# *MRM Pesticides that are difficult to analyse or problematic, due to degradation during the extraction or due to other causes: Question?*

**Italian official Laboratories and IT-NRL Experiences**

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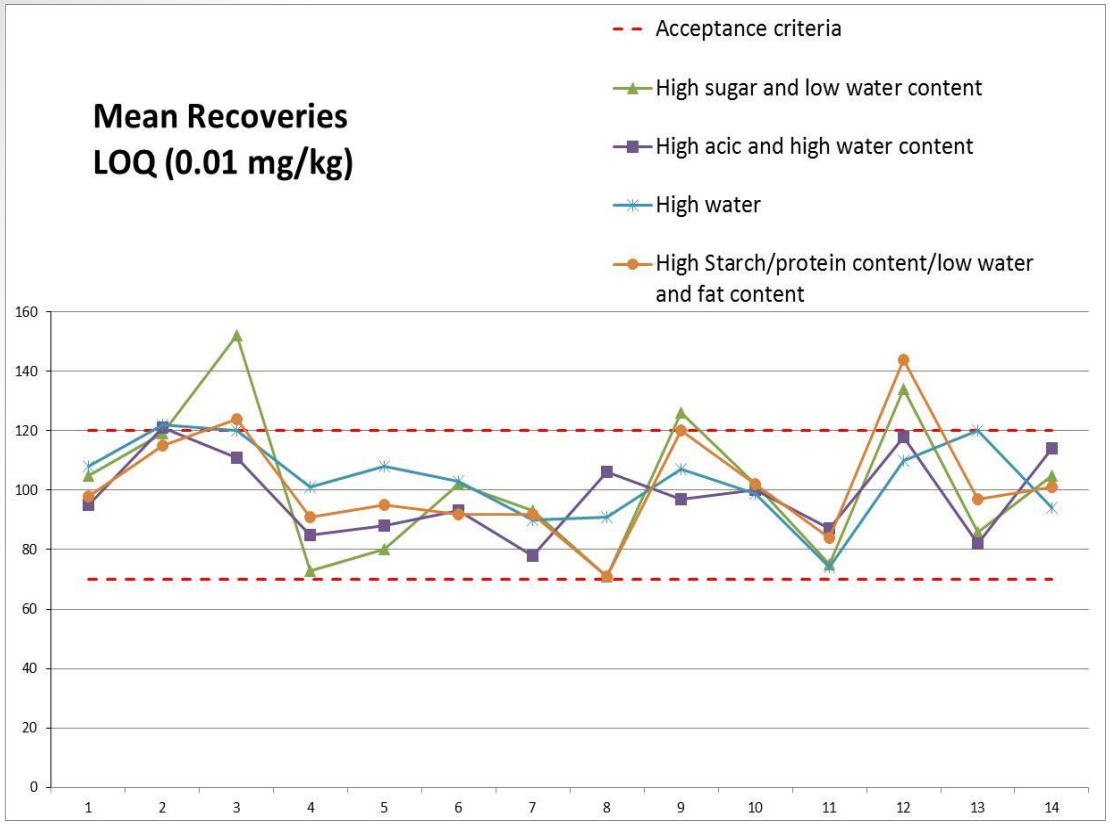
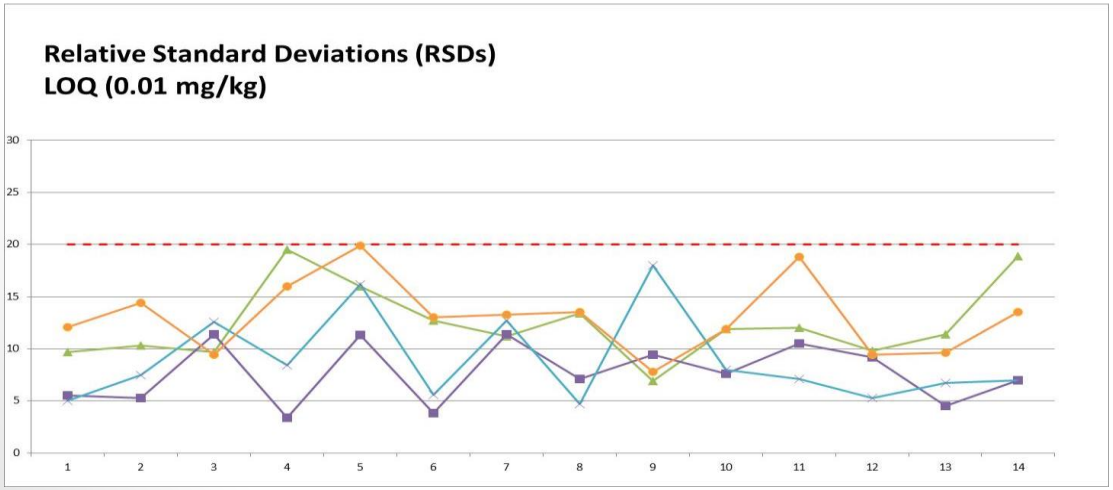
***Pesticide Section – National Institute of Health (Istituto Superiore di Sanità)***



