Supervised trials median residue – processed (STMR-P) (new definition)

The STMR-P is the expected residue in a processed commodity calculated by multiplying the STMR of the raw agricultural commodity by the corresponding processing factor, or derived directly from a series of processing trials The STMR-P is expressed in units of mg/kg

Temporary MRL (TMRL) or Temporary EMRL (TEMRL)

A TMRL or a TEMRL is an MRL or EMRL established for a specified, limited period and is recommended under either of the following conditions

1 Where a temporary acceptable daily intake has been estimated by the Joint FAO/WHO Meeting on Pesticide residues for the pesticide or contaminant of concern, or

2 Where, although an acceptable daily intake has been estimated, the good agricultural practice is not sufficiently known or residue data are inadequate for proposing an MRL or ERL by the Joint FAO/WHO Meeting on Pesticide Residues (Codex Alimentarius Vol 2A)

Note TMRLs and TEMRLs are not to be advanced further than Step 7 of the Codex Procedure

The 1992 JMPR gave the following definition (Report, section 2 8)

> A temporary maximum residue limit is a maximum residue limit for a specified, limited period, which is clearly related to required information

Comments

The "temporary maximum residue limit" is a successor of the "temporary tolerance" introduced by the 1966 JMPR, which was changed to "temporary maximum residue limit" in 1975

At the 1988 JMPR the decision was taken not to establish Temporary Acceptable Daily Intakes any longer for new compounds

According to the Report of 1992 JMPR, there is still a possibility that TMRLs may be recommended when the information lacking on some residue aspects is unlikely to affect the validity of an estimated maximum residue level and would be available shortly Each recommended TMRL will be directly related to an item of required information

See also Chapter 5 section, "Recommendation of temporary MRLs "

Appendix III

CIPAC CODES FOR PESTICIDE FORMULATIONS

AB	Grain bait
AE	Aerosol dispenser
AL	Other liquids to be applied undiluted
AP	Other powders to be applied undiluted**
BB	Block bait
BR	Briquette
CB	Bait concentrate
CF	Capsule Suspension for Seed Treatment**
CG	Encapsulated granule
CL	Contact liquid or gel**
CP	Contact powder**
CS	Capsule suspension
DC	Dispersible concentrate
DP	Dustable powder
DS	Powder for dry seed treatment
DT	Tablet for direct application**
EC	Emulsifiable concentrate
ED	Electrochargeable liquid
EG	Emulsifiable Granule**
EO	Emulsion, water in oil
EP	Emulsifiable powder**
ES	Emulsion for seed treatment
EW	Emulsion, oil in water
FD	Smoke tin
FG	Fine granule
FK	Smoke candle
FP	Smoke cartridge
FR	Smoke rodlet
FS	Flowable concentrate for seed treatment
FT	Smoke tablet
FU	Smoke generator
FW	Smoke pellet
GA	Gas
GB	Granular bait
GE	Gas generating product
GF	Gel for Seed Treatment**
GG	Macrogranule
GL	Emulsifiable gel
GP	Flo-dust
GR	Granule
GS	Grease
GW	Water soluble gel
HN	Hot fogging concentrate
KK	Combi-pack solid/liquid*
KL	Combi-pack liquid/liquid*
KN	Cold fogging concentrate
KP	Combi-pack solid/solid*
LA	Lacquer
LS	Solution for seed treatment
LV	Liquid vapouriser**
MC	Mosquito coil**
ME	Micro-emulsion**
MG	Microgranule
MV	Vapourizing mats**
OF	Oil miscible flowable concentrate (oil miscible suspension)
OL	Oil miscible liquid
OP	Oil dispersible powder
PA	Paste
PB	Plate bait
PC	Gel concentrate or paste concentrate
PO	Pour-on
PR	Plant rodlet
PS	Seed coated with a pesticide
RB	Bait (ready to use)
SA	Spot-on
SB	Scrap bait
SC	Suspension concentrate (= flowable concentrate)
SD	Suspension concentrate for direct application**
SE	Suspo-emulsion
SG	Water soluble granule
SL	Soluble concentrate
SO	Spreading oil
SP	Water soluble powder
SS	Water soluble powder for seed treatment
ST	Water soluble tablet**
SU	Ultra-low volume (ULV) suspension
TB	Tablet
TC	Technical material
TK	Technical concentrate
TP	Tracking powder***
UL	Ultra-low volume (ULV) liquid
VP	Vapour releasing product
WG	Water dispersible granule
WP	Wettable powder
WS	Water dispersible powder for slurry seed treatment
WT	Water dispersible tablet**
XX	Others

* Special two-letter codes for twin-packs

** Manual on Development and Use of FAO and WHO Specifications for Pesticides, FAO/WHO Joint Meeting on Pesticide Specifications (JMPS), Rome, 2002

*** Discontinued term – refer to CP (Contact Powder)

Appendix IV

MRL PERIODIC REVIEW PROCEDURE BY CCPR
(ALINORM 97/24 APPENDIX III)

