | -7,3 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A4 | - | -2,64 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - A4 | - | -5,78 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A6 | - | -5,35 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - A8 | - | -7,02 | - |

Date : 20/07/2020

Oxytetracycline 80 ppb

| Samples | Batch | Validated | | Visual reading | Delvoscan reading | |
|-------------------------|-------|------------|--|----------------|-------------------|---|
| | | Code | | | | |
| Milk 1 Oxytetra80 - 62° | 5 | 62°C - A7 | | + | 5,72 | + |
| Milk 1 Oxytetra80 - 66° | 5 | 66°C - A2 | | + | 4,07 | + |
| Milk 2 Oxytetra80 - 62° | 5 | 62°C - A5 | | + | 5,19 | + |
| Milk 2 Oxytetra80 - 66° | 5 | 66°C - A10 | | + | 3,34 | + |
| Milk 3 Oxytetra80 - 62° | 5 | 62°C - A9 | | + | 5,2 | + |
| Milk 3 Oxytetra80 - 66° | 5 | 66°C - A6 | | + | 4,84 | + |

2.1.3.2.2. Chlortetracycline

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -3,33 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -7,05 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - A1 | - | -2,37 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - A7 | - | -2,82 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - A4 | - | -2,52 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - A5 | - | -3,13 | - |

Date : 22/07/2020

Chlortetracycline 150 ppb

| Samples | Batch | Validated | | Visual reading | Delvoscan reading | |
|-----------------------------|-------|-----------|--|----------------|-------------------|---|
| | | Code | | | | |
| Milk 1 Chlortetra150 - 62°C | 5 | 62°C - B9 | | + | 6,95 | + |
| Milk 1 Chlortetra150 - 66°C | 5 | 66°C - C7 | | + | 5,74 | + |
| Milk 2 Chlortetra150 - 62° | 4 | 62°C - A6 | | + | 7,11 | + |
| Milk 2 Chlortetra150 - 66° | 4 | 66°C - A2 | | + | 6,35 | + |
| Milk 3 Chlortetra150 - 62° | 4 | 62°C - A8 | | + | 6,84 | + |
| Milk 3 Chlortetra150 - 66° | 4 | 66°C - A8 | | + | 4,82 | + |

2.1.3.3. Sulfonamides

2.1.3.3.1. Sulfadimethoxine

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -3,33 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -7,05 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - A1 | - | -2,37 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - A7 | - | -2,82 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - A4 | - | -2,52 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - A5 | - | -3,13 | - |

Date : 22/07/2020

Sulfadimethoxine 40 ppb

| Samples | Batch | Validated | | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|--|----------------|-------------------|---|
| | | Code | | | | |
| Milk 1 Sulfadimet40 - 62°C | 5 | 62°C - B2 | | + | 4,86 | + |
| Milk 1 Sulfadimet40 - 66°C | 5 | 66°C - C1 | | + | 3,81 | + |
| Milk 2 Sulfadimet40 - 62° | 4 | 62°C - A9 | | + | 5,69 | + |
| Milk 2 Sulfadimet40 - 66° | 4 | 66°C - A9 | | + | 5,05 | + |
| Milk 3 Sulfadimet40 - 62° | 4 | 62°C - A5 | | + | 5,88 | + |
| Milk 3 Sulfadimet40 - 66° | 4 | 66°C - A1 | | + | 5,56 | + |

2.1.3.3.2. Sulfadiazine

Date : 24/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - B1 | - | -3,33 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - C2 | - | -7,05 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - B4 | - | -3,86 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - C3 | - | -7,99 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - B6 | - | -2,7 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - C4 | - | -7,28 | - |

Date : 24/08/2020

Sulfadiazine 50 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 62°C | 5 | 62°C - B8 | + | 5,35 | + |
| Milk 1 Sulfadiaz50 - 66°C | 5 | 66°C - C9 | + | 3,77 | + |
| Milk 2 Sulfadiaz50 - 62°C | 5 | 62°C - B11 | + | 5,83 | + |
| Milk 2 Sulfadiaz50 - 66°C | 5 | 66°C - C5 | + | 2,82 | + |
| Milk 3 Sulfadiaz50 - 62°C | 5 | 62°C - B10 | + | 6,23 | + |
| Milk 3 Sulfadiaz50 - 66°C | 5 | 66°C - C10 | + | 4,04 | + |

2.1.3.4. Macrolides

2.1.3.4.1. Tylosin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A2 | - | -5,75 | - |
| Milk 1 Neg - 66°C | 4 | 66°C - C4 | - | -7,77 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A6 | - | -3,37 | - |
| Milk 2 Neg - 66°C | 4 | 66°C - C1 | - | -10,76 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A3 | - | -5,68 | - |
| Milk 3 Neg - 66°C | 4 | 66°C - C2 | - | -10,28 | - |

Date : 25/08/2020

Tylosine 35 ppb and 42 ppb

Validated 66°C +2

Validated 62°C

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - 62°C | 5 | 62°C - A1 | + | 1,35 | + |
| Milk 1 Tylo42 - 66°C | 4 | 66°C - C6 | + | 4,03 | + |
| Milk 2 Tylo35 - 62°C | 5 | 62°C - A5 | + | 2,73 | + |
| Milk 2 Tylo42 - 66°C | 4 | 66°C - C8 | + | 2,94 | + |
| Milk 3 Tylo35 - 62°C | 5 | 62°C - A4 | + | 2,89 | + |
| Milk 3 Tylo42 - 66°C | 4 | 66°C - C7 | + | 3,63 | + |

2.1.3.4.2. Erythromycin A

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A2 | - | -5,75 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - B1 | - | -9,08 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A6 | - | -3,37 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - B10 | - | -8,32 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A3 | - | -5,68 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - B7 | - | 7,53 | - |

Date : 25/08/2020

Erythromycine A (6 éch) 200 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|--------------------------|-------|------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Erythro200 - 62°C | 5 | 62°C - A7 | + | 5,5 | + |
| Milk 1 Erythro200 - 66°C | 5 | 66°C - B4 | + | 1,85 | + |
| Milk 2 Erythro200 - 62°C | 5 | 62°C - A9 | + | 5,17 | + |
| Milk 2 Erythro200 - 66°C | 5 | 66°C - B9 | + | 2,34 | + |
| Milk 3 Erythro200 - 62°C | 5 | 62°C - A11 | + | 5,26 | + |
| Milk 3 Erythro200 - 66°C | 5 | 66°C - B11 | + | 3,16 | + |

2.1.3.5. Aminoglycosides : dihydrostreptomycin

Date : 25/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|------------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - A2 | - | -5,75 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - B1 | - | -9,08 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - A6 | - | -3,37 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - B10 | - | -8,32 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - A3 | - | -5,68 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - B7 | - | 7,53 | - |

Date : 25/08/2020

Dihydrostreptomycine 800 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|---------------------------------|-------|------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Dihydrostrepto800 - 62°C | 5 | 62°C - A14 | + | 1,99 | + |
| Milk 1 Dihydrostrepto800 - 66°C | 5 | 66°C - B17 | + | 4,46 | + |
| Milk 2 Dihydrostrepto800 - 62°C | 5 | 62°C - A17 | + | 1,96 | + |
| Milk 2 Dihydrostrepto800 - 66°C | 5 | 66°C - B13 | + | 4,69 | + |
| Milk 3 Dihydrostrepto800 - 62°C | 5 | 62°C - A19 | + | 2,54 | + |
| Milk 3 Dihydrostrepto800 - 66°C | 5 | 66°C - B18 | + | 5,17 | + |

2.1.3.6. Cephalosporins : céfalexine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 4 | 62°C - B1 | - | -5,87 | - |
| Milk 1 Neg - 66°C | 8 | 66°C-E5 | - | -7,25 | - |
| Milk 2 Neg - 62°C | 4 | 62°C - B2 | - | -6,7 | - |
| Milk 2 Neg - 66°C | 8 | 66°C-E1 | - | -7,1 | - |
| Milk 3 Neg - 62°C | 4 | 62°C - B3 | - | -7,77 | - |
| Milk 3 Neg - 66°C | 8 | 66°C-E3 | - | -6,39 | - |

Date : 26/08/2020

Cefalexine 30 ppb and 36 ppb

Validated 62°C

Validated +20% 66°C

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cefal30 - 62°C | 4 | 62°C - B4 | + | 6,88 | + |
| Milk 1 Cefal36 - 66°C | 8 | 66°C-E2 | + | 4,44 | + |
| Milk 2 Cefal30 - 62°C | 4 | 62°C - B8 | + | 7,48 | + |
| Milk 2 Cefal36 - 66°C | 8 | 66°C-E6 | + | 7,96 | + |
| Milk 3 Cefal30 - 62°C | 4 | 62°C - B6 | + | 7,76 | + |
| Milk 3 Cefal36 - 66°C | 8 | 66°C-E4 | + | 4,27 | + |

2.1.3.7. Lincosamides : lincomycine

Date : 12/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 62°C | 5 | 62°C - D8 | - | -0,17 | - |
| Milk 1 Neg - 66°C | 5 | 66°C - E1 | - | -4,85 | - |
| Milk 2 Neg - 62°C | 5 | 62°C - D1 | - | -2,21 | - |
| Milk 2 Neg - 66°C | 5 | 66°C - E6 | - | -5 | - |
| Milk 3 Neg - 62°C | 5 | 62°C - D3 | - | -1,03 | - |
| Milk 3 Neg - 66°C | 5 | 66°C - E9 | - | -4,86 | - |

Date : 12/08/2020

Lincomycine 220 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco220 - 62°C | 5 | 62°C - D2 | + | 3,46 | + |
| Milk 1 Linco220 - 66°C | 5 | 66°C - E4 | + | 1,72 | + |
| Milk 2 Linco220 - 62°C | 5 | 62°C - D5 | + | 3,98 | + |
| Milk 2 Linco220 - 66°C | 5 | 66°C - E5 | + | 2,26 | + |
| Milk 3 Linco220 - 62°C | 5 | 62°C - D6 | + | 4,62 | + |
| Milk 3 Linco220 - 66°C | 5 | 66°C - E3 | + | 1,04 | + |