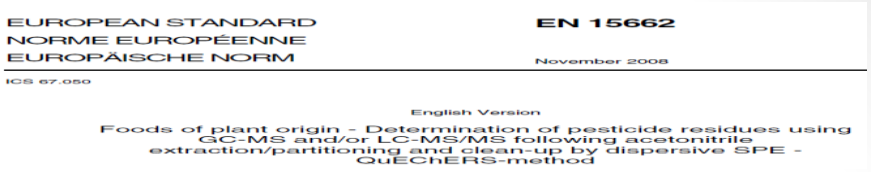

- **Method EN 15662:2009**
- **GC/MS/MS**

  (Agilent Technologies GC 7890 A equipped with a 7000 Triple Quad MS)
- **Single-level calibration**
- **Matrix matched calibration**
- **Internal standard**
- **Replicate experiments n ≥ 6**
- **List of investigated compounds:**

Bifenthrin, Chlorpyriphos, Diazinon, Endosulfan alpha, Endosulfan beta, Endosulfan sulfate, gamma-HCH, Phosalone, Indoxacarb, Kresoxim-methyl, lambda-Cyhalothrin, Pirimiphos-methyl, Procymidone, tau-Fluvalinate

Results from IT-NRL

**QuEChERS Multi Residue Method**
**EN 15662:2009**


EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15662

November 2008

ICS 67.050

English Version

Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE - QuEChERS-method

# Commodity Groups

- ✓ High acid and high water content
- ✓ High water

- ✓ High sugar and low water content
- ✓ High starch/protein content/low water and fat content

## *Analitycal Procedure – main differences*

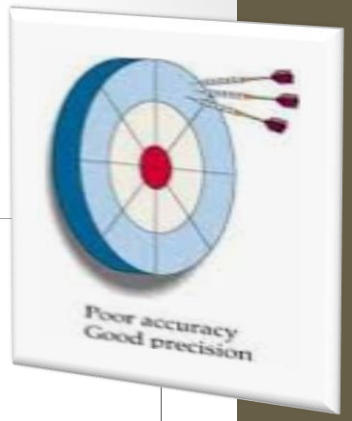
- Weighing: in the case of fruits and vegetables weigh 10

