

# Preliminary Results

## EU-RT-FV16

European Union  
Ring Test FV16  
Certified Standard  
Solutions



## DG SANCO

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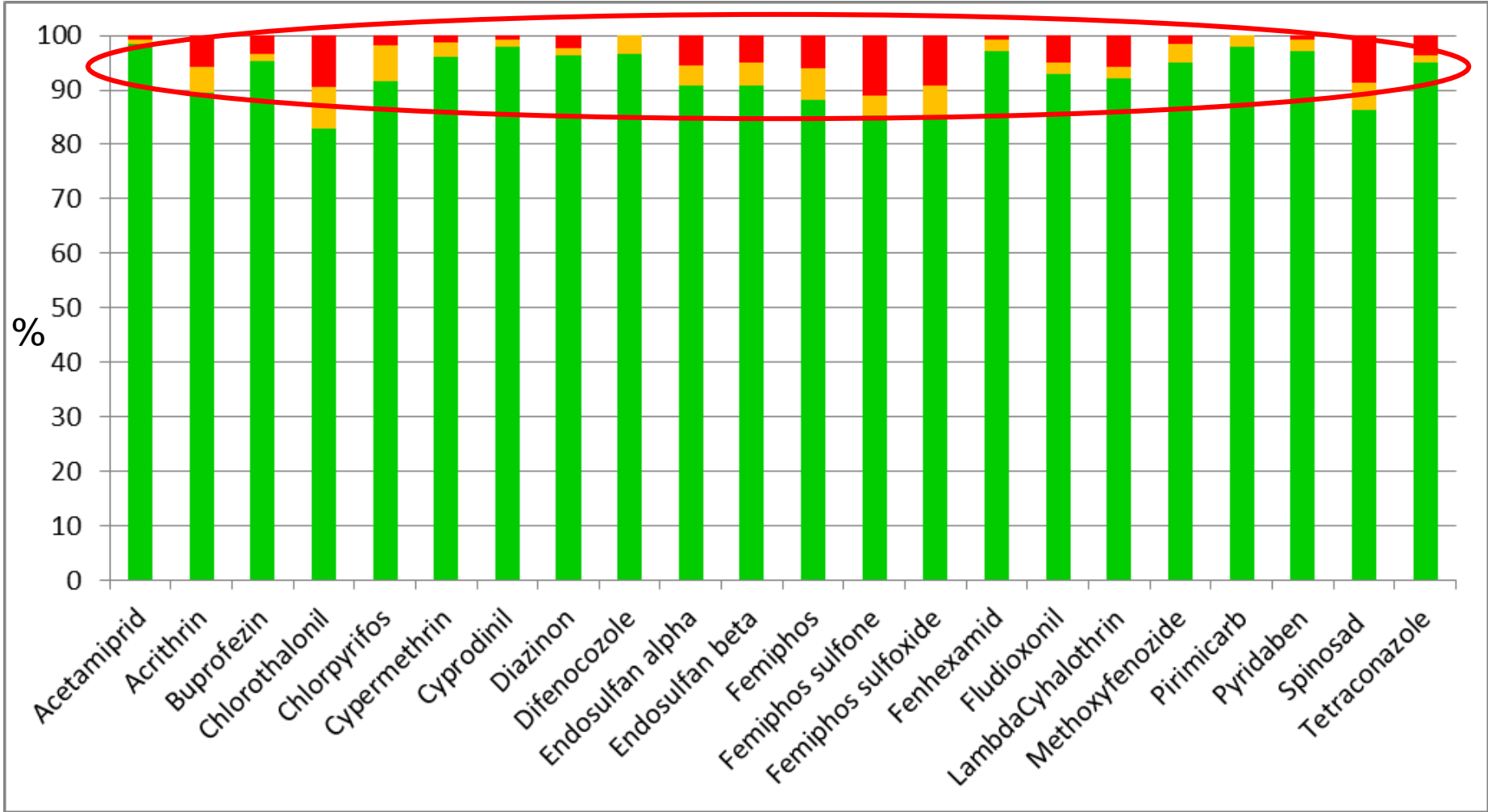
Dr. Sonja Masselter

Dr. Tuija Pihlström

Dr. Mette Erecius Poulsen

Dr. Darinka Stajnbaher

# Participation



**Acceptable**

**Questionable**

**Unacceptable**



## Pesticides evaluated in EUPT-FV16

Acetamiprid	Fenamiphos
Acrinathrin	Fenamiphos sulfone
Buprofezin	Fenamiphos sulfoxide
Chlorothalonil	Fenhexamid
Chlorpyrifos	Fludioxonil
Cypermethrin	Lambda-Cyhalothrin
Cyprodinil	Methoxyfenozide
Diazinon	Pirimicarb
Difenoconazole	Pyridaben
Endosulfan alpha	Spinosad
Endosulfan beta	Tetraconazole

**Total: 21**

# Preparation of the Standard solutions



External specialised firm

Certified Standard solutions

Acetonitrile

**CPAchem** Standard Solution 22 components  
 LOT No.: Z75861  
 Ref. No.: 7DD9.10.1NtrA.2.75  
 Expiry date: 07.2016

**C.P.A. Ltd.**  
 7-b-4 Han Asparuh Str.  
 6000 Stara Zagora, Bulgaria  
 tel/fax +359 42 607 716 www.cpachem.com

**PACKED UNDER NITROGENE**

Concentration 10 µg/ml each of:

Acetamidrid	Fludioxonil	Methoxyfenozide
Acrinathrin	Diazinon	Pyrimicarb
Buprofezin	Difenoconazole	Pyridaben
Fenamiphos-sulfone	Endosulfan-alpha	Spinosad
Chlorothalonil	Endosulfan-beta	Tetraconazole
Chlorpyrifos	Fenamiphos	Fenamiphos-sulfoxide
Cypermethrin	Fenhexamid	
Cyprodinil	lambda-Cyhalothrin	

Solv.: Acetonitrile / Acetone tr. Vol.: 2 ml

**FLASK DILUTION**  
 (fill here if diluted)

# Calendar

ACTIVITY	DATE
- Sample distribution.	23rd -27th June 2014
- Deadline for receiving results	21st July 2014
- Preliminary Report: only results.	30th July 2014
- Final Report	November 2014



[www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)



## PROTOCOL

### Ring Test Certified Standard Solutions FV16 (2014)

#### **Introduction**

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPTs) for Pesticide Residues in Food and Feed. This Proficiency Test is organised by the EURL for Pesticide Residues in Fruit and Vegetables covering Multiresidue Methods (MRM) of analysis. The aim of this ring test is to help the participant laboratories in EUPT-FV16 to check if their working standard solutions have affected the z scores obtained in EUPT

## Protocol-Steps to follow

The standard solutions have to be diluted 100 times (that way the concentration values will be between 10 and 500 µg/l). This dilution has to be performed following the laboratory's own procedures using the appropriate solvents for each analytical technique.





# Participation

**Total No. of Labs = 66**

**EU/EFTA Labs = 66**

**Other countries Labs = 0**

**Total No. of Countries = 23**

**EU/EFTA countries = 23**

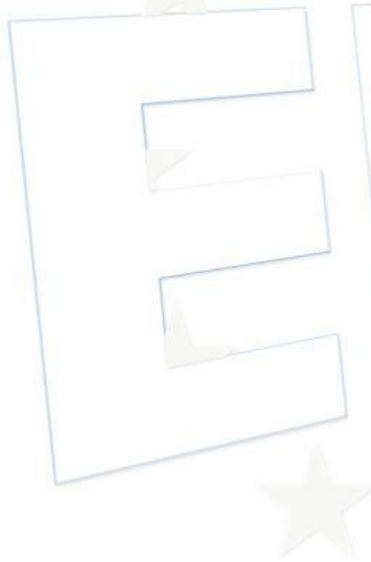
**Other countries = 0**

Austria	2
Belgium	2
Bulgaria	1
Croatia	3
Denmark	2
Estonia	1
Finland	1
France	4
Germany	7
Greece	4
Hungary	1

Iceland	1
ITALY	11
Latvia	1
Poland	3
Portugal	1
Romania	2
Slovakia	1
Spain	13
Switzerland	3
The Netherlands	1
UK	1

# Certified values

Approximately 10 mg/l

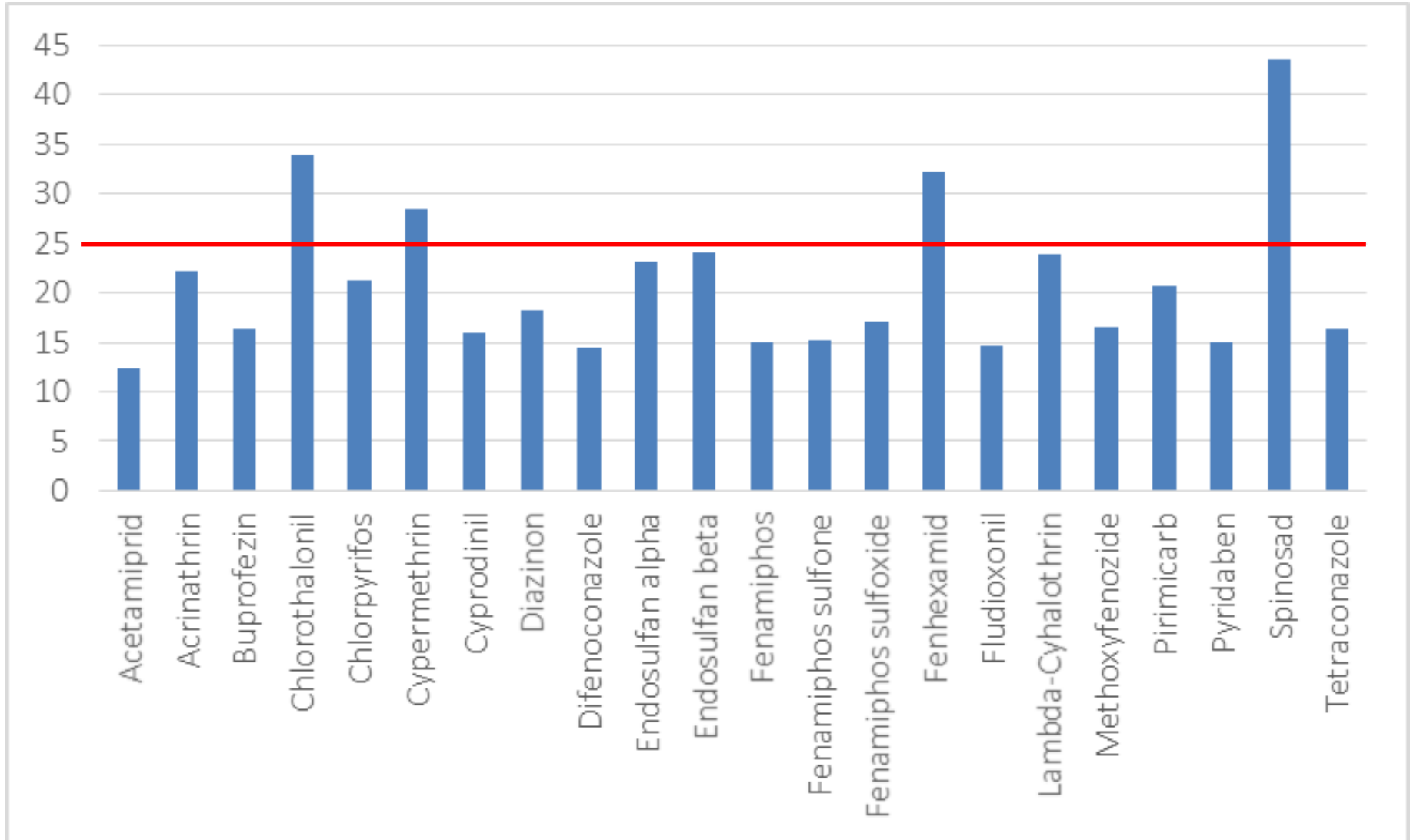


Pesticides	Certified value(mg/l)
Acetamiprid	9,972
Acrinathrin	10,021
Buprofezin	10,071
Chlorothalonil	10,044
Chlorpyrifos	9,975
Cypermethrin	10,030
Cyprodinil	10,012
Diazinon	10,026
Difenoconazole	10,035
Endosulfan alpha	9,995
Endosulfan beta	10,025
Fenamiphos	9,980
Fenamiphos sulfone	10,016
Fenamiphos sulfoxide	9,997
Fenhexamid	9,999
Fludioxonil	10,126
Lambda-Cyhalothrin	9,992
Methoxyfenozide	10,011
Pirimicarb	9,952
Pyridaben	10,043
Spinosad	10,045
Tetraconazole	10,073

