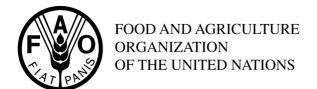
codex alimentarius commission





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Agenda Item 2

CX/MMP 02/2 January 2002

JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON MILK AND MILK PRODUCTS Fifth Session

Wellington, New Zealand, 8-12 April 2002

MATTERS REFERRED BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES

CONSIDERATION OF THE DRAFT STRATEGIC FRAMEWORK, PROPOSED DRAFT MEDIUM PLAN 2003-2007 AND THE CHAIRPERSON'S ACTION PLAN

- 1. The 24th Session (July 2001) of the Codex Alimentarius Commission adopted¹ the draft Strategic Framework, including the Strategic Vision Statement. It agreed that the draft Medium-Term Plan should be revised by the Secretariat in the light of the Strategic Framework, the Commission's discussion and the written comments received, and should incorporate the elements of the Chairperson's Action Plan agreed to by the Commission. The Commission agreed that the activities envisaged in the Medium Term Plan should include cost estimates to determine whether the objectives could be achieved within available resources and that the revised draft Medium-Term-Plan be circulated for the inputs of the Codex Coordinating Committees, other Codex Committees, Member governments and international organizations for further consideration and finalization at the 25th Session of the Commission.
- 2. The 49th Session (September 2001) of the Executive Committee of the Codex Alimentarius Commission noted² that Circular Letter CL 2001/26-EXEC had been sent to Members of the Commission on 14 August 2001. Governments and interested international organizations had been being invited to comment on the revised Draft Medium-Term Plan and also to propose or suggest new activities. Following the deadline for comments (30 November 2001) the Revised Draft Medium-Term Plan will be up-dated and placed on the Codex Website. The Plan will be up-dated following each Codex Committee/Task Force session to include new proposals as they arise.
- 3. This Plan will then be submitted to the 50th Session of the Executive Committee (26-28 June 2002) for review and then to governments and interested international organizations for comments. Those Codex Committees (especially Regional Committees) that had not previously commented will also have to opportunity to contribute to the development of the Medium-Term Plan. The Revised Draft Medium-Term Plan together with the various proposals made by Codex Committees and other interested parties will be considered by the 51st

ALINORM 01/41, paras. 46-70 and Appendix II

² ALINORM 03/3, paras. 37-41.

Session of the Executive Committee and then submitted to the 25th Session of the Codex Alimentarius Commission for adoption.

RISK ANALYSIS POLICIES OF THE CODEX ALIMENTARIUS COMMISSION

- 4. The 24th Session of the Codex Alimentarius Commission confirmed³ its initial mandate to the Committee on General Principles to complete the principles for risk analysis within Codex as a high priority, with a view to their adoption in 2003.
- 5. The Commission adopted the position, in regard to the consideration of precaution, that:
- "When there is evidence that a risk to human health exists but scientific data are insufficient or incomplete, the Commission should not proceed to elaborate a standard but should consider elaborating a related text, such as a code of practice, provided that such a text would be supported by the available scientific evidence."
- 6. The Commission also recommended that relevant Codex Committees should continue to develop and document the application of risk analysis in their work. It was agreed that the risk analysis policies developed by the Committees would be presented in a single document to the next session of the Commission.

CONSIDERATION OF PROPOSED DRAFT STANDARDS AND RELATED TEXTS

Decision of the 24th Session of the Codex Alimentarius Commission(Geneva, 2-7 July 2001)

<u>Draft Group Standard for Unripined Cheese Including Fresh Cheese</u>⁴

7. The Commission adopted the Draft Group Standard with pimaricin temporarily endorsed for surface/rind treatment only.

Proposed Draft Revised Standard for Edible Casein Products⁵

8. The Commission deleted the draft maximum level of Lead in accordance with its previous decisions concerning levels of Lead in milk and milk products (see para. 12 below) and **adopted** the Draft Revised Standard at Steps 5 and 8.