2.1.4. Delay of reading

2.1.4.1. Penicillins

2.1.4.1.1. Amoxicillin

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - A9 | - | -9,21 | - |
| Milk 1 Neg - TA | 5 | TA - A1 | - | -10,56 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - A11 | - | -8,68 | - |
| Milk 2 Neg - TA | 5 | TA - A4 | - | -9,1 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - A14 | - | -10,6 | - |
| Milk 3 Neg - TA | 5 | TA - A9 | - | -10,38 | - |

Date : 20/07/2020

Amoxicilline 2 ppb

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Amox2 - 4°C | 5 | 4°C - A7 | + | 6,38 | + |
| Milk 1 Amox2 - TA | 5 | TA - A2 | + | 8,06 | + |
| Milk 2 Amox2 - 4°C | 5 | 4°C - A1 | + | 5,13 | + |
| Milk 2 Amox2 - TA | 5 | TA - A6 | + | 7,31 | + |
| Milk 3 Amox2 - 4°C | 5 | 4°C - A12 | + | 6,28 | + |
| Milk 3 Amox2 - TA | 5 | TA - A3 | + | 7,04 | + |

2.1.4.1.2. Cloxacillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020
Cloxacilline 12 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa12 - 4°C | 5 | 4°C - C11 | + | 6,7 | + |
| Milk 1 Cloxa12 - TA | 5 | TA - D11 | + | 7,34 | + |
| Milk 2 Cloxa12 - 4°C | 5 | 4°C - C12 | + | 6,38 | + |
| Milk 2 Cloxa12 - TA | 5 | TA - D13 | + | 6,56 | + |
| Milk 3 Cloxa12 - 4°C | 5 | 4°C - C13 | + | 6,74 | + |
| Milk 3 Cloxa12 - TA | 5 | TA - D12 | + | 5,96 | + |

2.1.4.2. Tetracyclines

2.1.4.2.1. Oxytetracycline

Date : 20/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - A9 | - | -9,21 | - |
| Milk 1 Neg - TA | 5 | TA - A1 | - | -10,56 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - A11 | - | -8,68 | - |
| Milk 2 Neg - TA | 5 | TA - A4 | - | -9,1 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - A14 | - | -10,6 | - |
| Milk 3 Neg - TA | 5 | TA - A9 | - | -10,38 | - |

Date : 20/07/2020

Oxytetracycline 80 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Oxytetra80 - 4°C | 5 | 4°C - A8 | + | 3 | + |
| Milk 1 Oxytetra80 - TA | 5 | TA - A5 | + | 5,08 | + |
| Milk 2 Oxytetra80 - 4°C | 5 | 4°C - A15 | + | 2,2 | + |
| Milk 2 Oxytetra80 - TA | 5 | TA - A10 | + | 2,74 | + |
| Milk 3 Oxytetra80 - 4°C | 5 | 4°C - A10 | + | 2,72 | + |
| Milk 3 Oxytetra80 - TA | 5 | TA - A11 | + | 3,48 | + |

2.1.4.2.2. Chlortetracycline

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - B1 | - | -7,71 | - |
| Milk 1 Neg - TA | 5 | TA - C4 | - | -5,22 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - A6 | - | -4,37 | - |
| Milk 2 Neg - TA | 4 | TA - A7 | - | -4,56 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - A2 | - | -4,8 | - |
| Milk 3 Neg - TA | 4 | TA - A3 | - | -4,88 | - |

Date : 22/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - 4°C | 5 | 4°C - B12 | + | 4,68 | + |
| Milk 1 Chlortetra150 - TA | 5 | TA - C13 | + | 5,36 | + |
| Milk 2 Chlortetra150 - 4°C | 4 | 4°C - A4 | + | 6,57 | + |
| Milk 2 Chlortetra150 - TA | 4 | TA - A4 | + | 5,39 | + |
| Milk 3 Chlortetra150 - 4°C | 4 | 4°C - A7 | + | 6,21 | + |
| Milk 3 Chlortetra150 - TA | 4 | TA - A5 | + | 5,91 | + |

2.1.4.3. Sulfonamides

2.1.4.3.1. Sulfadimethoxine

Date : 22/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - B1 | - | -7,71 | - |
| Milk 1 Neg - TA | 5 | TA - C4 | - | -5,22 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - A6 | - | -4,37 | - |
| Milk 2 Neg - TA | 4 | TA - A7 | - | -4,56 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - A2 | - | -4,8 | - |
| Milk 3 Neg - TA | 4 | TA - A3 | - | -4,88 | - |

Date : 22/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - 4°C | 5 | 4°C - C9 | + | 4,95 | + |
| Milk 1 Sulfadimet40 - TA | 5 | TA - D10 | + | 5,6 | + |
| Milk 2 Sulfadimet40 - 4°C | 4 | 4°C - A3 | + | 5,48 | + |
| Milk 2 Sulfadimet40 - TA | 4 | TA - A8 | + | 5,18 | + |
| Milk 3 Sulfadimet40 - 4°C | 4 | 4°C - A5 | + | 5,61 | + |
| Milk 3 Sulfadimet40 - TA | 4 | TA - A2 | + | 4,87 | + |

2.1.4.3.2. Sulfadiazine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 5 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 5 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 5 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 5 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 5 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 5 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - 4°C | 5 | 4°C - C2 | + | 2,36 | + |
| Milk 1 Sulfadiaz50 - TA | 5 | TA - D5 | + | 3,69 | + |
| Milk 2 Sulfadiaz50 - 4°C | 5 | 4°C - C7 | + | 3,88 | + |
| Milk 2 Sulfadiaz50 - TA | 5 | TA - D3 | + | 5,07 | + |
| Milk 3 Sulfadiaz50 - 4°C | 5 | 4°C - C10 | + | 4,91 | + |
| Milk 3 Sulfadiaz50 - TA | 5 | TA - D7 | + | 4,49 | + |

2.1.4.4. Macrolides

2.1.4.4.1. Tylosin A

Date : 26/08/2020

Tylosine 42 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Tylo42 - 4°C | 4 | 4°C - E9 | + | 3,46 | + |
| Milk 1 Tylo42 - TA | 4 | TA - D7 | + | 2,67 | + |
| Milk 2 Tylo42 - 4°C | 4 | 4°C - E7 | + | 5,09 | + |
| Milk 2 Tylo42 - TA | 4 | TA - D8 | + | 3,07 | + |
| Milk 3 Tylo42 - 4°C | 4 | 4°C - E8 | + | 2,96 | + |
| Milk 3 Tylo42 - TA | 4 | TA - D9 | + | 3,71 | + |

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - E1 | - | -6,84 | - |
| Milk 1 Neg - TA | 4 | TA - D1 | - | -6,45 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - E4 | - | -6,81 | - |
| Milk 2 Neg - TA | 4 | TA - D6 | - | -7,59 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - E6 | - | -5,91 | - |
| Milk 3 Neg - TA | 4 | TA - D3 | - | -6,86 | - |

2.1.4.4.2. Erythromycin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 5 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 5 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 5 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 5 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 5 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 5 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Erythromycine 200 ppb

| Samples | Batch | Validated | | Delvoscan reading | |
|-------------------------|-------|-----------|----------------|-------------------|---|
| | | Code | Visual reading | | |
| Milk 1 Erythro200 - 4°C | 5 | 4°C - B13 | + | 3,79 | + |
| Milk 1 Erythro200 - TA | 5 | TA - C16 | + | 3,54 | + |
| Milk 2 Erythro200 - 4°C | 5 | 4°C - B15 | + | 4,27 | + |
| Milk 2 Erythro200 - TA | 5 | TA - C18 | + | 4,8 | + |
| Milk 3 Erythro200 - 4°C | 5 | 4°C - B17 | + | 4,52 | + |
| Milk 3 Erythro200 - TA | 5 | TA - C17 | + | 4,58 | + |

2.1.4.5. Aminoglycosides : dihydrostreptomycin

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - B1 | - | -7,71 | - |
| Milk 1 Neg - TA | 5 | TA - C4 | - | -5,22 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - B5 | - | -5,85 | - |
| Milk 2 Neg - TA | 5 | TA - C5 | - | -4,87 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - B6 | - | -7,07 | - |
| Milk 3 Neg - TA | 5 | TA - C6 | - | -5,66 | - |

Date : 19/08/2020

Dihydrostreptomycine 800 ppb

| Samples | Batch | Validated | | Delvoscan reading | |
|--------------------------------|-------|-----------|----------------|-------------------|---|
| | | Code | Visual reading | | |
| Milk 1 Dihydrostrepto800 - 4°C | 5 | 4°C - B2 | + | 1,53 | + |
| Milk 1 Dihydrostrepto800 - TA | 5 | TA - C12 | + | 2,02 | + |
| Milk 2 Dihydrostrepto800 - 4°C | 5 | 4°C - B9 | + | 4,08 | + |
| Milk 2 Dihydrostrepto800 - TA | 5 | TA - C10 | + | 3,81 | + |
| Milk 3 Dihydrostrepto800 - 4°C | 5 | 4°C - B11 | + | 2,67 | + |
| Milk 3 Dihydrostrepto800 - TA | 5 | TA - C7 | + | 3,27 | + |

2.1.4.6. Cephalosporins : céfalexine

Date : 19/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 5 | 4°C - B1 | - | -7,71 | - |
| Milk 1 Neg - TA | 5 | TA - C4 | - | -5,22 | - |
| Milk 2 Neg - 4°C | 5 | 4°C - B5 | - | -5,85 | - |
| Milk 2 Neg - TA | 5 | TA - C5 | - | -4,87 | - |
| Milk 3 Neg - 4°C | 5 | 4°C - B6 | - | -7,07 | - |
| Milk 3 Neg - TA | 5 | TA - C6 | - | -5,66 | - |

Date : 19/08/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Cefal30 - 4°C | 5 | 4°C - B3 | + | 4,78 | + |
| Milk 1 Cefal30 - TA | 5 | TA - C2 | + | 6,43 | + |
| Milk 2 Cefal30 - 4°C | 5 | 4°C - B7 | + | 6,68 | + |
| Milk 2 Cefal30 - TA | 5 | TA - C3 | + | 6,48 | + |
| Milk 3 Cefal30 - 4°C | 5 | 4°C - B8 | + | 5,69 | + |
| Milk 3 Cefal30 - TA | 5 | TA - C1 | + | 5,38 | + |

