lach : bruns

Taking a closer look.

Pesticides and contaminants in organic products:

Herbicides

Dr. Günter Lach, February 2019

HERBICIDES

HERBICIDES

2,4-D

Glyphosate

Paraquat

Prosulfocarb

MCPA

Dichlorprop

Haloxyfop

Pendimethalin

Imazamox

Propyzamid

……

RESIDUE DEFINITON

How is the MRL definition of the pesticide?

Does it cover only **one analyte** or also **metabolites** / **degradation products** / **conjugates**?

Example: Phenoxycarboxylic acids like f. ex. ***Haloxyfop*** or ***2,4-D***

The **MRL definitions** are complex:

Haloxyfop = sum of **Haloxyfop,** its' **esters,** its' **salts** and its' **conjugates**

2,4-D = sum of **2,4-D,** its' **salts,** its **esters** and its' **conjugates**

ANALYTICAL APPROACH

Questioning the analytical results

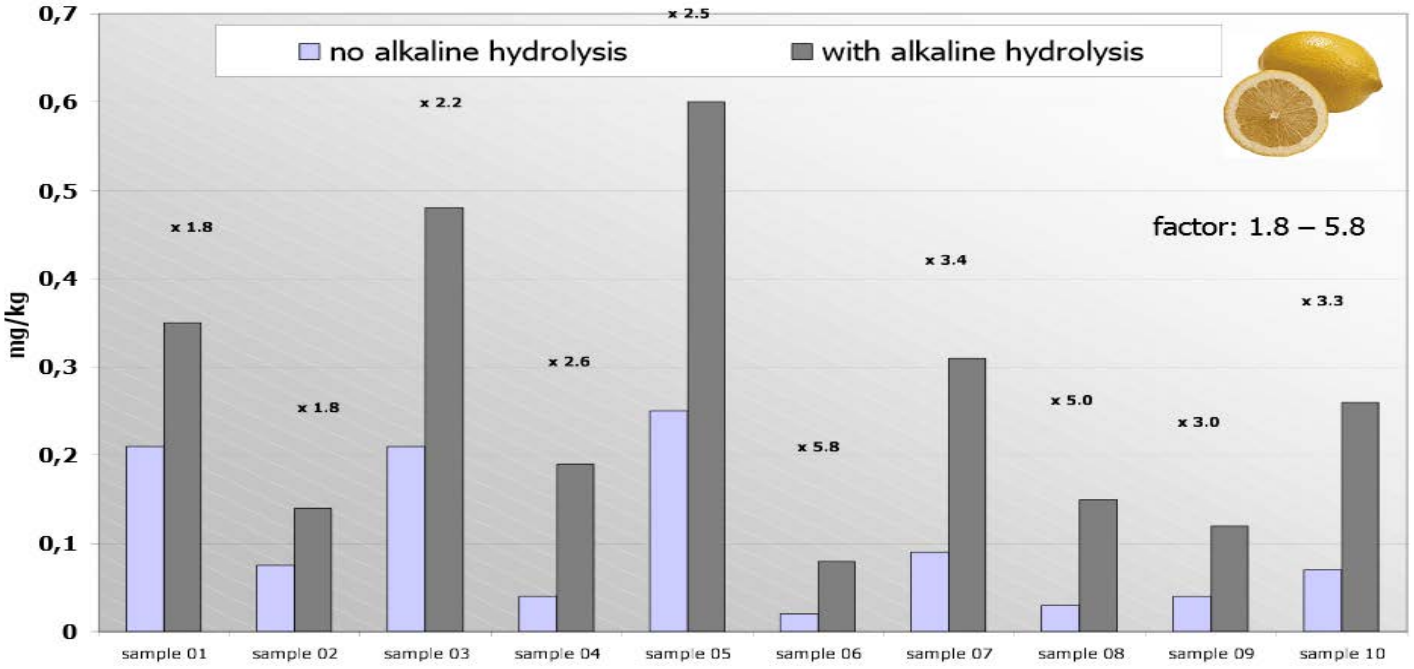
Can the "**entire**" **pesticide** be analysed by a multi-method? Or is a single residue method or an **additional analytical** step necessary?

Example **Haloxyfop**: This pesticide requires an extra step ("**Hydrolysis**") to **cover all components** which are relevant like free acid, esters, and **conjugates**.

What influence does this aspect have on the reported laboratory result?

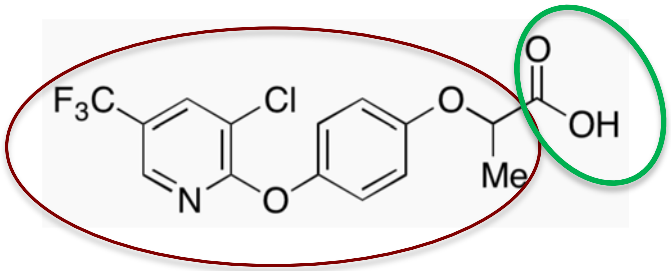
And: what are "conjugates"?