Proposed Draft Amendment to the Codex General Standard for Cheese (Description) ⁶

9. The Commission adopted the Draft Amendment at Steps 5 and 8 and was informed that the issue of the minimum protein level would be discussed further at the next session of the Committee on Milk and Milk Products.

Proposed Draft Amendment to the Codex Group Standard for Cheeses in Brine (Sampling)⁷

10. The Commission adopted the Draft Revised Standard at Steps 5 and 8.

Codex General Standard for Food Additives: Proposed Draft and Draft Food Additive Provisions in Table 1⁸

11. The Commission noted that the use of Pimaricin in Category 1.6 (Cheese) at a level of 40 mg/kg was based on the qualification that it was used for surface treatment only and was equivalent to 2 mg/dm² surface application to a maximum depth of 5 mm. However, as the provisions for the use of Pimaricin in sliced, cut shredded and grated products in the Draft Group Standard for Unripened Cheese, including Fresh Cheese were only temporarily endorsed by the Committee on Food Additives and Contaminants pending reevaluation by the JECFA, the Commission agreed that the provision in the Codex General Standard for Food Additives should remain as temporarily endorsed.

³ ALINORM 01/41, paras. 71-85.

⁴ ALINORM 01/41 paras 106-107; ALINORM 01/11 Appendix II

⁵ ALINORM 01/41 paras 108; ALINORM 01/11, Appendix III

⁶ ALINORM 01/41 paras 109; ALINORM 01/11, Appendix IV.

ALINORM 01/41 paras 110; ALINORM 01/11, Appendix V.

⁸ ALINORM 01/41 paras 114; ALINORM 01/12, Appendix III and ALINORM 01/12A Appendix II;

Draft Maximum Levels for Lead⁹

12. The Commission adopted the levels for lead in milk (0.02 mg/kg) and milk fat (0.1 mg/kg) as proposed, and requested the Committee on Food Additives and Contaminants to re-evaluate the levels.

<u>Draft Maximum Level for Aflatoxin M1 in Milk</u>¹⁰

13. The Commission could not reach a consensus on this issue. However, in view of the importance of establishing a level for the health protection of consumers, and in consideration that the higher level provided an adequate level of protection as determined by the Committee on Food Additives and Contaminants, the Commission adopted the maximum level of $0.5~\mu g/kg$ in milk. It was agreed that data supporting the lower level, if and when available, could be examined by the Committee on Food Additives and Contaminants at a future meeting if necessary.

Decision of the 47th Session of the Executive Committee(Geneva, 28-30 June 2000) 11

Proposed Draft Standards and related Texts Advanced to Step 6

- 14. The Executive Committee advanced to step 6 the following draft standards and related texts:
- Proposed Draft Revised Standard for Creams, Whipped Creams and Fermented Creams¹²;
- Proposed Draft Revised Standard for Fermented Milks¹³;
- Proposed Draft Revised Standard for Whey Powders¹⁴;

Note from the Secretariat - the proposed draft standards listed above are going to be discussed under Agenda Item 3

CONSIDERATION OF NEW WORK PROPOSALS

Decision of the 47th Session of the Executive Committee (Geneva, 28-30 June 2000)¹⁵

Approved proposals for new work:

15. The Executive Committee approved the following proposals for new work:

- Proposed Draft Standard for Evaporated Skimmed Milk with Vegetable Fat (tentative title proposed by the CCMMP) / Proposed Draft Standard for Evaporated Filled Milk Fat (tentative title proposed by the CCASIA)
- Proposed Draft Standard for Sweetened Condensed Milk with Vegetable Fat (tentative title proposed by the CCMMP) / Proposed Draft Standard for Sweetened Condensed Filled Milk (tentative title proposed by the CCASIA)
- Proposed Draft Standard for Skimmed Milk Powder with Vegetable Fat (tentative title proposed by the CCMMP) / Proposed Draft Standard for Filled Milk Powder (tentative title proposed by the CCASIA)