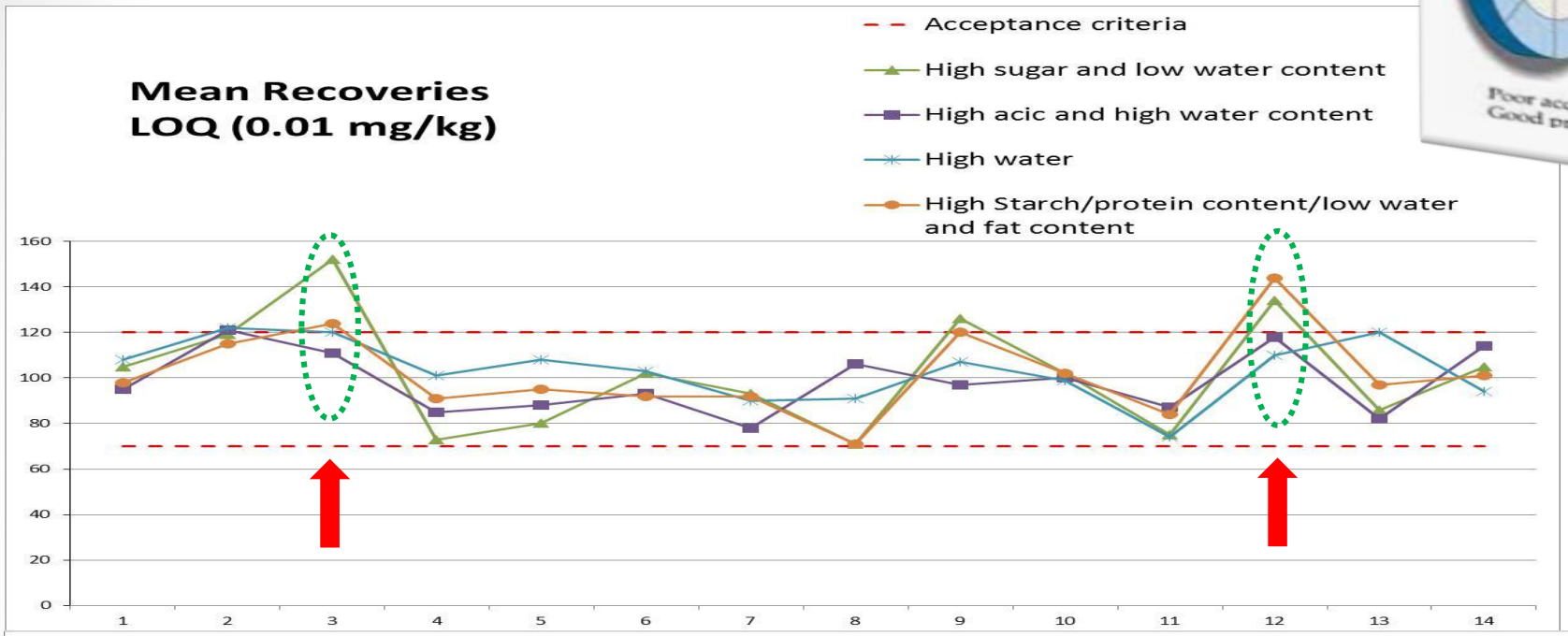
- Weighing: for dry sample materials like cereal products and honey weigh 5g ± 0.05g