2.1.4.7. Lincosamides : lincomycine

Date : 26/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Neg - 4°C | 4 | 4°C - E1 | - | -6,84 | - |
| Milk 1 Neg - TA | 4 | TA - D1 | - | -6,45 | - |
| Milk 2 Neg - 4°C | 4 | 4°C - E4 | - | -6,81 | - |
| Milk 2 Neg - TA | 4 | TA - D6 | - | -7,59 | - |
| Milk 3 Neg - 4°C | 4 | 4°C - E6 | - | -5,91 | - |
| Milk 3 Neg - TA | 4 | TA - D3 | - | -6,86 | - |

Date : 26/08/2020

Lincomycine 264 ppb

Validated +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Linco264 - 4°C | 4 | 4°C - E2 | + | 3,53 | + |
| Milk 1 Linco264 - TA | 4 | TA - D2 | + | 2,05 | + |
| Milk 2 Linco264 - 4°C | 4 | 4°C - E5 | + | 1,98 | + |
| Milk 2 Linco264 - TA | 4 | TA - D5 | + | 1,04 | + |
| Milk 3 Linco264 - 4°C | 4 | 4°C - E3 | + | 2,31 | + |
| Milk 3 Linco264 - TA | 4 | TA - D4 | + | 0,77 | + |

2.2. Matrix quality

2.2.1. pH

2.2.1.1. Penicillins

2.2.1.1.1. Amoxicillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -11,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,55 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,04 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Amoxicilline 2 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|------------------------|-------|-----------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Amox2 - weak pH | 5 | pH faible - A5 | + | 3,66 | + |
| Milk 1 Amox2 - high pH | 5 | pH fort - B4 | + | 11,4 | + |
| Milk 2 Amox2 - weak pH | 5 | pH faible - A12 | + | 2,47 | + |
| Milk 2 Amox2 - high pH | 5 | pH fort - B25 | + | 11,85 | + |
| Milk 3 Amox2 - weak pH | 5 | pH faible - A14 | + | 2,35 | + |
| Milk 3 Amox2 - high pH | 5 | pH fort - B26 | + | 12,21 | + |

2.2.1.1.2. Cloxacillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -11,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,55 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,04 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Cloxacilline 10 ppb

| Samples | Batch | Code | Visual reading | Validated | |
|--------------------------|-------|----------------|----------------|-------------------|---|
| | | | | Delvoscan reading | |
| Milk 1 Cloxa10 - weak pH | 5 | pH faible - A7 | + | 1,97 | + |
| Milk 1 Cloxa10 - high pH | 5 | pH fort - B23 | + | 11,4 | + |
| Milk 2 Cloxa10 - weak pH | 5 | pH faible - A4 | + | 1,95 | + |
| Milk 2 Cloxa10 - high pH | 5 | pH fort - B3 | + | 10,96 | + |
| Milk 3 Cloxa10 - weak pH | 5 | pH faible - A9 | + | 1,59 | + |
| Milk 3 Cloxa10 - high pH | 5 | pH fort - B21 | + | 3,46 | + |

2.2.1.2. Tetracyclines

2.2.1.2.1. Oxytetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 8 | pH-D5 | - | -9,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 8 | pH-D9 | - | -7,85 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 8 | pH-D1 | - | -8,28 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Oxytetracycline 80 ppb and 96 ppb

Validated high pH and weak pH +20%

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Oxytera96 - weak pH | 8 | pH-D11 | + | 1,96 | + |
| Milk 1 Oxytetra80 - high pH | 5 | pH fort - B1 | + | 4,58 | + |
| Milk 2 Oxytera96 - weak pH | 8 | pH-D14 | + | 2,5 | + |
| Milk 2 Oxytetra80 - high pH | 5 | pH fort - B13 | + | 5,47 | + |
| Milk 3 Oxytera96 - weak pH | 8 | pH-D10 | + | 3,44 | + |
| Milk 3 Oxytetra80 - high pH | 5 | pH fort - B5 | + | 5,68 | + |

2.2.1.2.2. Chlortetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 5 | pH faible - A1 | - | -11,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 5 | pH faible - A13 | - | -8,55 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 5 | pH faible - A15 | - | -9,04 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - weak pH | 5 | pH faible - A16 | + | 2,99 | + |
| Milk 1 Chlortetra150 - high pH | 5 | pH fort - B18 | + | 8,3 | + |
| Milk 2 Chlortetra150 - weak pH | 5 | pH faible - A23 | + | 3,37 | + |
| Milk 2 Chlortetra150 - high pH | 5 | pH fort - B6 | + | 6,51 | + |
| Milk 3 Chlortetra150 - weak pH | 5 | pH faible - A21 | + | 3,46 | + |
| Milk 3 Chlortetra150 - high pH | 5 | pH fort - B20 | + | 8,11 | + |

2.2.1.3. Sulfonamides

2.2.1.3.1. Sulfadimethoxine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 8 | pH-D5 | - | -9,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 8 | pH-D9 | - | -7,85 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 8 | pH-D1 | - | -8,28 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Sulfadimethoxine 40 ppb and 48ppb

Validated high pH and +20% weak pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Sulfadimet48 - weak pH | 8 | pH-D15 | + | 0,19 | + |
| Milk 1 Sulfadimet40 - high pH | 5 | pH fort - B10 | + | 9,26 | + |
| Milk 2 Sulfadimet48 - weak pH | 8 | pH-D2 | + | 1,3 | + |
| Milk 2 Sulfadimet40 - high pH | 5 | pH fort - B16 | + | 10,23 | + |
| Milk 3 Sulfadimet48 - weak pH | 8 | pH-D12 | + | 2,86 | + |
| Milk 3 Sulfadimet40 - high pH | 5 | pH fort - B9 | + | 8,97 | + |

2.2.1.3.2. Sulfadiazine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 8 | pH-D5 | - | -9,14 | - |
| Milk 1 Neg - high pH | 8 | pH fort - B2 | - | -7,47 | - |
| Milk 2 Neg - weak pH | 8 | pH-D9 | - | -7,85 | - |
| Milk 2 Neg - high pH | 8 | pH fort - B13 | - | -5,43 | - |
| Milk 3 Neg - weak pH | 8 | pH-D1 | - | -8,28 | - |
| Milk 3 Neg - high pH | 8 | pH fort - B18 | - | -3,35 | - |

Date : 01/09/2020

Sulfadiazine 50 ppb

Validated high pH and +20% weak pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|--------------|----------------|-------------------|---|
| Milk 1 Sulfadiaz60 - weak pH | 8 | pH-D21 | + | 1,21 | + |
| Milk 1 Sulfadiaz50 - high pH | 8 | pH fort - B5 | + | 9,08 | + |
| Milk 2 Sulfadiaz60 - weak pH | 8 | pH-D7 | + | 0,09 | + |
| Milk 2 Sulfadiaz50 - high pH | 8 | pH fort - B4 | + | 7,26 | + |
| Milk 3 Sulfadiaz60 - weak pH | 8 | pH-D20 | + | 1,28 | + |
| Milk 3 Sulfadiaz50 - high pH | 8 | pH fort - B6 | + | 9,33 | + |

2.2.1.4. Macrolides

2.2.1.4.1. Tylosin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -11,16 | - |
| Milk 1 Neg - high pH | 8 | pH fort - B2 | - | -7,47 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,18 | - |
| Milk 2 Neg - high pH | 8 | pH fort - B13 | - | -5,43 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -10,67 | - |
| Milk 3 Neg - high pH | 8 | pH fort - B18 | - | -3,35 | - |

Date : 01/09/2020

Tylosine 35 ppb and 42ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Tylo42 - weak pH | 4 | pH-D11 | - | -5,38 | - |
| Milk 1 Tylo35 - high pH | 8 | pH fort - B8 | + | 8,99 | + |
| Milk 2 Tylo42 - weak pH | 4 | pH-D5 | - | -5,47 | - |
| Milk 2 Tylo35 - high pH | 8 | pH fort - B10 | + | 9,13 | + |
| Milk 3 Tylo42 - weak pH | 4 | pH-D9 | - | -4,33 | - |
| Milk 3 Tylo35 - high pH | 8 | pH fort - B9 | + | 9,84 | + |

2.2.1.4.2. Erythromycin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -11,16 | - |
| Milk 1 Neg - high pH | 8 | pH fort - B2 | - | -7,47 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,18 | - |
| Milk 2 Neg - high pH | 8 | pH fort - B13 | - | -5,43 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -10,67 | - |
| Milk 3 Neg - high pH | 8 | pH fort - B18 | - | -3,35 | - |

Date : 01/09/2020

Erythromycine 200 ppb and 240 ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Erythro240 - pH weak | 4 | pH-D6 | - | -4,77 | - |
| Milk 1 Erythro200 - pH high | 8 | pH fort - B15 | + | 9,06 | + |
| Milk 2 Erythro240 - pH weak | 4 | pH-D15 | - | -3,13 | - |
| Milk 2 Erythro200 - pH high | 8 | pH fort - B22 | + | 11,03 | + |
| Milk 3 Erythro240 - pH weak | 4 | pH-D3 | - | -4,21 | - |
| Milk 3 Erythro200 - pH high | 8 | pH fort - B17 | + | 10,59 | + |

2.2.1.5. Aminoglycosides : dihydrostreptomycin

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 4 | pH-D1 | - | -11,16 | - |
| Milk 1 Neg - high pH | 8 | pH fort - B2 | - | -7,47 | - |
| Milk 2 Neg - weak pH | 4 | pH-D8 | - | -9,18 | - |
| Milk 2 Neg - high pH | 8 | pH fort - B13 | - | -5,43 | - |
| Milk 3 Neg - weak pH | 4 | pH-D2 | - | -10,67 | - |
| Milk 3 Neg - high pH | 8 | pH fort - B18 | - | -3,35 | - |