# Dispersion of Results

Pesticides	Robust mean (mg/kg)	CV (%)
Acetamiprid	10,14	12,3
Acrinathrin	9,63	22,2
Buprofezin	9,9	16,4
Chlorothalonil	8,88	34,0
Chlorpyrifos	8,78	21,3
Cypermethrin	8,01	28,4
Cyprodinil	9,75	16,0
Diazinon	9,64	18,3
Difenoconazole	9,52	14,5
Endosulfan alpha	9,84	23,2
Endosulfan beta	9,38	24,0
Fenamiphos	10,18	15,1
Fenamiphos sulfone	10,55	15,2
Fenamiphos sulfoxide	10,60	17,1
Fenhexamid	8,98	32,3
Fludioxonil	9,59	14,6
Lambda-Cyhalothrin	9,15	23,9
Methoxyfenozide	9,72	16,5
Pirimicarb	10,13	20,7
Pyridaben	9,02	15,1
Spinosad	11,58	43,6
Tetraconazole	10,14	16,4



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Acetamiprid	10,14	12,3
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# Dispersion of Results

Pesticides	CV (%) 	CV (%) EUPT-FV16 
Acetamiprid	12,3	15,9
Acrinathrin	22,2	18,6
Buprofezin	16,4	13,6
Chlorothalonil	34,0	34,4
Chlorpyrifos	21,3	20,4
Cypermethrin	28,4	16,4
Cyprodinil	16,0	14,6
Diazinon	18,3	16,9
Difenoconazole	14,5	18,7
Endosulfan alpha	23,2	17,3
Endosulfan beta	24,0	18,4
Fenamiphos	15,1	21,0
Fenamiphos sulfone	15,2	19,3
Fenamiphos sulfoxide	17,1	23,6
Fenhexamid	32,3	19,8
Fludioxonil	14,6	18,9
Lambda-Cyhalothrin	23,9	19,7
Methoxyfenozide	16,5	21,1
Pirimicarb	20,7	14,6
Pyridaben	15,1	18,2
Spinosad	43,6	25,2
Tetraconazole	16,4	16,0

Taking into account all EUPT-FV16 population (169 participants)

# Dispersion of Results

Pesticides	CV (%)	CV (%) EUPT-FV16
Acetamiprid	12,3	
Acrinathrin	22,2	
Buprofezin	16,4	
Chlorothalonil	34,0	
Chlorpyrifos	21,3	
Cypermethrin	28,4	21,6
Cyprodinil	16,0	
Diazinon	18,3	
Difenoconazole	14,5	
Endosulfan alpha	23,2	
Endosulfan beta	24,0	
Fenamiphos	15,1	
Fenamiphos sulfone	15,2	
Fenamiphos sulfoxide	17,1	
Fenhexamid	32,3	19,5
Fludioxonil	14,6	
Lambda-Cyhalothrin	23,9	
Methoxyfenozide	16,5	
Pirimicarb	20,7	
Pyridaben	15,1	
Spinosad	43,6	
Tetraconazole	16,4	

Taking into account only EU-RT-FV16 participants

# PRELIMINARY RESULTS





## Z-Scores

$$z_i = (x_i - \mu_i) / \delta_i$$

$x_i$  is the value reported by the laboratory

$\mu_i$  the assigned value

$\delta_i$  the standard deviation for each pesticide

$$\delta_i = b * \mu_i \text{ with } b = 0.25 \text{ (25 \% FFP-RSD)}$$



For EUPTs

## Z-Scores

$$z_i = (x_i - \mu_i) / \delta_i$$

$x_i$  is the value reported by the laboratory

$\mu_i$  the assigned value

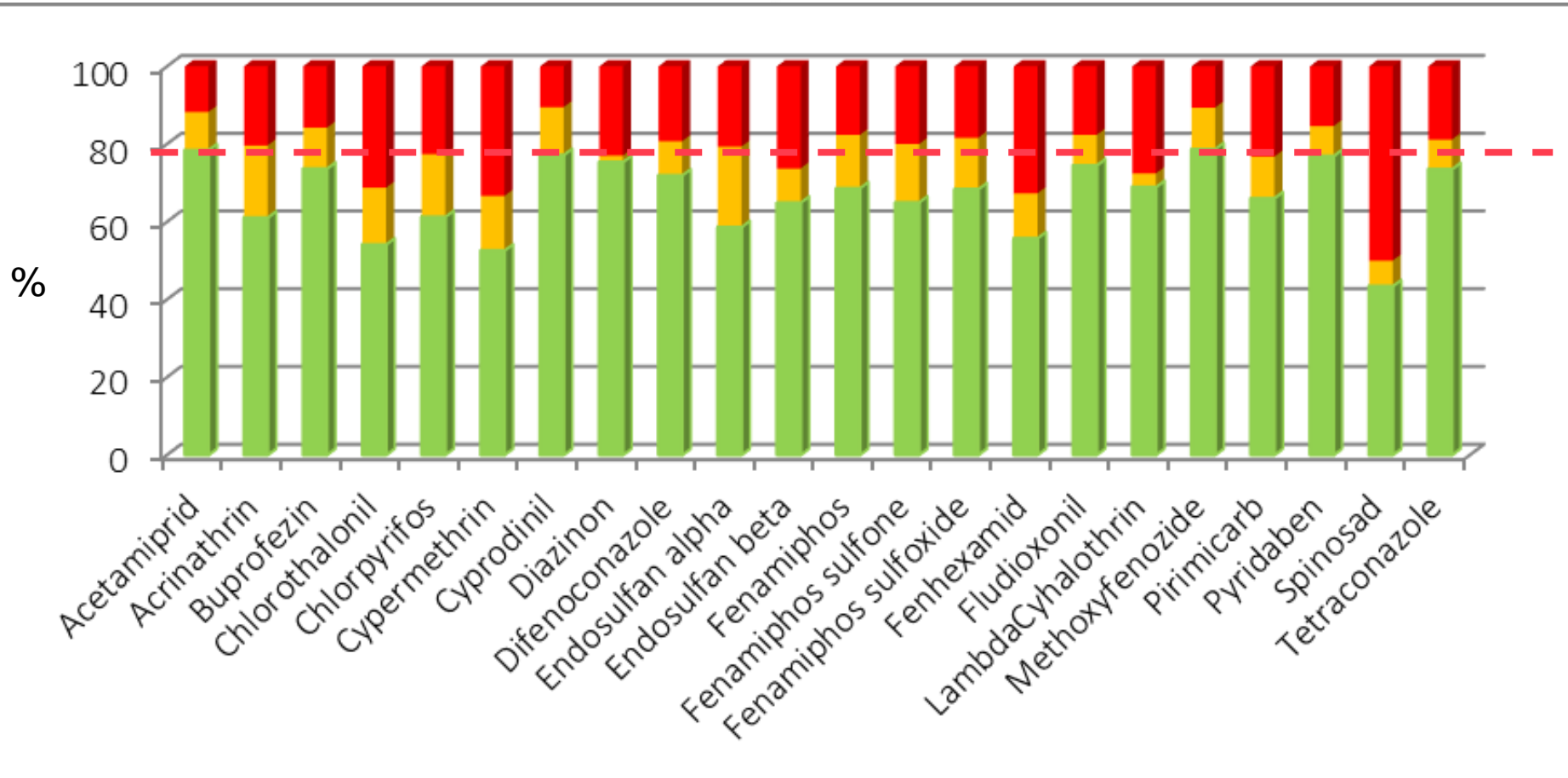
$\delta_i$  the standard deviation for each pesticide

