Current Challenges in the Analysis of Copper Pesticide Residues

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INTRODUCTION

Copper (Cu) compounds are widely used in the EU, both in conventional and in organic farming. Being classified as pesticides, Cu compounds technically fall within the responsibility area of pesticide residue laboratories. In current practice, however, Cu analysis is mainly run by laboratories responsible for elemental analysis. This has mainly practical reasons, as Cu is efficiently covered by methods focusing on multi-metal elements analysis, which employ instrumentation uncommon to most pesticide residue laboratories. Such laboratories, mostly follow their own rules as regards sample handling prior to analysis. With Cu being classified as a pesticide, however, both sampling and sample handling need to actually follow the pesticide residue rules. This ambiguous situation has created some uncertainty among the Member States, which was also confirmed in a survey run by the EURL-SRM, which revealed great differences among the laboratories within the EU (even within Member States) in the way samples for Cu analysis are handled. With Cu being included in the EU-Coordinated Multi-Annual Control Program (MACP) for pesticides from 2024 onwards (Reg. (EU) 2023/731), solving this ambiguity became a priority.

„PESTICIDES TRACK“ (to be followed)		„CONTAMINANTS TRACK“ (common so far)
≥ 1 kg (small sized) **≥ 1 kg and ≥10 units** (medium sized) **≥ 2 kg and ≥ 5 units** (large sized)	**SAMPLING RULES** for fresh produce [1] - [2]	**≥ 1 kg,** **≥ 3 to ≥ 10 incremental samples** (depending on weight or volume of lot)
Whole products should be prepared **Specific products:** Remove e.g. stems, crowns, easy detachable soil,… **Stone fruits**: Remove kernels but record and consider kernel weight.	**SAMPLE PRE-TREATMENT RULES** for fresh produce [1, 3] - [2, 4]	*“**only the edible part should be investigated** and **the results should refer to this part**”* *„**Surface contamination** like soil **should be thoroughly removed by rinsing** with drinking water“*

LITERATURE

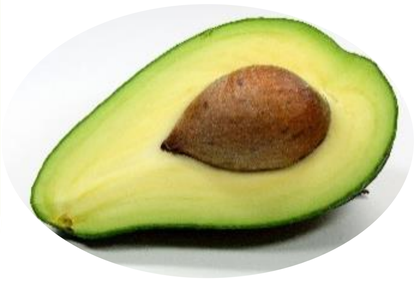
[1] Dir. 2002/63/EC
[2] Reg. (EC) No. 333/2007
[3] Reg. (EU) No. 396/2005
[4] EN 13804:2013
[5] Document Nº SANTE/11312/2021v2
[6] DIN 32645:2008-11
[7] www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM19_Preliminary_Report.pdf

Natural content: 2.8 mg/kg
Spiking level for validation: 10 mg/kg

Specificity	**26 %** of blank signal (≤ 30 %)	✓
Recovery	**99.3 %** (70-120 %)	✓
Precision	**± 2.0 %** (≤ 20 %)	✓

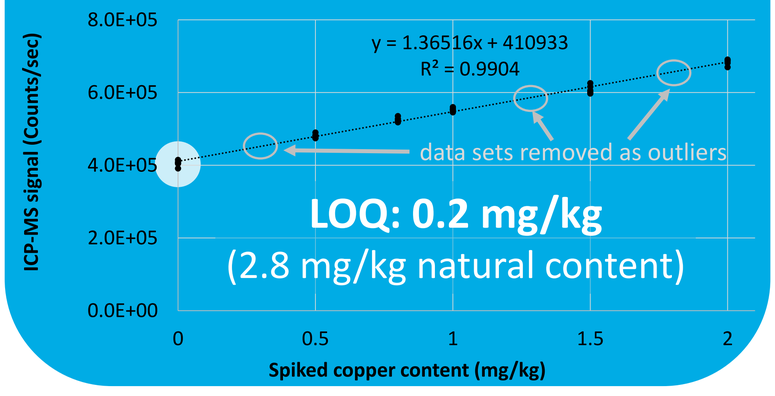
- **Validation at levels <10 mg/kg not foreseen in AQC-Guidelines currently** (due to specificity criteria)
- **Lowest successfully validated level = LOQ: 10 mg/kg**

METHOD VALIDATION
exemplary for avocado
[5] - [4, 6]



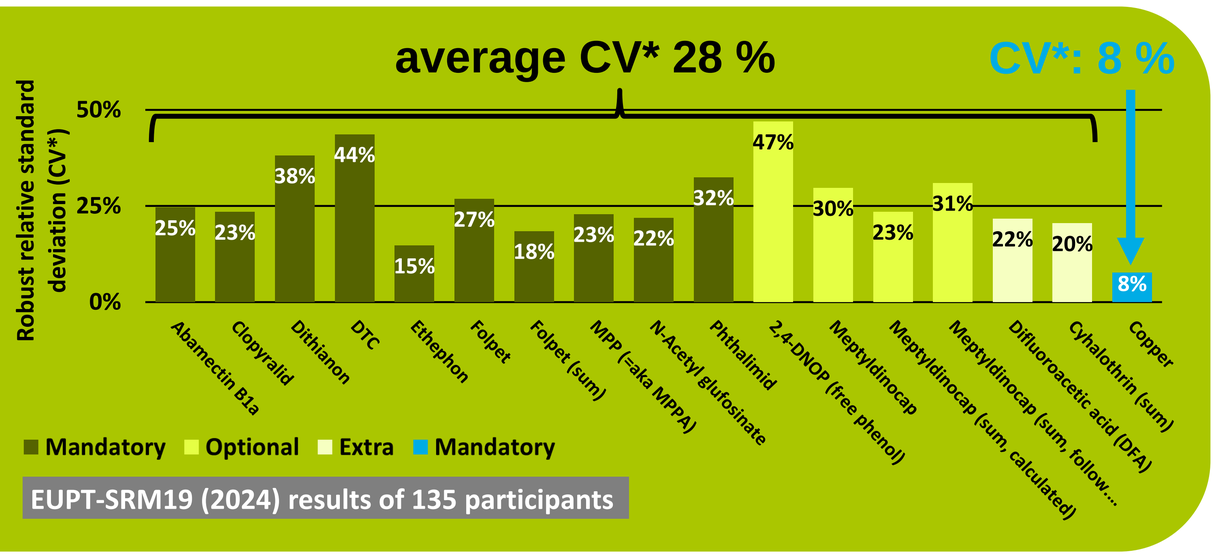
Indirect calibration curve method:

- spike the sample at 10 equidistant conc. up to 10x of the expected LOD, e.g. n = 3
- LOQ = 10x StDev of intercepts of derived calibration curves



ASSESSMENT OF RESULTS & MEASUREMENT UNCERTAINTY [5, 7]

- PT-data show that **the robust RSD** (CV*) **of Cu is much smaller than that of most other pesticides**
- **the default expanded measure-ment uncertainty of ±50 % applying for pesticides does not seem appropriate for Cu residues**



SUMMARY

There are multiple discrepancies between the procedures followed for Cu analysis in fields of pesticides and contaminants. The data of Cu contents in samples resulting from both approaches must therefore not be mixed to prevent wrong assessments on the natural background, wrong intake calculations as well as wrong conclusions as regards the MRL conformity. An EU-wide consolidation and harmonization is needed !

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