Note from the Secretariat - the three proposed draft standards listed above are going to be discussed under Agenda Item 4 e

• Proposed Draft Amendment to the Codex General Principle for Cheeses in Brine (Sampling)

⁹ ALINORM 01/41 paras 121; ALINORM 01/12, Appendix XI

ALINORM 01/41 paras 127-129; ALINORM 01/12, Appendix X

ALINORM 01/3, Appendix IV

ALINORM 01/11, Appendix VI

ALINORM 01/11, Appendix VII

ALINORM 01/11, Appendix VIII

ALINORM 01/3, Appendix III

 Proposed Draft Amendment to the Codex General Standard for Cheese (Appendix on cheese rind, surface and coatings)

Note from the Secretariat - the above draft amendments is going to be discussed under Agenda Item 4 a

Codex General Standard for the Use of Dairy Term - filled milk¹⁶

16. The Executive Committee noted the concern expressed by the Representative of Asia on the use of the term "filled milk" which was not allowed under the codex general standard for the Use of Dairy Term and therefore could cause problems in the trade in such products. The Executive Committee requested the next Session of the Committee on Milk and Milk Products to re-examine the matter.

DISCONTINUATION OF WORK

17. The 47th Session of the Executive Committee noting that the matter of heart treatment definition had been transferred to the work programme of the Codex Committee on food hygiene, endorsed the proposal to discontinue the work on the proposed Draft Definition for Heat Treatment.¹⁷

CODEX GUIDELINES FOR THE PRESERVATION OF RAW MILK BY USE IF THE LACTOPEROXIDASE SYSTEM - CAC/GL 13-1991

- 18. Since 1997 FAO has been involved in promoting the use and application of the Lactoperoxidase system of raw milk preservation in developing countries according to the 1991 Codex Guidelines (CAC GL. 13/91). Main activities have included formation of an international experts panel on LP-s, many of whom were involved in the proposal of the system to Codex and JECFA in the 1980's.
- 19. Over 80 member countries of FAO have requested FAO assistance in determining the suitability and applicability of the system in their respective countries. A limited number of demonstrations have been carried out in 12 countries to date.
- 20. As a result of these field level demonstrations, feedback from country standards and food regulation bodies, and in consultation with the Group of Experts of the FAO led Global Lactoperoxidase Programme, a number of improvements in the guideline are suggested for the consideration of the Committee. The proposed amendments (see Appendix I) are primarily suggested to facilitate the uptake of this safe and effective methods of raw milk preservation which is badly needed in many developing countries.

PROPOSED DRAFT CODE OF HYGIENIC PRACTICE FOR MILK AND MILK PRODUCTS¹⁸

21. The 34th Session of the Codex Committee on Food Hygiene (Bangkok, 8-13 October 2001) agreed to return the Proposed Draft Code to Step 2 for revision by a drafting group. The revised code will be circulated for further comments at Step 3 in advance before the next Session of the Committee.

ALINORM 03/13, paras. 129-134

16

ALINORM 01/3, para. 45

ALINORM 01/3, para 50 and Appendix III

PROPOSED DRAFT AMENDMENTS* GUIDELINES FOR THE PRESERVATION OF RAW MILK BY USE OF THE LACTOPEROXIDASE SYSTEM

CAC/GL 13-1991

INTRODUCTION

Milk is an easily perishable raw material. Contaminating bacteria *micro-organisms* may multiply rapidly and render it unsuitable for processing and/or unfit for human consumption. Bacterial growth can be retarded by refrigeration, thereby slowing down the rate of deterioration. Under certain conditions refrigeration may not be feasible *or sufficient* due to economical and/or technical reasons. Difficulties in applying refrigeration are specially a problem for certain areas in countries setting up or expanding their milk production. In these situations, it would be beneficial to have access to a method, other than refrigeration, for retarding bacterial *microbial* growth in raw milk during collection and transportation to the dairy processing plant.