$$\delta_i = b * \mu_i \text{ with } b = 0.1 \text{ (10 \% FFP-RSD)}$$



For Standard Solutions

## z-Scores classification



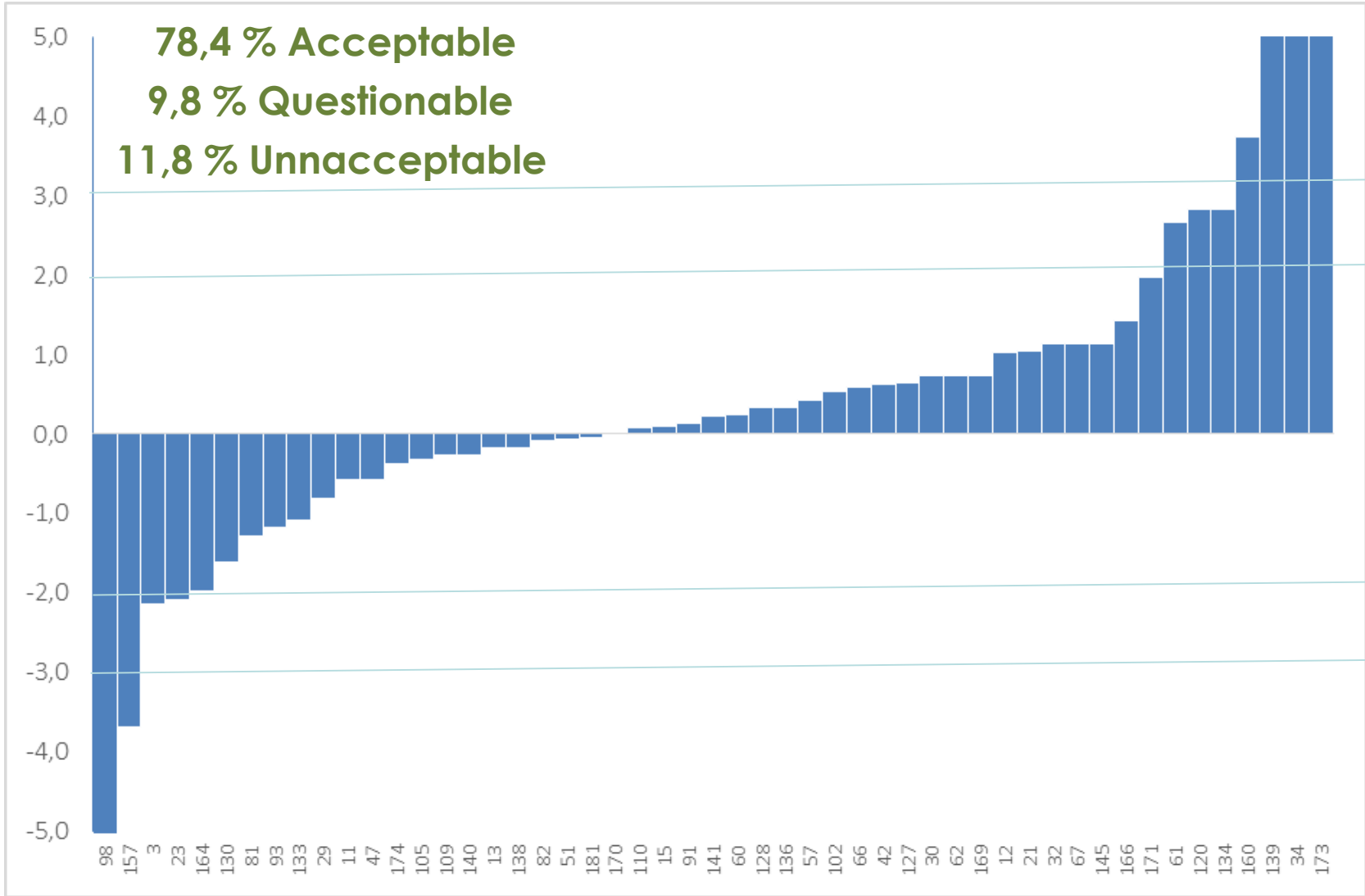
Acceptable

Questionable

Unacceptable

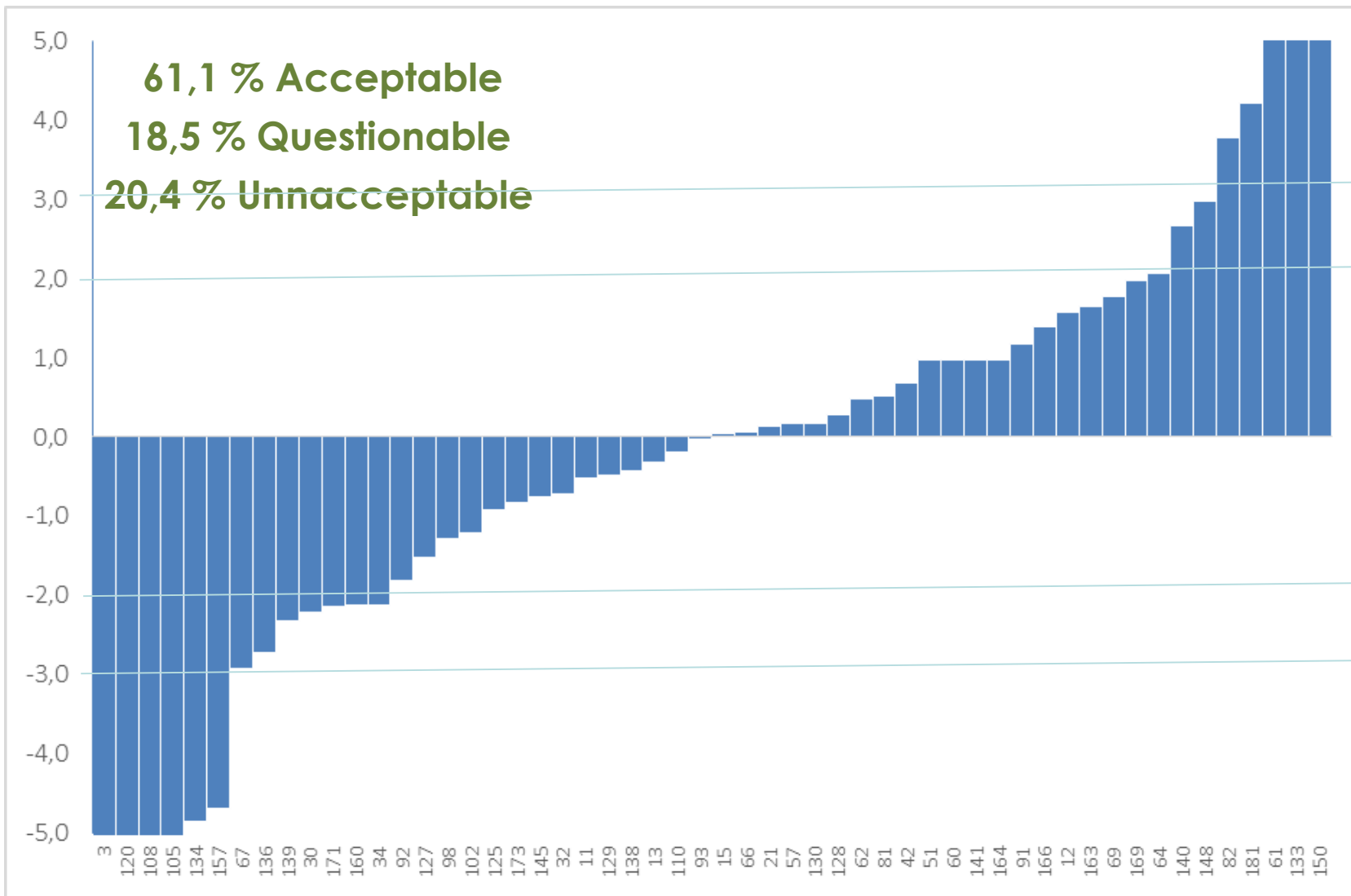
Pesticides	% Acceptable z-scores	% Questionable z-scores	% Unacceptable z-scores
Acetamiprid	78,4	9,8	11,8
Acrinathrin	61,1	18,5	20,4
Buprofezin	73,7	10,5	15,8
Chlorothalonil	54,2	14,6	31,3
Chlorpyrifos	61,3	16,1	22,6
Cypermethrin	52,6	14,0	33,3
Cyprodinil	77,2	12,3	10,5
Diazinon	75,4	1,6	23,0
Difenoconazole	71,9	8,8	19,3
Endosulfan alpha	58,6	20,7	20,7
Endosulfan beta	64,9	8,8	26,3
Fenamiphos	68,6	13,7	17,6
Fenamiphos sulfone	65,0	15,0	20,0
Fenamiphos sulfoxide	68,4	13,2	18,4
Fenhexamid	55,8	11,5	32,7
Fludioxonil	74,5	7,8	17,6
LambdaCyhalothrin	69,0	3,4	27,6
Methoxyfenozide	78,8	10,6	10,6
Pirimicarb	66,1	10,7	23,2
Pyridaben	76,9	7,7	15,4
Spinosad	43,5	6,5	50,0
Tetraconazole	73,6	7,5	18,9

