Accelerated Storage Test

CIPAC MT 46.4

(CIPAC 5217/R)

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on behalf of DAPF (German Speaking Council for Plant Protecting Formulations)

Advancing Together

Introduction

CIPAC Analytical and Miscelleneous Test (MT) Methods

- CIPAC methods are used for…
  - Registering
  - Controlling
  - Specifying

    …. Plant Protection, Public Health, Home & Garden and Professional Use products


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- Methods for content of active ingredients and impurities are generally substance specific and published as **CIPAC Methods**, e.g. deltamethrin (333/TC/M2/2, CIPAC Handbook L, p.46, 2006)
- Methods for physical parameter methods are applicable to all products and published as **CIPAC MT (Miscellaneous Test) Methods**, e.g. pH value, Water by Karl Fischer or Viscosity.

Introduction

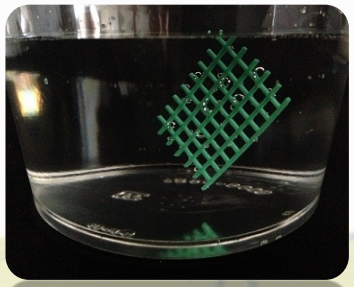
Acceptance of CIPAC MT Methods

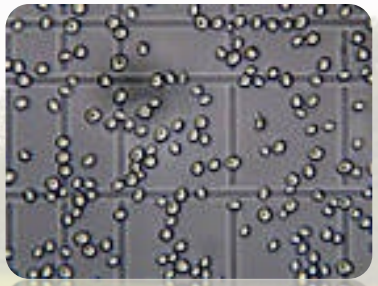
- CIPAC MT methods are accepted in most countries worldwide.
- Examples of references:
  - FAO/WHO Manual on development and use of FAO and WHO specifications for pesticides (1st edition – 3rd revision, Rome 2016)
  - International points of reference: FAO / WHO Specifications
  - National and international guidelines for registration, e.g.
    - Regulation (EC) No. 1107/2009
    - NY/T 1427 – 2016 (China)
    - …
- Acceptance of CIPAC methods for registration purpose continues to grow, cf. Japan

Introduction

Reasons for New or Revised CIPAC MT Methods

- Most CIPAC MTs have been developed more than 50 years ago but agriculture is changing at high speed:
  - New application technologies, e.g. drones
  - Novel formulation types, e.g. Matrix Release - MR, Gel for Direct Application - GD
  - Innovative biological concepts for active ingredients, e.g. mircoorganisms & RNAi



- As a consequence, there is always work to do to provide up-to-date CIPAC MT Methods …