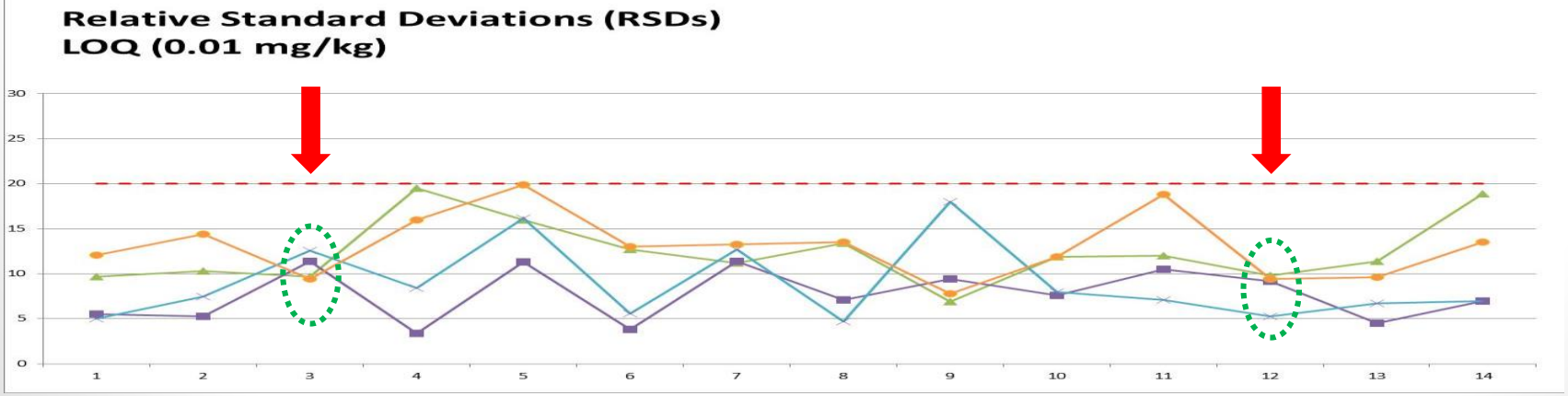
- Addition water
- Additional clean up: freezing + Dispersive SPE C18