# Acetamiprid

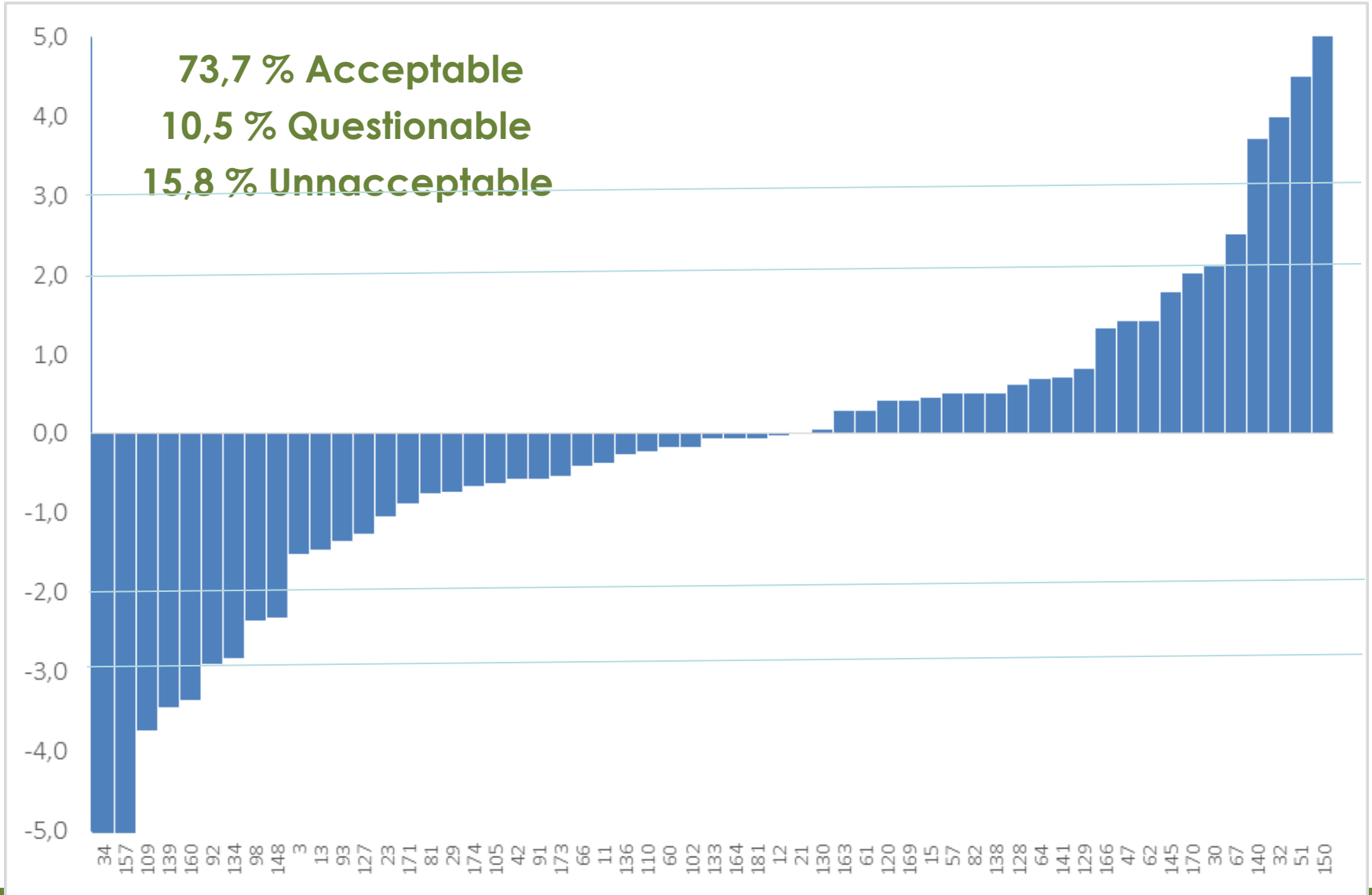




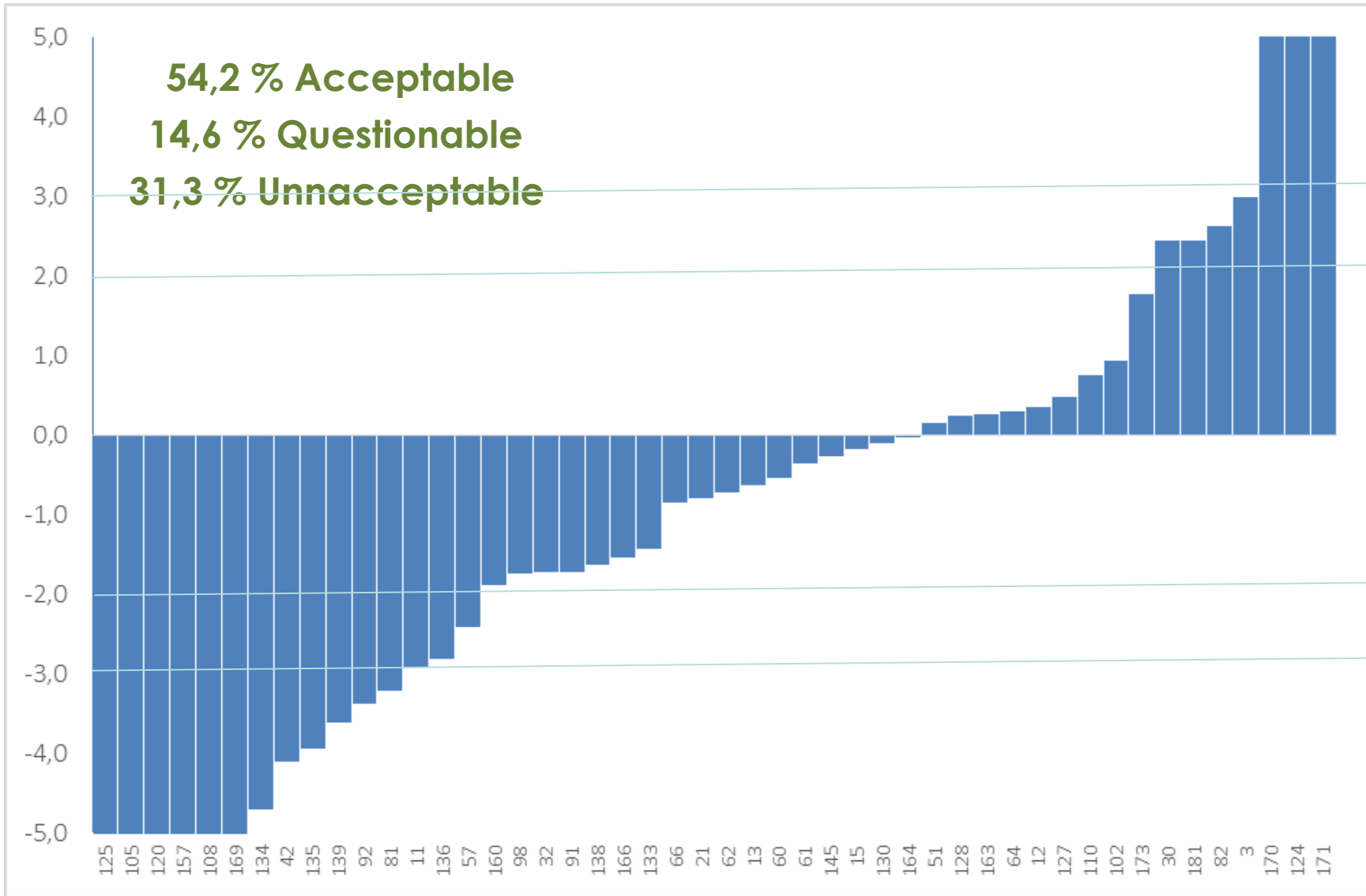
# Acrinathrin



# Buprofezin

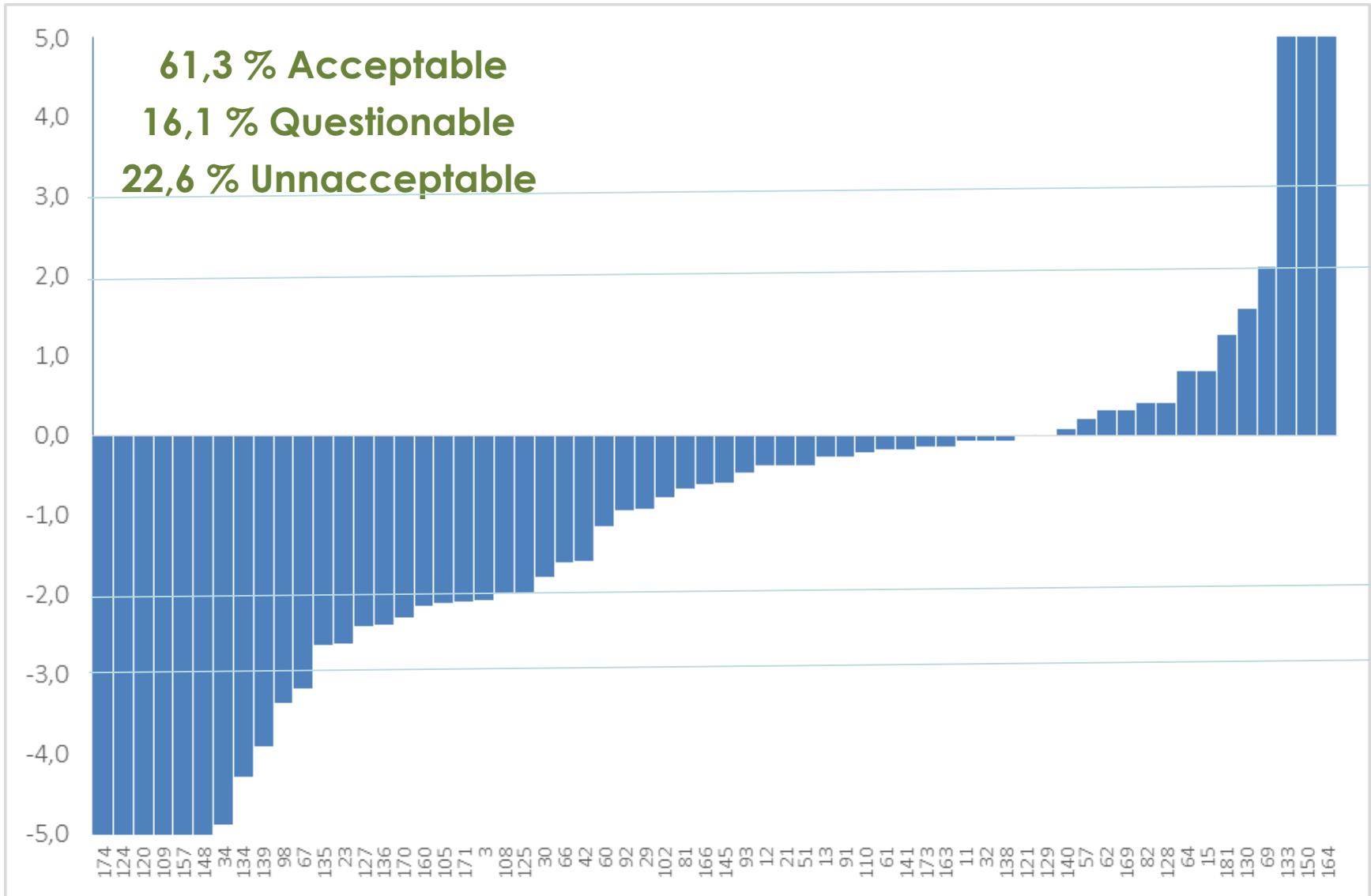


