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FOOD AND AGRICULTURE  
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**Agenda Item 4**

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**JOINT FAO/WHO FOOD STANDARDS PROGRAMME**

**CODEX COMMITTEE ON CONTAMINANTS IN FOODS**

**4<sup>th</sup> Session**

**Izmir, Turkey, 26 – 30 April 2010**

**PROPOSED DRAFT CODE OF PRACTICE FOR THE REDUCTION OF ETHYL CARBAMATE  
IN STONE FRUIT DISTILLATES (N11-2009)**

*Comments at Step 3 submitted by Brazil, Canada, Chile, Japan, United States of America, CIAA and FAO*

## **BRAZIL**

First of all, we would like to thank Germany for the work on the PROPOSED DRAFT CODE OF PRACTICE FOR THE PREVENTION AND REDUCTION OF ETHYL CARBAMATE CONTAMINATION IN STONE FRUIT SPIRITS AND STONE FRUIT MARC SPIRITS. We appreciate the opportunity to express our opinion.

We believe the inclusion of some items as scope, definitions (head, tail, heart, copper catalytic converters, etc) and a flow with the process steps, including storage (and a description of each step) could bring more clarity to the Code. The flow should include a phase to remove the stone, if the production technology permits this. If the stones are removed, the recommendations for other steps may not be applied. In this case, should be provided some care to avoid mechanical damage at the stone, beyond the time for processing, protection against light, cooling, etc..

The steps in part II should be harmonized with the steps of part I and practices should be recommended for each step.

It should be emphasized practices to minimize the Ethyl carbamate formation as light and temperature.

It is important to highlight the ideal conditions to storage the fermented mash for further distillation process. Only the light is important?

We would like some clarification about copper catalytic converters and the way to assure that the acid is linked before the distillation process.

It is necessary to describe how to clean the distillation equipment in case of discontinuous distillation.

We have a concern regarding the use of Copper agents that were not specified and assessed by JECFA, because buying them in specialized shops does not assure a good quality product and a safe use. Which copper agents and what level of these Copper agents are appropriate to be added in the fermented fruit mash? After this addition, there is any recommendation as to protect against light?

There is any recommendation on how to remove the stones before the distillation process?

Criteria should be established to redistilling the tail. Just redistilling separately is enough to obtain a product with safe level of the contaminant?

The establishment of Ethyl carbamate or Hydrocyanic acid levels in the final product is out of the scope of this document. The performance objective could be established by monitoring the process and taking a decision to redistillate the product, but it is important to highlight that there is no consensus regarding the correlation between the level of hydrocyanic acid and ethyl carbamate.

It is necessary to have some provisions to storage the final product, including recommendations to the storage by the consumers in order to prevent the ethyl carbamate formation at this stage. The color of the bottle is important? Other point to be assessed is the control during the aging process.

The paragraph 9 is not appropriated for a Code a Practice. The Committee may assess if it could be included as a recommendation in the report.

## CANADA

Canada wishes to thank Germany for developing the Proposed Draft Code of Practice for the Prevention and Reduction of Ethyl Carbamate Contamination in Stone Fruit Spirits and Stone Fruit Marc Spirits (CX/CF 10/4/4).

Canada would recommend adding a note to sections 7, 30 and/or 31 reiterating that "According to the group of experts for spirit drinks of the German Society for Food Chemistry based on practical experiences it can be assumed that from 1 mg of hydrocyanic acid up to 0.4 mg ethyl carbamate potentially can be formed in a non-equimolar relationship (German Society for Food Chemistry, 2006)". This was mentioned in the Discussion Paper on Ethyl Carbamate in Alcoholic Beverages, agenda item 9(e), CX/CF 09/3/13. We believe the statement will allow jurisdictions to better understand where the 1 mg of hydrocyanic acid threshold originates, and to adjust the guide to their particular situation, as needed.