Date : 01/09/2020

Dihydrostreptomycine 800 ppb and 960 ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto960 - weak pH | 4 | pH-D16 | - | -5,65 | - |
| Milk 1 Dihydrostrepto800 - high pH | 8 | pH fort - B19 | + | 10,52 | + |
| Milk 2 Dihydrostrepto960 - weak pH | 4 | pH-D10 | - | -4,46 | - |
| Milk 2 Dihydrostrepto800 - high pH | 8 | pH fort - B16 | + | 10,1 | + |
| Milk 3 Dihydrostrepto960 - weak pH | 4 | pH-D17 | - | -3,35 | - |
| Milk 3 Dihydrostrepto800 - high pH | 8 | pH fort - B23 | + | 10,84 | + |

2.2.1.6. Cephalosporins : céfalexine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|----------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 8 | pH faible - A1 | - | -11,04 | - |
| Milk 1 Neg - high pH | 8 | pH fort - B2 | - | -7,47 | - |
| Milk 2 Neg - weak pH | 8 | pH faible - A5 | - | -9,23 | - |
| Milk 2 Neg - high pH | 8 | pH fort - B13 | - | -5,43 | - |
| Milk 3 Neg - weak pH | 8 | pH faible - A2 | - | -10,68 | - |
| Milk 3 Neg - high pH | 8 | pH fort - B18 | - | -3,35 | - |

Date : 01/09/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------------|----------------|-------------------|---|
| Milk 1 Cefal30 - weak pH | 8 | pH faible - A25 | + | 2 | + |
| Milk 1 Cefal30 - high pH | 8 | pH fort - B26 | + | 7,86 | + |
| Milk 2 Cefal30 - weak pH | 8 | pH faible - A26 | + | 3,32 | + |
| Milk 2 Cefal30 - high pH | 8 | pH fort - B25 | + | 8,12 | + |
| Milk 3 Cefal30 - weak pH | 8 | pH faible - A27 | + | 3,47 | + |
| Milk 3 Cefal30 - high pH | 8 | pH fort - B27 | + | 8,95 | + |

2.2.1.7. Lincosamides : lincomycine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Neg - weak pH | 8 | pH-D5 | - | -9,14 | - |
| Milk 1 Neg - high pH | 5 | pH fort - B7 | - | -4,58 | - |
| Milk 2 Neg - weak pH | 8 | pH-D9 | - | -7,85 | - |
| Milk 2 Neg - high pH | 5 | pH fort - B8 | - | -4,2 | - |
| Milk 3 Neg - weak pH | 8 | pH-D1 | - | -8,28 | - |
| Milk 3 Neg - high pH | 5 | pH fort - B27 | - | -3,25 | - |

Date : 02/09/2020

Lincomycine 220 ppb

Not Validated weak pH

Validated high pH

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|---------------|----------------|-------------------|---|
| Milk 1 Linco264 - weak pH | 8 | pH-D7 | - | -6,41 | - |
| Milk 1 Linco220 - high pH | 5 | pH fort - B11 | + | 6,98 | + |
| Milk 2 Linco264 - weak pH | 8 | pH-D4 | - | -3,49 | - |
| Milk 2 Linco220 - high pH | 5 | pH fort - B19 | + | 8,62 | + |
| Milk 3 Linco264 - weak pH | 8 | pH-D8 | - | -4,91 | - |
| Milk 3 Linco220 - high pH | 5 | pH fort - B15 | + | 8,77 | + |

2.2.2. Total Bacteria Count

2.2.2.1. Penicillins

2.2.2.1.1. Amoxicillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Amox2 - high TBC | 5 | Mat - C2 | + | 8,13 | + |
| Milk 2 Amox2 - high TBC | 5 | Mat - C8 | + | 9,77 | + |
| Milk 3 Amox2 - high TBC | 5 | Mat - C7 | + | 10,43 | + |

2.2.2.1.2. Cloxacillin

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Cloxacilline 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - high TBC | 5 | Mat - C14 | + | 8,34 | + |
| Milk 2 Cloxa10 - high TBC | 5 | Mat - C6 | + | 10,02 | + |
| Milk 3 Cloxa10 - high TBC | 5 | Mat - C12 | + | 7,91 | + |

2.2.2.2. Tetracyclines

2.2.2.2.1. Oxytetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Oxytetracycline 80 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Oxytetra80 - high TBC | 5 | Mat - C4 | + | 3,57 | + |
| Milk 2 Oxytetra80 - high TBC | 5 | Mat - C13 | + | 5,09 | + |
| Milk 3 Oxytetra80 - high TBC | 5 | Mat - C9 | + | 2,34 | + |

2.2.2.2.2. Chlortetracycline

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - high TBC | 5 | Mat - C20 | + | 5,74 | + |
| Milk 2 Chlortetra150 - high TBC | 5 | Mat - C5 | + | 7,06 | + |
| Milk 3 Chlortetra150 - high TBC | 5 | Mat - C24 | + | 5,61 | + |

2.2.2.3. Sulfonamides

2.2.2.3.1. Sulfadimethoxine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - high TBC | 5 | Mat - C25 | + | 3,92 | + |
| Milk 2 Sulfadimet40 - high TBC | 5 | Mat - C18 | + | 5,32 | + |
| Milk 3 Sulfadimet40 - high TBC | 5 | Mat - C22 | + | 3,13 | + |

2.2.2.3.2. Sulfadiazine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 8 | Mat - C14 | - | -7,86 | - |
| Milk 2 Neg - high TBC | 8 | Mat - C1 | - | -8,72 | - |
| Milk 3 Neg - high TBC | 8 | Mat - C11 | - | -7,11 | - |

Date : 01/09/2020
Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - high TBC | 8 | Mat - C4 | + | 4,51 | + |
| Milk 2 Sulfadiaz50 - high TBC | 8 | Mat - C8 | + | 4,79 | + |
| Milk 3 Sulfadiaz50 - high TBC | 8 | Mat - C3 | + | 3,83 | + |

2.2.2.4. Macrolides

2.2.2.4.1. Tylosin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 8 | Mat - C14 | - | -7,86 | - |
| Milk 2 Neg - high TBC | 8 | Mat - C1 | - | -8,72 | - |
| Milk 3 Neg - high TBC | 8 | Mat - C11 | - | -7,11 | - |

Date : 01/09/2020
Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - high TBC | 8 | Mat - C12 | + | 4,49 | + |
| Milk 2 Tylo35 - high TBC | 8 | Mat - C13 | + | 2,41 | + |
| Milk 3 Tylo35 - high TBC | 8 | Mat - C7 | + | 4,71 | + |

2.2.2.4.2. Erythromycin A

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 8 | Mat - C14 | - | -7,86 | - |
| Milk 2 Neg - high TBC | 8 | Mat - C1 | - | -8,72 | - |
| Milk 3 Neg - high TBC | 8 | Mat - C11 | - | -7,11 | - |

Date : 01/09/2020
Erythromycine A (3 éch) 200 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Erythro200 - high TBC | 8 | Mat - C19 | + | 6,59 | + |
| Milk 2 Erythro200 - high TBC | 8 | Mat - C9 | + | 5,58 | + |
| Milk 3 Erythro200 - high TBC | 8 | Mat - C16 | + | 5,79 | + |

2.2.2.5. Aminoglycosides : dihydrostreptomycin

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 8 | Mat - C14 | - | -7,86 | - |
| Milk 2 Neg - high TBC | 8 | Mat - C1 | - | -8,72 | - |
| Milk 3 Neg - high TBC | 8 | Mat - C11 | - | -7,11 | - |

Date : 01/09/2020
Dihydrostreptomycine 800 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto800 - high TBC | 8 | Mat - C21 | + | 6,77 | + |
| Milk 2 Dihydrostrepto800 - high TBC | 8 | Mat - C20 | + | 6,25 | + |
| Milk 3 Dihydrostrepto800 - high TBC | 8 | Mat - C22 | + | 7,42 | + |

2.2.2.6. Cephalosporins : céfalexine

Date : 01/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 8 | Mat - C14 | - | -7,86 | - |
| Milk 2 Neg - high TBC | 8 | Mat - C1 | - | -8,72 | - |
| Milk 3 Neg - high TBC | 8 | Mat - C11 | - | -7,11 | - |

Date : 01/09/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cefal30 - high TBC | 8 | Mat - C25 | + | 6,4 | + |
| Milk 2 Cefal30 - high TBC | 8 | Mat - C27 | + | 6,81 | + |
| Milk 3 Cefal30 - high TBC | 8 | Mat - C24 | + | 5,47 | + |

2.2.2.7. Lincosamides : lincomycine

Date : 02/09/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - high TBC | 5 | Mat - C10 | - | -6,99 | - |
| Milk 2 Neg - high TBC | 5 | Mat - C1 | - | -7,98 | - |
| Milk 3 Neg - high TBC | 5 | Mat - C3 | - | -6,77 | - |

Date : 02/09/2020

Lincomycine 220 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco220 - high TBC | 5 | Mat - C16 | + | 1,56 | + |
| Milk 2 Linco220 - high TBC | 5 | Mat - C21 | + | 0,47 | + |
| Milk 3 Linco220 - high TBC | 5 | Mat - C23 | + | 1,16 | + |

2.2.3. Frozen milk

2.2.3.1. Penicillins

2.2.3.1.1. Amoxicillin

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------|-------|------|----------------|-------------------|---|
| Milk 1 Amox2 - frozen | 4 | 1-A1 | + | 7,48 | + |
| Milk 2 Amox2 - frozen | 4 | 2-B4 | + | 7,83 | + |
| Milk 3 Amox2 - frozen | 4 | 3-C9 | + | 7,81 | + |

2.2.3.1.2. Cloxacillin

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-D3 | - | -5,91 | - |
| Milk 2 Neg - frozen | 4 | 2-E5 | - | -6,54 | - |
| Milk 3 Neg - frozen | 4 | 3-F2 | - | -7,17 | - |

Date : 14/08/2020
Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Cloxa10 - frozen | 4 | 1-D2 | + | 5,86 | + |
| Milk 2 Cloxa10 - frozen | 4 | 2-E9 | + | 6,52 | + |
| Milk 3 Cloxa10 - frozen | 4 | 3-F9 | + | 5,77 | + |