# Chlorothalonil

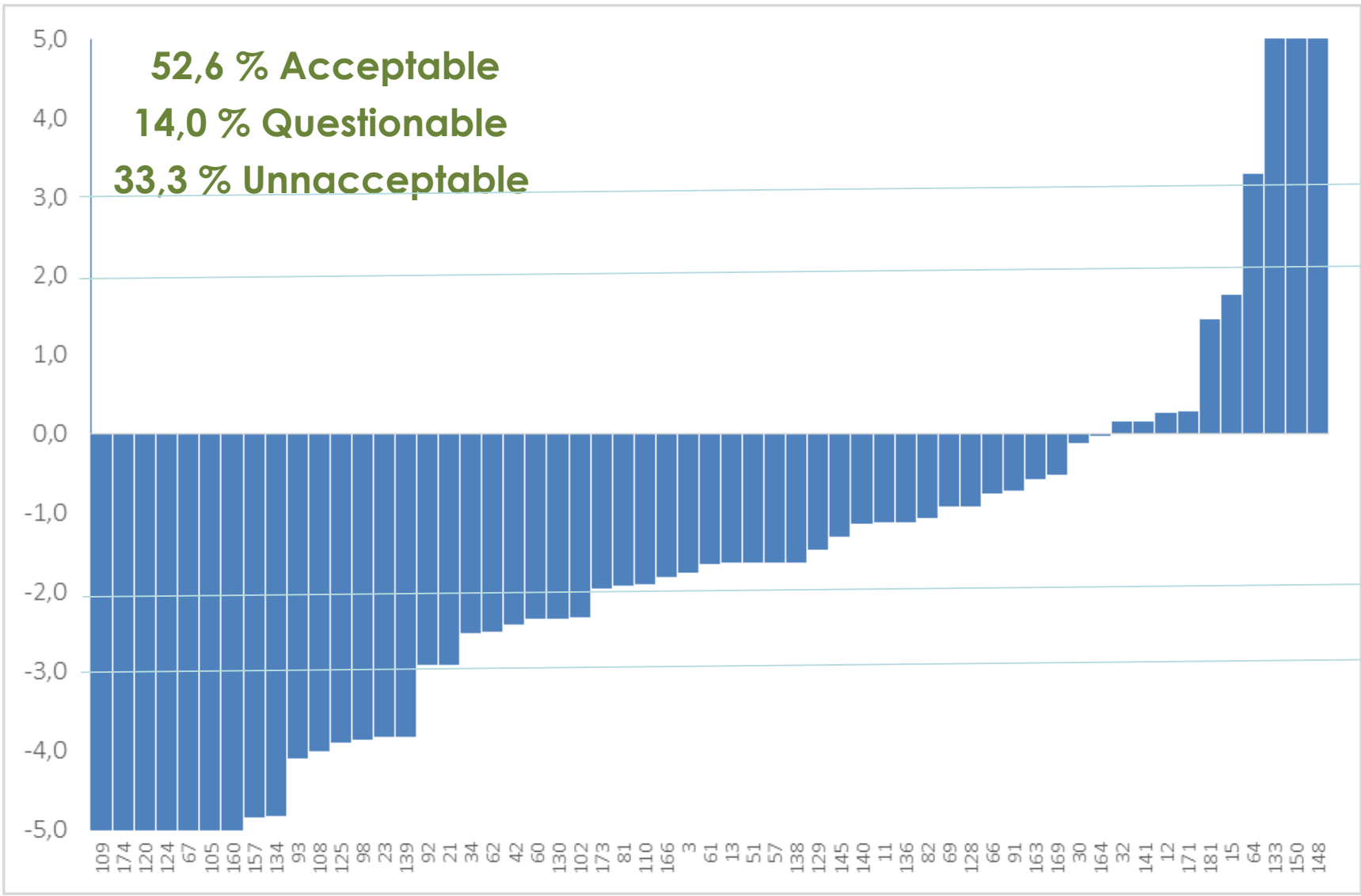




# Chlorpyrifos

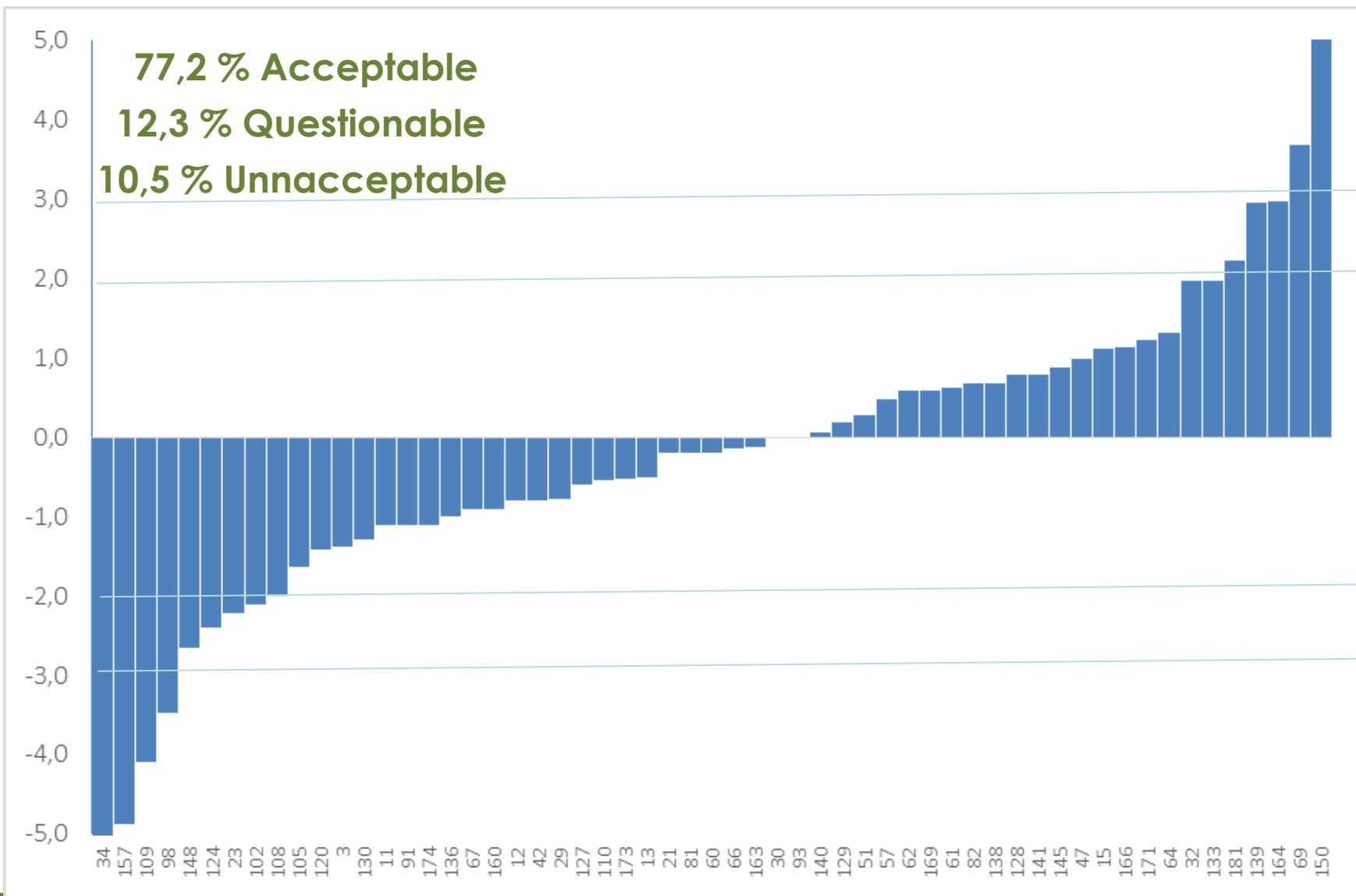


# Cypermethrin

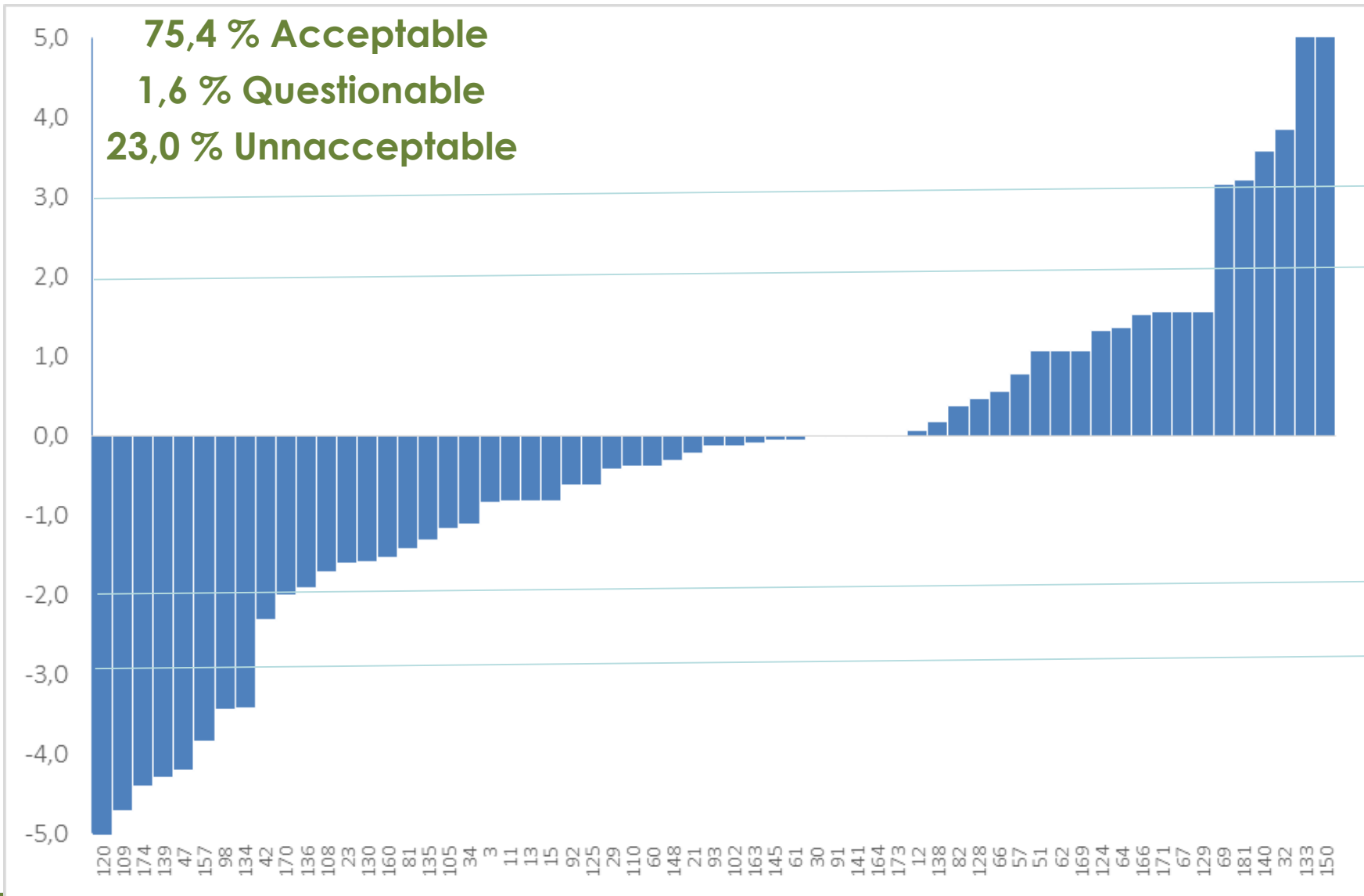




# Cyprodinil

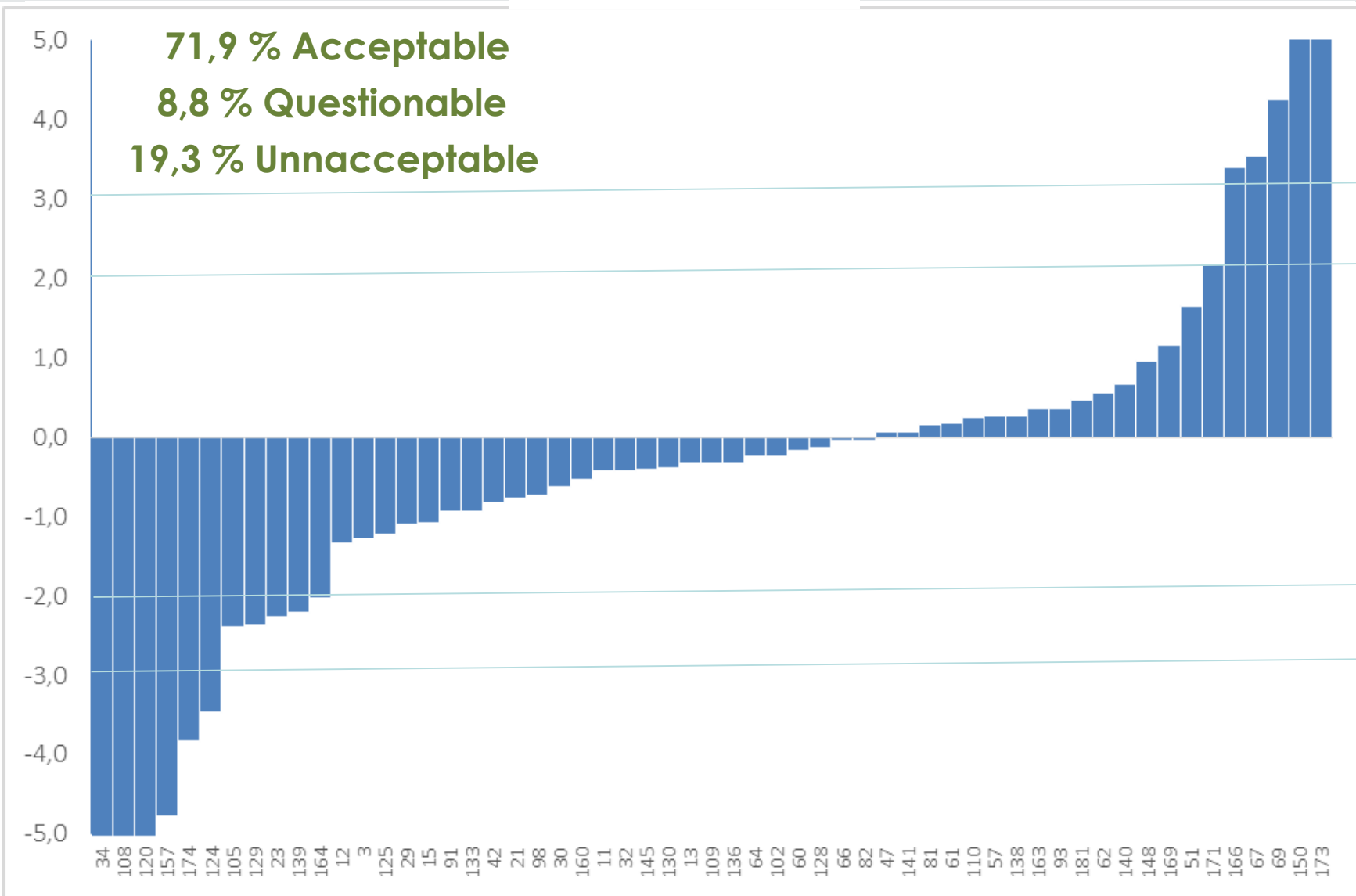