CODEX COMMITTEE ON PESTICIDE RESIDUES
MRL PERIODIC REVIEW PROCEDURE

Periodic review may also be referred to as periodic re-evaluation The two terms are synonymous "Periodic review programme" and "periodic review procedure" also mean the same thing

The periodic review programme was initiated to ensure that the support for Codex MRLs would be brought up to modern standards A complete data submission is requested for old compounds Recommendations to confirm, amend or delete old MRLs or to introduce new MRLs arise from the new data The periodic review procedure consists of two distinct phases as described below

PHASE I

IDENTIFY PERIODIC REVIEW CHEMICALS AND SOLICIT DATA COMMITMENTS
(Year 1, CCPR Meeting)

Identify candidate chemicals for re-evaluation

On an annual basis the CCPR (Working Group on Priorities) lists chemicals meeting the following criteria

- pesticide chemicals for which MRLs were first estimated more than 10 years ago,

or

- pesticide chemicals for which a periodic review was conducted more than 10 years ago

A list of candidate compounds is prepared for the Working Group on Priorities report – Annex 1, List of candidate compounds for periodic re-evaluation –not yet scheduled

Notify data owners or other parties of candidate list

Governments and international organizations represented at the annual CCPR Meeting expeditiously notify current data owners (or other interested parties) of the candidate list for periodic reviews, and when available, tentative lists for the following years A copy of the most recent procedure for periodic review is also included

Invite commitment to support continued (or new) codex maximum residue limits (CXLs)

With their notification to data owners (or other interested parties) on the candidacy of chemicals for periodic review, governments and international organizations inquire of these parties their willingness to provide data for that review and also to advise them of the implications if they choose not to

The invitation for a commitment will request a written response within six months to be provided to

- Chairman, CCPR
- Chairman, Priorities Working Group
- JMPR Secretariats
- the requester (government or international organization representative) Names, titles and addresses will be provided

The invitation will request that the following information be provided in the response

(i) A list of all commodities for which interested parties are willing to support CXLs

(ii) A brief summary of all current Good Agricultural Practice (GAP) which they are willing to provide and which is pertinent to residue data they are willing to provide (e g commodities and countries for which detailed GAP summaries and representative labels can be provided)

(iii) A lists of all chemistry (residue, metabolism, animal transfer, processing, analytical sample storage stability, analytical methods etc) and toxicology studies and other data that they are willing to provide (regardless of whether previously provided) and the complete data package submissions to the JMPR Comments on the status of registrations for the chemicals at the national level are encouraged Data for which a submission is committed should be identified in the response by study or report title and number, author and date

Note Data should be submitted in both paper and electronic form

Repeat the notification and invitation

By means of a Codex Circular Letter to accompany the report of the Meeting the Secretariat will repeat the notification and request On receipt of the request by the Circular Letter, governments and international organizations will immediately repeat their notification and invitation to identified interested parties who may not have been represented at the CCPR (they would not have received the report of the Meeting or the accompanying Circular Letter) Interested parties need only respond to one of the requests, but should copy addressees listed under "Invite commitment to support continued (or new) codex maximum residue limits"

PHASE II

STATUS REPORT ON DATA COMMITMENTS AND CCPR FOLLOW-UP
(Year 2, CCPR Meeting)

Status report on data commitments

The Priorities Working Group will provide a report and room document to the CCPR on the status of commitments received to provide data for each compound identified in year 1 This information will be used to schedule JMPR reviews or to make other recommendations such as withdrawal of CXLs

Response to data commitments

a) If there is no commitment to provide and identify or develop data to support current CXLs, the CXL(s) will be recommended by the CCPR for withdrawal by the next session of the Codex Commission

b) If a commitment is made to provide and identify or develop data to support current CXLs, the MRL(s) are scheduled for JMPR review The JMPR review will result in one of the following scenarios

- Sufficient data are submitted to confirm the CXL and it remains in place

- Sufficient data are submitted to support a new proposed MRL, it enters the process at Step 3 and the existing CXL is deleted automatically after no more than 4 years

- If insufficient data have been submitted to support a new MRL or to confirm the existing CXL, data submitters are so advised by written notification from the FAO Joint Secretary or by issuance of the JMPR Report

  On being advised of the data inadequacy, data submitters may by the next CCPR Meeting, provide to the FAO and CCPR Secretaries a written commitment to generate and submit a complete dossier of required data for review within 4 years The CXL is maintained for no more than 4 years following advice of data inadequacy (by direct notification or by issuance of the JMPR Report) The 4-year period may be extended by the CCPR only to the extent necessary for the JMPR to schedule and complete review of the available new data

  The new data are scheduled for the second JMPR review and the first part of the PHASE II "if a commitment is made" procedure is repeated

