Guideline on managing dairy material or product potentially exposed to chemical residues - Part B: Dairy material and product

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Version 4

Important Disclaimer

Every effort has been made to ensure the information in this report is accurate.

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A copy of this document can be found at: http://www.nzfsa.govt.nz/dairy/publications/guidelines/index.htm

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1. Purpose

This guideline sets out a process for assessing disposition options for dairy material or dairy product manufactured from raw milk which contained, or may have contained, residues of an inhibitory substance, veterinary medicine or agricultural compound.

2. Scope

This guideline is provided to assist NZFSA and Recognised Agencies in making decisions on disposition options for non-conforming dairy material or product in the event it is found to contain antimicrobial residues. The steps set out in this guideline are also applicable to assessing other compounds, but is not intended to cover situations where the presence of a compound resulted from use of a compound not permitted for use on milking animals.

3. References

The following references are applicable to this document:

DPC1 General Dairy Processing: Animal Products (Dairy) Approved Criteria

DPC2 Farm Dairies, Animal Products (Dairy) Approved Criteria

DPC3 Manufacturing of Dairy Material and Products, Animal Products (Dairy) Approved Criteria

Animal Products (Dairy Processing Specifications) Notice 2006

Animal Products (Export Requirements – Dairy Products) Notice 2005

Animal Products (Official Assurance Specifications – Dairy Products) Notice 2005

Animal Products (Dairy) Regulations 2005

Guideline for managing dairy material or product potentially exposed to chemical residues – Part A: Raw milk and raw material.

4. Interpretation

For the purposes of this document:

- **Low sampling frequency** means sampling at the start, middle and end of each affected, discrete, homogenous lot,
- **High sampling frequency** means sampling at a rate sufficient to ensure that affected material/product would be detected within a lot if it were present and takes into account concentration during processing. The frequency of sampling will be determined by the volume of affected raw material within the lot (refer to Appendix 6.2),
- **Restricted markets** means those markets that do not require an assurance on the residue status of the raw milk from which the product was manufactured.

5. Guidelines

When referring to residues the scope applies to vet medicines registered for use on milking animals (or used under written vet instruction) or agricultural compounds used according to label instructions. If the chemical concerned is found to be one not permitted for the identified use then this procedure ceases to apply and all resultant product is deemed to be non-conforming. In such situations the appropriate actions will be determined on a case by case basis.

When assessing inhibitory substances and compound residue levels predicted through silo dilution calculations, NZFSA will require the calculated level to be below all applicable limits (i.e. not at the limit). This is on the basis that the base milk may have contained very low levels of the same compound.

Consideration must be given to the cumulative effect when more than 1 consignment received contained inhibitory substance or the residue/chemical contaminant implicated.

When considering dilution under sections 5.2 to 5.4 the inhibitory substances result and the specific compound level must be considered separately. Inhibitory substance testing can be used to cover both the inhibitory substance and compound specific MRL limits provided the inhibitory substances method used is sufficiently sensitive. In any case the method used must be an NZFSA approved method for product and be accepted by the intended market(s).

5.1 Affected consignment information

Calculate the residue concentration using the initial raw milk result from the laboratory If the concentration in the tanker cannot be shown to be at or below 0.006 iu/ml then identify and provide;

i. Litres supplied and date and time (supply date and actual date if the two differ),

ii. Inhibitory substance level,

iii. Confirmation from the laboratory that the result is within method calibration,

iv. Penicillinase sensitivity[1],

v. B-lactam and Tetracycline biochemical test results and AsureQuality Bioassay if the result from (iv) is "not penacillinase sensitive"[1],

vi. Results (compound and concentration level) from LCMS or HPLC testing, refer to AsureQuality Wellington[1],

vii. Whether the medicines/chemicals containing the compound implicated were available for use on farm (from farm traceback),

viii. List of all treatments administered about the time of affected supply (from farm traceback),

ix. Date of last known unaffected supply from the farm. All milk supplied subsequently is suspect unless there is clear identification of on-farm use of the implicated medicine/chemical or other evidence that the cause is a 'one-off' event [1]. Refer to Appendix 6.4 for further clarification on the steps to follow and the information that is required. Where any FMCG (fast moving consumer good) products are affected these must be tested with urgency, therefore recipients of all potentially affected material must be notified without delay,

x. Was there an opportunity to intervene prior to the tanker unloading at the receiving site,

xi. Findings from the on-farm traceback which must be thorough and clearly legible, including all relevant information and the assessors opinion on

- Cause

[1] only required when the inhibitory substance level in the silo exceeds 0.003 iu/ml based on dilution calculations.