# Trueness ⟶ Case Studies

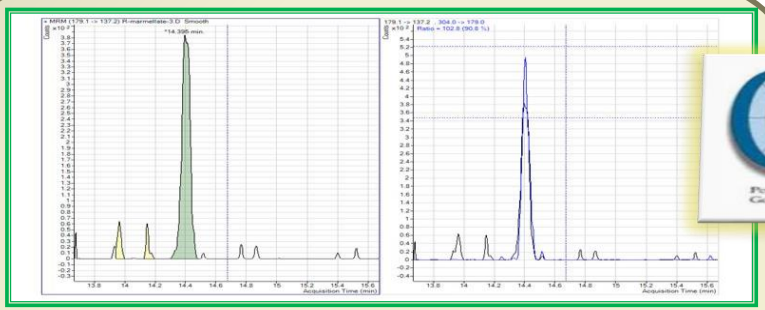
## Diazinon and Pirimiphos-methyl

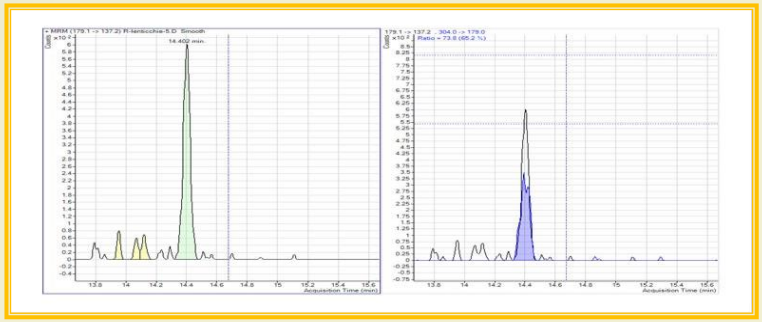


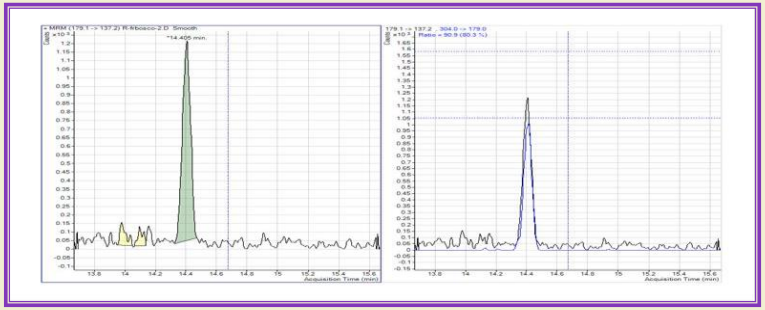



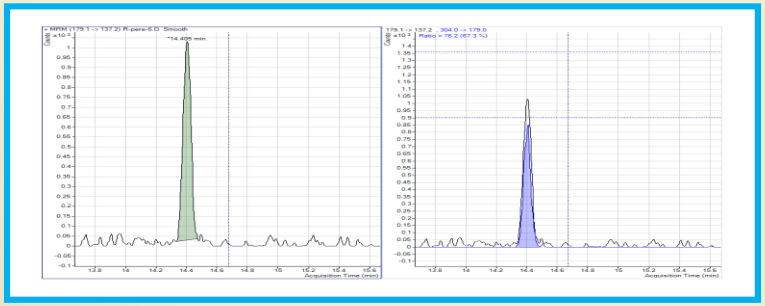



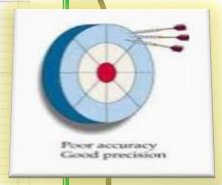
# Case Study: Diazinon



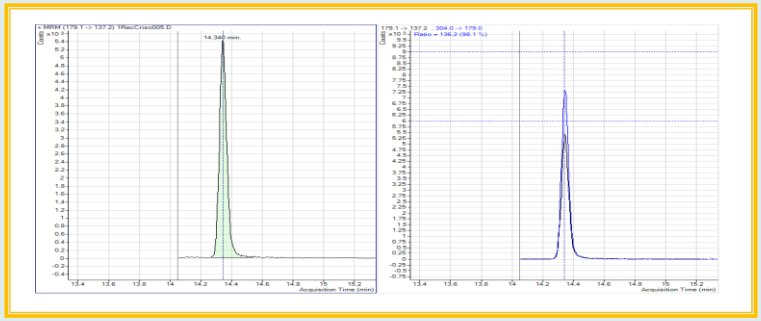








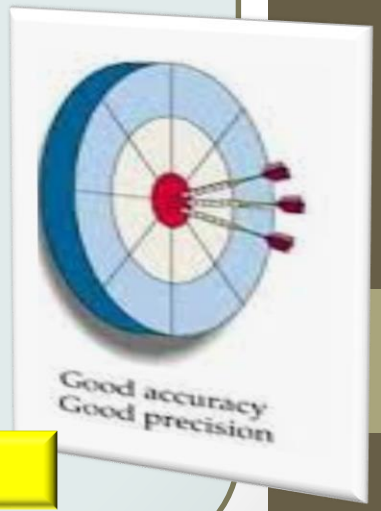

- High sugar and low water content
- High acic and high water content
- High water
- High Starch/protein content/low water and fat content