Analysis of **Acidic Herbicides** including a "**hydrolysis step**"



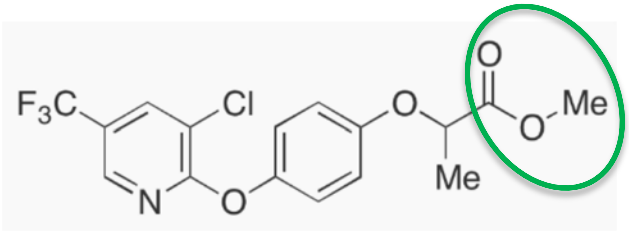
Levels of 2,4-D in lemons without and with hydrolysis
(Data: Acidic pesticides using QuEChERS method, CVUA Stuttgart)

Residues of Haloxyfop in food products



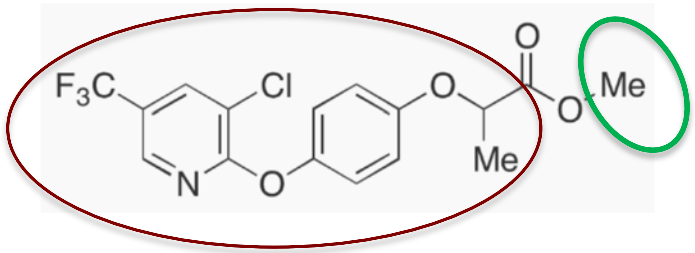
Haloxyfop-moiety

Laboratories are usually able to detect only **Haloxyfop** **(acid)**



and …
particular **esters**
(f.ex. Haloxyfop-**methyl**)

Conjugates of Haloxyfop



Haloxyfop-moiety

There might be much more "conjugates", which we don't know ➔ nature is very creative!

Haloxyfop-glucoside:

Substitute "-Me" by "Glucoside"

Haloxyfop-glutamate:

Substitute "-Me" by "Glutamate"

Haloxyfop-aspartate:

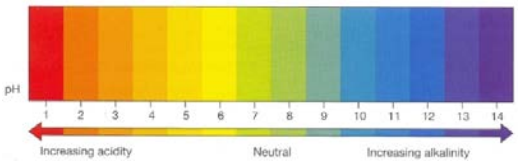
Substitute "-Me" by "Aspartate"

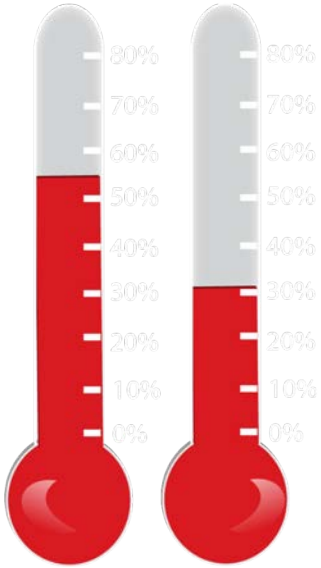
Haloxyfop-???:

Substitute "-Me" by "**???**"

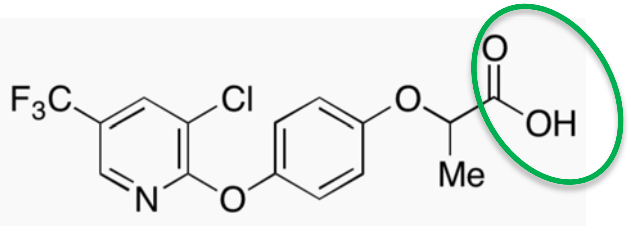
ANALYTICAL APPROACH

The laboratories are required to analyse the samples in a different way ➔ **alkaline hydrolysis** (H_2O, NaOH, time, high temperatures)



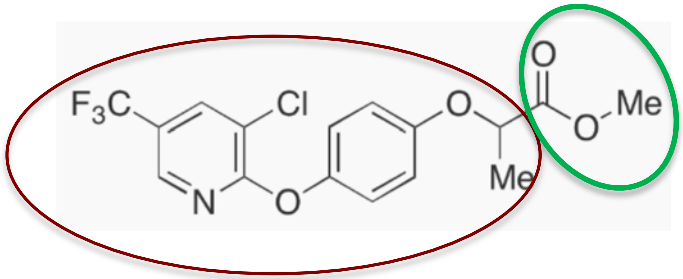


Residues of Haloxyfop in food products



Laboratories are usually able to detect only **Haloxyfop (acid)**

H_2O, NaOH, time, high temperatures = "Hydrolysis"