- Date milk affected from

In cases of extreme contamination by a veterinary medicine (eg above 0.100 iu/ml penicillin equivalent) the veterinarian should be advised and consulted if the supplier permits. In any case a statement should be made on the traceback confirming whether the veterinarian was consulted and any relevant information obtained (eg medicines purchased but not evident from farm records).

5.2 Factory requirements if dilution in tanker exceeds 0.006 IU/ML

If the calculated concentration in the tanker exceeds 0.006 IU/ml then:

i. Calculate dilution calculation at first receiving silo and identify;

- time in silo,
- whether it was a feed and bleed situation [2],
- whether there was agitation and if so the number of agitator blades [3],
- percentage of silo filled (or volume filled and capacity),
- the volume or % fill required to reach first agitator blade.

This information must be supplied to the RA as justification for any mixing efficiency applied. The RA must confirm (to NZFSA) if the mixing efficiency is supported.

If pumping to more than 1 silo then run the calculation for each silo. If any silo exceeds the MRL then supporting evidence is required to confirm the quantity of affected milk that went into that silo.

ii. If the silo calculation shows the inhibitory substance level to be:

[2] In the case of a feed and bleed situation, NZFSA will consider dilution of the affected milk with half the volume of the unaffected milk situated in the section of the silo below the inlet (applicable when the inlet is on the side of the silo and the outlet is on the base of the silo).

[3] For non agitated silos mixing factor to be 0.5 (provided time in silo was a minimum of 1 hour) unless the operator can provide data to justify a higher value.

- < MRL for the compound (if identified), and either
- <0.006 IU/ml (if B-lactam or penicillinase sensitive) or <0.003 IU/ml (if not B-lactam or penicillinase sensitive) then;

a. Raise an exception identifying 'no product affected'. Note that this is on the condition that any mixing factor greater than 0.5 has sufficient justification. If, in the opinion of the recognised person, there is not sufficient justification then continue with the procedures under sections 5.3 and 5.4 to determine if the material/product is conforming.

b. Test final product at low frequency to confirm the conclusion.

c. If any results show detections at any level then further, more frequent testing will be required to establish if a spike occurred (ie a small parcel of affected material within the wider lot). Refer to Appendix 6.3.

d. If all test results are within limits then no further action is required.

iii. If it is determined from 5.5ii that the material/product does not comply then proceed to step 5.3. This applies even if tanker screening is in place and failed to identify the milk as above limits.

5.3 Dilution calculations prior to heating milk to above 40°C

When identifying what dilution has occurred, the minimum dilution volumes must be used. If further dilution can be shown to have occurred prior to heating the milk above 40°C then;

i. Calculate concentrations (both inhibitory substance and compound level) prior to heating.

ii. If the calculated levels both meet requirements then:

a. Test product at low sampling frequency to confirm assumptions made by calculations. Note that if any results show detections at any level then further, more frequent testing will be required to establish if a spike occurred (ie a small parcel of affected material within the wider lot). Refer to Appendix 6.3.

b. Submit justification and test results to support unrestricted release.

iii. If either of the calculated levels from 5.3 i. above exceed applicable limits then proceed to 5.4.

5.4 Dilution calculations post heating milk above 40°C

i. For each product stream calculate Inhibitory substance and compound levels taking into account dilution of the liquid material through processing (using minimum volumes) and document all calculations.

ii. If necessary make adjustment for concentration occurring through the process for each final product.

iii. If the calculations under (i) and (ii) show the inhibitory substance and compound level meet applicable limits then:

a. Document and provide details to the recognised agency. Test product at low sampling frequency to confirm. Note that if any results show detections at any level then further, more frequent testing will be required to establish if a spike occurred (ie a small parcel of affected material within the wider lot). Refer to Appendix 6.3.

b. Supplementary (derived/downstream) material streams are deemed to be conforming.

c. Nominated product disposition purpose

- Product that was not shown to be diluted sufficiently prior to heating above 40°C but can be shown to be diluted sufficiently and to meet the applicable limits after heating (ie through the process) is able to be directed to restricted markets.
- No attestations on raw milk residue status will be issued by NZFSA.

iv. If all calculations under (i) or (ii) show that either the inhibitory substance or the compound level do not meet limits then proceed to section 5.5.