## Cereals

**Mean Recs: 100%**

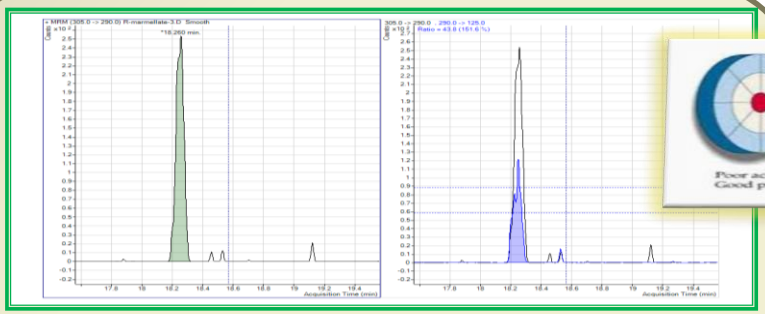
**RSDs: 2,7%**

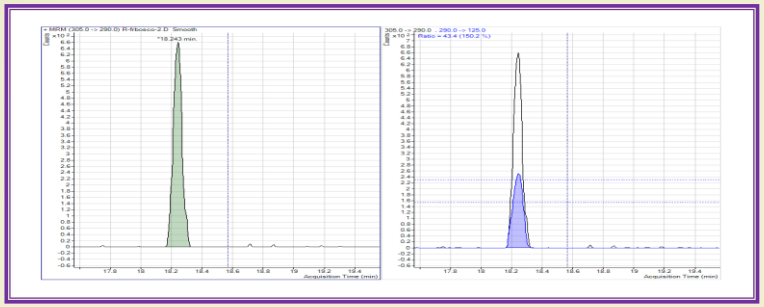


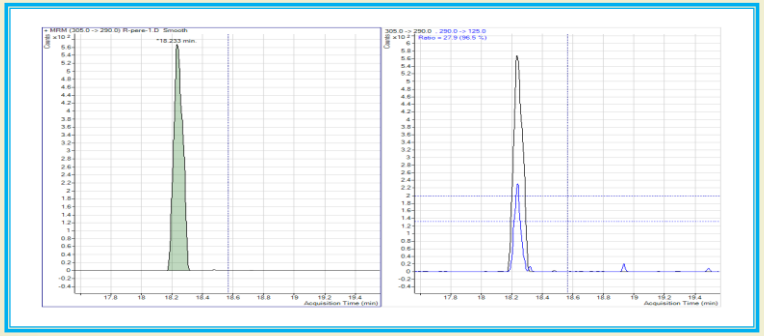

**Low Conc Level : 0.05 mg/kg**

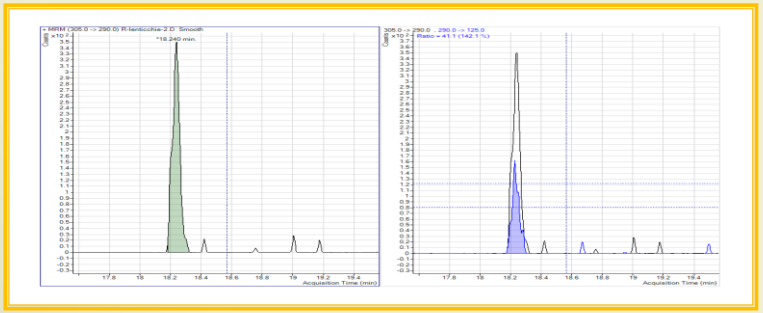
**LOQ: 0.01 mg/kg**

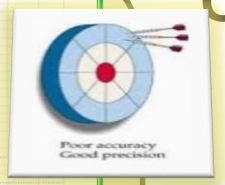
# Case Study: Pirimiphos-methyl









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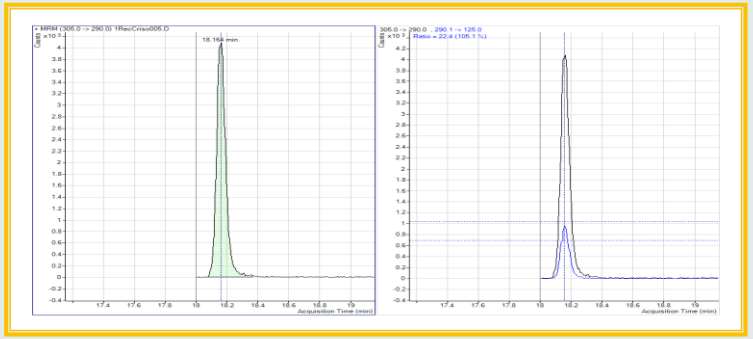
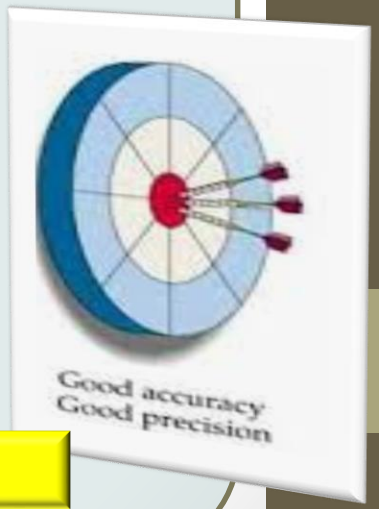