In 1967 the FAO/WHO Expert Panel on Milk Quality concluded that the use of hydrogen peroxide might be an acceptable alternative in the early stages of development of an organized dairy industry, provided that certain conditions were complied with. However, this method has not achieved any general acceptance as it has several drawbacks, most important of which is the difficulty of controlling its use: it may be misused to disguise milk of basic hygienic quality produced under poor hygienic conditions. The toxicological aspects of the use of relatively high concentrations of hydrogen peroxide in milk have also been questioned. *Difficulties in clothing of milk treated with hydrogen peroxide have also been observed*.

A *bio*chemical method for preserving milk would still be of great advantage in certain situations. The search for such a method has therefore continued. Interest has recently been focused on the indigenous antibacterial systems in milk to determine if these could be applied practically to preserve raw milk. During the last decade, basic and applied research has demonstrated that one of these systems, the Lactoperoxidase/thiocyanate/hydrogen peroxide system (LP-system) can be used successfully for this purpose.

1. SCOPE

1.1 This Code of Practice describes the use of the Lactoperoxidase system for preventing **bacterial** *micro-organisms* spoilage of raw milk (bovine and buffalo other mammal milk used for human consumption). It describes the principles of the method, in what situations it can be used, its practical application and control of the method. It should be stressed that this method should be utilized when refrigeration of the raw milk is not feasible or sufficient.

2. PRINCIPLES OF THE METHOD

2.1 The enzyme peroxidase in combination with thiocyanate and hydrogen peroxide is an indigenous antibacterial system, which is present in raw milk and some other body fluids (including saliva). In milk the enzyme is called lactoperoxidase and is found in relatively high concentrations. Lactoperoxidase has no antibacterial effect on its own, but it can oxidise thiocyanate ions in the presence of hydrogen peroxide. By this reaction antibacterial compounds are created which will inactivate with the metabolism of bacteria.

(LP) $(CSN) + H_2O_2 \leftrightarrow HOSCN$ $HOSCN \leftrightarrow H^+ + OSCN$

2.2 The effect on bacteria is both species and strain dependent. Against a mixed raw milk flora, dominated by mesophilic bacteria, the effect is bacteriostatic (inhibitory). Against some gram-negative bacteria, i.e. pseudomonads, Escherichia coli, the effect is bactericidal. Due to the mainly bacteriostatic effect

^{*} Proposed Draft Amendments: New text in bold italics (xxx); deleted text in strikethrough (xxx)

of the system it is not possible to disguise poor quality milk, which originally contained a high bacterial population, by applying this method.