5.5 Suspect product due to dilution calculations

Product failing under section 5.4 is deemed to be non-conforming due to the calculated levels. However the calculations are conservative and incorporate an allowance for uncertainty. As such an operator is able to test product in order to prove conformance via high frequency sampling/testing for each product stream. The high frequency testing is recommended to be performed using an approved inhibitory substance test (if available) in conjunction with low frequency LCMS testing provided the compound is detectable by LCMS

(which can be ascertained from the raw milk LCMS result or knowledge of the compound implicated). Outcomes from this testing will be as follows:

i. All material/product confirmed not detected then submit all results and justification for sampling frequency. Derived/downstream material/product deemed okay for restricted markets. No attestations on raw milk residue status will be issued by NZFSA.

ii. If there is a detection (above or below limits) in any material/product then:

   a. Increased sampling/testing required for material/product between sampling points to show more definitively that all material/product conforms. This is to ensure that highest actual level present within the affected range is within limits. The frequency must be sufficient to give confidence that any peak concentration would be observed. As a guide a further 5 samples will be required between each of the initial sampling points. See Appendix 6.3 for more details on required sampling.

   b. Downstream product is also non-conforming and must be subject to same test frequency.

   c. If all test results are within limits proceed as per 5.5 i. above.

   d. If any results exceed applicable limits then the lot fails, refer to sublotting guidelines to determine if suitability is an option http://www.nzfsa.govt.nz/dairy/publications/approved-criteria/dpc1-approvedcriteriaforgeneraldairyprocessing.pdf.

6. Appendix I

6.1 Working example

Raw Milk:

- 8000 litres at Farm
- 5% Fat
- Into tanker filler to 26,000 litres
- Into silo with 400,000 litre capacity and filled to 200,000 litres (no agitation)
- Inhibitory substance estimated: 0.250 IU/ml penicillin equivalent
- LCMS: Penicillin G at 0.150 mg/kg.

Considerations

1. Estimated concentration in the tanker:
    - (8,000 litres x 0.25 iu/ml)/26,000 litres = 0.077 iu/ml versus 0.006 iu/ml limit.
    - (8,000 litres x 0.15 mg/l)/26,000 litres = 0.046 mg/l versus 0.004 mg/l limit. As both exceed limits then must consider silo consolidation.
2. Estimated level in initial receiving silo:
    - (8,000 litres x 0.025 iu/ml)/200,000 litres = 0.010 iu/ml, divided by mixing factor 0.5^4 (no agitation) = 0.020 iu/ml versus 0.006 iu/ml limit.
    - (8,000 litres x 0.015 mg/kg)/200,000 litres = 0.006 mg/kg, divided by mixing factor 0.5^3 (no agitation) = 0.012 mg/kg versus 0.004 mg/kg limit.
3. No further dilution occurred prior to pre heat and separation therefore market restrictions apply.

[4] Mixing factor to be 0.5 (provided time in silo was a minimum of 1 hour) unless the operator can provide data to justify a higher value. In this case there is holding of cream for a sufficient period of time to allow for full mixing (maximum 90% or 0.9 mixing factor).

4. The raw milk from the silo was separated and:

   - All the skimmilk (175,000 litres) went into a 400,000 litre silo with no agitation, filled to a total of 325,000 litres then dried as skim milk powder,
   - 25,000 litres of cream went into an agitated silo with 50,000 of unaffected cream (ie 75,000 litres in total), held for sufficient time to allow mixing and was then processed into butter.

Each product stream should be considered separately. Unless a compound is known to concentrate in the fat assume that the residue is evenly distributed across all milk components from the farm milk supply. In this case both the skim and cream from the farm are assumed to contain 0.25 iu/ml penicillin equivalent inhibitory substances or 0.15mg/l penicillin-G.

Stream A - Cream

- Milkfat from farm = 8000L at 5% fat = 400 kgs
- Total milkfat in 75,000L cream at 40% fat = 30,000kgs
- (400 kgs x 0.25 iu/ml)/30,000 litres = 0.0033 iu/ml, divide by mixing factor 0.9[5] (agitated silo) = 0.0037 iu/ml versus 0.006iu/ml limit
- (400 kgs x 0.15 mg/kg)/30,000 litres = 0.002mg/kg or 2 ug/kg, divide by mixing factor 0.9[4] (agitated silo) = 2.2ug/kg versus 4 ug/kg limit.

On the basis of this calculation the butter is predicted to meet both limits, therefore the operator tests the butter at the low sampling frequency to confirm.

Stream B - Skim

- Skim milk silo prior to drying = 175,000 litres at plus 150,000L unaffected = 325,000litres

[5] Mixing factor to be 0.5 (provided time in silo was a minimum of 1 hour) unless the operator can provide data to justify a higher value. In this case there is holding of cream for a sufficient period of time to allow for full mixing (maximum 90% or 0.9 mixing factor).