Haloxyfop-moiety

Haloxyfop-esters
Haloxyfop-glucoside / -glutamate / -aspartate / -…

ANALYTICAL APPROACH

1. relana® Method Ring Test June 2018

Acidic Herbicides
in Clementine and Flaxseed

2. relana® Method Ring Test December 2018

Acidic Herbicides (Conjugates)
in Clementine

ANALYTICAL APPROACH

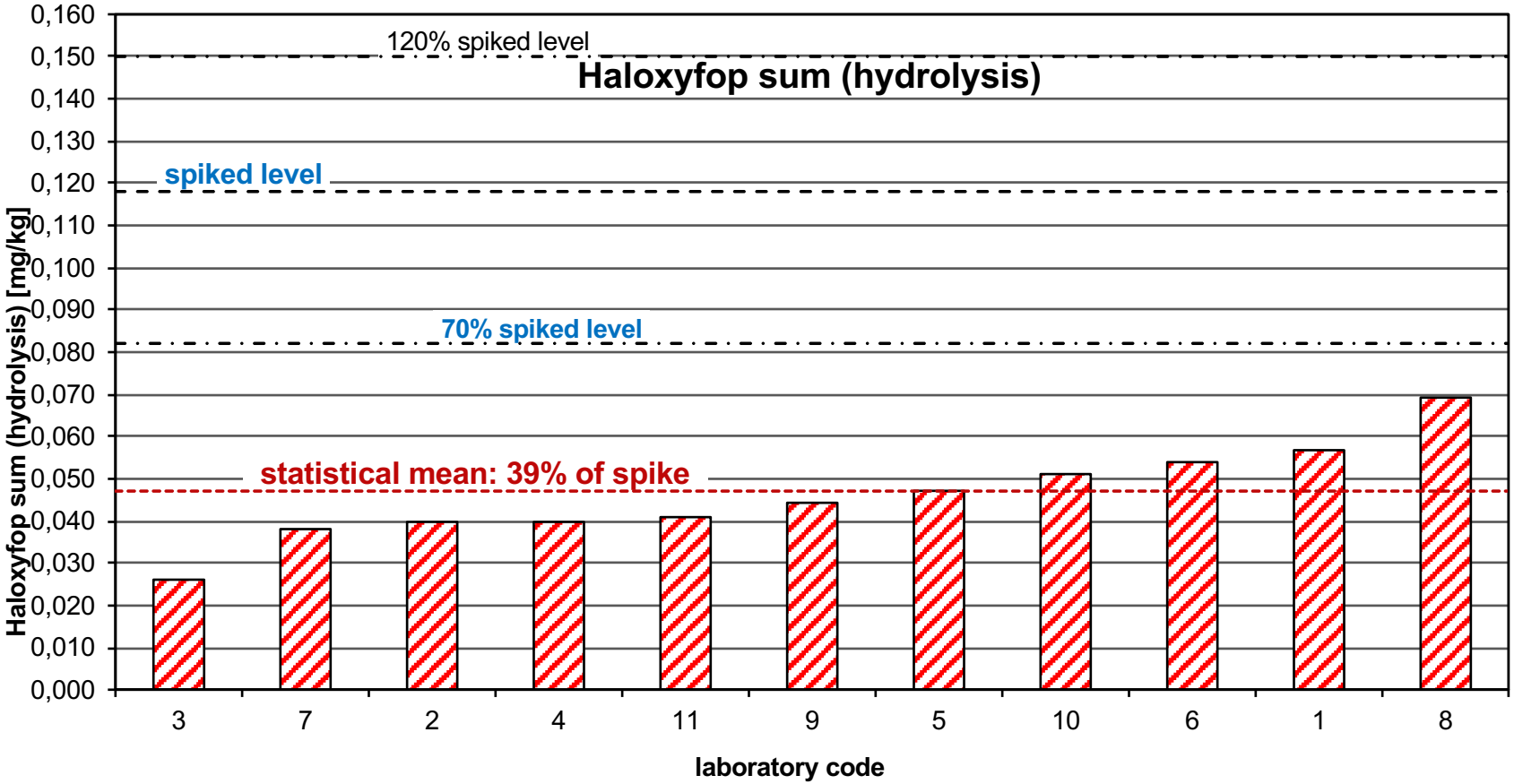
relana® Method Ring Test June 2018

Acidic Herbicides
in Clementine and Flaxseed

- **Haloxyfop** (acid)
- Haloxyfop-methyl (ester)
- **MCPA** (acid)
- MCPA-2-ethylhexyl (ester)
- **2,4-D** (acid)
- 2,4-D-2-ethylhexyl (ester)
- **Dichlorprop** (acid)
- Dichlorprop-butotyl (ester)