- 2.3 The antibacterial oxidation products of thiocyanate are not stable at neutral pH. Any surplus of these are reduced spontaneously to thiocyanate. The velocity of this reaction is temperature dependent, i.e. more rapid at higher temperatures. Pasteurisation of the milk will ensure a complete removal of any residual concentrations of the active oxidation products.
- 2.4 The natural oxidation of the thiocyanate in milk is limited. It can, however, be catalysed through addition of small concentrations of hydrogen peroxide (9 ppm; see section 4 Practical application of the method). The high concentrations of hydrogen peroxide previously used to preserve milk (300-800 ppm), destroy the enzyme lactoperoxidase and thereby preclude the oxidation of thiocyanate. With that method the antibacterial effect was thus an effect of hydrogen peroxide itself. The Third Meeting of the Mixed Committee on Milk and Milk Products Experts of the Codex Alimentarius held in Montevideo, Uruguay in 1998 agreed on the elimination of the use of high concentrations of hydrogen peroxide to preserve milk.
- 2.5 The antibacterial effect of the natural LP-system is, within certain limits, proportional to the thiocyanate concentration in the milk (provided that an equimolar amount of hydrogen peroxide is added). The level of thiocyanate in fresh milk varies depending on the feeding of the animals and is generally from x to y ppm.. The practical use of the method to enhance the antibacterial property of the natural LP-system consequently requires addition of some thiocyanate to ensure that a level necessary to achieve the desired effect is present in the milk.
- 2.6 The levels of thiocyanate resulting from this treatment are within the physiological levels reported to occur in milk under certain circumstances and feeding regimes. They are also far below the thiocyanate levels known to exist in human saliva and certain common vegetables, e.g. cabbage and cauliflower. In addition, results from clinical experiments have clearly demonstrated that milk treated according to this method will not cause any interference of the iodine uptake of the thyroid gland, neither in persons with a normal iodine status nor in cases of iodine deficiency.
- 2.1 The Lactoperoxidase/thiocyanate/hydrogen peroxide system is an indigenous antibacterial system in milk and human saliva. The enzyme Lactoperoxidase is present in bovine and buffalo milk in relatively high concentrations. It can oxidise thiocyanate ions in the presence of hydrogen peroxide. By this reaction, thiocyanate is converted into hypothiocyanous acid (HOSCN). At the pH of milk HOSCN is dissociated and exists mainly in the form of hypothiocyanate ions (OSCN-). This agent reacts specifically with free sulphydryl groups, thereby inactivating several vital metabolic bacterial enzymes, consequently blocking their metabolism and ability to multiply. As milk proteins contain very few sulphydryl groups and those that are present are relatively inaccessible to OSCN- (masked), the reaction of this compound is in milk quite specific and is directed against the bacteria present in the milk.
- 2.2 Concerning the characteristics of the microorganisms, the antimicrobial effect depends on the species and the strain. The effect against is both species and strain dependent. Against a mixed raw milk flora, dominated by mesophilic bacteria, the effect is bacteriostatic (predominantly inhibitory). Against some gram-negative bacteria, i.e. pseudomonads, Escherichia coli, the effect is bactericidal. Due to the mainly bacteriostatic effect of the system it is not possible to disguise poor quality milk, which originally contained a high bacterial population, by applying this method.
- 2.3 The antibacterial oxidation products of thiocyanate are not stable at neutral pH. Any surplus of these decomposes spontaneously to thiocyanate. The velocity of this reaction is temperature dependent, i.e. more rapid at higher temperatures. Pasteurisation of the milk will ensure a complete removal of any residual concentrations of the active oxidation products.
- 2.4 Oxidation of thiocianate does not cour to any great extent in milk when it has left the udder. *The natural oxidation of the thyocianate in the milk is limited.* It can, however, be initiated through addition of small concentrations of hydrogen peroxide (see Section 4). The high concentrations of hydrogen peroxide used to preserve milk (300-800 ppm), destroy the enzyme Lactoperoxidase and thereby preclude the oxidation of thiocyanate. With this method the antibacterial effect is thus an effect of hydrogen peroxide itself

- 2.5 The antibacterial effect of the LP-system is, within certain limits, proportional to the thiocyanate concentration in the milk (provided that an equimolar amount of hydrogen peroxide is provided). The level thiocyanate in milk is related to the feeding of the animals and can thus vary from *1 to 10 ppm*. The practical use of the method consequently requires addition of some thiocyanate to ensure that a level necessary to achieve the desired effect, is present in the milk.
- 2.6 The levels of thiocyanate resulting from this treatment are within the physiological levels reported to occur in milk under certain circumstances and feeding regimes. They are also far below the thiocyanate levels known to exist in human saliva and certain common vegetables, e.g. cabbage and cauliflower. In addition, results from clinical experiments have clearly demonstrated that milk treated according to this method will not cause any interference of the iodine uptake of the thyroid gland, neither in persons with a normal iodine status nor in cases of iodine deficiency.

