Laboratory challenges in the quality control of public health pesticides in the framework of international tenders

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CIPAC Symposium, Liège, June 24, 2014

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Quality Control of pesticide formulations

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Importance to control the quality of pesticides

Poor-quality pesticides :

- are unlikely to serve the intended purpose;
- are likely to provide poor value to users;
- are likely to be more harmful, directly or indirectly, to humans and the environment.

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Importance to control the quality of pesticides

Examples of adverse effects of poor-quality pesticides :

- An excessive level of a hazardous impurity increases risks of adverse effects on users and/or the environment;
- Insoluble particulates present in products intended for spray application may block nozzles and/or filters;
- A poor suspensibility of dispersions may produce uneven distribution of active ingredient in the spray tank and uneven application;
- Granular formulations that are too fragile may produce respirable dust when handled and applied;

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Importance to control the quality of pesticides

Examples of adverse effects of poor-quality pesticides :

- Poor retention/ migration of insecticide through successive washes in a long-lasting insecticidal net (LN) leads to reduced personal protection of the user;
- If poor quality leads to poor efficacy, users may increase doses rates or the number of applications and unknowingly increase other risks…

→ Any of the above consequences will usually have a negative impact on the marketability of a pesticide product and its registration could be withdrawn or restricted.

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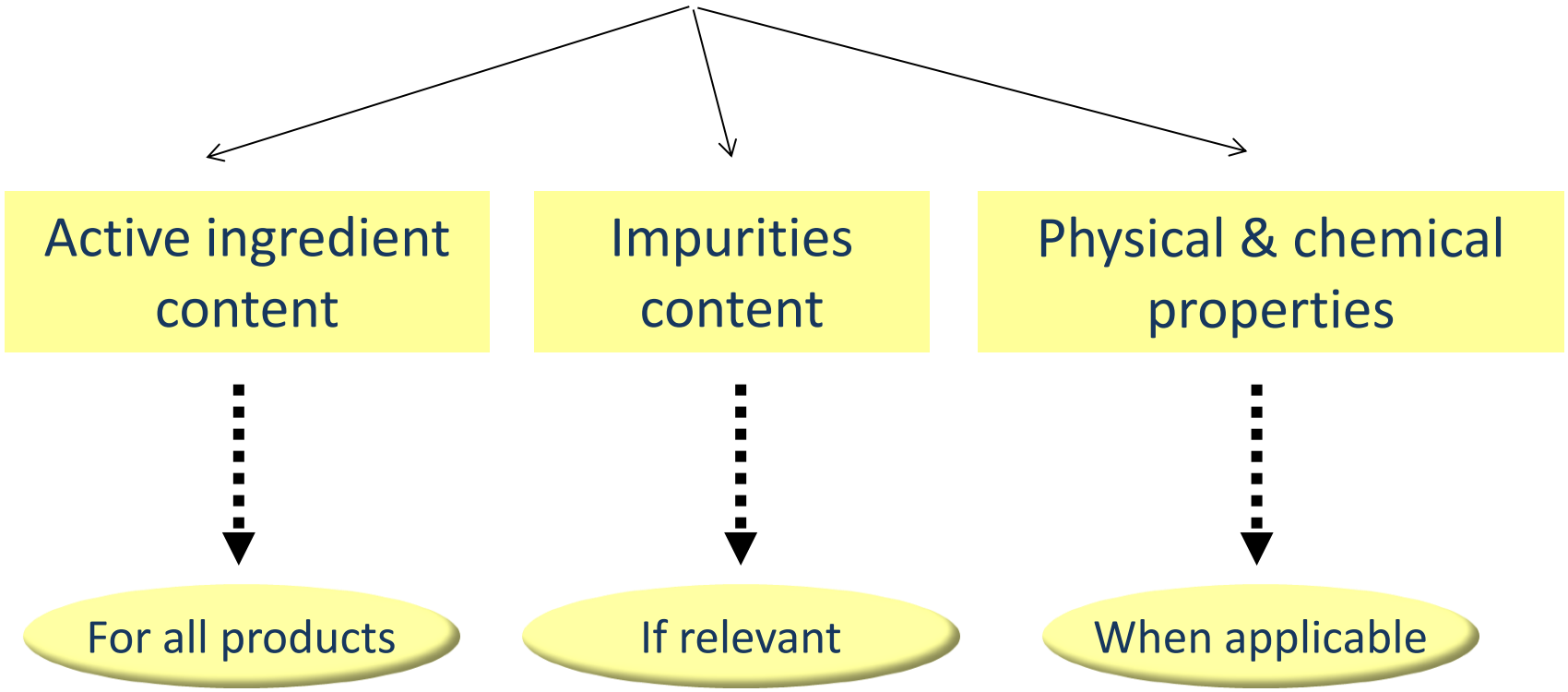
What does pesticide quality control involves ?