## CHILE

1. Targets and scope	In their 3 <sup>rd</sup> meeting, the CCCF decided to initiate a new work on a Code of Practices on the theme indicated in the title of this position paper and also decided that the delegation from Germany would prepare a proposed draft document to collect information in Step 3
2. Relevance and topicality. Evaluate whether the proposed text contributes health measures towards impacting on the problem.	The revision of the proposed text allows for a revision of the manufacture of stone fruit spirits and fruit marc spirits, and how to prevent and reduce the formation of ethyl carbamate in the production of these drinks.
3. Another viewpoint is that of estimating whether the standard will improve or deteriorate international exchange flow of this type of foods in the medium or long term.	The Code will improve the production of the drinks indicated, especially in non-industrial production.
4. Examine whether the proposed measures in the preliminary draft can be feasibly applied by developing countries. This can	The recommended practices, based on GMP are easier to apply in industries but it is unknown whether they can be applied at the non-industrial level. The analytical capacity has not been developed by laboratories

be looked at from the point of view of technical complexity, the installed laboratory capacity and the economic cost, among others.	specializing in analysis of ethyl carbamate in the country. (gas chromatography/mass spectrometry)
5. That it not be a repetition of other already existing standards.	It is not a repetition since it corresponds to a Code of Practice which is beginning as a new work.
6. In the case where there are shortcomings, as for example aspects that are lacking or in existence, but insufficiently treated, propose how this could be improved or complemented by national and regional contributions.	Attached Text where the addition of the point, GENERAL RECOMMENDATIONS, is suggested, taking into consideration the fact that in other codes of practices this is included.
<b>7. Proposal of national position.</b>	Support the Proposed draft with the suggested corrections.

Attached Text

#### **GENERAL RECOMMENDATIONS**

1. The national, state and local governments as well as the non-governmental organizations (NGOs, commercial associations and cooperatives) should provide their own basic training and update the information on the dangers associated with contamination by ethyl carbamate in stone fruit spirits and fruit marc spirits.
2. The non-industrial, small-scale preparation of these drinks should have available material with information on the specific recommendations based on good manufacturing practices and guidance on prevention and reduction of ethyl carbamate in the stone fruit distillates.
3. Verification is recommended of the level of ethyl carbamate in the distillates where the compound could be present (e.g. distillates with an unknown history or storage under light), and this should be carried out in a specialized laboratory.

#### **JAPAN**

Japan supports this proposed draft document, which provides useful guidance comprising comprehensive measures to prevent and reduce ethyl carbamate contamination in stone fruit spirits and stone fruit marc spirits. However, in order to clearly identify the scope of the document and to make some improvement, Japan would like to propose the following specific comments:

## Paras 6 and 7

In order to clearly identify the Scope of the products covered in this Code, Japan proposes to create a new Section “SCOPE AND DEFINITIONS” after Introduction Section. Japan has a question whether or not this Code covers the distillates obtained through the distillation of alcoholic beverages in which stone fruits have been macerated. In this moment, Japan believes that such distillates can be included in the Scope of this Code and would like to propose the insertion of the Scope Section to address them. In addition, one minor modification in para. 6 should be made to delete the reference to “stone fruit distillates” as it is unnecessary. The proposals are as follows:

6. ... In ~~stone fruit distillates~~ (stone fruit spirits and stone fruit marc spirits) ethyl carbamate ...

### SCOPE AND DEFINITIONS

7bis. This Code of Practice intends to provide national and local authorities, manufactures and other relevant bodies with guidance to prevent and/or reduce formation of ethyl carbamate in stone fruit spiris and stone fruit marc spirits. Ethyl carbamate formation in other alcoholic beverages and foods is not covered in this Code.

7ter. The definitions below apply to this Code:

STONE FRUIT means a fruit which is produced on trees belonging to the genus *Prunus* of the rose family (*Rosaceae*).

DISTILLATES means alcohol-rich products obtained after the distillation process.

STONE FRUIT SPIRITS means the distillates for consumption, obtained after distillation process of the mash prepared by fermentation of crushed stone fruit (so-called “-wasser” in Germany, “eau-de-vie” in France) or by maceration of crashed and/or whole stone fruit in alcoholic beverages (so-called “-geist” in Germany).