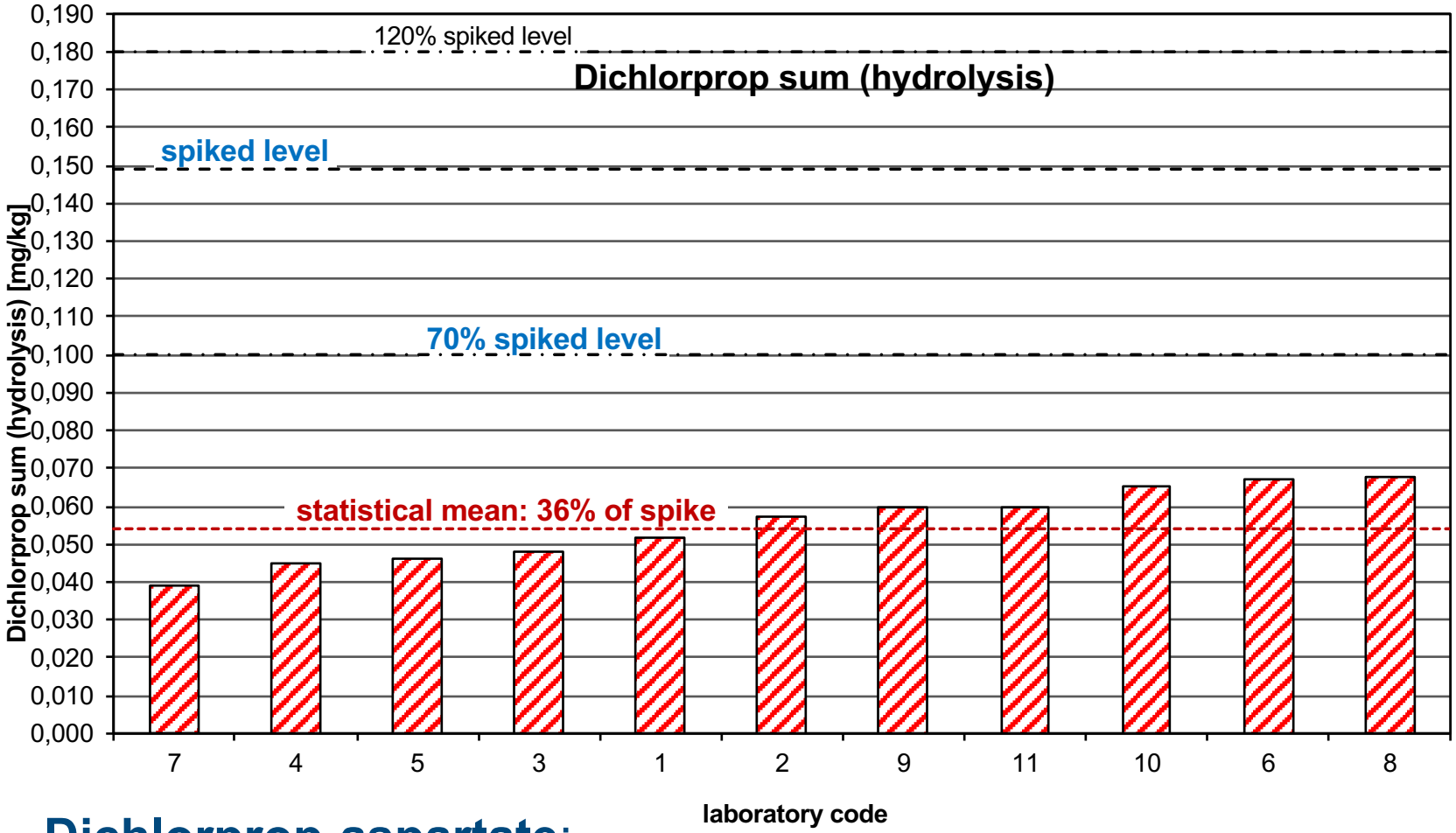
- Haloxyfop-**glutamate** (conjugate)
- MCPA-**glucoside** (conjugate)
- no conjugate
- Dichlorprop-**aspartate** (conjugate)

Haloxyfop-glutamate