According to WHO specifications

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Quality Control of pesticide formulations

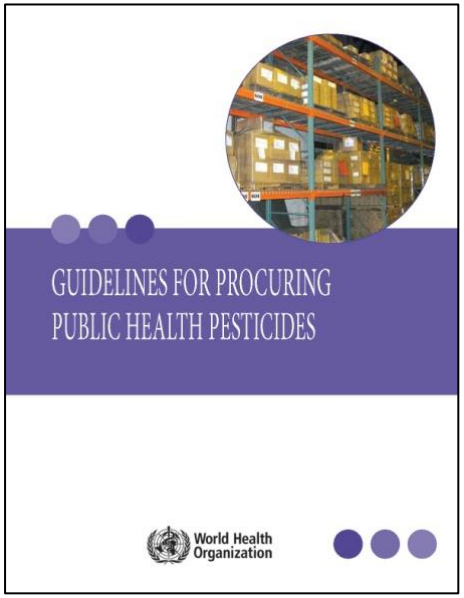
References and guidelines

- WHO specifications for pesticides
- Manual on development and use of FAO and WHO specifications for pesticides
- Specifications for pesticides : a training manual
- Quality control of pesticides products : Guidelines for national laboratories

Quality Control of pesticide formulations

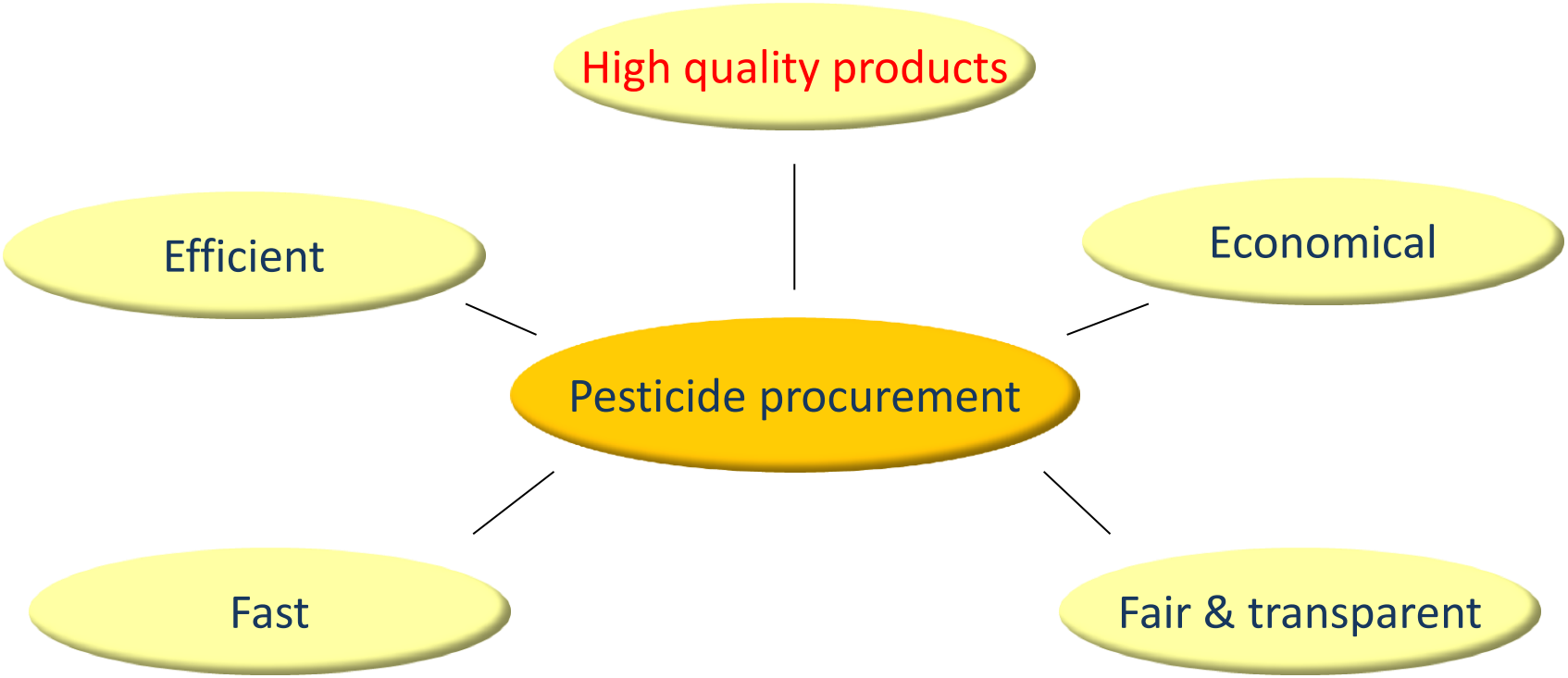
References and guidelines

- Guidelines for procuring public health pesticides



Pesticide procurement

Pesticide procurement is a highly specialized and complex subject



Pesticide procurement

Pesticide procurement is a highly specialized and complex subject

Public health pesticides ≠ agricultural pesticides

Pesticide procurement

Large quantities of public health pesticides are procured annually

During the period 2000-2009 :