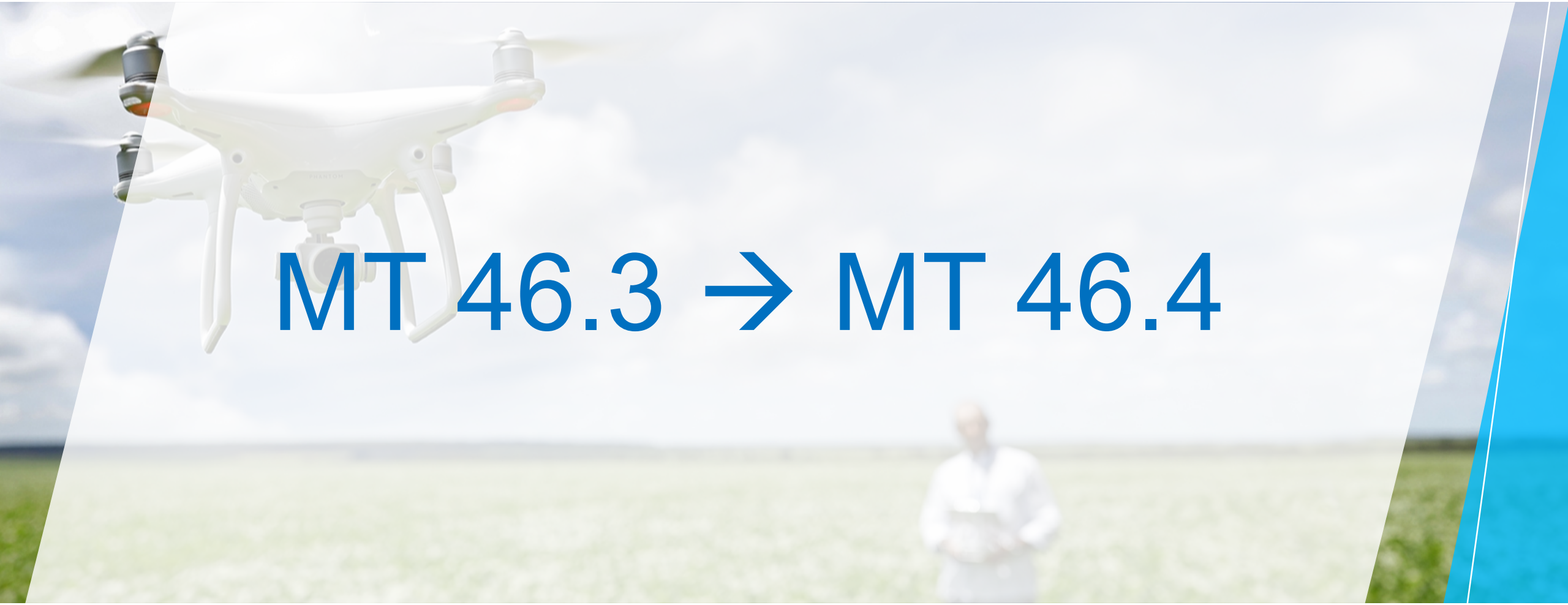
Introduction

Process of Revision

- **DAPF** (German speaking working group for pesticide formulations) closely collaborates with **CIPAC** to revise **MT methods**.
- Our process:
  - Identify need for new methods or revision
  - Draft new MT method (version)
  - Perform ring trials as required
  - Present in annual CIPAC meeting
  - Decision by CIPAC
    - Request for further clarification / ring test / … or:
    - Provisional MT methods which will become full MT method the following year in case of no further comments

Introduction

Revision of CIPAC MT 46.3



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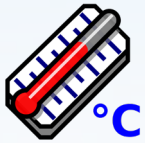
Scope of Method and Goal of Revision

// Scope



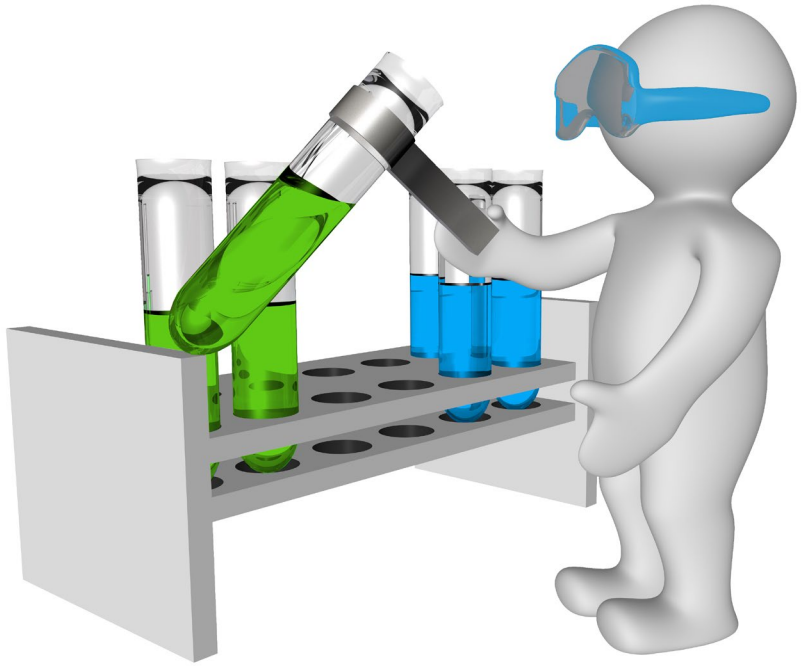
- // The objective of the method is to simulate the long-term aging of a formulation under conditions accelerated by heating.
- // The method describes conditions for storage and does not give results.
- // Tests performed after accelerated storage are not part of this method.