2.2.3.2. Tetracyclines

2.2.3.2.1. Oxytetracycline

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Oxytetracycline 80 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Oxytetra80 - frozen | 4 | 1-A2 | + | 4,35 | + |
| Milk 2 Oxytetra80 - frozen | 4 | 2-B1 | + | 2,41 | + |
| Milk 3 Oxytetra80 - frozen | 4 | 3-C5 | + | 4,14 | + |

2.2.3.2.2. Chlortetracycline

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-D3 | - | -5,91 | - |
| Milk 2 Neg - frozen | 4 | 2-E5 | - | -6,54 | - |
| Milk 3 Neg - frozen | 4 | 3-F2 | - | -7,17 | - |

Date : 14/08/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - frozen | 4 | 1-D9 | + | 5,92 | + |
| Milk 2 Chlortetra150 - frozen | 4 | 2-E8 | + | 5,46 | + |
| Milk 3 Chlortetra150 - frozen | 4 | 3-F8 | + | 5,82 | + |

2.2.3.3. Sulfonamides

2.2.3.3.1. Sulfadimethoxine

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - frozen | 4 | 1-A3 | + | 5,4 | + |
| Milk 2 Sulfadimet40 - frozen | 4 | 2-B5 | + | 5,5 | + |
| Milk 3 Sulfadimet40 - frozen | 4 | 3-C3 | + | 5,3 | + |

2.2.3.3.2. Sulfadiazine

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-D3 | - | -5,91 | - |
| Milk 2 Neg - frozen | 4 | 2-E5 | - | -6,54 | - |
| Milk 3 Neg - frozen | 4 | 3-F2 | - | -7,17 | - |

Date : 14/08/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - frozen | 4 | 1-D5 | + | 4,62 | + |
| Milk 2 Sulfadiaz50 - frozen | 4 | 2-E3 | + | 4,5 | + |
| Milk 3 Sulfadiaz50 - frozen | 4 | 3-F4 | + | 4,29 | + |

2.2.3.4. Macrolides

2.2.3.4.1. Tylosin A

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Tylo35 - frozen | 4 | 1-A9 | + | 3,31 | + |
| Milk 2 Tylo35 - frozen | 4 | 2-B7 | + | 1,84 | + |
| Milk 3 Tylo35 - frozen | 4 | 3-C1 | + | 2,71 | + |

2.2.3.4.2. Erythromycin A

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-D3 | - | -5,91 | - |
| Milk 2 Neg - frozen | 4 | 2-E5 | - | -6,54 | - |
| Milk 3 Neg - frozen | 4 | 3-F2 | - | -7,17 | - |

Date : 14/08/2020

Erythromycine A (3 éch) 200 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Erythro200 - frozen | 4 | 1-D1 | + | 4,37 | + |
| Milk 2 Erythro200 - frozen | 4 | 2-E4 | + | 3,34 | + |
| Milk 3 Erythro200 - frozen | 4 | 3-F7 | + | 4,59 | + |

2.2.3.5. Aminoglycosides : dihydrostreptomycin

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Dihydrostreptomycine 800 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto800 - frozen | 4 | 1-A6 | + | 5,08 | + |
| Milk 2 Dihydrostrepto800 - frozen | 4 | 2-B8 | + | 4,71 | + |
| Milk 3 Dihydrostrepto800 - frozen | 4 | 3-C4 | + | 4,89 | + |

2.2.3.6. Cephalosporins : céfalexine

Date : 07/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-A4 | - | -4,71 | - |
| Milk 2 Neg - frozen | 4 | 2-B2 | - | -5,26 | - |
| Milk 3 Neg - frozen | 4 | 3-C8 | - | -8,02 | - |

Date : 07/08/2020

Cefalexine 30 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Cefal30 - frozen | 4 | 1-A7 | + | 6,42 | + |
| Milk 2 Cefal30 - frozen | 4 | 2-B3 | + | 5,35 | + |
| Milk 3 Cefal30 - frozen | 4 | 3-C2 | + | 5,53 | + |

2.2.3.7. Lincosamides : lincomycine

Date : 14/08/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------|-------|------|----------------|-------------------|---|
| Milk 1 Neg - frozen | 4 | 1-D3 | - | -5,91 | - |
| Milk 2 Neg - frozen | 4 | 2-E5 | - | -6,54 | - |
| Milk 3 Neg - frozen | 4 | 3-F2 | - | -7,17 | - |

Date : 14/08/2020

Lincomycine 220 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------|-------|------|----------------|-------------------|---|
| Milk 1 Linco220 - frozen | 4 | 1-D7 | + | 2,05 | + |
| Milk 2 Linco220 - frozen | 4 | 2-E1 | + | 0,49 | + |
| Milk 3 Linco220 - frozen | 4 | 3-F6 | + | 1,74 | + |

2.2.4. Milk temperature

2.2.4.1. Penicillins

2.2.4.1.1. Amoxicillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020

Amoxicilline 2 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Amox2 - room temperature | 6 | TA - C1 | + | 8,73 | + |
| Milk 1 Amox2 - cold | 6 | 4°C - D6 | + | 9,55 | + |
| Milk 2 Amox2 - room temperature | 6 | TA - C8 | + | 8,29 | + |
| Milk 2 Amox2 - cold | 6 | 4°C - D4 | + | 8 | + |
| Milk 3 Amox2 - room temperature | 6 | TA - C13 | + | 9,15 | + |
| Milk 3 Amox2 - cold | 6 | 4°C - D7 | + | 8,63 | + |

2.2.4.1.2. Cloxacillin

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020

Cloxacilline 10 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Cloxa10 - room temperature | 6 | TA - C12 | + | 7,33 | + |
| Milk 1 Cloxa10 - cold | 6 | 4°C - D16 | + | 8,49 | + |
| Milk 2 Cloxa10 - room temperature | 6 | TA - C5 | + | 6,69 | + |
| Milk 2 Cloxa10 - cold | 6 | 4°C - D2 | + | 5,96 | + |
| Milk 3 Cloxa10 - room temperature | 6 | TA - C3 | + | 7,24 | + |
| Milk 3 Cloxa10 - cold | 6 | 4°C - D9 | + | 7,25 | + |

2.2.4.2. Tetracyclines

2.2.4.2.1. Oxytetracycline

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020

Oxytetracycline 80 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Oxytetra80 - room temperature | 6 | TA - C17 | + | 4,73 | + |
| Milk 1 Oxytetra80 - cold | 6 | 4°C - D8 | + | 5,29 | + |
| Milk 2 Oxytetra80 - room temperature | 6 | TA - C14 | + | 4,51 | + |
| Milk 2 Oxytetra80 - cold | 6 | 4°C - D18 | + | 5,6 | + |
| Milk 3 Oxytetra80 - room temperature | 6 | TA - C16 | + | 5,05 | + |
| Milk 3 Oxytetra80 - cold | 6 | 4°C - D14 | + | 3,97 | + |

2.2.4.2.2. Chlortetracycline

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Chlortetracycline 150 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---|-------|-----------|----------------|-------------------|---|
| Milk 1 Chlortetra150 - room temperature | 6 | TA - C27 | + | 5,78 | + |
| Milk 1 Chlortetra150 - cold | 6 | 4°C - D27 | + | 5,46 | + |
| Milk 2 Chlortetra150 - room temperature | 6 | TA - C24 | + | 6,41 | + |
| Milk 2 Chlortetra150 - cold | 6 | 4°C - D26 | + | 5,35 | + |
| Milk 3 Chlortetra150 - room temperature | 6 | TA - C26 | + | 6,67 | + |
| Milk 3 Chlortetra150 - cold | 6 | 4°C - D21 | + | 5,8 | + |

2.2.4.3. Sulfonamides

2.2.4.3.1. Sulfadimethoxine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020

Sulfadimethoxine 40 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--|-------|-----------|----------------|-------------------|---|
| Milk 1 Sulfadimet40 - room temperature | 6 | TA - C2 | + | 6,78 | + |
| Milk 1 Sulfadimet40 - cold | 6 | 4°C - D12 | + | 6,25 | + |
| Milk 2 Sulfadimet40 - room temperature | 6 | TA - C4 | + | 6,39 | + |
| Milk 2 Sulfadimet40 - cold | 6 | 4°C - D17 | + | 7,29 | + |
| Milk 3 Sulfadimet40 - room temperature | 6 | TA - C9 | + | 6,53 | + |
| Milk 3 Sulfadimet40 - cold | 6 | 4°C - D22 | + | 6,52 | + |

2.2.4.3.2. Sulfadiazine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Sulfadiazine 50 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---------------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Sulfadiaz50 - room temperature | 6 | TA - C3 | + | 3,79 | + |
| Milk 1 Sulfadiaz50 - cold | 6 | 4°C - D1 | + | 4,44 | + |
| Milk 2 Sulfadiaz50 - room temperature | 6 | TA - C1 | + | 4,51 | + |
| Milk 2 Sulfadiaz50 - cold | 6 | 4°C - D4 | + | 4,26 | + |
| Milk 3 Sulfadiaz50 - room temperature | 6 | TA - C14 | + | 4,7 | + |
| Milk 3 Sulfadiaz50 - cold | 6 | 4°C - D8 | + | 5,42 | + |

2.2.4.4. Macrolides

2.2.4.4.1. Tylosin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Tylosine 35 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|----------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Tylo35 - room temperature | 6 | TA - C13 | + | 2 | + |
| Milk 1 Tylo35 - cold | 6 | 4°C - D5 | + | 2,7 | + |
| Milk 2 Tylo35 - room temperature | 6 | TA - C12 | + | 2,85 | + |
| Milk 2 Tylo35 - cold | 6 | 4°C - D14 | + | 1,82 | + |
| Milk 3 Tylo35 - room temperature | 6 | TA - C4 | + | 2,74 | + |
| Milk 3 Tylo35 - cold | 6 | 4°C - D11 | + | 2,98 | + |