STONE FRUIT MARC SPIRITS means the distillates for consumption, obtained after distillation process of fermented stone fruit residues of juice or wine (so-called “pomace brandies”).

### After para. 7

Japan proposes to create a new Section “GENERAL REMARKS” before Part I as follows, aimed to provide a remark on the use of this Code. In addition, Japan proposes to transfer paras 8 and 9 from the INTRODUCTION to this new Section with some minor modifications for clarity.

### GENERAL REMARKS

7quat. This Code covers all possible measures that have been proven to prevent and/or reduce high levels of ethyl carbamate in stone fruit spirits and stone fruit marc spirits. When applying the Code for specific stone fruit spirits and stone fruit marc spirits, measures should be carefully chosen from the viewpoint of benefit and feasibility. In addition, measures should be implemented in accordance with the relevant national and international legislation and standard.

8. It is recognised that reasonably applicable technological measures - Good Manufacturing Practices - can be taken to prevent and reduce significantly high ethyl carbamate levels in stone fruit ~~distillates~~ spirits and stone fruit marc spirits. The prevention and reduction of ethyl carbamate could be achieved using two different approaches: first, by reducing the concentration of the main precursor substances; second, by reducing the tendency of these substances to react to form ~~cyanate~~ ethyl carbamate. The main influencing factors on ethyl carbamate formation are the concentration of precursors (e.g. hydrocyanic acid and cyanides) and storage conditions, such as light exposure and temperature.

9. It is recommended that ~~the~~ levels of ethyl carbamate in stone fruit spirits and stone fruit marc spirits ~~should be~~ are monitored after implementation of measures in the Code ~~during a time period of three years and the results be used to assess their effects~~ this Code of Practice after three years of implementation. Thereafter, the possibility of setting a maximum level should be assessed.

**After para. 9**

Two sentences in paragraph with no number (after para. 9) are not necessary, hence they should be deleted.

**Part I**

The reference to “Part I” in Section title should be removed as it is unnecessary. In addition, Japan proposes to amend the text in para. 10 as follows, in order to properly address both stone fruit spirits and stone fruits marc spirits:

**~~PART I~~-TYPICAL PRODUCTION PROCESS**

10. The production process for stone fruit spirits and stone fruit marc spirits involves preparing mashing by using and fermentation of the whole stone fruits or their residues as ingredients, followed by distillation. The process typically follows the steps listed below:
  - o preparing the mash by crushing the whole ripe fruit (for stone fruit spirits) or by using stone fruit residues of juice or wine (for stone fruit marc spirits);
  - o fermenting the mash in stainless steel tanks or other suitable fermentation vessels;
  - o in the case of using maceration process, the mash is prepared by macerating crashed or whole fruit into alcoholic beverages and store for a period, without fermentation process;
  - o transferring the ~~fermented~~-mash containing alcohol into the distillation device, often a copper pot;
  - o heating the ~~fermented~~-mash by a suitable heating method in order to slowly boil off the alcohol;
  - o cooling the alcohol vapour in an appropriate (e.g. stainless steel) column where it condenses and is collected;
  - o if needed, the collected distillates may be distilled again in order to concentrate alcohol;
  - o separation of three different fractions of alcohol in the process of final distillation: ‘heads’, ‘hearts’ and ‘tails’;
  - o dilution to the final alcoholic grade

**Part II**

The reference to “Part II” in Section title should also be removed as it is unnecessary. Japan proposes to amend para. 14 as follows:

**~~PART II~~-RECOMMENDED PRACTICES BASED ON GOOD MANUFACTURING PRACTICES (GMP)****Raw materials and preparation of ~~fruit~~ mash**

14. The raw materials and preparation of the ~~fruit~~-mash should be suitable to avoid the release of hydrocyanic acid.

**Para. 17**

Japan proposes the following modification:

17. If ~~the fruits are not~~ de-stoned fruits and/or the residues of fruits are used for preparing mash, they should be mashed gently avoiding the crushing of stones. If possible, stones should be removed from the mash.