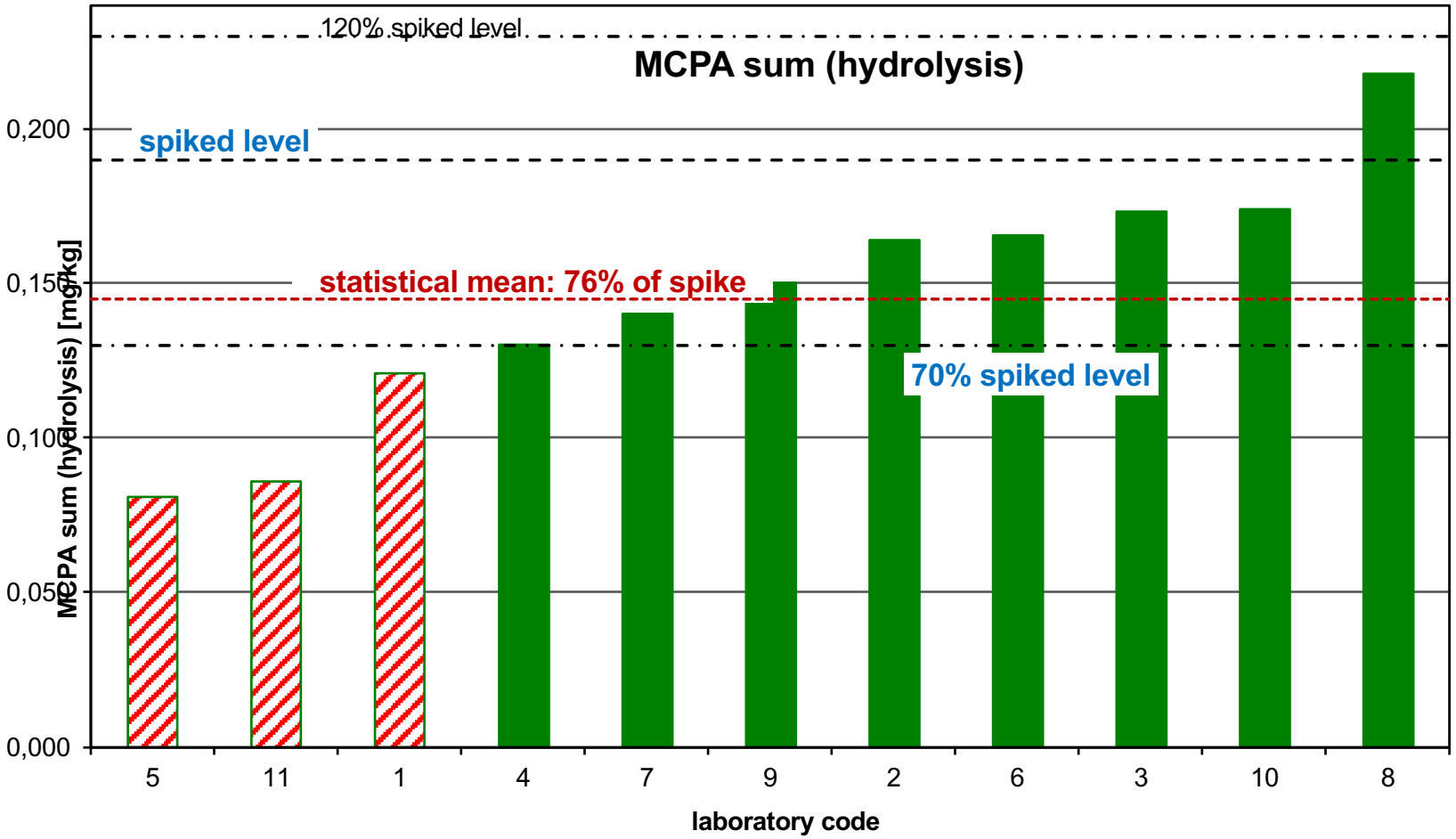
Haloxyfop-glutamate:
Conditions of the alkaline hydrolysis are not appropriate to cover glutamate-conjugates! ➔ results are (much) too low!