## Cereals

**Mean Recs: 108%**

**RSDs: 2,1%**




**Low Conc Level: 0.05 mg/kg**

**LOQ: 0.01 mg/kg**

For Commodity Groups of  high sugar and low water content , and high fat content and high starch/protein content at LOQ 0.01 mg/Kg  it is necessary to stress the clean up of the Standard Method EN 15662

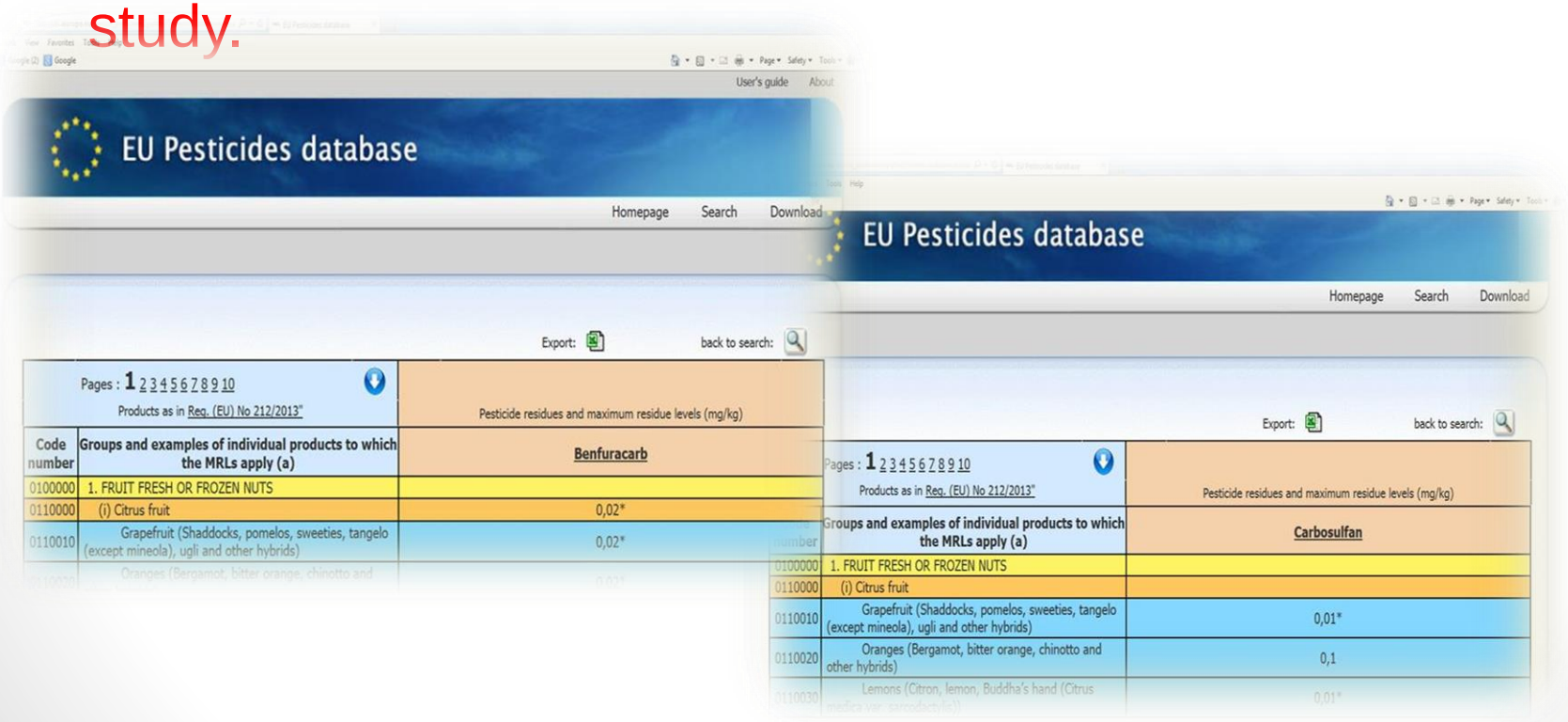
# Question?