# Diazinon



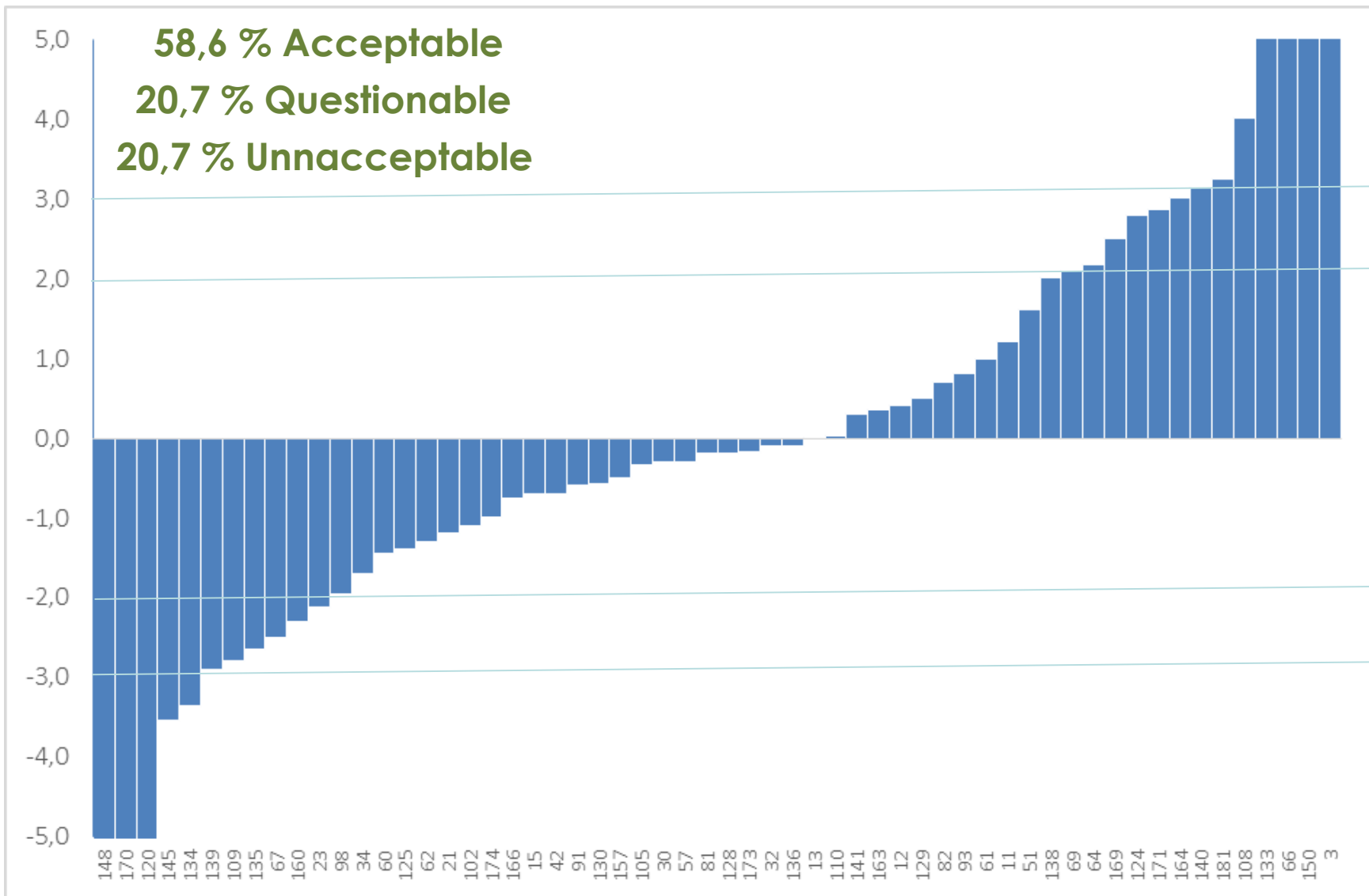


# Difenoconazole



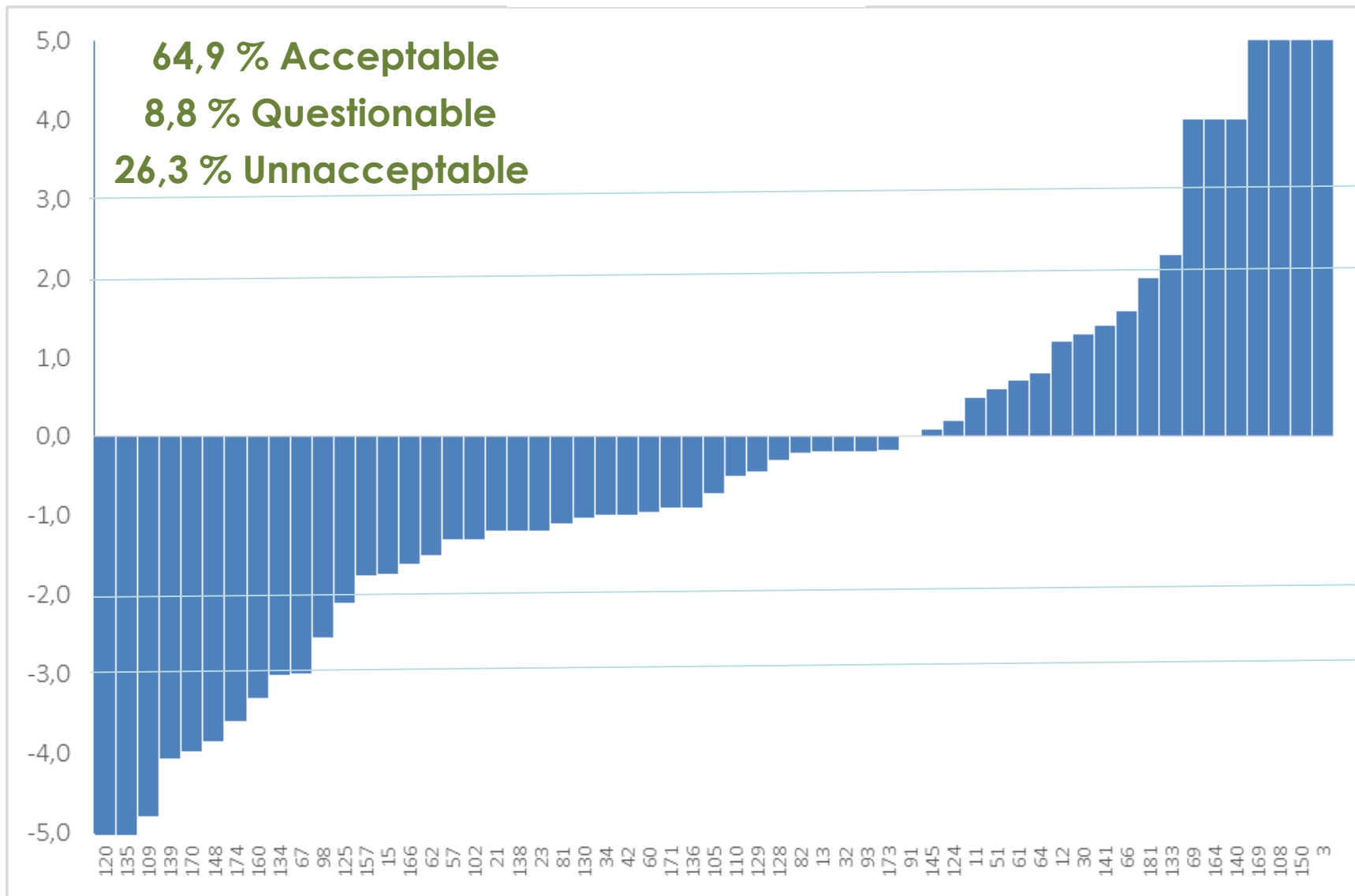


# Endosulfan alpha



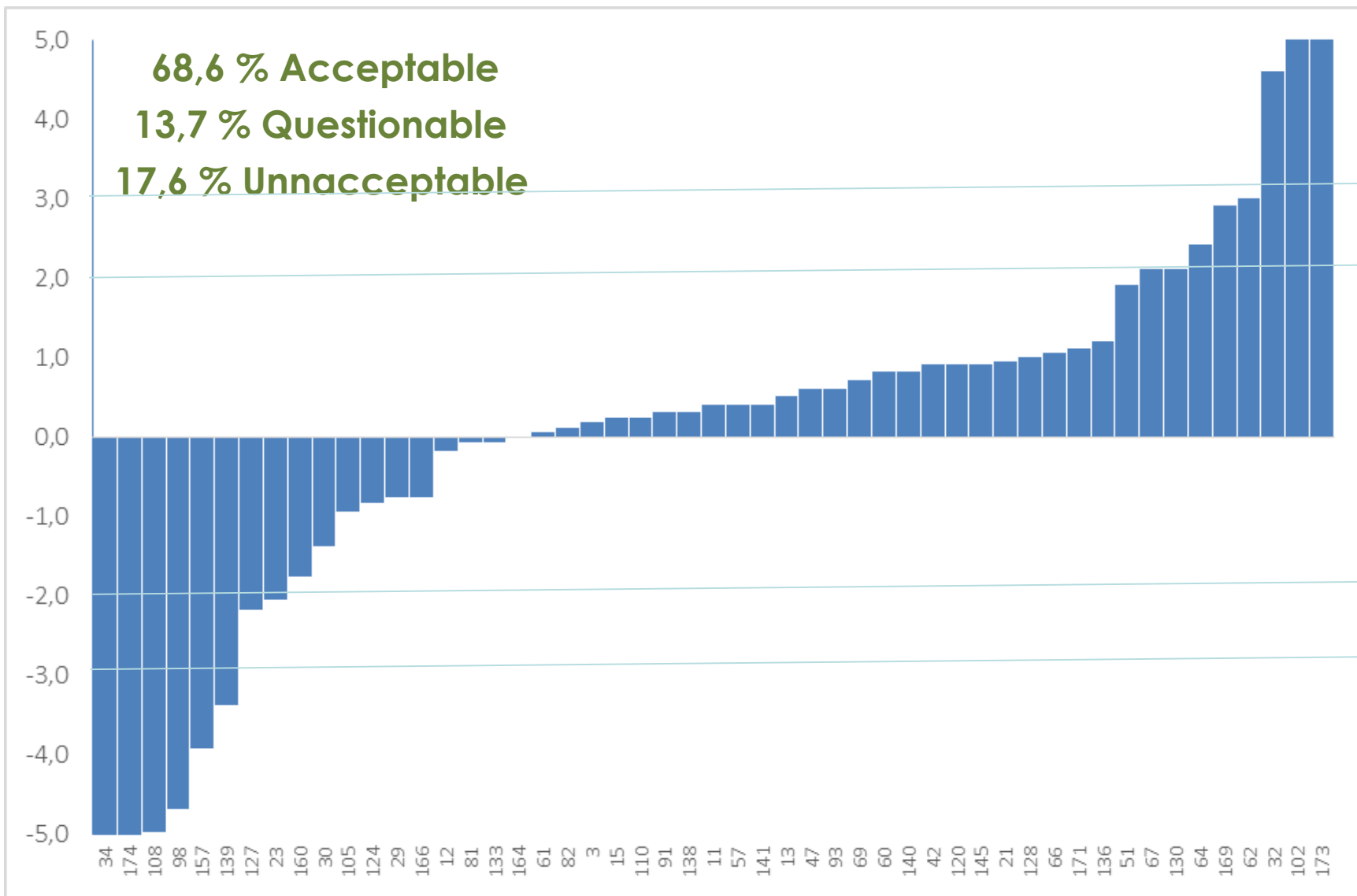