Dichlorprop-aspartate



Dichlorprop-aspartate:
Conditions of the alkaline hydrolysis are not appropriate to cover aspartate-conjugates! ➔ results are (much) too low!

MCPA-glucoside



MCPA-glucoside:
Conditions of the **alkaline hydrolysis** are **appropriate** to cover **glucoside-conjugates**! ➔ results are OK!

ANALYTICAL APPROACH

relana® Method Ring Test December 2018

Acidic Herbicides (conjugates) part 2 in Clementine

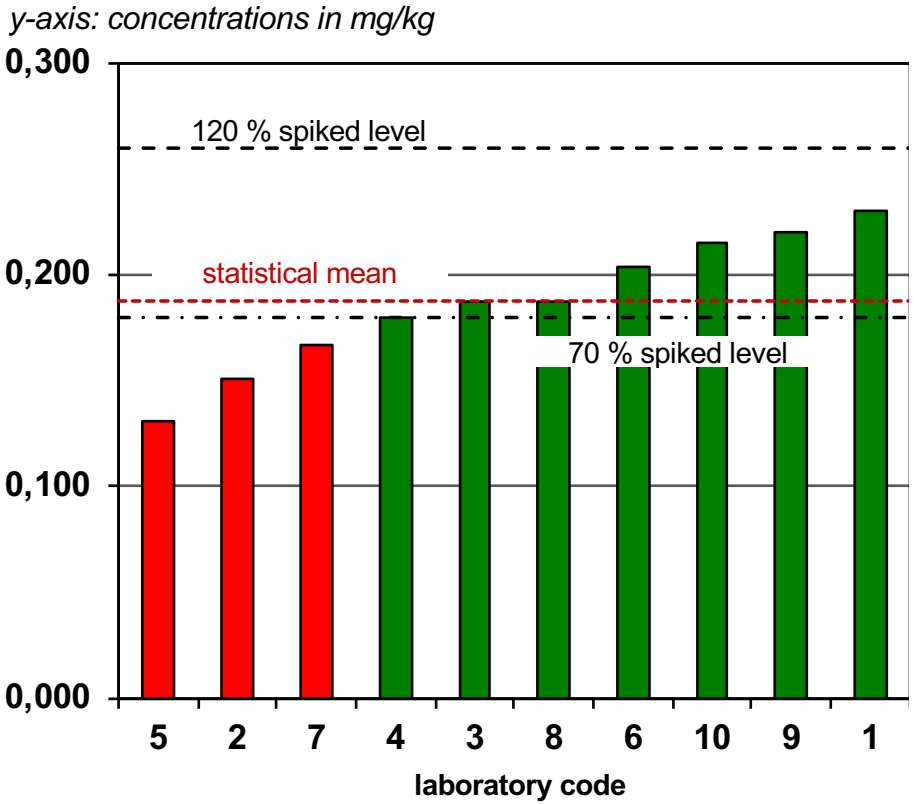
Part 1

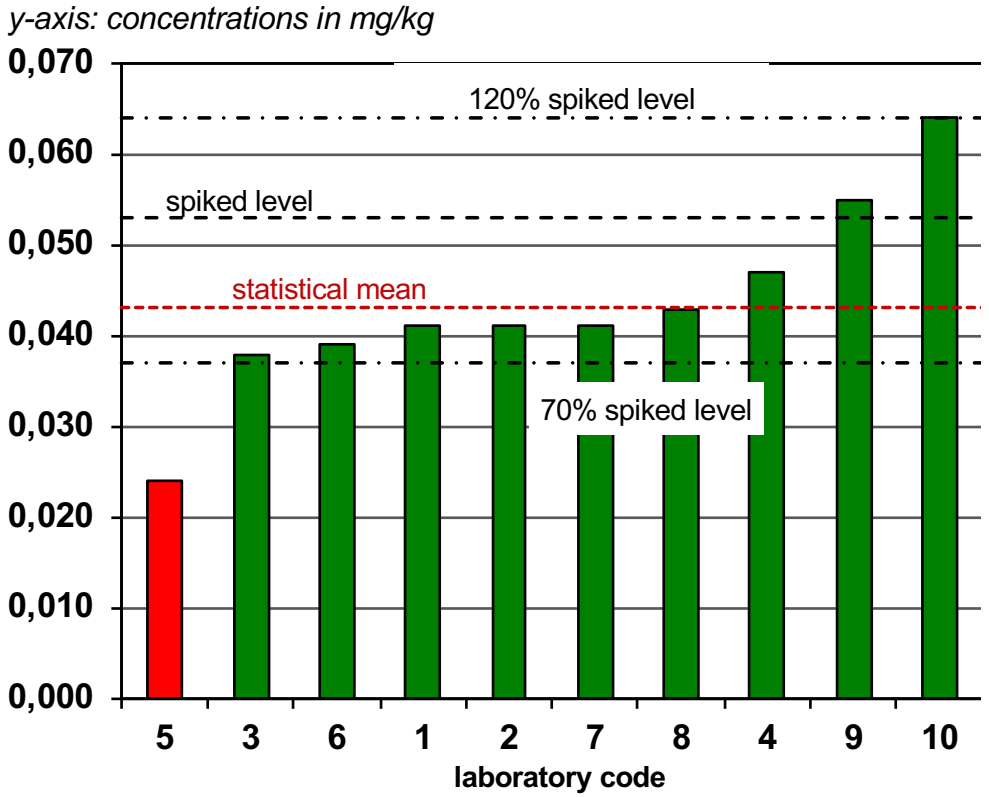
- Haloxyfop-**glutamate**
- MCPA-**glucoside**
- 2,4-D **no** conjugate
- Dichlorprop-**aspartate**

Part 2

- Haloxyfop-**glucoside**
- MCPA-**glucoside**
- 2,4-D-**glucoside**
- Dichlorprop-**glucoside**