3. INTENDED UTILIZATION OF METHOD

- 3.1 This method should only be used in situations when technical, economical and/or practical reasons do not allow the use of *appropriate* cooling facilities for maintaining the quality of raw milk. Use of the LP-system in areas which currently lack an adequate infrastructure for collection of liquid milk, would ensure the production of milk as a safe and wholesome food, which otherwise would be virtually impossible.
- 3.2 The method should not be used by the individual farmers trained persons but at a suitable collecting point/centre. These centres must be equipped with proper facilities for cleaning and sanitising the vessels used to hold and transport milk.
- 3.3 The personnel responsible for the collection of milk should be in charge for the treatment of the milk. They should be given appropriate training, including training in general milk hygiene, to enable them fulfil this in a correct way.
- 3.4 The dairy *unit* processing the milk collected by use of the Lactoperoxidase system should be made responsible for ensuring that the method is used as intended. This dairy should set up appropriate control methods (see Section 5) to monitor usage of the method, raw milk quality and quality of the milk prior to processing.
- 3.5 The method should primarily be used to prevent undue bacterial multiplication in raw milk during collection, **storage** and transportation to the dairy processing plant under conditions stated in 3. 1. The inhibitory effect of the treatment is dependent on the temperature of the stored milk **and on its microbiological quality**. and It has been found to act for the following periods of time in laboratory and field-experiments carried out in different countries with raw cow milk of an initial good hygienic standard:

Temperature, °C	Time, h
30	7-8
25	11 - 12
20	<i>16-17</i>
15	24-26

3.6 The use of the Lactoperoxidase method does not exclude the necessity of pasteurization of the milk before human consumption. Neither does it exclude the normal precautions and handling routines applied to ensure a high hygienic standard of the raw milk.

4. PRACTICAL APPLICATION OF THE METHOD

4.1 The Lactoperoxidase system can be activated in raw milk to give the above stated antibacterial effect by an addition of thiocyanate as sodium thiocyanate and hydrogen peroxide in the form of sodium percarbonate by the following procedure:

14 mg of NaSCN or equivalent quantities according to the content of the ion in the milk, but never surpassing the quantity indicated of 14 mg of the reactant is added per litre of milk. The milk should then be mixed to ensure an even distribution of the SCN-. Plunging for about 1 minute with a clean plunger is normally satisfactory.

Secondly, 30 mg of sodium percarbonate (containing 32% of free oxygen or an equivalent weight per litre of milk) is added per litre of milk. The milk is then stirred for another 2-3 minutes to ensure that the sodium percarbonate is completely dissolved and the hydrogen peroxide is evenly distributed in the milk. The utilization of formulations containing the activator substances suitably dosed for determined milk volumes, facilitate the application of the method and reduce the possible errors that could occur when this doses are conformed by the direct weighing of the salts.

- 4.2 It is essential that the sodium thiocyanate and sodium percarbonate are added in the *correct* order *as* stated above *on the label of the product*. The enzymatic reaction is started in the milk when the *sodium* percarbonate (hydrogen peroxide *source*) (sodium percarbonate) is added. It is completed within about 5 minutes from the addition of H202; thereafter, no hydrogen peroxide is present in the milk.
- 4.3 The *optimal* activation of the Lactoperoxidase system should be carried out within 2-3 hours from the time of milking. The activation after this time could reduce the beneficial effect, due to the increasing number of bacteria and the acidity of the milk.
- 4.4 Quantities of sodium thiocyanate and sodium bicarbonate needed for the treatment of a certain volume of milk, or example 40 or 50 litres milk clums, should be distributed to the collecting centre/point in prepapeked ammounts lasting for a few weeks at a time. The technical specifications of the thiocyanate and sodium percarbonate which should be used as states in Appendices 1 and 11. The label of the pre-packed activators of the LP system should have all the specifications of use in a clear and direct manner, including the specific amounts of activators and product shelf-life.