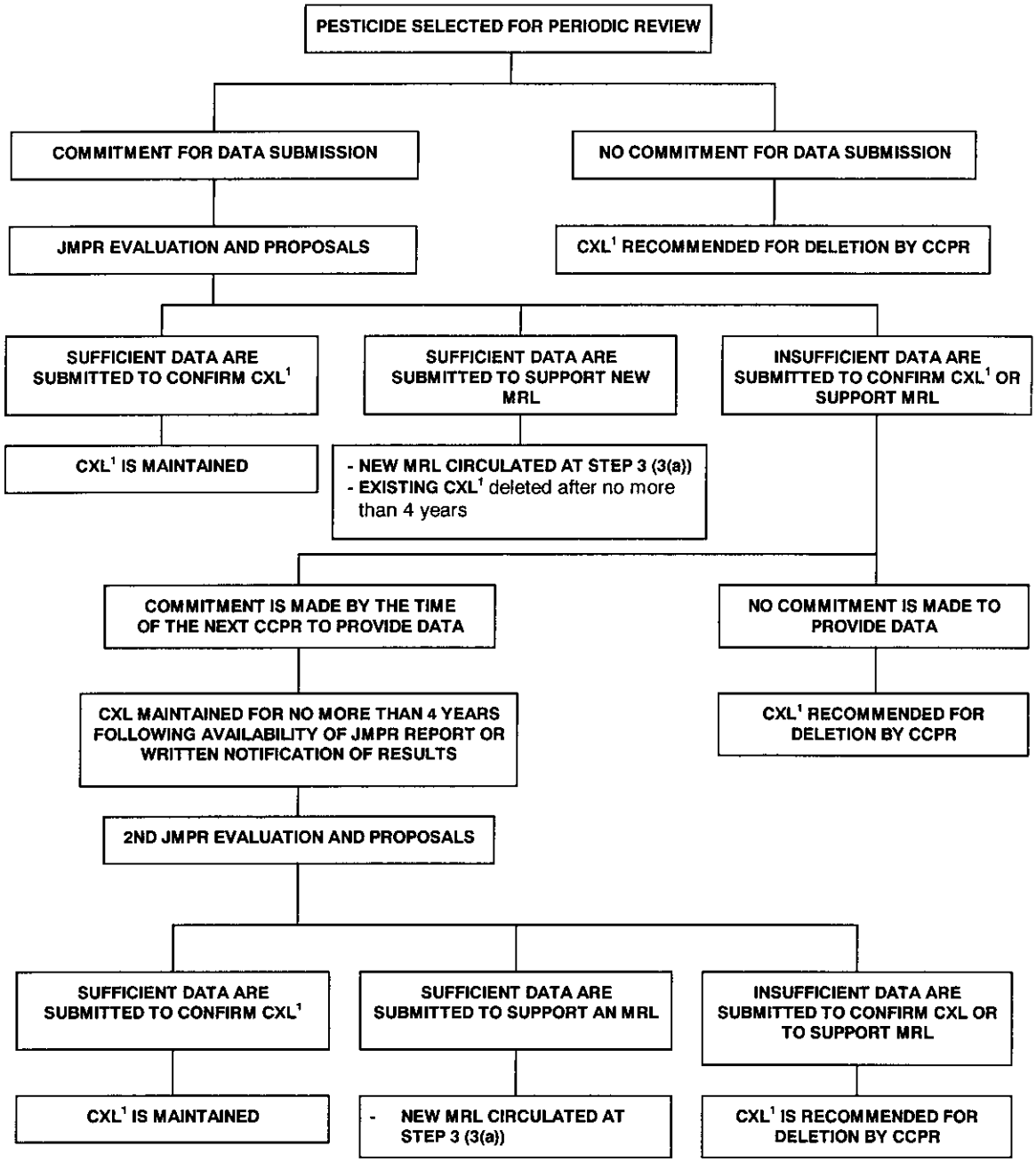
- Sufficient data are submitted to confirm the CXL and it remains in place

- Sufficient data are submitted to support a new proposed MRL and it enters the process at Step 3 The CXL is automatically deleted no more than 4 years after the new proposal enters the process

- Insufficient data are submitted to confirm the CXL or support a proposed MRL and the CCPR recommends deletion of the CXL

c) If the committed data are not submitted, or if the data submitted for the initial periodic review are insufficient and no commitment is made by the next CCPR Meeting to generate new data, the CCPR recommends deletion of the CXL

SUMMARY OF PERIODIC REVIEW PROCEDURE FOR CODEX MRLs



[1]Codex MRL adopted by the Codex Alimentarius Commission The Codex Alimentarius Commission may decide to delete certain Codex MRLs based on the recommendations made to it by the Codex Committee on Pesticide Residues

Appendix V

RECOMMENDED SAMPLING METHODS FOR SUPERVISED FIELD TRIALS[17]

CONTENTS

GENERAL RECOMMENDATIONS

The best information about the residue behaviour of the pesticide under study would be obtained by the analysis of the entire yield of a plot Since this is not practicable, representative samples have to be taken Careful attention to the details of sampling is essential if worthwhile samples are to be obtained Valid analytical results can only be obtained if the samples have been properly taken, despatched and stored before analysis

In selecting sampling points and the sampling method, all factors that control the residue distribution over the entire experimental plot must be considered The best approach for any given plot can only be determined by a sufficiently trained person who is capable of recognising the importance and usefulness of the residue data sought, and who can interpret the results

