INSTRUCTIONS FOR WRITING CIPAC METHODS

Introduction

CIPAC methods are intended to be used by many people whose native language is not English. Writers of methods should keep this in mind and should use straightforward wording and give clear concise, but sufficiently detailed and unambiguous description of the procedures to be carried out.

General instruction

CIPAC methods should be written in the imperative mode except for the section OUTLINE OF METOD, which should be written in the passive voice.
Use no abbreviations unless they are internationally accepted, or are fully explained when used for the first time.

Use of notes

Avoid the use of notes; they will distract the attention of the reader. Incorporate their content in the text or use a special section or sub-section. Footnotes should not be used; the only exception is the footnote giving the status of the CIPAC method, with the year of acceptance and the committee that prepared the method.

Units, symbols and nomenclature

Use SI units, symbols and prefixes e.g. mass instead of weight, Pa instead of mm Hg or Torr, mg/kg (not µg/g) instead of ppm. Express quantity concentrations (amount-of-substance concentra-tions) in the following way (IUPAC rules):
c(NaOH) = 1 mol/l (previously 1 N),
c(1/2 H_2SO_4) = 2 mol/l (previously 2 N),
c(1/5 $KMnO_4$) = 0.1 mol/l (previously 0.1 N)

Express the concentration of other solutions as:
% (m/m), % (v/v), or g/l (or sub-multiples thereof).
When a solution is prepared by dilution of another solution observe the ISO convention, i.e.
$v_1 \rightarrow v_2$ means that solution v_1 is diluted to give a final volume of v_2
$v_1 + v_2$ means that volume v_1 of the specified solution is added to volume v_2 of the solvent.
Do not use $v_1 : v_2$, or v_1/v_2.
Use the ISO common names of pesticides. Chemicals should be named according to the IUPAC rules for nomenclature. In the descriptive section of the active ingredient the name according to Chemical Abstracts should be added. Do not use empirical formulae in the text of the method. Long names may be abbreviated or substituted by trivial names. In that case the abbreviation or the trivial name should be explained in the reagent section.

Format

The best way to get familiar with the CIPAC format is to take an example from one of the latest volumes of the CIPAC Handbook. Keep in mind that CIPAC methods are analytical methods and not specifications.

Coding of methods and cross reference

Methods should be identified by the CIPAC coding system, which makes use of the GCPF two-letter codes for formulations. So the code for ampropylofos technical is: **500**/TC/M/-

In principle each formulation type should have its own method. If there are no differences between the methods for the respective formulations it is sufficient to refer to the basic method. In other cases where the differences are partial or small, shorten the text by using sentences like:

As for **500**/TC/3.c except:……..

As for **500**/TC/3.c except substitute……..for…….

As for **500**/TC/3.c together with:……

Proceed as for **500**/TC/3.c beginning at "……...

Continue as for **500**/TC/3.c from "……..."

The order of the methods for the different formulations should be: technical, technical concentrates, wettable powders, water dispersible granules, emulsion concentrates, suspension concentrates, solutions, granules, dustable powders and further in decreasing order of the usual concentration, whereby similar formulations should be grouped together.

Lay-out and arrangement of the sections

A short descriptive section that gives information about the active ingredient concerned should precede new compounds for which no method has been published before. This section should include things like the structural formula, the chemical name(s), CAS no, empirical formula, rel. molecular mass, together with the most important physical constants such as: m.p., b.p., vapour pressure, solubility in water and in the most important solvents. Some chemical characteristics like hydrolysis and stability should be added as well. Complete the list with a description of the appearance of the pure and/or technical material and specify which formulations are available.

Methods will among others have the following sections in the order given, but always use a continuous numbering:

1 Sampling. In this section the amount of material should be specified. If special precautions should be taken e.g. to obtain homogeneity of the sample, they should be mentioned in this section.

2 Identity tests. At least two identity tests, based on different analytical techniques, are required, one of which may be the check of the (relative) retention time(s) of the chromatographic method used for quantification. If necessary refer to example chromatograms or spectra. For spectroscopic techniques its is necessary to separate the active ingredient from other components of the formulation. Use standard wording wherever possible (See annex).

3 Active ingredient. In this section a accurate description of the procedure must be given together with a list of the materials and apparatus needed. Use the following sub-headings:

OUTLINE OF METHOD Give the analytical principles of the method, important conditions (e.g. the wavelength at which the absorption is determined, the use of internal or external standardisation) and a short description of the most important manipulations. Mention chemicals that play a key-role in the procedure, avoiding at the same time to present too many details. Use the passive voice.

SCOPE This subsection can usually be omitted, because the scope is sufficiently clear from the code above each method. Use this section only if there are other limitations to the applicability of the method.

REAGENTS Give a list of the reagents required in the method. It is understood that all chemicals are analytical grade unless otherwise specified. Refer to the respective RE sections (CIPAC Handbook E) whenever possible. Use the ISO notation to indicate the strength of standard solutions (see above). Use IUPAC names for all chemicals. Give here the abbreviations to be used for long names in the rest of the method.
To this section also belong the preparation of the internal standard solution and the calibration solution. In CIPAC methods "internal standard solution" denotes the solution that solely contains the internal standard, whereas the "calibration solution" means the solution containing the internal standard plus the pure standard compound (or in cases of external standardisation the pure standard compound only).

APPARATUS In this section a list of the most important apparatus is given. Describe apparatus in terms of performance rather than mentioning brands and manufacturers. There is no need to mention the usual laboratory equipment like balances, glassware (beakers, Erlenmeyer flasks pipettes, burettes and volumetric flasks) etc.