**Para. 19**

Japan proposes to revise the second sentence as follows:

19. ... The finally fermented fruit mash should be stored as short as possible before distillation since hydrocyanic acid may also be released from intact stones during longer storage of the mash in case the fruit was not de-stoned.

**After para. 19**

Japan proposes to add a new Subsection after para. 19 to describe the practices for preparing mash using maceration process of fruit, if the product using such technique is covered by this Code.

**Maceration**

19bis. If the mash is prepared by macerating stone fruit into alcoholic beverages, the stone fruit should be removed soon after the aroma of stone fruit is adequately extracted.

**Paras 21 and 22**

Japan proposes to modify the first sentence of para. 21 because it is not practical to introduce automatic rinsing devices and copper catalytic converters into the distillation equipment that is already working without them. Along with this modification, Japan proposes to modify para. 22 as follows:

21. The distillation equipment should preferably include automatic rinsing devices and copper catalytic converters. The automatic rinsing devices will keep the copper stills cleaned while the copper catalytic converters will bind hydrocyanic acid before it passes into the distillate.
22. If it is difficult to include automatic rinsing devices are not in the necessary in the case of discontinuous distillation equipment, the distillation equipment should be cleaned by systematic and thorough cleaning procedures.

**Paras 23 and 30**

Japan would like to request for clarification on the term “copper agents” used in paras 23 and 30. If the term means some copper-containing chemicals, we believe that they should be limited to those which JECFA assessed.

**Paras 30 and 31**

Japan is of the opinion that, following the agreement of the previous Session (see paragraph 115 of ALINORM 09/32/41) to start the new work on the elaboration of this Code which will not include a signal value, this Code should not mention the level of 1 mg/l for hydrocyanic acid, a precursor of ethyl carbamate. Therefore, Japan proposes the following amendments:

30. If the concentration of hydrocyanic acid in the distillate is relatively high exceeds a level of 1 mg/l, re-distillation with catalytic converters or copper agents is recommended (see points 20, 21 and 23).
31. Distillates should be stored in lightproof bottles or covering boxes and storage time should be kept as short as possible, particularly if the distillates are close to a level of 1 mg/l hydrocyanic acid.

**Para. 32**

Japan proposes to add a new paragraph after para. 32 as follows:

32bis. Additional distillation is effective in order to reduce ethyl carbamate in distillates.

**USA**

The United States of America (U.S.) appreciates the efforts of the German delegation to prepare the draft Code of Practice (COP). The U.S. offers the following comments:

**Paragraph 1**

The U.S. suggests modifying this sentence to read “soy sauce” instead of “sauce” as follows (change underlined): “Ethyl carbamate is a compound that occurs naturally in fermented foods and alcoholic beverages such as bread, yoghurt, soy sauce, wine, beer, and particularly in stone fruit spirits and stone fruit marc spirits, mainly those made from cherries, plums, mirabelles and apricots.”

**Paragraph 5**

The U.S. suggests deleting the phrase “Based on a broader number of samples” in the first sentence, so that the sentence reads as follows: “The Scientific Panel on Contaminants in the Food Chain of the European

Food Safety Authority (EFSA) adopted on 20 September 2007 a scientific opinion on ethyl carbamate and hydrocyanic acid in food and beverages and concluded that ethyl carbamate in alcoholic beverages indicates a health concern, particularly with respect to stone fruit brandies, and recommended taking mitigation measures to reduce the levels of ethyl carbamate in these beverages.”

#### **Paragraph 6**

For clarity, the U.S. suggests modifying the first sentence to read as follows (changes underlined): “Stone fruit and stone fruit marc spirits, in particular, contain ethyl carbamate in many fold higher concentrations than other fermented foods and beverages.”

For clarity, the U.S. suggests revising sentences five, six, and seven of this paragraph to read as follows (changes underlined): “Hydrocyanic acid may also be released from intact stones during prolonged storage of the fermented mash. During the distillation process, hydrocyanic acid may be enriched in certain fractions of the distillate. Ultimately, cyanide may be oxidized to cyanate, which can react with ethanol to form ethyl carbamate. Certain environmental conditions, such as exposure to light or the presence of copper ions in the distillate, may promote the formation of ethyl carbamate in the distillate.”