The samples must be representative to enable the analytical result to be applied to the entire experimental unit The greater the number of plants sampled in a field plot, the more representative the sample will be However, economics and the practical problems involved in handling large samples affect the magnitude of the sampling programme The size of sample suggested is the minimum that experience has shown is needed to give a representative, valid sample The sizes are not usually dictated by the analytical method, which can often determine minute amounts of pesticides in small amounts of sample

Method of sampling

Generally, the selection of the portions that make up the field sample should be made depending on the circumstances

- randomly, e g by the use of random numbers
- systematically, e g in the case of field crops on a diagonal ("X"or an "S" course)

[17] This procedure is extracted with some modifications form FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials, FAO Rome 1990

- selectively from predetermined sampling-points, e g in the case of tree fruits, take both exposed samples and those covered by foliage so that each fruit has an equal chance of being taken

Points to be borne in mind are

- Avoid taking samples at the beginning or at the extreme end of plots (start and finish of spraying)
- Take and bag the required weight or number of samples in the field and do not subsample until the samples are in a clean field laboratory or in the analytical laboratory
- Sample all parts of the crop that can be consumed by humans or livestock
- Sample the parts of the crop that normally constitute the commercial commodity as described in Appendix VI
- Where appropriate, consider commercial harvesting practice which reflects normal "Good Agricultural Practice" (see also this appendix section "Contamination")

Replication

In certain cases where there is likely to be considerable within-plot variation, such as orchard and glasshouse trials, three sample replicates per plot may be taken at or near harvest [18] Sample integrity should be maintained throughout the procedure

Sample handling

Take care not to remove surface residues during handling, packing or preparation

Avoid any damage to or deterioration of the sample which might affect residue levels

To provide a representative sample of the raw commodity, adhering soil may have to be removed from some crops, such as root crops This may be done by brushing and, if necessary, gentle rinsing with cold running water (see also this appendix section "Bulb vegetables, root vegetables, tuber vegetables")

Sample control plots before treated plots (see also this appendix sections "Contamination" and "Control samples")

CONTAMINATION

It is vital to avoid any contamination with the pesticide under study or with other chemicals during sampling, transportation or subsequent operations Special attention should, therefore, be paid to the following

- Ensure that sampling tools and bags are clean To avoid contamination use new bags and containers of suitable size and adequate strength The bags or containers should be made of materials which will not interfere with the analysis

Avoid contamination of the sample by hands and clothes which may have been in contact with pesticides

[18] The Study Plan should prescribe when replicate samples are needed Replicate samples should be clearly indicated in the sampling and analytical reports

Do not allow the samples to come into contact with containers or equipment (including vehicles) that have been used for transporting or storing pesticides

Avoid sampling at the plot borders because the residue deposit may not be representative[19]

- Take special care to avoid contamination when commercial mechanical harvesting practices are used (see also this appendix sections "Cereals", "Seeds" and "Herbs and spices tea leaves hops, beer")

Avoid cross-contamination of crop and soil samples

Sampling should proceed from the control to the lowest treatment and so on to the highest treatment

CONTROL SAMPLES

Control samples are in every way as important as samples from test plots The quality of control samples should be similar to that of the test samples, e g maturity of fruit, type of foliage, etc

Always take control samples In decline studies of up to 14 days' duration, control samples from the start and from the end of the study may suffice (see also this appendix section "Sampling in decline studies ")

SAMPLING IN DECLINE STUDIES AND AT NORMAL HARVEST TIME

Representative and valid sampling protocols might be different for decline studies and residue trials at normal harvest time

Sampling in decline studies

The first sampling may take place on the day of application These samples have to be taken immediately after application or, in the case of spray application, immediately after the spray has dried (approx 2 hours)

Take great care to avoid contamination

Take samples so as to be representative of the average size or weight of crop on the plot

Sampling at normal harvest time

Take samples so as to be representative of typical harvesting practice

- Avoid taking diseased or undersized crop parts or commodities at a stage when they would not normally be harvested

Detailed sampling procedures

The following recommendations refer to the sampling of mature crops at normal harvest time, unless otherwise stated The classification of the crops is contained in Section 2 of Codex Alimentarius Volume 2A[20]

[19] The possibility of spray drift or overlap, especially where the plot is small and particularly when various pesticides and dosages are applied to adjacent areas should be considered and avoided when the experimental plots are marked

Fruits and tree nuts

Circle each tree or bush and select fruit from all segments of the tree or plant, high and low, exposed and protected by foliage For small fruits grown in a row, select fruit from both sides, but not within 1 metre of the end of the row

- Select the quantity of the fruit according to its density on the tree or plant, i e take more from the heavily laden parts
- Take both large and small fruits where appropriate, but not so small or damaged that they could not be sold (except when taking immature samples for a residue decline study)

Take samples of fruit juices, cider and wine in a manner reflecting common practice