- Mixing factor: as there is no agitation it can be assumed that the affected material (175,000 litres) was mixed with 50% of the unaffected material (150,000 x 0.5 = 75,000). Therefore the mixing factor is (175+75)/325 = 0.77
- (175,000 x 0.020 iu/ml)/325,000 = 0.011 iu/ml, divided by mixing factor 0.77 = 0.014 iu/ml versus 0.006 iu/ml limit
- (175,000 x 0.012 mg/kg)/325,000 = 0.0065 mg/kg = 6.5 ug/kg, divided by mixing factor 0.77 = 8.4 ug/kg versus 4 ug/kg limit.

On the basis of this calculation it is predicted that at least some of the skimmilk powder will fail to meet both the Inhibitory substance and penicillin MRL limits.

To clear any of the product the operator must test across the affected cypher(s) at the high sampling frequency (refer to Appendix 6.2) using an approved inhibitory substances test method, and test by LCMS or HPLC for penicillin at the low sampling frequency.

6.2 Determine high frequency sampling – working example

The frequency of sampling will be determined by the volume of affected raw material within the lot. The purpose is to ensure that sampling is at a frequency that will ensure that any affected product will be detected.

Using the previous working example 325,000 litres of skim was dried into skimmilk powder, some or all of which is likely to exceed residue limits. For the purpose of this example it can be assumed that all this powder went into one cypher comprising 240 MT in total.

The steps are:

i. The “affected material” prior to drying is the total skim in the affected silo multiplied by the mixing factor, in this case:

325,000 litres x 0.77 = 250,250 litres

ii. Allowance must then be made for concentration[6]. For simplicity it will be assumed in this example that 10 litres skim will yield 1 kg skimmilk powder (1/10):

250,250 x 1/10 = 25,025 kg powder

[6] Note that in practice the concentration factor must be corrected for actual yield.

iii. Lastly, an adjustment must be made to provide a 10% margin of safety and provide for a small overlap:

25,025 x 0.9 = 22,522.5

Thus one sample must be taken at intervals not exceeding 22,523 kgs. This would require a minimum of 11 samples across a cypher of 240 MT.

In a feed and bleed situation it should be assumed that the quantity of affected raw material remains unchanged. That is, the quantity from the previous step should be assumed.

Using the previous example, no dilution would be applied, so the calculation becomes 175,000L of affected material * 1/10 (product concentration) *0.9 (10 % margin of safety) = 15,750kgs of powder.

6.3 Sampling frequency clarification

When high frequency testing identifies a detection at any level additional samples will be required to confirm the maximum concentration in the product. Typically 5 samples taken at evenly distributed intervals between the product units previously sampled at the high frequency will suffice, extending from the “no detection” found immediately before the first detection through to the “no detection” found immediately after the last detection within the batch or cypher.

For example, in the table below units 31 and 51 had a detection, therefore additional samples are required from unit 22 though to 60, with 5 samples between 21 and 31, 5 between 31 and 41 etc. This will be a total of 20 samples.

Product unit	1	11	21	31	41	51	61	71	81	90
Level	nd	nd	nd	1	nd	1	nd	nd	nd	nd

If the results are all within applicable limits and there is no indication from the result trends of a “spike” then no further testing would be required.

6.4 Farm traceback requirements.

Where the traceback at farm has identified that previous supply from the farm was affected (as opposed to suspect) high frequency testing of the primary product [7] manufactured from all the affected milk is to be conducted (section 5.5 ii applies).This may span more than one day.

In instances where it is only suspected that the previous supply is affected (for instance no clear event was identified from the traceback) the following process applies:

- Farm - assume the level in the milk is the same as the known affected day.
- Calculate dilution level in tanker. If this result is below the limit then stop and record. Product is eligible for unrestricted use. If the result is above the limit then calculate the dilution level in the silo.
- If the dilution level in the silo is below the limit then stop and record. Product is eligible for unrestricted use. If the result is above the limit then calculate the dilution level through the process (as per above).
- If the dilution level through the process is below the limit then stop and record. Product is eligible for unrestricted use. If the result is above the limit then test the primary product[5] at high frequency.
- If the high frequency testing returns no detectable level then stop and record. Product is eligible for unrestricted use. If any results have detection then:
    - section 5.5 ii applies,
    - a product disposition must be submitted to NZFSA for consideration,
    - repeat the process for the next previous days consignment from farm (unless this supply is known to not be affected).

[7] Primary product is defined as the first level of products produced from the affected milk.