# Endosulfan Beta





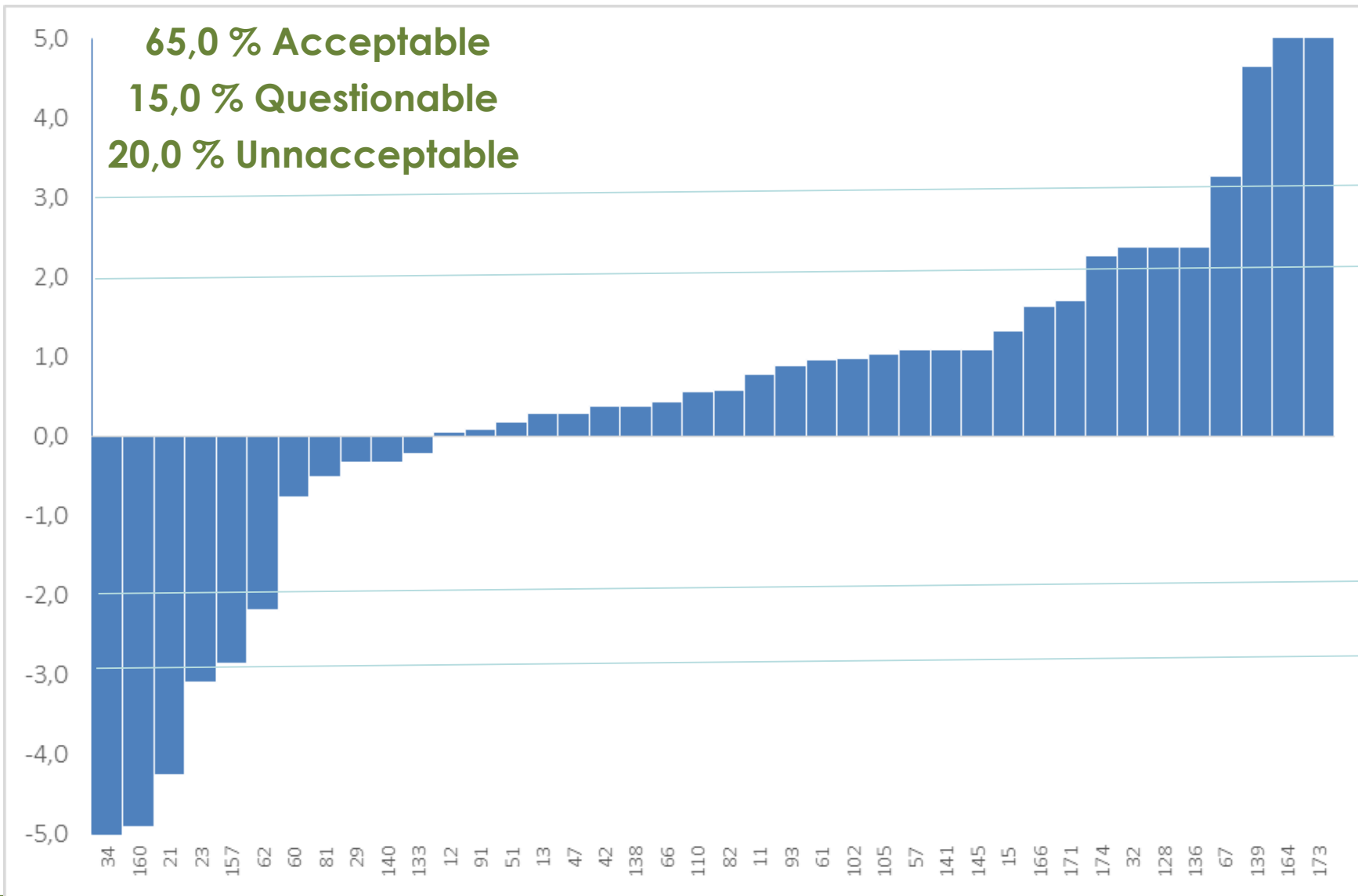
# Fenamiphos





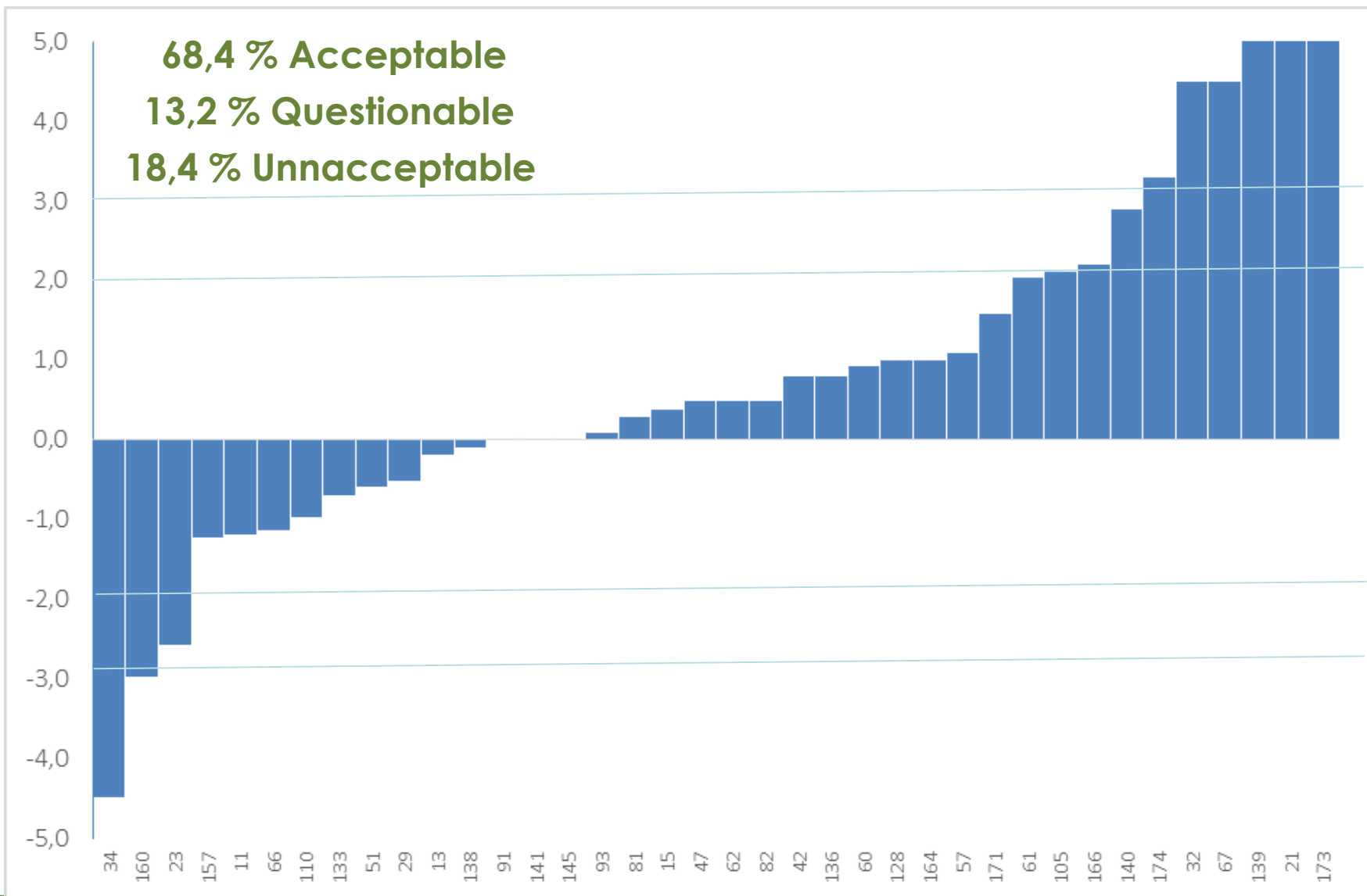


# Fenamiphos sulfone



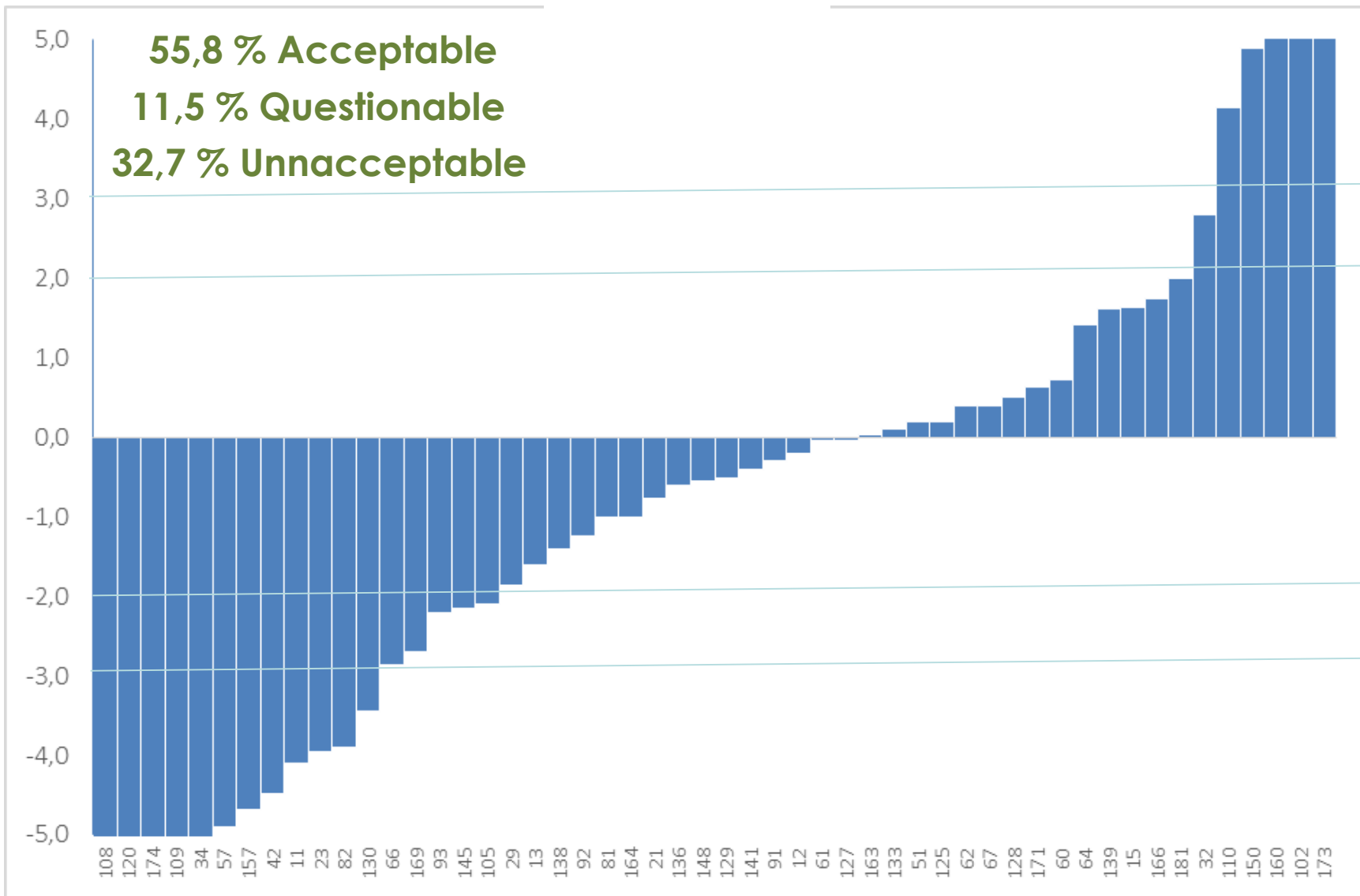