Table V 1 Sampling of fruits

Commodity	Codex Code No	Quantity, method of collection
Citrus fruits e g orange, lemon, mandarin, pomelo, grapefruit, clementine, tangelo, tangerine	Group 001	12 fruits from several places on 4 individual trees (If this produces a sample weight of less than 2 kg, more fruit should be taken to yield a 2 kg sample)
Pome fruits e g apples, pears, quinces, medlars	Group 002	
Large stone fruit e g apricots, nectarines, peaches, plums	Group 003	
Miscellaneous fruit e g avocados, guavas, mangoes, papayas, pomegranates, persimmons, kiwifruit, litchi	Group 006	
Small stone fruit e g cherries	Group 003	1 kg from several places on 4 trees
Grapes	FB 0269	12 bunches, or parts of 12 bunches, from separate vines to give at least 1 kg
Currants, raspberries and other small berries	Group 004	0 5 kg from 12 separate areas or bushes
Strawberries, Gooseberries	FB 0275, FB 0276 FB 0268	1 kg from 12 separate areas or bushes
Miscellaneous small fruits e g olives, dates, figs	Group 005	1 kg from several places on 4 trees
Pineapples	FI 0353	12 fruits
Bananas	FI 0327	24 fruits Take two fingers each from top, middle and lowest hand of four harvestable bunches
Tree nuts e g walnuts, chestnuts, almonds	Group 022	1 kg
Coconut	TN 0655	12 nuts
Fruit juices, wine, cider	Group 070	1 litre

Vegetables

Bulb vegetables, root vegetables, tuber vegetables

Take samples from all over the plot, excluding 1 metre at the edges of the plot and the ends of the rows The number of sampling points depends on the sample size of the crop (see below)

- To provide a representative sample of the raw commodity, adhering soil may have to be removed This may be done by brushing and, if necessary, gentle rinsing with cold running water

[20] FAO/WHO 1993 Codex Classification of Foods and Animal Feeds in Codex Alimentarius, 2nd ed , Volume 2 Pesticide Residues, Section 2 Joint FAO/WHO Food Standards Programme FAO, Rome

- Trim off tops according to local agricultural practice Details of any trimming should be recorded Where the tops are not used as animal feed (carrots, potatoes) they should be discarded, otherwise (e g turnips, beets) they should be bagged separately

Table V 2 Sampling of bulb, root and tuber vegetables

Commodity	Codex Code No	Quantity, method of collection
Fodder beets, Sugar beets	AM 1051 VR 0596	12 plants
Potatoes	VR 0589	12 tubers (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Other root crops e g carrots, red beet, Jerusalem artichoke, sweet potato, celeriac, turnip, swede, parsnip, horseradish, salsify, chicory, radish, scorzonera	Group 016	12 roots (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Leeks, Bulb onions	VA 0384 VA 0385	12 plants
Spring onions	VA 0389	24 plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)
Garlic, Shallots	VA 0381 VA 0388	12 bulbs from 12 plants (the sample should weigh at least 2 kg - where necessary, take a larger number to produce a 2 kg sample)

Brassica vegetables, leafy vegetables, stalk and stem vegetables, legume vegetables and fruiting vegetables

Take the sample from all parts of the plot, leaving 1 metre at the edges and ends of rows The number of sampling points depends on the sample size of the crop (see below)

Sample items of crops such as peas or beans protected from the spray by foliage and also from parts exposed to the spray

To provide a representative sample of the raw commodity, adhering soil may have to be removed This may be done by brushing and, if necessary, gentle rinsing with cold running water

Do not trim except for the removal of obviously decomposed or withered leaves Details of any trimming should be recorded

The quantities to be taken are shown in Table V 3

Cereals

If the plot is small, cut the whole yield

If the plot is large but mechanical harvesting is not carried out, cut not less than twelve short lengths of row chosen from all over the plot Cut stalks 15 cm above the ground and remove the grain from the straw

- Care should be taken to avoid contamination when mechanical methods are used to separate the parts of the crop The operation is best carried out in the laboratory

If the plots are harvested mechanically, take not less than twelve grab samples of grain and straw from the harvester at uniform intervals over the plot

Do not sample within 1 metre of the edges of the plot

Grasses, forage and animal feed

Cut with shears at normal harvest height (usually 5 cm above the ground) the vegetation from not less than twelve areas uniformly spaced over the entire plot, leaving 1 metre at the edges of the plot

Record height of cutting and avoid soil contamination

- Crops which are harvested mechanically can be sampled from the harvester as it proceeds through the crop

The quantities to be taken are shown in Table V 5

Sugar cane (GS 0659)

Select whole canes from 12 areas of the plot and take short (e g 20 cm) sections from all parts of the length of the canes Care is necessary owing to the rapid changes which normally occur in cane juices If required, 1 litre samples of juice should be taken and frozen immediately and then shipped in cans