Glucoside-conjugates

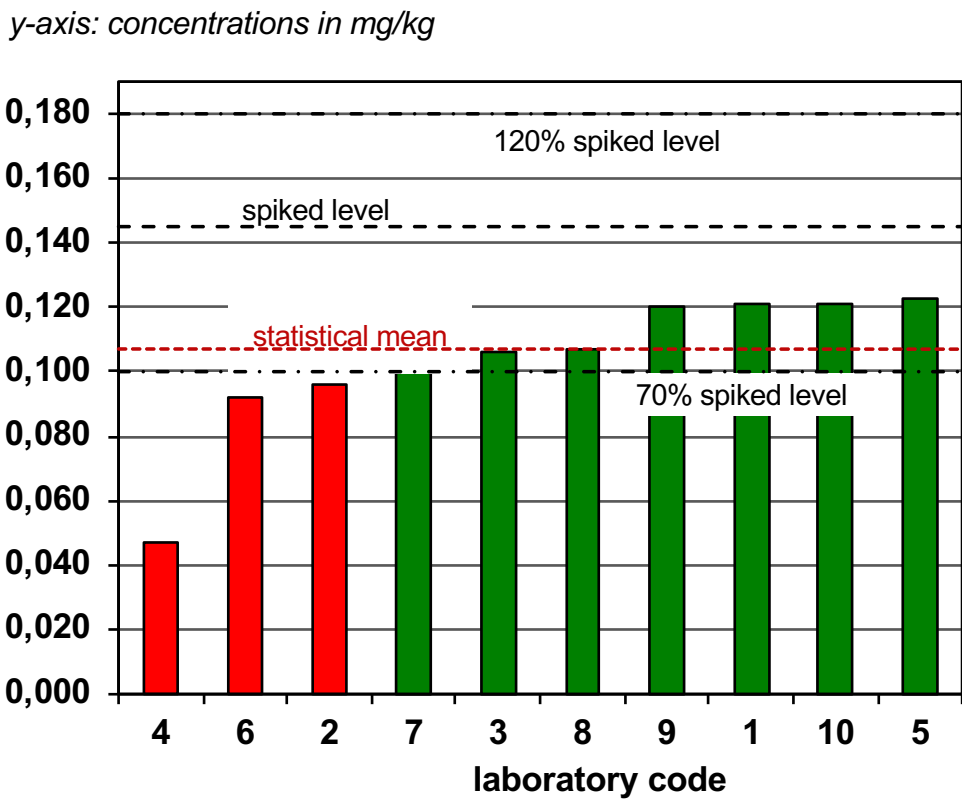


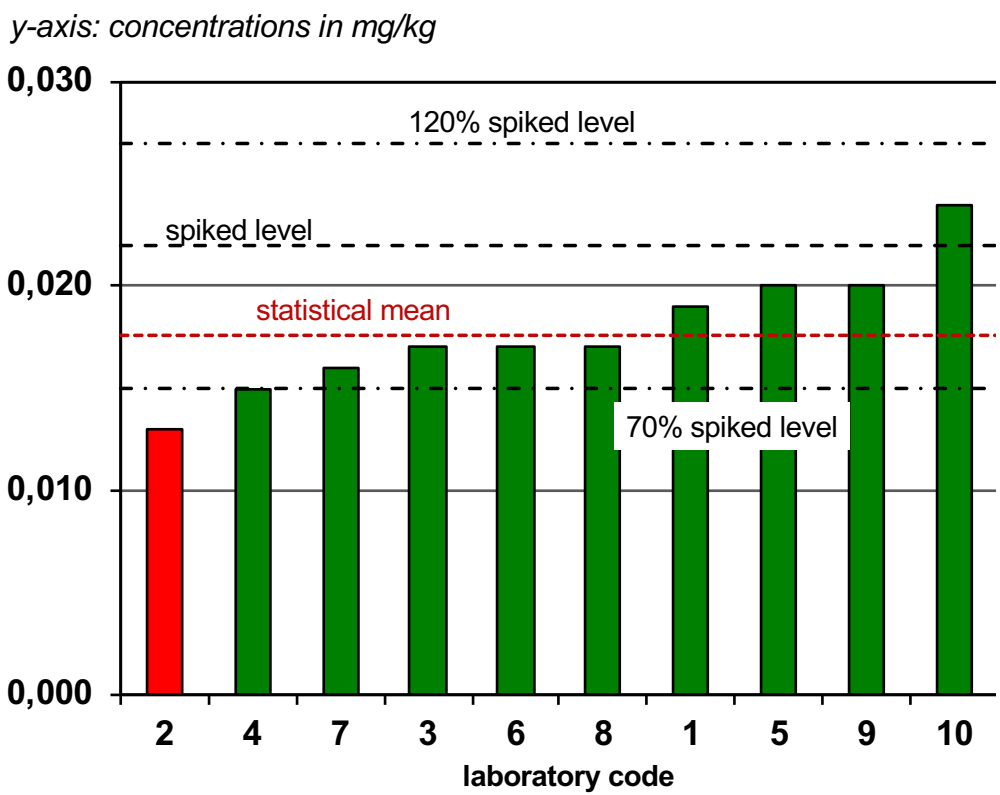


2,4-D-glucoside:
statistical mean =
72% of spiked level

Dichlorprop-glucoside:
statistical mean =
81% of spiked level

Glucoside-conjugates





Haloxyfop-glucoside:
statistical mean =
74% of spiked level

MCPA-glucoside:
statistical mean =
80% of spiked level

Acidic Herbicides

Overall result of the method ring tests:

Glucosides:
Conditions of the alkaline hydrolysis **are appropriate** to cover **glucoside-conjugates**! ➔ results are OK!

Other conjugates like aspartates, glutamates and perhaps others are not covered by the alkaline hydrolysis approach!

➤ more research necessary!

➤ in the meantime:
Results of acidic herbicides measured by applying the alkaline hydrolysis approach are often closer to the true value than the results of the sum of free acids + esters only.

But bear in mind, that the entire levels might be even higher, considering the fact, that not all conjugates might be covered!

Imazamox – an “alternative” herbicide

- *Commercial introduction: 2001 (BASF: “Beyond®”, “Raptor®”)*
- *Contact and residual activity, inhibits plant amino acid synthesis*
- ***Targeted weeds:** Grass and broadleaf weeds, Common reed (Phragmites australis), Flowering rush (Butomus umbellatus), Curly-leaf pond weed (Potamogeton crispus)*

- ***Uses**: **Pre- or post-emergence** control of weeds in **maize**, **rape**, alfalfa, **peas** and **beans (edible legumes)**, at 36–56 g/ha; pre-emergence control of weeds in ‘Clearfield’ imidazolinone-resistant **wheat**, rape and **sunflowers**.*

Sources: PPDB of the Universtity of Herfordshire (UK); The Pesticide Manual, BCPC (UK)

Imazamox – an “alternative” herbicide

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imazamox supplier! imazamox 40g/l sl with good price

US $1.5-1.5 / Liters
1000 Liters (Min. Order)

2 YRS Shanghai Sinogreatland Indu...
78.0%

Herbicide Imazamox 95%,98% TC, systemic selective pesticide...