The clean up of the Standard Method could be improved

Could the procedure be revised?

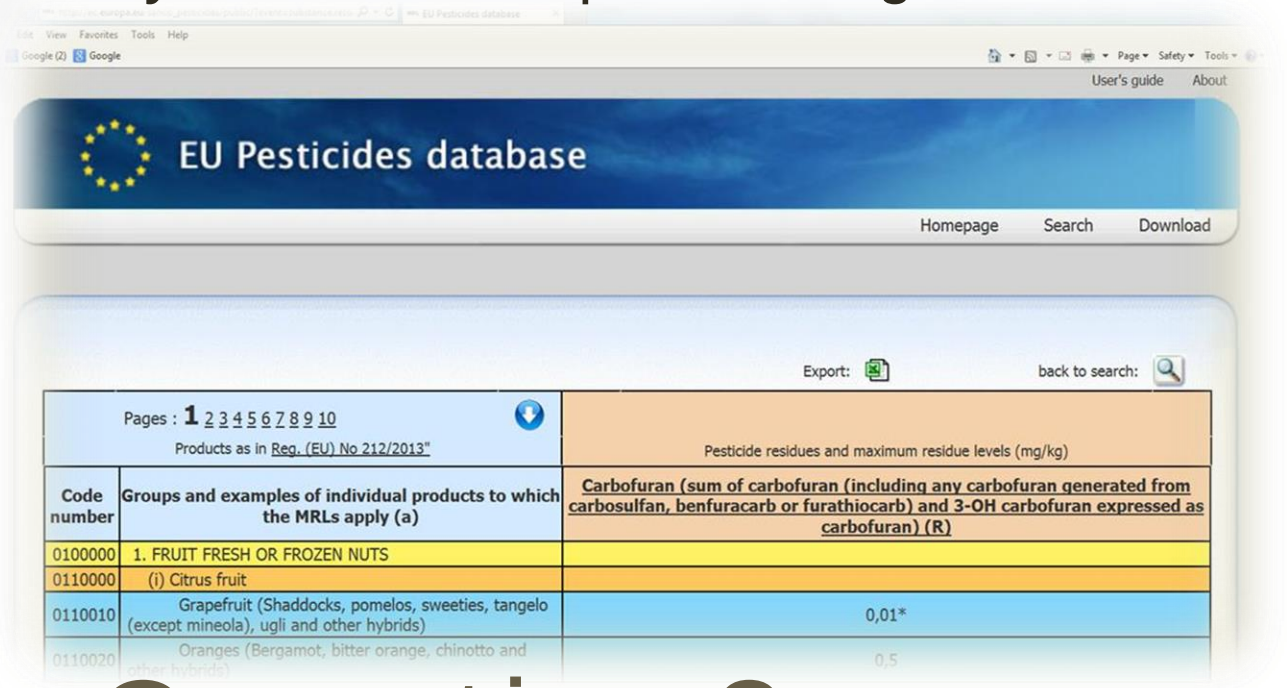
# Case study: Benfuracarb and Carbosulfan

- Some laboratories have highlighted problems of poor accuracy and sensitivity for the Benfuracarb and Carbosulfan.
- Not be capable of providing acceptable validation study.



# Case study: Benfuracarb and Carbosulfan

Maybe, these compounds degrade in Carbofuran (?)




# Question?

Could the Benfuracarb and Carbosulfan be included only in Carbofuran MRL residue definition?

# Case study: Quinalphos

Some laboratories have highlighted changes in colour and physical state of the pure reference standard


First preparation of the stock solution

White crystalline powder

Other preparation of stock solution

***Time***

# Question?

Could pure reference standard degrade?

Thanks for your attention!!!!