Table V 3 Sampling of other vegetables

Commodity	Codex Code No	Quantity, method of collection
Large Brassica crops e g cabbage, cauliflower, kohlrabi	Group 010	12 plants
Broccoli	VB 0400	1 kg from 12 plants
Brussels sprouts	VB 0402	1 kg from 12 plants Buttons to be taken from at least two levels on each plant
Cucumbers	VC 0424	12 fruits from 12 separate plants
Gherkins, courgettes, squash	Group p 011	12 fruits from 12 plants (the sample should weigh at least 2 kg - where necessary take a larger number of fruit to produce a 2 kg sample)
Melons, gourds, pumpkins, watermelons[21]	Group 011	12 fruits from 12 separate plants
Egg plants (aubergines)	VO 0440	12 fruits from 12 separate plants
Sweet corn	VO 0447	12 ears (the sample should weigh at least 2 kg - where necessary take a larger number of items to produce a 2 kg sample)
Mushrooms	VO 0450	12 items (the sample should weigh at least 0 5 kg - where necessary take a larger number of items to produce a 0 5 kg sample)
Tomatoes, Peppers	VO 0448 VO 0051	24 fruits from small-fruiting varieties, 12 from large fruiting varieties From 12 plants in all cases (The sample should weigh a minimum of 2 kg - where necessary take a larger number of items to produce a 2 kg sample)
Endive[a]	VL 0476	12 plants
Lettuce[a]	VL 0482, VL 0483	12 plants
Spinach[a], Chicory leaves[a]	VL 0502 VL 0469	1 kg from 12 plants
Kale	VL 0480	2 kg from 12 plants sampled from two levels on the plant
Small-leaf salad crops e g cress, dandelion, corn salad	Group 013	0 5 kg from 12 plants (or sites in plot)
Peas, Phaseolus beans e g French, kidney,	Group 014	1 kg (fresh green or dry seed as appropriate)

[21] In case of large crops, a sample consisting of 12 units could be 50-100 kg or more In such cases the sample size may be reduced to 5 units which conforms with the sample size recommended by the CCPR for enforcement

Commodity	Codex Code No	Quantity, method of collection
runner		
Pulses e g dried broad beans, field beans, lentils, soya beans	Group 015	1 kg
Celery	VS 0624	12 plants
Asparagus, Rhubarb	VS 0621 VS 0627	12 sticks from 12 separate plants (The sample should weigh a minimum of 2 kg where necessary take a larger number of sticks to produce a 2 kg sample)
Globe artichoke	VS 0620	12 heads
Fodder crops	Groups 050, 051, 052	2 kg from 12 separate areas of plot (Crops harvested mechanically can be sampled from the harvester as it proceeds through the crop)
Oilseed e g rape seed, mustard seed, poppy seed	Group 023	

Note (a) also at immature stages during decline studies

Table V 4 Sampling of cereals

Commodity	Codex Code No	Quantity, method of collection
Cereal grains e g wheat, barley, oats, rye, triticale and other small grain cereals, maize (off the cob), rice, sorghum	Group 020	1 kg
Straw of the above crops	Group 051	0 5 kg
Maize straw, fodder and forage (mature plants excluding cobs)	AF 0645 (forage) AS 0645 (fodder)	12 plants (Cut each stem into three equal lengths (with leaves attached) Take top portion from stems 1 to 4, middle portion from stems 5 to 8 and bottom portion from stems 9 to 12, thus ensuring that parts of all 12 stems are included in the sample)
Green or silage maize	Group 051	12 plants (Cut each stem and subsample as in previous item, retaining any cobs present on the appropriate portions of stem)
Maize cobs	Group 051	12 ears (The sample should weigh at least 2 kg - where necessary, take a larger number of ears to produce a 2 kg sample)

Table V 5 Sampling of forage crops and animal feed

Commodity	Codex Code No	Quantity, method of collection
Green forage or silage crops of alfalfa, clover, pea and bean forage, vetch, sainfoin, lotus, soya bean fodder and forage, rye forage, fodder cereals, sorghum forage	Group 050, 051	1 kg
Dry hay of the above crops	Group 050, 051	0 5 kg

Seeds

Use essentially the same technique as for cereals, taking samples of mature seed from at least twelve parts of the plot Where the sample is harvested by hand, seed should normally be sent to the laboratory in the pod Where mechanical harvesting is used, only the seed should normally be supplied

- Cotton seed (Codex Code No SO 0691)

Pick the cotton at the normal stage of harvesting Take 1 kg, with or without fibre

- Peanuts (Codex Code No SO 0697)

Collect at the normal stage of harvesting Take 1 kg

- Sesame seed, rape seed (Codex Code Nos SO 0700, SO 0495)

· Collect the pods when they have reached the stage of maturity at which they are normally harvested Take 0 5 kg

- Sunflower seed, safflower seed (Codex Code Nos SO 0702, SO 0699)

· Where the sampling is done by hand select ripe heads Where it is done mechanically submit the seed to the laboratory Take 12 heads or 1 kg of seed

- Coffee and cacao beans (Codex Code Nos SB 0716, SB 0715)

Take samples in a manner reflecting common practice, quantity 1 kg - The freshly harvested produce is not normally required

Herbs and spices, tea leaves, hops, beer

Take samples in a manner reflecting common practice

The freshly harvested produce is not normally required for tea although herbs, such as parsley and chives, should be sampled fresh In the case of hops, both fresh and dried cones should be supplied

Table V 6 Sampling of herbs and spices, tea leaves, hops and beer

Commodity	Codex Code No	Quantity, method of collection
Garden herbs and medicinal plants e g parsley, thyme	Group 027 Group 028 Group 057	0 5 kg fresh 0 2 kg dry
Teas (dry leaves)	Group 066	0 2 kg
Hops (dry cones)	DH 1100	0 5 kg
Beer		1 litre