Organochlorines :
4429 tonnes

Organophosphates:
1375 tonnes

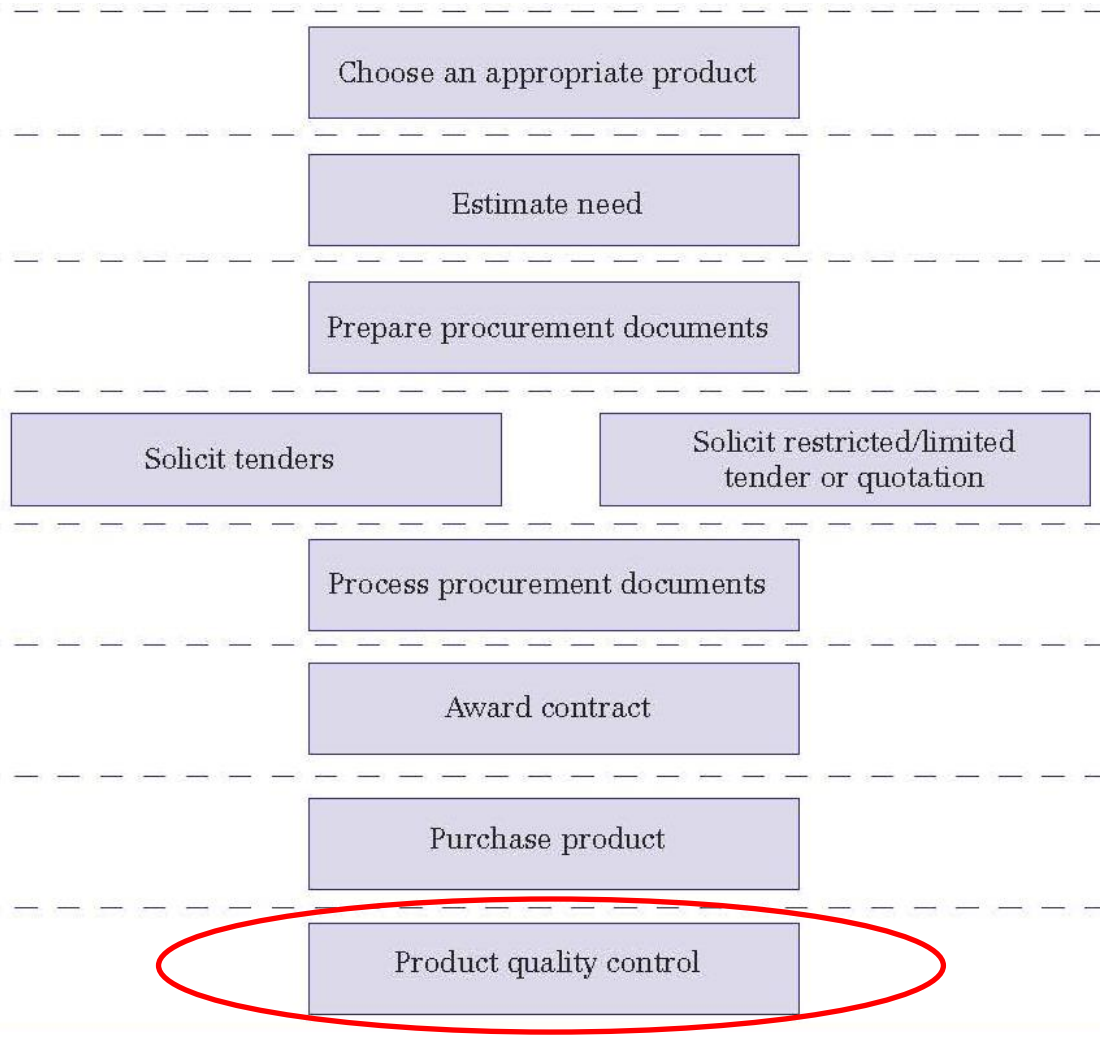
Carbamates :
30 tonnes

Pyrethroïds :
414 tonnes

By the end of 2010, approximately 298 million LNs were delivered to the African Region for malaria prevention

Pesticide procurement

Stages in procurement of public health pesticides



Source : WHO, Guidelines for procuring public health pesticides

Contribution of CRA-W to the Quality Control of pesticide formulations

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Plant Protection Products and Biocides
Physico-chemistry and Residues Unit (U10)

WHO Collaborating Center for
Quality Control of Pesticides

- ✓ has a long experience in pesticides physico-chemistry and residues ;
- ✓ gives support to WHO, FAO, CIPAC, ESPAC, GF, UNDP, PFSCM …

GLP Certified

ISO 17025 Accredited

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Laboratory challenges

Logistics challenges

- Large series of samples to analyze (up to 70 samples);
- Several series of samples to analyze at the same time (in spraying season);
- Difficulties to plan the testing : the arrival date of samples is unknown or vague;
- Incomplete information about the testing :
  - How many samples have to be analyzed?
  - Is the storage stability testing necessary?