Also in paragraph 6, the U.S. suggests deleting the final sentence, “Once the reaction has been triggered, it cannot be stopped.”

#### **Paragraph 8**

The last sentence of this paragraph cites temperature as a major factor influencing ethyl carbamate formation. The U.S. suggests that information should be included later in the COP on how temperature should be regulated to reduce ethyl carbamate formation (e.g., storing or not storing distillates at certain temperatures).

#### **Paragraph 9**

The U.S. suggests removing paragraph 9 as it contains a policy recommendation for the Committee and does not belong in a COP.

#### **Paragraph 14**

For clarity, the U.S. suggests adding the phrase “a precursor of ethyl carbamate” to the end of the sentence so that it reads as follows (changes underlined): “The raw materials and preparation of the fruit mash should be suitable to avoid the release of hydrocyanic acid, a precursor of ethyl carbamate.”

#### **Paragraph 15**

To explain why high-quality fruit is important, the U.S. suggests adding the phrase “as damaged and spoiled fruit may contain more free cyanide” to the end of the sentence, as follows: “The stone fruits should generally be of a high quality, not mechanically damaged and not microbiologically spoiled, as damaged and spoiled fruit may contain more free cyanide.”

#### **Paragraph 17**

Because damage short of crushing may release hydrocyanic acid, the U.S. suggests modifying this sentence to read as follows (changes underlined): “If the fruits are not de-stoned, they should be mashed gently to avoid damaging the stones.”

#### **Paragraph 18**

For clarity, the U.S. suggests rewording this paragraph as follows: “Spirit manufacturers should select appropriate yeast preparations and follow manufacturer instructions to allow a fast and clean fermentation.”

Also, it may also be useful to include a definition of “fast and clean fermentation” (e.g., length of time, indicators of “clean”).

**Paragraphs 18-19**

The U.S. suggests consideration of a new paragraph between paragraphs 18 and 19, based on an article by Schehl et al. (2007)<sup>1</sup>: “Some ethyl carbamate in spirits may result from urea production from yeast. Yeast strains that result in lower amounts of ethyl carbamate may be available now or in the future. Manufacturers should select yeast strains that result in lower levels of ethyl carbamate in spirits.”

**Paragraph 19**

For clarity, the U.S. suggests revising the second sentence as follows (changes underlined): “Fermented fruit mashes containing stones should be stored as briefly as possible before distillation since hydrocyanic acid may also be released from intact stones during prolonged storage.”

**Paragraphs 20-21**

The U.S. suggests addition of a new paragraph between paragraphs 20 and 21 as follows: “Use of a copper still will limit carryover of ethyl carbamate-forming precursors into the distillate.”

**Paragraphs 21-22**

The U.S. suggests addition of a new paragraph between paragraphs 21 and 22 as follows: “Use of a stainless steel condenser rather than a copper condenser will limit presence of copper in the distillate, where copper can promote formation of ethyl carbamate.”

**Paragraph 23**

The U.S. suggests the following changes to the first sentence for clarity (changes underlined): “When copper catalytic converters or other dedicated cyanide separators are not available, copper agents can be added to the fermented fruit mash before distillation.”

**Paragraph 28**

The U.S. suggests the following changes for clarity (changes underlined): “Some manufacturers may redistill the separated tails, possibly containing ethyl carbamate. If the tails are used for redistilling, they should be re-distilled separately.”

The U.S. also suggests adding the following sentence to the paragraph: “Another option for reducing ethyl carbamate concentration in spirits is not to redistill tailings.”

**Paragraph 29**

The U.S. suggests adding a new sentence to the beginning of the paragraph and adding the word “Therefore,” to the beginning of the second sentence as follows (changes underlined): “Testing for hydrocyanic acid may be used as a simple test for ethyl carbamate in distillates, since low hydrocyanic acid levels predict low ethyl carbamate levels. Therefore, distillates should be regularly checked for their levels of hydrocyanic acid.”