5. CONTROL OF USAGE

- 5.1 The use of the Lactoperoxidase system for preserving raw milk must be controlled by the dairy processing plant *unit* receiving the milk. This should be a combination of currently used acceptance tests, e.g. *pH measurement*, titratable acidity, methylene blue, resazurin, total viable count and analyses of the thiocyanate concentration in the milk. Since the thiocyanate is not *completely* consumed in the reaction, treated milk arriving at the dairy plant *unit* would contain approximately 10 mg above the natural amount of thiocyanate ion (the latter can be determined by analysing untreated milk from the same area) per litre of milk. The analytical method for SCN- is described in Appendix III 111 Testing should be undertaken at random. If the concentration of thiocyanate is too high (or too low), investigation must be carried out to determine why the concentration is outside specification. The dairy processing plant *unit* should also be responsible for the control of the chemicals to be used at the collection for the activation of the Lactoperoxidase system.
- 5.2 Analysis of the *acidity and of the* bacteriological quality of the milk (*Titratable acidity, pH measurement*, methylene blue, resazurin, total plate count) should also be carried out to ensure that good hygienic standards are not neglected. Since the effects of the system are predominantly bacteriostatic, an initial high bacterial population in the milk can still be revealed by such tests.

APPENDIX-1-1: TECHNICAL SPECIFICATION OF SODIUM THIOCYANATE

Definition

Chemical name Sodium thiocyanate

Chemical formula NaSCN
Molecular weight 81.1
Assay content 98-99%
Humidity 1-5 2%

Purity (according to JECFA * specification)

Heavy 1-leavymetals (as Pb)< 2 ppm</th>Sulphates (as S04)< 50 ppm</td>Sulphide (S)< 10 ppm</td>

^{*} Joint FAO/WHO Expert Committee on Food Additives.

APPENDIX II: TECHNICAL SPECIFICATION OF SODIUM PERCARBONATE

Definition

Chemical name Sodium percarbonate Chemical formula 2Na2C03.3 H-H202

Molecular weight 314.0 Assay content 85% Minimum concentration of peroxide 32%

Commercial available sodium percarbonate recommended to be used has the following specification:

Purity

Sodium carbonate peroxyhydrate > 85% Heavy metals (as Pb) < 10 ppm Arsenic (as As) < 3 ppm

For information where sodium percarbonate could be obtained commercially, please apply to the iDF General Secretariat, 41 Square Vergote, B-1040 Brussels, Belgium.

APPENDIX III: ANALYSIS OF THIOCYANATE IN MILK

Principle

Thiocyanate can be determined in milk, after deproteinization, with trichloroacetic acid (TCA) as the ferric complex by measuring the absorbance at 460 *nm* rim. The minimum level of detection by this method is 1 to 2 ppm of SCN-.

Reagent Solutions

- 1. 20% (w/w) trichloroacetic acid: 20 g TCA is dissolved in 100 ml of distilled water and filtered
- 2. Ferric nitrated reagent: $16.0 \text{ g Fe}(\text{N0}_3)_3.9\text{H}_2\text{O}$ is dissolved in 50 ml $2 M \text{ HNO}_3$ and then diluted with distilled water to 100 ml. The solution should be stored dark and cold.
 - * 2 M HNO₃ is obtained by diluting 138.5 ml 65% HNO₃ to 1000 ml with distilled water.

Determination

4.0 nil ml of milk is mixed with 2.0 nil ml of 20% TCA solution. The mixture is blended well and then allowed to stand for at least 30 minutes. It is thereafter filtered through a suitable filter paper (Whatman No. 40). 1.5 ml of the clear filtrate is then mixed with 1.5 nil ml of the ferric nitrate reagent and the absorbance measured at 460 nm. As a blank, a mixture of 1.5 ml of ferric nitrate solution and 1.5 ml of water is used. The concentration of thiocyanate is then determined by comparison with standard solutions of known thiocyanate concentration, e.g.