// Goals

- // Harmonize the accelerated storage procedure for all formulation types
- // Avoid adaption of method for future novel formulation types
- // Keep key parameter of method (storage conditions) unchanged
- // Editorial up-date

How to perform an …

Accelerated Storage Test ?



Accelerated Storage Test

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Prepare storage container with sample

Store container at elevated temperature

Remove container from storage

Equilibrate container with sample

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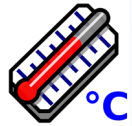
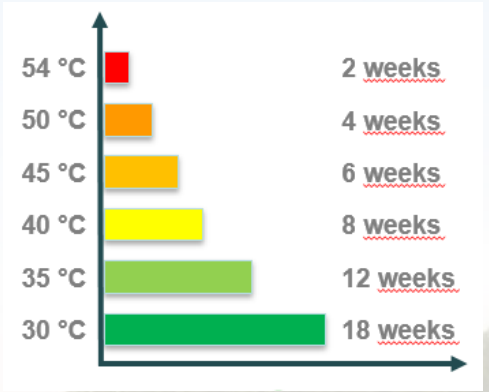
Conditions for storage - temperature / time

- Conditions for storage remain unchanged

Note 2 Using any of the following, alternative storage conditions is possible:
54 ± 2 °C for 14 days
50 ± 2 °C for 4 weeks
45 ± 2 °C for 6 weeks
40 ± 2 °C for 8 weeks
35 ± 2 °C for 12 weeks
30 ± 2 °C for 18 weeks
Where available, use storage condition as specified.





- Conclusion (storage conditions): no change
- General remarks
  - All conditions can be used as being equivalent for the purpose of the test method. Unless specified otherwise, there is no preferred condition.
  - There is no requirement to store for 2 weeks / 54 °C to support tropical conditions and storage at e.g. 40 °C may be fully sufficient.

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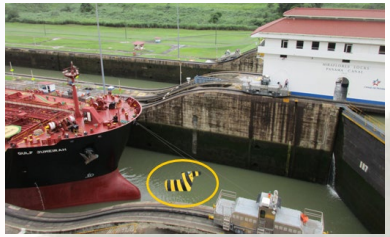
Accelerated storage under pressure

- Current MT 46.3 contains precedure for storage of granular formulations stored under pressure.
- This procedure is already described in test method for flowability (CIPAC MT 172.2).
- MT 172.2 also includes conditions for accelerated storage equivalent to MT 46.3 / 46.4
- MT 172.2 has been presented in Pananma 2018



- Proposal to omitt section on storage of solid formulations stored under pressure in revised CIPAC MT 46.4
- Conclusion: change acceptable as there is no impact on any other method and storage conditions are not needed in MT 46.4

CIPAC MT 46.4

Applicability to all Formulation Types

- MT 46.3
  - 1 - liquid formulations
  - 2 - solid formulations
  - 4 - LN formulations
  - 5 - MR formulations
- MT 46.4: Storage conditions (time / temperature) can be universally applied to any formulation type, therefore the method was opened to accomodate all existing formulation types (e.g. AE) but also newly developed ones such as GD, GB
- Harmonized Procedure:

PROCEDURE

Transfer the sample into the container for storage (Note 3) and seal tightly. Alternatively, formulations in commercial packs can be stored as delivered.

Put the container / commercial pack in an oven and keep it at the specified temperature for the defined period of time (Note 2). Afterwards, remove the container / commercial pack from the oven and allow it to reach room temperature before opening and carrying out the required tests.

- Conclusion: no relevant change

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Container for Storage: Material

- MT 46.3: According to the method, accelerated storage can be performed in a glass bottle, the commercial pack or a good representation thereof.
  - Commercial pack: available usually only for the Manufcaturer or when sampling products in original containers from the market.
  - Glass bottles or good representations of the original container (e.g. HDPE bottle): typically used when accelerated storage needs to be performed by e.g. control labs which do not have access to original containers.
- MT 46.4:
  - Use of commercial pack, a good representation thereof or glass bottle continues to be possible.
- Conclusion (container material): no relevant change

CIPAC MT 46.4

Container for Storage: Special containers

- MT 46.3 (Note 1)
  - Option for using sealed glass tubes or ampoules instead of glass bottles for storage of formulations containing volatile solvents.