**Paragraph 31**

The U.S. suggests changing the phrase “lightproof bottles” to “bottles that are lightproof (or filter ultraviolet light),” as shown in the following (changes underlined): “Distillates should be stored in bottles that are lightproof (or filter ultraviolet light) or in covering boxes, and storage time should be kept as short as possible, particularly if the distillates are close to a level of 1 mg/l hydrocyanic acid.”

The U.S. also suggests clarification of the phrase “keeping storage time as short as possible,” given that shops and consumers may store spirits for long periods of time.

**Paragraph 32**

The U.S. suggests that the phrase “or at high temperatures” be added to the following sentence (change underlined): “Testing of ethyl carbamate is recommended for distillates in which the compound may already

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<sup>1</sup> Schehl B, et al. Appl Microbiol Biotechnol (2007) 74:843–850.



have been formed (e.g. distillates with unknown history of production, distillates with higher levels of cyanide, or storage with exposure to light or at high temperatures).

The U.S. appreciates the opportunity to comment on this document.

## **CIAA**

We would like to thank you for allowing us the opportunity to comment on the Codex Draft Code of Practice the prevention and reduction of ethyl carbamate contamination in stone fruit spirits and stone fruit marc spirits (CX/CF 10/4/4).

We are glad to report that our members do not see difficulties in implementing the monitoring system and the 1mg/l target threshold suggested by the Code of Practice. In some Member States (e.g. Czech Republic), legal maximum levels for ethyl carbamate, equal or lower than 1 mg/l, are already applicable. Moreover, most of the EU stone-fruit spirits producers have commonly adopted very low standard levels (below 1 mg/l) as best practices.

In any case, CIAA welcomes the time period of three years to assess the implementation of the Code of Practice and that the possibility of setting a maximum level will be considered thereafter.

## **FAO**

Thank you for the opportunity to comment on the draft CoP for reduction of ethylcarbamate in stone fruit distillates.

I refer to the relevant paragraphs in the draft CoP in my comments below.

Scope: Why is the scope including also specifically marc (or pomace) stone fruit spirits, when no reference is made in the CoP either on how they are produced or how the cyanide levels may be reduced. This leads to the question if not marc (or pomace) spirits on the whole should be included in the CoP, as also pomace from wine production is distilled to marc spirits and these may also contain ethylcarbamate.

para 4. Please add the reference to the JECFA monograph on ethylcarbamate, which contains the complete evaluation. WHO Food Additive Series 55 and FAO Food and Nutrition Paper 82, 2006, p. 205-316. [http://whqlibdoc.who.int/publications/2006/9241660554\\_eng.pdf](http://whqlibdoc.who.int/publications/2006/9241660554_eng.pdf) with the ethylcarbamate monograph available at this link [http://whqlibdoc.who.int/publications/2006/9241660554\\_ETH\\_eng.pdf](http://whqlibdoc.who.int/publications/2006/9241660554_ETH_eng.pdf).

para 5. It is suggested to delete the introductory phrase "Based on a broader number of samples" as this is probably not the reason why a scientific opinion was asked of EFSA. Also, the JECFA evaluation contained substantial amounts of data on occurrence in foods and beverages.

para. 6. In the 3rd line from the bottom, copper ions are mentioned as catalysts for the formation of cyanide to cyanate. This has not been mentioned in the JECFA monograph, and furthermore, it is confusing with the recommendations and text in paragraphs 21 and 23. This should be checked.

para 21, 23 and 30. There are also possibilities to use ion-exchange resins and silver, in addition to copper to complex cyanide to prevent cyanate formation. Please see page 286 of the WHO FAS 55.

para 31. There is only one reference to the level of 1 mg/l of HCN as a cut-off level for increased formation of ethylcarbamate. As it is not generally the level of cyanide but cyanate that triggers the formation, should not a level of cyanate be more relevant. How robust is the data on this cut-off level?

para 29 and para. 32. It would be good to have references to recent analytical methods used, with detection limits etc. and not only refer to specialist laboratories. See e.g. the JECFA monograph pages 275 - 277 for information on methods and sampling.

The storage in dark bottles for the shelf-life of the products should be emphasized.