PROCEDURE Write this section in the imperative mode. Use standard wording for the weighing procedure and indicate the required accuracy (see annex). Express quantities in the following way:
"Dissolve in dichloromethane (40 ml), add by pipette internal standard solution (25.0 ml), and dilute to volume"
Examples of headings for sub-sections are:
(a) Operating conditions
Specify operating conditions such as: kind of column, column dimensions, column-, injection port-, and detector temperatures, injection volume, flow rates, approximate retention times, required number of theoretical plates, split- or splitless injection, mobile phase composition, wavelength settings.
(b) Preparation of calibration curve
(c) Linearity check
(d) System suitability check
(e) Calibration
(f) Preparation of sample
(g) Determination
(h) Calculation. For the sake of uniformity use, where possible, standard symbols in the formulae to calculate the content of the active ingredient (see annex). Keep the final calculation formula as simple as possible. If e.g. the dilutions of the calibration solution and the sample solution are the same, omit any dilution factors. If the volumes of the internal standard solution added to the calibration and the sample solutions are equal (and thus $r = q$, see annex), r can be omitted from the formula for the response factor and q from the formula for the calculation of the content. Express the content always in units of g/kg; mg/kg may be used if too many decimal places are needed.
Close this section with the repeatability and reproducibility figures that were calculated in the study with the method.
Repeatability r = ….g/kg at …g/kg active ingredient content
Reproducibility R = ….g/kg at …g/kg active ingredient content

4 Impurities. Add methods for impurities if requested, e.g. by FAO specifications.

5 Determination of the active ingredient after a physical test. If after the application of a physical test (e.g. suspensibility, sieve test) the concentration or the amount of the active

ingredient has to be determined in the processed formulation or in a fraction thereof, the method to be used should be mentioned, either by referring to the method for that particular formulation, or by giving a modified method. In particular in the case of suspensions the regular method may not be directly applicable. In other cases the method may have to be adapted to a different concentration level. Do not repeat the physical test itself, but refer to the MT methods concerned.

Typing

This section gives instructions for the final typescript only. Type the text in 14 pt Times New Roman font. Use single spacing between the lines, leave a margin of 3.5 cm on the left-hand side and fill out the text. The centred heading will be in bold type capitals. Other headings will be bold type or in italics as shown in this document. Use indentation sparingly. Start a new paragraph only when it follows logically from the text.

Present the final typescript on a 3.5" diskette preferably in Word 97 for Windows. Add two hard copies on A4 size paper.

Graphs and line drawings

Add drawings and calibration curves only in those cases where they are indispensable or when a description becomes too lengthy or complicated. Infrared spectra are welcomed as long as they are the spectra of the active ingredient and not of a particular formulation. Simple chromatograms with well-defined and completely resolved peaks usually give little additional information and can easily be replaced by a short list of retention times together with chromatographic parameters like number of theoretical plates, peak separation, etc. Give only chromatograms in complex situations, in cases with deviating peak shapes and when details are important.

Chromatograms and spectra should be submitted as a file that can be embedded into a WORD document. The software of modern analytical instruments provides the tools for converting the chromatograms or spectra to formats for inserting the graphs into WORD documents, e.g. the Report Manager from Perkin Elmer. Graphs produced by a scanner are in most cases of poor quality and - if scanned at a high resolution - the files produced are often too large to be stored on a 1.4 MB diskette.

Annex

1. Examples of standard wording for Identity tests

GLC (HPLC). Use the GLC (HPLC) method below. The relative retention time of …(*a.i.*)…. with respect to the internal standard for the sample solution should not deviate by more than .. % from that of the calibration solution.

or:

Use the GLC (HPLC) method below. The difference between the retention time of …(*a.i.*)…. and of the internal standard for the sample solution should not deviate by more than .. % (should be less than ..s) from that of the calibration solution.

or in case of external standardisation:

Use the GLC (HPLC) method below. The retention time of …(*a.i.*)…. for the sample solution should not deviate by more than .. % from that of the calibration solution.

or:

Use the GLC (HPLC) method below. The difference between the retention time of …(*a.i.*)…. for the sample solution and that of the calibration solution should be less than .s.

Infrared. Prepare potassium bromide discs from the sample and from …(*a.i.*)…. standard using approximately *x* mg material an *X* mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard.

2. Example of standard wording to be used in *Preparation of sample*

Weigh (to the nearest 0.1 mg) into a volumetric/Erlenmeyer/round-bottomed flask (*xx* ml) sufficient sample to contain about *x* mg (*or x* ± …mg) …(*a.i.*)…. (*w* mg).

3. Standard symbols to be used in the calculation formulae

t	= ml required for the sample determination
b	= ml required for the blank determination
a	= ml required for the back titration
N	= normality of the standard solution
A	= absorbance of the sample solution
$A¢$	= absorbance of the calibration solution
A_o	= absorbance of the background
f_i	= single response factor, i = 1,…
f	= average response factor
H_s	= area (height) of the….(*a.i.*)….peak in the calibration solution
H_w	= area (height) of the….(*a.i.*)….peak in the sample solution
I_r	= area (height) of internal standard peak in the calibration solution
I_q	= area (height) of internal standard peak in the sample solution
s	= mass of ….(*a.i*)…. in the calibration solution (mg)
w	= mass of sample taken (mg)
r	= mass of internal standard in the calibration solution (mg)
q	= mass of internal standard in the calibration solution (mg)
P	= purity of …(*a.i.*)…. reference substance (g/kg)
R	= …(*a.i.*)…. to internal standard peak area (height) ratio for the sample solution
$R¢$	= …(*a.i.*)…. to internal standard peak area (height) ratio for the calibration solution
Vi	= dilution factor(s), i = 1,…