- General comments:
  - Cracking of glass can cause severe injuries. In addition, sealing of glass ampoulas required open fire which is against QHSE rules of most analytical labs nowadays.
  - However, some products are sold in ampoulas which then represent the commercial pack.
- MT 46.4:
  - Sealed glass tubes or ampoules are not any more explicitely mentioned in this method, however, they still can be used when commercialized.
- Conclusion: no relevant change

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Container for Storage: General remarks

- Avoid storage of unrepresentative sample size as storage of e.g. 20 mL in a large bottle may lead to artificial effects on formulation
  - Additon to Notes: MT 46.4)

> Preferably, the container should contain the amount representative for the market to reflect a representative ratio of sample volume to headspace volume (where applicable). Storage containers and sealing mechanisms may need to be adapted individually for size and / or shape, e.g. in case the formulation type is a device.

- How to get a Mosquito Coil (MC) into a bottle ?
  → some formulation types may need adaptation of either the container (e.g. wrapping of Mosquitro Coil) or sample size and shape.
- However, this is not part of the Accelerated Storage Test.
- Proposal to include relevant information e.g. in Specification Templates in the FAO/WHO Manual (e.g. for LN or MR).

Recommendation

CIPAC MT 46.4

Conclusion

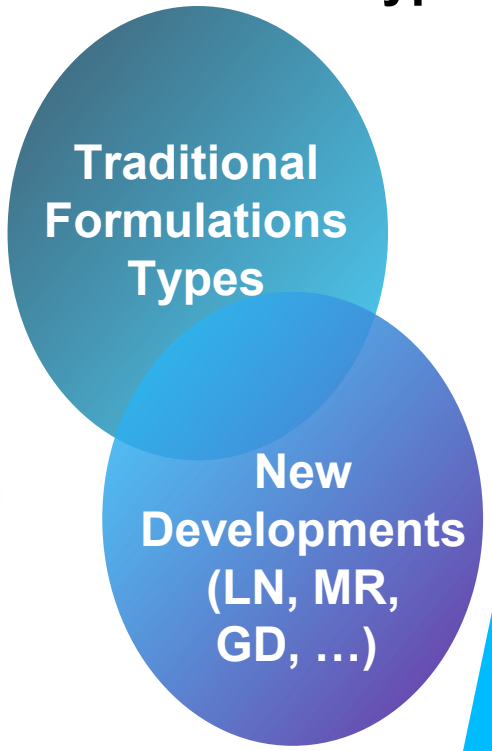
- CIPAC MT 46.4 has been revised by DAPF (German speaking working group for pesticide formulations) with the intention to provide a universal procedure for the storage of any formulation type under acelerated storage conditions.
- Key parameter of the method such as storage conditions & packaging materials remain unchanged.
- DAPF therefore proposes:
  - MT 46.4 as provisional CIPAC MT.
  - MT 46.4 to supersede MT 46.3 (incl. 46.3.4 for LN / 46.3.5 for MR).
- Recommendation:
  - For some formulation types having inherent properties of devices (e.g. bags, nets, coils, matrices) thorough thought should be given how to create and store representative samples.

Formulation Types

Diversity to Meet Customer Needs

	Formulation Cluster		Formulation type
Liquids	EC	Solutions/Emulsions	AL, EC, EO, ES, EW, ME, OL, SL, UL
	SC	Dispersions	CS, DC, FS, LS, OD, OF, SC, SD, SE, SU, ZC, ZE, ZW
Solids	WP	Powders	AP, DP, DS, EP, GP, OP, SP, SS, WP, WS
	WG	Granules (dispersible)	EG, SG, WG
	GR	Granules	GR
Others	TB	Tabletts	DT, ST,TB, WT
	AE	Sprays	AE (packed in Aerosol cans), TD (new)
	Others	Divers other formulation types	BR, CB, FU, GA, GD (new), GE, GL, GS, GW, HN, KK, KL, KN, KP, LB (new), LN, MC, MR (new), PR, SO, VP, XX……
Baits	RB	Ready Baits	RB (Gel) RB (Grain), CP (Contact Powder) RB (Block, Paste, Pellets)

Formulation Types



We are happy to answer your questions

Thank you!

Bye-Bye