US $190000-250000 / Ton
1 Ton (Min. Order)

8 YRS Shanghai Mingdou Chemical ...
66.7%

imazamox 40g/l sl

US $4.5-20 / Kilogram
500 Kilograms (Min. Order)

4 YRS Shanghai Sinogreatland Indu...
75.0%

seller agrochemical herbicide Imazamox 4%SL best price

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6 YRS Shijiazhuang Awiner Biotech ...
97.6%

high quality low price imazamox 114311-32-9

US $100-3900 / Gram
1 Gram (Min. Order)

9 YRS Haihang Industry (Jinan) Co.,...
"Excellent customer service" (3)

Imazamox – an “alternative” herbicide

Product	Imazamox concentration in mg/kg
Sunflower seeds (bio)	0,003 up to **0,021** **(0,042*)**
Lentils (bio)	0,026 up to **0,035**
Lentils (conv.)	0,010 up to 0,032
Rice (bio)	0,012
Rice (conv.)	0,003 up to 0,007

Analytical data provided by

**source: SKAL (NL)*

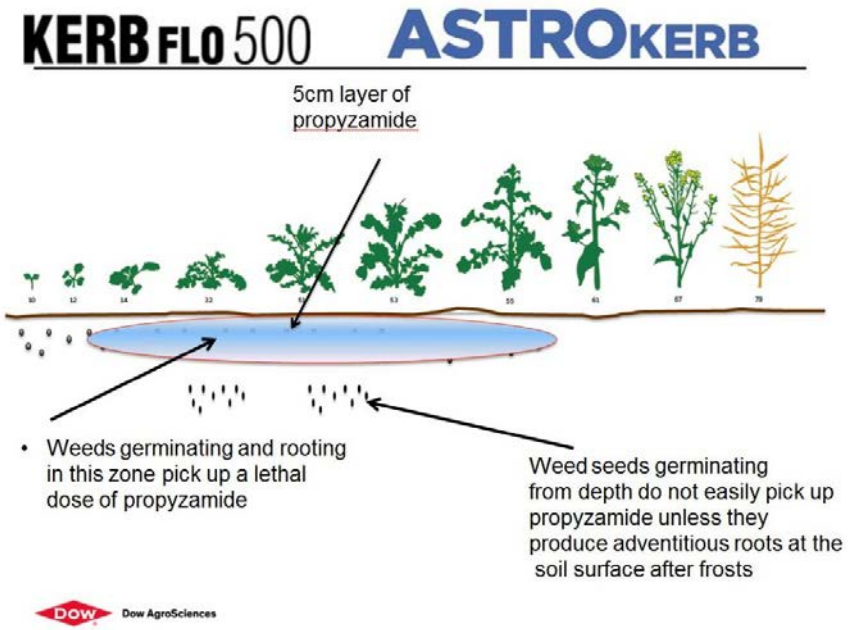
Propyzamide – an “alternative” herbicide

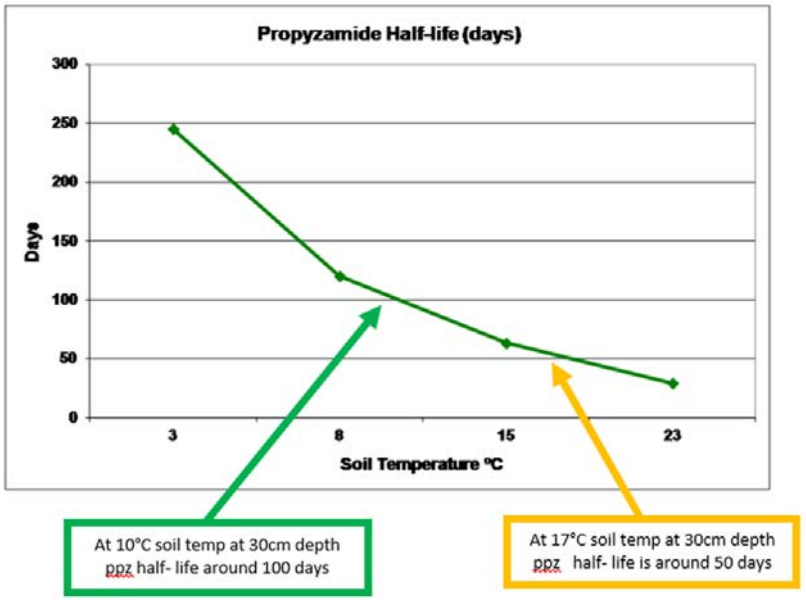
- *Commercial introduction: 1969 (Dow AgroScience: “Kerb®”)*
- *Selective systemic herbicide, absorbed by the roots*
- ***Targeted weeds:** Certain grasses; broad-leaved weeds including chickweed, mayweed, poppy, black bindweed, fat hen, speedwells*

- ***Uses**: Pre- and early post-emergence control of annual and perennial grass and some broad-leaved weeds, in fruit, vines, lettuce, endive, chicory, brassicas, oilseed rape, legumes, alfalfa, clover, ..., artichokes, sugar beets, ..., fallow land and forestry*

Sources: PPDB of the Universtity of Herfordshire (UK); The Pesticide Manual, BCPC (UK)

Propyzamide – an “alternative” herbicide





- *Actual results: f. ex. 0,02 mg/kg in kohlrabi (stem turnip)*

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Taking a closer look.

… keep an eye on …

… Herbicides …