2.2.4.4.2. Erythromycin A

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Erythromycine 200 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|--------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Erythro200 - room temperature | 6 | TA - C5 | + | 4,85 | + |
| Milk 1 Erythro200 - cold | 6 | 4°C - D7 | + | 5,32 | + |
| Milk 2 Erythro200 - room temperature | 6 | TA - C7 | + | 5 | + |
| Milk 2 Erythro200 - cold | 6 | 4°C - D10 | + | 4,92 | + |
| Milk 3 Erythro200 - room temperature | 6 | TA - C8 | + | 5,45 | + |
| Milk 3 Erythro200 - cold | 6 | 4°C - D6 | + | 5,08 | + |

2.2.4.5. Aminoglycosides : dihydrostreptomycin

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Dihydrostreptomycine 800 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|---|-------|-----------|----------------|-------------------|---|
| Milk 1 Dihydrostrepto800 - room temperature | 6 | TA - C15 | + | 4,32 | + |
| Milk 1 Dihydrostrepto800 - cold | 6 | 4°C - D12 | + | 4,28 | + |
| Milk 2 Dihydrostrepto800 - room temperature | 6 | TA - C10 | + | 4,36 | + |
| Milk 2 Dihydrostrepto800 - cold | 6 | 4°C - D18 | + | 3,58 | + |
| Milk 3 Dihydrostrepto800 - room temperature | 6 | TA - C18 | + | 4,69 | + |
| Milk 3 Dihydrostrepto800 - cold | 6 | 4°C - D20 | + | 5,04 | + |

2.2.4.6. Cephalosporins : céfalexine

Date : 15/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C28 | - | -5,27 | - |
| Milk 1 Neg - cold | 6 | 4°C - D29 | - | -5,71 | - |
| Milk 2 Neg - room temperature | 6 | TA - C30 | - | -4,22 | - |
| Milk 2 Neg - cold | 6 | 4°C - D28 | - | -5,58 | - |
| Milk 3 Neg - room temperature | 6 | TA - C29 | - | -4,98 | - |
| Milk 3 Neg - cold | 6 | 4°C - D30 | - | -4,91 | - |

Date : 15/07/2020

Cefalexine 30 ppb and 36 ppb

Validated +20% 4°C

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-----------------------------------|-------|----------|----------------|-------------------|---|
| Milk 1 Cefal30 - room temperature | 6 | TA - C23 | + | 7,43 | + |
| Milk 1 Cefal36 - cold | 6 | 4°C - D3 | + | 6,88 | + |
| Milk 2 Cefal30 - room temperature | 6 | TA - C25 | + | 6,99 | + |
| Milk 2 Cefal36 - cold | 6 | 4°C - D1 | + | 6,28 | + |
| Milk 3 Cefal30 - room temperature | 6 | TA - C22 | + | 7,1 | + |
| Milk 3 Cefal36 - cold | 6 | 4°C - D6 | + | 7,23 | + |

2.2.4.7. Lincosamides : lincomycine

Date : 13/07/2020

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|-------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Neg - room temperature | 6 | TA - C7 | - | -6,13 | - |
| Milk 1 Neg - cold | 6 | 4°C - D1 | - | -6,91 | - |
| Milk 2 Neg - room temperature | 6 | TA - C21 | - | -6,74 | - |
| Milk 2 Neg - cold | 6 | 4°C - D10 | - | -6,06 | - |
| Milk 3 Neg - room temperature | 6 | TA - C19 | - | -5,72 | - |
| Milk 3 Neg - cold | 6 | 4°C - D3 | - | -5,12 | - |

Date : 13/07/2020

Lincomycine 220 ppb

Validated

| Samples | Batch | Code | Visual reading | Delvoscan reading | |
|------------------------------------|-------|-----------|----------------|-------------------|---|
| Milk 1 Linco220 - room temperature | 6 | TA - C11 | + | 2,68 | + |
| Milk 1 Linco220 - cold | 6 | 4°C - D15 | + | 4,17 | + |
| Milk 2 Linco220 - room temperature | 6 | TA - C15 | + | 2,37 | + |
| Milk 2 Linco220 - cold | 6 | 4°C - D20 | + | 2,18 | + |
| Milk 3 Linco220 - room temperature | 6 | TA - C22 | + | 1,35 | + |
| Milk 3 Linco220 - cold | 6 | 4°C - D23 | + | 6,95 | + |

Appendix 3: Results of preliminary and interlaboratory studies in 2013 (ANSES)

Table 3A. Detection capabilities of antibiotics on raw cow milk.

| Antibiotic family | Antibiotic | LMR in milk (ppb) | AMPOULES (ppb) | PLATES (ppb) | |
|-------------------|--------------|-------------------|----------------|----------------|-------------------|
| | | | | Visual reading | Delvoscan reading |
| Penicillins | Penicillin G | 4 | ≤ 4 | ≤ 2 | ≤ 2 |
| | Amoxicilline | 4 | > 6 | ≤ 4 | ≤ 4 |
| | Ampicilline | 4 | ≤ 6 | ≤ 4 | ≤ 4 |
| | Cloxacilline | 30 | ≤ 30 | ≤ 30 | ≤ 30 |
| Cephalosporins | Cefquinome | 20 | ≤ 40 | ≤ 40 | ≤ 40 |
| | Cefalonium | 20 | ≤ 30 | ≤ 30 | ≤ 30 |
| | Cefapirine | 60 | ≤ 15 | ≤ 15 | ≤ 15 |
| | Ceftiofur | 100 | ≤ 20 | ≤ 20 | ≤ 20 |
| Tetracyclines | Tetracycline | 100 | ≤ 200 | ≤ 200 | ≤ 200 |
| Macrolides | Tylosin A | 50 | ≤ 50 | ≤ 50 | ≤ 50 |
| Aminoglycosides | Gentamycine | 100 | ≤ 80 | ≤ 100 | ≤ 100 |

Table 3B. Results of interlaboratory study on raw cow milk for ampoules.

| Lab | TTC | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|-----|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|---|---|---------|--|--|--|----|--|--|--|---------|--|--|--|----|--|--|--|
| | 0 | | | | 40 | | | | 200 | | | | 300 | | | | | | | | | | | | | | | | | | | |
| | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | | | | | | | | | | | | | | | | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AE | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AH | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | |
| | nb + L0 | | | | 0 | | | | nb + L1 | | | | 5 | | | | nb + L2 | | | | 32 | | | | nb + L3 | | | | 32 | | | |

| Lab | Pénicilline G | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|-----|---------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|---------|--|--|--|----|--|--|--|---------|--|--|--|----|--|--|--|
| | 0 | | | | 1 | | | | 4 | | | | 6 | | | | | | | | | | | | | | | | | | | |
| | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | | | | | | | | | | | | | | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| | nb + L0 | | | | 0 | | | | nb + L1 | | | | 2 | | | | nb + L2 | | | | 32 | | | | nb + L3 | | | | 32 | | | |

| Lab | Tylosine | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|-----|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|---|---|---------|--|--|--|----|--|--|--|---------|--|--|--|----|--|--|--|
| | 0 | | | | 20 | | | | 50 | | | | 300 | | | | | | | | | | | | | | | | | | | |
| | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | | | | | | | | | | | | | | | | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AD | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AE | 0 | 0 | 0 | 0 | 1 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AG | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AH | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| | nb + L0 | | | | 0 | | | | nb + L1 | | | | 15 | | | | nb + L2 | | | | 32 | | | | nb + L3 | | | | 32 | | | |

| Lab | Cefquinome | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|-----|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|---------|--|--|--|----|--|--|--|---------|--|--|--|----|--|--|--|
| | 0 | | | | 20 | | | | 80 | | | | 300 | | | | | | | | | | | | | | | | | | | |
| | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | | | | | | | | | | | | | | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AH | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | | | | | | | |
| | nb + L0 | | | | 0 | | | | nb + L1 | | | | 2 | | | | nb + L2 | | | | 32 | | | | nb + L3 | | | | 32 | | | |

Table 3C. Results of interlaboratory study on raw cow milk for plates (visual reading)

| | | Pénicilline G | | | | | | | | | | | | | | | |
|-----|---|---------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 1 | | | | 4 | | | | 6 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AI | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 6 | | | | nb + L2 32 | | | | nb + L3 36 | | | |

| | | TTC | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 40 | | | | 200 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AI | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 14 | | | | nb + L2 36 | | | | nb + L3 36 | | | |

| | | Cefquinome | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 20 | | | | 80 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AI | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 11 | | | | nb + L2 36 | | | | nb + L3 36 | | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |

| | | Tylosine | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 20 | | | | 50 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AI | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 24 | | | | nb + L2 34 | | | | nb + L3 36 | | | |

Table 3D. Results of interlaboratory study on raw cow milk for plates (Delvo®Scan)

| | | Pénicilline G | | | | | | | | | | | | | | | |
|-----|---|---------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 1 | | | | 4 | | | | 6 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 2 | | | | nb + L2 32 | | | | nb + L3 32 | | | |

| | | TTC | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 40 | | | | 200 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AI | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 5 | | | | nb + L2 32 | | | | nb + L3 32 | | | |

| | | Cefquinome | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 20 | | | | 80 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 2 | | | | nb + L2 32 | | | | nb + L3 32 | | | |

| | | Tylosine | | | | | | | | | | | | | | | |
|-----|---|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--------------|------------|--|--|
| | | 0 | | | | 20 | | | | 50 | | | | 300 | | | |
| Lab | | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | 1ere analyse | 2e analyse | | |
| AA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AB | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AD | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AE | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AF | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AG | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AH | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| AK | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| | | nb + L0 0 | | | | nb + L1 15 | | | | nb + L2 32 | | | | nb + L3 32 | | | |

Appendix 4: Details on antibiotics used in interlaboratory study (2021)

| Antibiotic | Brand | Reference | Batch |
|------------------|---------------|-------------|-------------|
| Tetracycline | Sigma-Aldrich | T7660-5G | 0000115278V |
| Gentamycin | Sigma-Aldrich | G1914-250MG | 0000110726 |
| Sulfadimethoxine | Sigma-Aldrich | S7007-10G | 059M4032V |

Appendix 5: Raw data for homogeneity (2021)