  - What is/are the use rate(s)?
  - Will the product be packed in soluble bags? ...

Laboratory challenges

Logistics challenges

- Shipments are sometimes poorly made
  - leak of product out of the package, hazardous for handlers;
  - package retained by the customs;
  - package improperly labelled;
  - lack of information about the supplier …

- Most (≈ all) of the testing is extremely urgent

- In quality control, all the tests listed in the related WHO specification have to be performed (except in some cases the tests after accelerated storage).

Laboratory challenges

Logistics challenges

Example : WHO specification 333/WG
for deltamethrin water dispersible granules

1 **Description**
2 **Active ingredient**
- 2.1 Identity tests
- 2.2 Deltamethrin content

3 **Relevant impurities**
4 **Physical properties**
- 4.1 Acidity
- 4.2 Wettability
- 4.3 Wet sieve test
- 4.4 Degree of dispersion
- 4.5 Suspensibility
- 4.6 Persistent foam
- 4.7 Dustiness
- 4.8 Flowability

5 **Storage stability**
5.1 **Stability at elevated temperature**
- – deltametrhin content
- – acidity
- – wet sieve test
- – degree of dispersion
- – suspensibility
- – dustiness

Laboratory challenges

Logistics challenges

Example : WHO specification 463/CS
for lambda-cyhalothrin slow-release capsules suspension