SAMPLING PROCESSED COMMODITIES

Where a commodity is normally processed between harvest and marketing, for example by milling, pressing, fermentation, drying or extraction, data may be required on the processed crop or its products Details of the processing method should be supplied with the samples together with storage and handling histories In such cases, the trials should be designed to provide samples with appropriate residue levels so that the fate of residues can be studied during the processing Sample separately any cleanings, husks or by-products which could be used for animal feed

SAMPLING STORED COMMODITIES

Supervised trials of post-harvest treatments of stored products should be carried out over a wide range of storage facilities, and the sampling technique must be carefully chosen if valid samples are to be obtained Procedures for taking valid samples from most commodities in storage units are well established Such procedures are acceptable in sampling for pesticide residue analysis and may be used if adequate references are given

The sampling procedures are usually designed for three kinds of storage conditions

Sampling from bulk

Obtaining a representative sample from a (large) bulk container (e g of cereal grains) is difficult if possible, samples should be taken at frequent intervals from the stream during transfer into another container A probe sample is not representative but may be acceptable if

it is possible to reach every part of the storage container, and

- a larger number of individual samples are taken before mixing and reducing to produce a final sample

Pesticide residues are normally higher in the dust fraction and this should be recognised in the sampling procedure

Sampling bagged commodities

Sampling of the commodity within a bag must be random A representative sample from a large stack of bags can be obtained only if every bag is accessible This is not always possible in practice and the alternative is to obtain a sample from a number of randomly chosen bags by probing Since pesticide treatments are often directed to the surface of the bag, selective sampling to show the effect of the position of the bag in the stack and the penetration of the pesticide into the bag may be necessary

Sampling fruit and vegetables in packing houses

Where post-harvest treatments are applied to fruit and vegetables in packing houses, an adequate number of samples must be taken to determine the range of residue levels resulting from variations in the treatment process The effects on residue levels of concentration, temperature, duration of treatment, drying (after dip treatments) and subsequent handling may need to be considered

Post-harvest treated fruit and vegetables should be kept in, or packed in, commercial containers or punnets and stored at ambient or cool-room temperature according to normal commercial practice Samples should then be drawn for analysis from the commercial containers at suitable intervals representing the time expected between treatment and subsequent marketing The rate of disappearance or degradation of some residues depends on whether the commodity is held in a sealed or partly sealed container or is open to the air

The sizes of samples are the same as shown in Tables V 1 - V 3

SAMPLE SIZE REDUCTION

Large samples cannot be handled economically, especially if freezing and long transport are involved Take only that amount prescribed in the Study Plan

Except for cereal grains sampled on the conveyor belt or from the stream of material transferred from one large container to another, mixing of samples and sample size reduction at the field site is not recommended and should be avoided

SAMPLE PACKING AND STORAGE

Once packed and labelled, samples may be stored or immediately sent to the residue laboratory according to the nature of the sample, the stability of the residue and the kind of study undertaken

It is important that packing and shipment are carried out in such a way that the samples arrive as soon as possible (normally within 24-36 hours) after being taken and without change of any kind, e g deterioration, physical damage, contamination, loss of residue, or change in moisture content

Storage and shipping should always be under deep-frozen conditions

Packing

Containers

Individual samples should be placed in suitable containers, e g heavy polyethylene bags, and then put inside additional heavy paper bags and, where necessary, frozen or refrigerated as soon as possible after sampling according to the nature of the chemical involved Polyethylene bags alone may become brittle in contact with dry ice and therefore there is a risk of breakage and subsequent loss of the sample

Avoid other plastic containers, or plastic-lined caps, unless made of "Teflon" or other inert plastic which does not interfere with the analytical method, laboratories have frequently experienced such interference, and PVC bags should be avoided If cans are used, they should first be checked to demonstrate the absence of materials such as oil films, lacquers or resin from soldered joints that could interfere with analyses

Glass containers should be used for liquid samples and should be thoroughly cleaned and rinsed with one or more suitable pesticide-free solvents such as acetone, isopropyl alcohol or hexane, and dried before use Pesticides can migrate to the walls of a container and be adsorbed, hence even a glass container, after the sample is poured out, should be rinsed with solvent if the extraction is not made in the container itself

In summary, any type of container or wrapping material should be checked before use for possible interference with the analytical method and at the limit of determination of the analysis

Fasten boxes securely with strong twine, rope or tape

Shipment of samples

Non-perishable commodities containing residues that are known to be stable over the period required to reach the laboratory can be shipped in a non-frozen state, but samples should be protected against any effects which might cause degradation or contamination

Where samples need to be frozen, use shipping containers of polystyrene foam, if available, as they are excellent for this purpose If not available, use two cardboard boxes of slightly different size with insulation between Proper insulation is essential to ensure samples arrive at the residue laboratory still frozen Sufficient dry ice must be used for some to remain when samples are received at the residue laboratory This usually requires a minimum of one kg of dry ice per kg of sample For journeys lasting more than two days, two kg of dry ice or more

per kg of sample may be required Poorly insulated containers require more dry ice Use caution in handling dry ice (gloves and ventilated work area) Packages must of course comply with transport regulations