AMPOULES

| Samples | Code | HOMOGENEITY - BLANK MILK - AMPOULE | | | |
|---------|--------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -7,35 | - | -6,45 |
| Ctrl + | Ctrl + | + | 6,16 | + | 7,01 |

| Samples | Code | HOMOGENEITY - GENTAMYCIN - AMPOULE | | | |
|---------|------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-G01 | 1 | + | 5,92 | + | 5,96 |
| H-G02 | 2 | + | 6,33 | + | 5,95 |
| H-G03 | 3 | + | 6,11 | + | 6,15 |
| H-G04 | 4 | + | 6,56 | + | 6,39 |
| H-G05 | 5 | + | 6,66 | + | 6,64 |
| H-G06 | 6 | + | 6,08 | + | 5,73 |
| H-G07 | 7 | + | 5,54 | + | 5,75 |
| H-G08 | 8 | + | 6,35 | + | 6,27 |
| H-G09 | 9 | + | 6,52 | + | 6,5 |
| H-G10 | 10 | + | 6,7 | + | 6,07 |

| Samples | Code | HOMOGENEITY - SULFADIMETHOXINE - AMP | | | |
|---------|------|--------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-S01 | 21 | + | 3,2 | + | 3,42 |
| H-S02 | 22 | + | 3,07 | + | 3,48 |
| H-S03 | 23 | + | 2,6 | + | 3,5 |
| H-S04 | 24 | + | 3,21 | + | 3,12 |
| H-S05 | 25 | + | 4 | + | 3,85 |
| H-S06 | 26 | + | 3,35 | + | 3,82 |
| H-S07 | 27 | + | 3,56 | + | 3,91 |
| H-S08 | 28 | + | 3,25 | + | 3,29 |
| H-S09 | 29 | + | 3,36 | + | 4,11 |
| H-S10 | 30 | + | 3,69 | + | 3,64 |

| Samples | Code | HOMOGENEITY - TETRACYCLINE - AMPOULE | | | |
|---------|------|--------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-Ta01 | 41 | + | 4,49 | + | 4,15 |
| H-Ta02 | 42 | + | 4,9 | + | 4,81 |
| H-Ta03 | 43 | + | 4,47 | + | 4,78 |
| H-Ta04 | 44 | + | 4,72 | + | 4,62 |
| H-Ta05 | 45 | + | 5,18 | + | 4,38 |
| H-Ta06 | 46 | + | 4,17 | + | 4,07 |
| H-Ta07 | 47 | + | 4,83 | + | 4,68 |
| H-Ta08 | 48 | + | 4,35 | + | 4,17 |
| H-Ta09 | 49 | + | 4,51 | + | 4,02 |
| H-Ta10 | 50 | + | 5,28 | + | 3,96 |

PLATES

| Samples | Code | HOMOGENEITY - BLANK MILK - PLATE | | | |
|---------|--------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -6,96 | - | -7,51 |
| Ctrl + | Ctrl + | + | 5,56 | + | 6,41 |

| Samples | Code | HOMOGENEITY - GENTAMYCIN - PLATE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-G01 | 1 | + | 4,6 | + | 6,1 |
| H-G02 | 2 | + | 5,34 | + | 5,69 |
| H-G03 | 3 | + | 5,75 | + | 5,99 |
| H-G04 | 4 | + | 5,5 | + | 6,65 |
| H-G05 | 5 | + | 5,62 | + | 6,95 |
| H-G06 | 6 | + | 6,07 | + | 6,38 |
| H-G07 | 7 | + | 6,28 | + | 5,28 |
| H-G08 | 8 | + | 5,95 | + | 5,83 |
| H-G09 | 9 | + | 4,92 | + | 5,81 |
| H-G10 | 10 | + | 5,62 | + | 6,29 |

| Samples | Code | HOMOGENEITY - SULFADIMETHOXINE - PLATE | | | |
|---------|------|--|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-S01 | 21 | + | 4,71 | + | 5,27 |
| H-S02 | 22 | + | 4,77 | + | 4,87 |
| H-S03 | 23 | + | 5,14 | + | 3,53 |
| H-S04 | 24 | + | 4,28 | + | 4,46 |
| H-S05 | 25 | + | 4,17 | + | 4,52 |
| H-S06 | 26 | + | 4,24 | + | 4,72 |
| H-S07 | 27 | + | 4,75 | + | 4,35 |
| H-S08 | 28 | + | 5,42 | + | 4,94 |
| H-S09 | 29 | + | 4,87 | + | 4,78 |
| H-S10 | 30 | + | 4,85 | + | 3,97 |

| Samples | Code | HOMOGENEITY - TETRACYCLINE - PLATE | | | |
|---------|------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| H-Tm01 | 61 | + | 4,6 | + | 5,73 |
| H-Tm02 | 62 | + | 5,66 | + | 5,73 |
| H-Tm03 | 63 | + | 5,66 | + | 5,51 |
| H-Tm04 | 64 | + | 5,98 | + | 5,98 |
| H-Tm05 | 65 | + | 5,84 | + | 5,83 |
| H-Tm06 | 66 | + | 5,88 | + | 5,26 |
| H-Tm07 | 67 | + | 5,71 | + | 5,22 |
| H-Tm08 | 68 | + | 5,13 | + | 6,36 |
| H-Tm09 | 69 | + | 4,49 | + | 5,8 |
| H-Tm10 | 70 | + | 5,47 | + | 6,16 |

Appendix 6: Raw data for stability study

AMPOULES

PLATES

T0 = after 24h in freezer

| Samples | Code | STABILITY - BLANK MILK - AMPOULE | | | |
|---------|--------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -6,63 | - | -6,51 |
| Ctrl + | Ctrl + | + | 5,97 | + | 7,06 |

| Samples | Code | STABILITY - BLANK MILK - PLATE | | | |
|---------|--------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -7,5 | - | -8,19 |
| Ctrl + | Ctrl + | + | 6,82 | + | 6,09 |

| Samples | Code | STABILITY - GENTAMYCIN - AMPOULE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-G01 | 81 | + | 6,17 | + | 7,3 |
| S1-G02 | 82 | + | 5,52 | + | 6,03 |
| S1-G03 | 83 | + | 5,57 | + | 5,68 |

| Samples | Code | STABILITY - GENTAMYCIN - PLATE | | | |
|---------|------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-G01 | 81 | + | 6,13 | + | 6,69 |
| S1-G02 | 82 | + | 6,79 | + | 6,6 |
| S1-G03 | 83 | + | 6,37 | + | 6,78 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - AMPOULE | | | |
|---------|------|--|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-S01 | 87 | + | 2,66 | + | 3,2 |
| S1-S02 | 88 | + | 3,7 | + | 3,4 |
| S1-S03 | 89 | + | 3,46 | + | 3,97 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - PLATE | | | |
|---------|------|--------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-S01 | 87 | + | 5,76 | + | 5,96 |
| S1-S02 | 88 | + | 5,05 | + | 5,77 |
| S1-S03 | 89 | + | 5,63 | + | 5,55 |

| Samples | Code | STABILITY - TETRACYCLINE - AMPOULE | | | |
|---------|------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-Ta01 | 93 | + | 4,69 | + | 4,95 |
| S1-Ta02 | 94 | + | 4,01 | + | 4,91 |
| S1-Ta03 | 95 | + | 3,56 | + | 4,67 |

| Samples | Code | STABILITY - TETRACYCLINE - PLATE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S1-Tm01 | 99 | + | 5,69 | + | 5,5 |
| S1-Tm02 | 100 | + | 6,03 | + | 5,94 |
| S1-Tm03 | 101 | + | 6,69 | + | 6,98 |

AMPOULES

PLATES

T1 = day of shipment

| Samples | Code | STABILITY - BLANK MILK - AMPOULE | | | |
|---------|--------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -9,72 | - | -10,31 |
| Ctrl + | Ctrl + | + | 6,72 | + | 6,82 |

| Samples | Code | STABILITY - BLANK MILK - PLATE | | | |
|---------|--------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -4,67 | - | -7,46 |
| Ctrl + | Ctrl + | + | 5,84 | + | 6,39 |

| Samples | Code | STABILITY - GENTAMYCIN - AMPOULE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-G01 | 1 | + | 6,57 | + | 6,39 |
| S2-G02 | 2 | + | 6,71 | + | 6,63 |
| S2-G03 | 3 | + | 6,73 | + | 6,66 |

| Samples | Code | STABILITY - GENTAMYCIN - PLATE | | | |
|---------|------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-G01 | 1 | + | 5,77 | + | 6,68 |
| S2-G02 | 2 | + | 6,51 | + | 7,08 |
| S2-G03 | 3 | + | 6,96 | + | 7,31 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - AMPOULE | | | |
|---------|------|--|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-S01 | 7 | + | 4,74 | + | 4,62 |
| S2-S02 | 8 | + | 4,37 | + | 4,44 |
| S2-S03 | 9 | + | 4,39 | + | 4,66 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - PLATE | | | |
|---------|------|--------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-S01 | 7 | + | 6,84 | + | 6,62 |
| S2-S02 | 8 | + | 5,57 | + | 5,67 |
| S2-S03 | 9 | + | 5,38 | + | 6,01 |

| Samples | Code | STABILITY - TETRACYCLINE - AMPOULE | | | |
|---------|------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-Ta01 | 13 | + | 4,96 | + | 4,79 |
| S2-Ta02 | 14 | + | 4,78 | + | 4,91 |
| S2-Ta03 | 15 | + | 5,02 | + | 5,25 |

| Samples | Code | STABILITY - TETRACYCLINE - PLATE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S2-Tm01 | 19 | + | 6,07 | + | 6,6 |
| S2-Tm02 | 20 | + | 6,53 | + | 7,48 |
| S2-Tm03 | 21 | + | 6,26 | + | 7,03 |