1 **Description**
2 **Active ingredient**
- 2.1 Identity tests
- 2.2 Total lambda-cyhalothrin content
- 2.3 "Free" ("non-encapsulated") lambda-cyhalothrin content
- 2.4 Release of lambda-cyhalothrin

3 **Physical properties**
- 3.1 pH range
- 3.2 Pourability
- 3.3 Spontaneity of dispersion
- 3.4 Suspensibility
- 3.5 Wet sieve test
- 3.6 Persistent foam

4 **Storage stability**
4.1 **Freeze/thaw stability**
- pH range
- pourability
- spontaneity of dispersion
- suspensibility
- wet sieve test

4.2 **Stability at elevated temperature**
- “free” lambda-cyhalothrin content
- release of lambda-cyhalothrin
- pH range
- pourability
- spontaneity of dispersion
- suspensibility
- wet sieve test

Laboratory challenges

Scientific challenges

The CIPAC methods mentioned in the WHO specification for physico-chemical tests have to be strictly observed. But…

Some information lacks sometimes in the method/specification.

Example :

WHO specification 454/SC for alpha-cypermethrin SC : test of pourability

- Temperature : Value by default? Provided by the manufacturer?

Laboratory challenges

Scientific challenges

The CIPAC methods referred in the WHO specification for physico-chemical tests have to be strictly observed. But…

For suspensibility test, the analysis of the remaining 1/10th are only « draft methods ».

Example :

Determination of the remaining 1/10th for deltamethrin WG and WP

- We have developed at CRA-W a simpler and more robust method.

  ! We had to verify and validate our method !

Laboratory challenges

Scientific challenges

Some methods referred in the WHO specification for impurities were developed by a manufacturer for its product, but…

they need be optimized for other products, containing other adjuvants.

Example :

Determination of *iso*-temephos and temephos-oxon content in temephos EC and GR

- Some peaks were not correctly separated.

  The method had to be optimized and validated.

Laboratory challenges

Scientific challenges

The CIPAC methods for active ingredient content suggest « *typical* » chromatographic conditions.

These conditions have to be optimized with respect to the apparatus used or the product analyzed.

Example :

Active ingredient content in pirimiphos-methyl EC

- The injection temperature was adapted.

Laboratory challenges

Scientific challenges

The application of CIPAC methods requires to have some « scientific common sense »!

Example :

Determination of active ingredient content in lambda-cyhalothrin WP and in deltamethrin incorporated LN

- We observed an interference with the peak of the internal standard mentioned in the CIPAC method.

« Carry out injections of internal standard and sample without internal standard and check whether there are any interfering peaks from impurities ».

Laboratory challenges

Scientific challenges

When is a product at the limit of the requirement of the WHO specification for one test…

The clauses of the WHO specifications

- include the analytical uncertainty;
- were established on basis of the data submitted by the manufacturers.

Laboratory challenges

Scientific challenges

Lack of WHO specification for products packaged in water soluble bags.

- WHO specification 463/WP for lambda-cyhalothrin WP packaged in water soluble bags;
- WHO specification 333/WG-SB for deltamethrin WG-SB, published in May 2014;
- WHO specification 232/WP-SB for bendiocarb WP-SB, published in June 2014.

Laboratory challenges

Scientific challenges

Methods and specifications evolve

Example :

Persistent foam : the CIPAC MT 47.2 became the CIPAC MT 47.3

Wash resistance index : the CIPAC MT 195 replaces the WHO washing method

- The laboratory has to be flexible and must be aware of these changes. The staff has to be continuously trained.

Laboratory challenges

“Consulting” challenges

Our job is not only to perform quality control testing

- We justify and interprete the results obtained
- We provide support to the procurement agencies

! Our role is to give advices to the procurement agency but not to take any final decision, especially on the use or not of a product !

Laboratory challenges

“Consulting” challenges

Our job is not only to perform quality control testing

- We give support to the manufacturer when a product is not in conformity with the WHO specification or in case of divergence with the results obtained

In any case, our ultimate goal is to improve de quality of the products

Thank you for your attention

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