Frozen samples must never be allowed to thaw, either before or during shipment They must be shipped under conditions that permit their arrival at the residue laboratory still solidly frozen

Advise the consignee by FAX or e-mail of the full details of shipment of samples, including shipping document numbers and flight numbers, so that delay in delivery to the laboratory is avoided

When samples have to be shipped across national boundaries, quarantine regulations must be observed and appropriate permits obtained well in advance of dispatching samples

Labels and records

Label each sample with the appropriate sample identification The label and ink should be such that the writing will not be illegible if the label becomes wet Attach the label securely so that it cannot come loose during shipment, and place the label so that it will not become wet from condensation

Complete the Sampling Report (residue data sheets) clearly and accurately with all the requested trial details Failure to do so may mean that data will not be acceptable The completed sheets should be protected by enclosing them in protective polythene bags which should be sent with the sample Duplicate sheets should be kept by the sender

Use a label on the outside of the shipping container stating the following "Perishable Goods Deliver immediately upon arrival" and 'This material is not fit for human consumption"

Sample reception and handling

Immediately upon arrival of the samples, the residue laboratory personnel should

- Verify that the copy of the Sampling Report is included with the samples
- Check and report on the condition of the samples
- Check to see that the samples match the details of the Sampling Report
- Check the Sampling Report for accuracy (especially the rate and interval data) and verify that the information is complete
- Check the Sampling Report to determine whether any special treatment or testing is indicated

If there are any deviations of any consequence, or the Sampling Report is not received or is incomplete (in such a way that a proper comparison is not possible), the samples should be stored in the simplest form that will preserve the residue and the crop The trial organizer should then be contacted immediately to determine how to proceed

Note it is dangerous to put packages containing dry ice into deep freeze

Storage

Samples should be analysed as quickly as possible after collection before physical and chemical changes occur If prolonged storage is unavoidable, it is usually preferable to store the samples at a low temperature, preferably at or below -20°C This removes the residue from contact with enzymes which might degrade the pesticide and also prevents further possibility of residues being "bound" in the tissue Do not store samples (whole or homogenized) for analysis unless an adequate check has been made on the stability of the residue Fumigant residue samples need special attention and ideally should be analysed immediately on receipt at the laboratory Storage at -20°C is likely to be inadequate to prevent loss of fumigant residues

Studies of the stability of residues in samples, over the time and at the temperature of storage, should be carried out with representative pesticides and substrates When there is doubt about the stability of residues in storage, spiked control samples should be held under the same conditions as the samples or extracts

Light degrades many pesticides, it is therefore advisable to protect the sample and any solutions or extracts from needless exposure Samples other than water should ordinarily be stored in a freezer, preferably at -20°C or below Even then, physical and chemical changes may occur either in the sample or in the residues sought Extended storage in freezers can cause moisture to migrate to the surface of the sample then to the freezer coils, slowly desiccating the sample This effect may be of importance if water content affects the subsequent analysis and can affect the calculated residue concentration Water samples should be stored slightly above freezing to avoid rupture of the container as a result of freezing

Appendix VI

PORTION OF COMMODITIES TO WHICH CODEX MAXIMUM RESIDUE LIMITS APPLY AND WHICH IS ANALYSED

INTRODUCTION

Codex Maximum Residue Limits are in most cases stated in terms of a specific whole raw agricultural commodity as it moves in international trade In some instances, a qualification is included that describes the part of the raw agricultural commodity to which the maximum residue limit applies, for example, almonds on a shell-free basis and beans without pods In other instances, such qualifications are not provided Therefore, unless otherwise specified, the portion of the raw agricultural commodity to which the MRL applies and which is to be prepared as the analytical sample for the determination of pesticide residues is as described in the following table

Classification of commodities	Portion of commodity to which the Codex MRL applies (and which is analysed)
Group 1 - ROOT AND TUBER VEGETABLES (Codex Classification[22] Group 016 Root and tuber vegetables)	
Root and tuber vegetables are starchy foods derived from the enlarged solid roots, tubers, corms or rhizomes, mostly subterranean, of various species of plants The entire vegetable may be consumed	
Root and tuber vegetables beets, carrots, celeriac, parsnips, potatoes, radishes, rutabagas, sugar beet, sweet potatoes, turnips, yams	Whole commodity after removing tops Wash the roots or tubers in cold running water, brushing gently with a soft brush to remove loose soil and debris, if necessary, and then dab lightly with clean tissue paper to dry For carrots, after drying, the tops are carefully cut off with a knife by cutting through the bottom of the stem at the lowest point of attachment of the outer petioles If an annulus of root tissue is thereby severed from hollow-crown roots, the material should be re-combined with the roots
Group 2 - BULB VEGETABLES (Codex Classification Group 009 Bulb vegetables)	

[22] The number and categories of groups for portion of commodities do not always correspond to the grouping used by the current Codex Classification of Foods and Animal Feeds The corresponding groups are given in brackets