AMPOULES

PLATES

T2 = day of sample analysis

| Samples | Code | STABILITY - BLANK MILK - AMPOULE | | | |
|---------|--------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -10,88 | - | -9,345 |
| Ctrl + | Ctrl + | + | 6,38 | + | 6,05 |

| Samples | Code | STABILITY - BLANK MILK - PLATE | | | |
|---------|--------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| Ctrl - | Ctrl - | - | -8,3 | - | -10,68 |
| Ctrl + | Ctrl + | + | 5,39 | + | 5,35 |

| Samples | Code | STABILITY - GENTAMYCIN - AMPOULE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-G01 | 33 | + | 6,77 | + | 6,93 |
| S3-G02 | 34 | + | 6,66 | + | 6,56 |
| S3-G03 | 35 | + | 7,09 | + | 6,71 |

| Samples | Code | STABILITY - GENTAMYCIN - PLATE | | | |
|---------|------|--------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-G01 | 33 | + | 3,78 | + | 4,65 |
| S3-G02 | 34 | + | 4,14 | + | 5,05 |
| S3-G03 | 35 | + | 4,52 | + | 5,44 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - AMPOULE | | | |
|---------|------|--|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-S01 | 39 | + | 4,28 | + | 5,27 |
| S3-S02 | 40 | + | 4,24 | + | 5,03 |
| S3-S03 | 41 | + | 4,69 | + | 4,31 |

| Samples | Code | STABILITY - SULFADIMETHOXINE - PLATE | | | |
|---------|------|--------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-S01 | 39 | + | 4,56 | + | 3,74 |
| S3-S02 | 40 | + | 4,5 | + | 4,3 |
| S3-S03 | 41 | + | 3,23 | + | 4,14 |

| Samples | Code | STABILITY - TETRACYCLINE - AMPOULE | | | |
|---------|------|------------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-Ta01 | 45 | + | 4,57 | + | 5,55 |
| S3-Ta02 | 46 | + | 5,21 | + | 4,85 |
| S3-Ta03 | 47 | + | 5,55 | + | 4,66 |

| Samples | Code | STABILITY - TETRACYCLINE - PLATE | | | |
|---------|------|----------------------------------|----------|----------------|----------|
| | | 1st analysis | | 2nd analysis | |
| | | Visual reading | Z-values | Visual reading | Z-values |
| S3-Tm01 | 51 | + | 3,9 | + | 4,77 |
| S3-Tm02 | 52 | + | 4,05 | + | 0,58 |
| S3-Tm03 | 53 | + | 4,47 | + | 2,15 |

Appendix 7: Results of interlaboratory study in 2021 (ACTALIA Cecalait)

Tables 7A. Number of positive results obtained with Delvotest® T in ampoules format for gentamycin

| | VISUAL | | | | DELVO®SCAN | | | | | |
|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|------------------|--------------|--------------|-----|
| | Labs | Levels | | | | Labs | Levels | | | |
| | L0 | L1 | L2 | L3 | L0 | L1 | L2 | L3 | | |
| GENTAMYCIN | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 |
| | 1 | 0/4 | 4/4 | 4/4 | 4/4 | 1 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 2 | 0/4 | 4/4 | 4/4 | 4/4 | 2 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 4/4 | 4/4 | 4/4 | 3 | 0/4 | 2/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 4/4 | 4/4 | 4/4 | 4 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | Software problem | | | |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 7 | 0/4 | 4/4 | 4/4 | 4/4 | 7 | 0/4 | 2/4 | 4/4 | 4/4 |
| | 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 9 | 0/4 | 4/4 | 4/4 | 4/4 | 9 | 0/4 | 0/4 | 4/4 | 4/4 |
| Total of positive results | 0/36 | 32/36 | 36/36 | 36/36 | Total of positive results | 0/32 | 20/32 | 32/32 | 32/32 | |

Tables 7B. Number of positive results obtained with Delvotest® T in ampoules format for sulfadimethoxine

| | VISUAL | | | | DELVO®SCAN | | | | | |
|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|------------------|--------------|--------------|-----|
| | Labs | Levels | | | | Labs | Levels | | | |
| | L0 | L1 | L2 | L3 | L0 | L1 | L2 | L3 | | |
| SULFADIMETHOXINE | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 |
| | 1 | 0/4 | 4/4 | 4/4 | 4/4 | 1 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 2 | 0/4 | 4/4 | 4/4 | 4/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 0/4 | 4/4 | 4/4 | 3 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 4/4 | 4/4 | 4/4 | 4 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | Software problem | | | |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 7 | 0/4 | 4/4 | 4/4 | 4/4 | 7 | 0/4 | 1/4 | 4/4 | 4/4 |
| | 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 9 | 0/4 | 4/4 | 4/4 | 4/4 | 9 | 0/4 | 0/4 | 4/4 | 4/4 |
| Total of positive results | 0/36 | 28/36 | 36/36 | 36/36 | Total of positive results | 0/32 | 13/32 | 32/32 | 32/32 | |

Tables 7C. Number of positive results obtained with Delvotest® T in ampoules format for tetracycline

| TETRACYCLINE | VISUAL | | | | | DELVO®SCAN | | | | |
|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|------------------|--------------|--------------|-----|
| | Labs | Levels | | | | Labs | Levels | | | |
| | | L0 | L1 | L2 | L3 | | L0 | L1 | L2 | L3 |
| | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 |
| | 1 | 0/4 | 4/4 | 4/4 | 4/4 | 1 | 0/4 | 1/4 | 4/4 | 4/4 |
| | 2 | 0/4 | 0/4 | 4/4 | 4/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 0/4 | 4/4 | 4/4 | 3 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 0/4 | 4/4 | 4/4 | 4 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | Software problem | | | |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| 7 | 0/4 | 3/4 | 4/4 | 4/4 | 7 | 0/4 | 0/4 | 4/4 | 4/4 | |
| 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 4/4 | 4/4 | 4/4 | |
| 9 | 0/4 | 0/4 | 4/4 | 4/4 | 9 | 0/4 | 0/4 | 4/4 | 4/4 | |
| Total of positive results | 0/36 | 15/36 | 36/36 | 36/36 | Total of positive results | 0/32 | 5/32 | 32/32 | 32/32 | |

Tables 7D. Number of positive results obtained with Delvotest® T in plates format for gentamycin

| GENTAMYCIN | VISUAL | | | | | DELVO®SCAN | | | | |
|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|-------------|--------------|--------------|-----|
| | Labs | Levels | | | | Labs | Levels | | | |
| | | L0 | L1 | L2 | L3 | | L0 | L1 | L2 | L3 |
| | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 |
| | 1 | 0/4 | 0/4 | 4/4 | 4/4 | 1 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 2 | 0/4 | 0/4 | 4/4 | 4/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 0/4 | 4/4 | 4/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 4/4 | 4/4 | 4/4 | 4 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| 7 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | 0/4 | 0/4 | 4/4 | 4/4 | |
| 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 1/4 | 4/4 | 4/4 | |
| 9 | 0/4 | 0/4 | 4/4 | 4/4 | 9 | 0/4 | 0/4 | 4/4 | 4/4 | |
| Total of positive results | 0/36 | 12/36 | 36/36 | 36/36 | Total of positive results | 0/36 | 5/36 | 36/36 | 36/36 | |

Tables 7E. Number of positive results obtained with Delvotest® T in plates format for sulfadimethoxine

| | VISUAL | | | | DELVO®SCAN | | | | | |
|-------------------------|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|--------------|--------------|--------------|
| | Labs | Levels | | | | Labs | Levels | | | |
| | L0 | L1 | L2 | L3 | L0 | L1 | L2 | L3 | | |
| SULFADIMETHOXINE | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 3/4 | 4/4 |
| | 1 | 0/4 | 0/4 | 4/4 | 4/4 | 1 | 0/4 | 0/4 | 3/4 | 4/4 |
| | 2 | 0/4 | 0/4 | 4/4 | 2/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 4/4 | 4/4 | 4/4 | 3 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 4/4 | 4/4 | 4/4 | 4 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 4/4 | 4/4 | 4/4 | 5 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 7 | 0/4 | 4/4 | 4/4 | 4/4 | 7 | 0/4 | 3/4 | 4/4 | 4/4 |
| | 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 2/4 | 4/4 | 4/4 |
| | 9 | 0/4 | 4/4 | 4/4 | 4/4 | 9 | 0/4 | 3/4 | 4/4 | 4/4 |
| | Total of positive results | 0/36 | 28/36 | 36/36 | 34/36 | Total of positive results | 0/36 | 16/36 | 35/36 | 36/36 |

Tables 7F. Number of positive results obtained with Delvotest® T in plates format for tetracycline

| | VISUAL | | | | DELVO®SCAN | | | | | |
|---------------------|----------------------------------|-------------|--------------|--------------|--------------|----------------------------------|-------------|-------------|--------------|--------------|
| | Labs | Levels | | | | Labs | Levels | | | |
| | L0 | L1 | L2 | L3 | L0 | L1 | L2 | L3 | | |
| TETRACYCLINE | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 | ACTALIA | 0/4 | 0/4 | 4/4 | 4/4 |
| | 1 | 0/4 | 0/4 | 4/4 | 4/4 | 1 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 2 | 0/4 | 0/4 | 4/4 | 4/4 | 2 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 3 | 0/4 | 0/4 | 4/4 | 4/4 | 3 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 4 | 0/4 | 4/4 | 4/4 | 4/4 | 4 | 0/4 | 4/4 | 4/4 | 4/4 |
| | 5 | 0/4 | 0/4 | 4/4 | 4/4 | 5 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 6 | 0/4 | 4/4 | 4/4 | 4/4 | 6 | 0/4 | 0/4 | 4/4 | 4/4 |
| | 7 | 0/2* | 1/4 | 4/4 | 4/4 | 7 | 0/2* | 0/4 | 4/4 | 4/4 |
| | 8 | 0/4 | 4/4 | 4/4 | 4/4 | 8 | 0/4 | 2/4 | 4/4 | 4/4 |
| | 9 | 0/4 | 0/4 | 4/4 | 4/4 | 9 | 0/4 | 0/4 | 4/4 | 4/4 |
| | Total of positive results | 0/34 | 13/36 | 36/36 | 36/36 | Total of positive results | 0/34 | 6/36 | 36/36 | 36/36 |

*: Exclusion of sample “26PLA” for the collaborative laboratory n°7 because they noticed a problem with this sample during the experiment.