# Fenamiphos sulfoxide

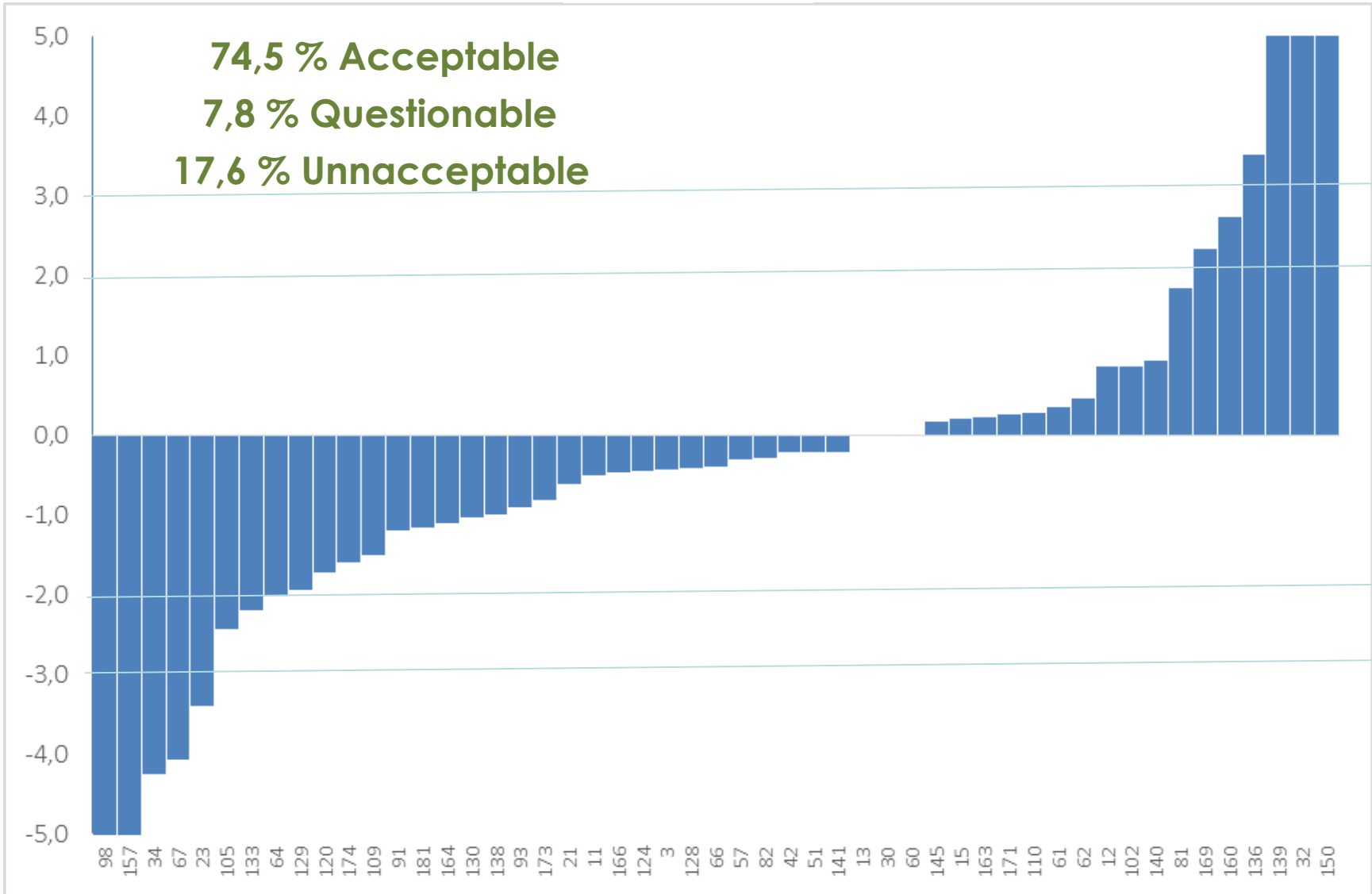




# Fenhexamid

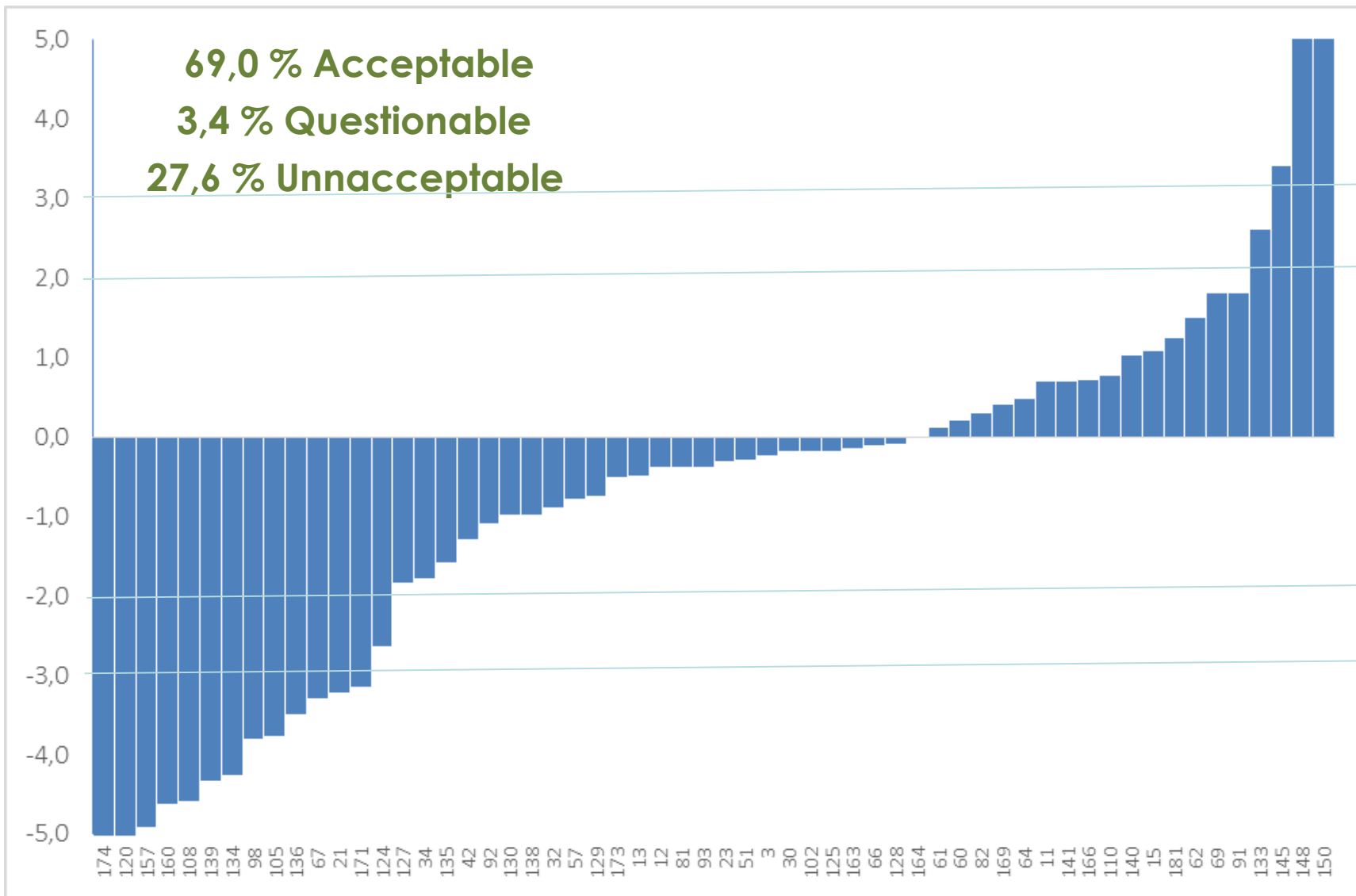


# Fludioxonil



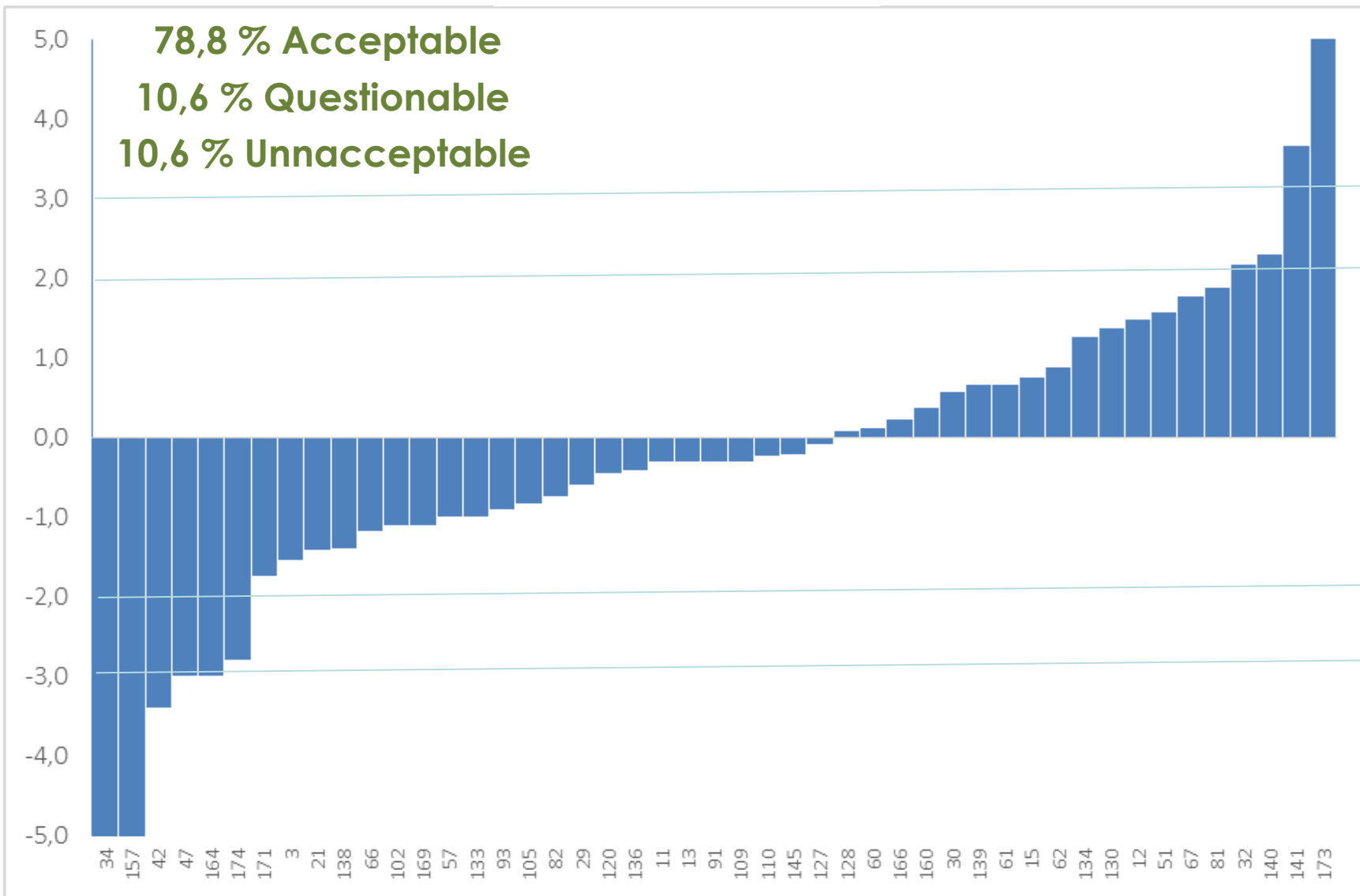


# Lambda-Cyhalothrin



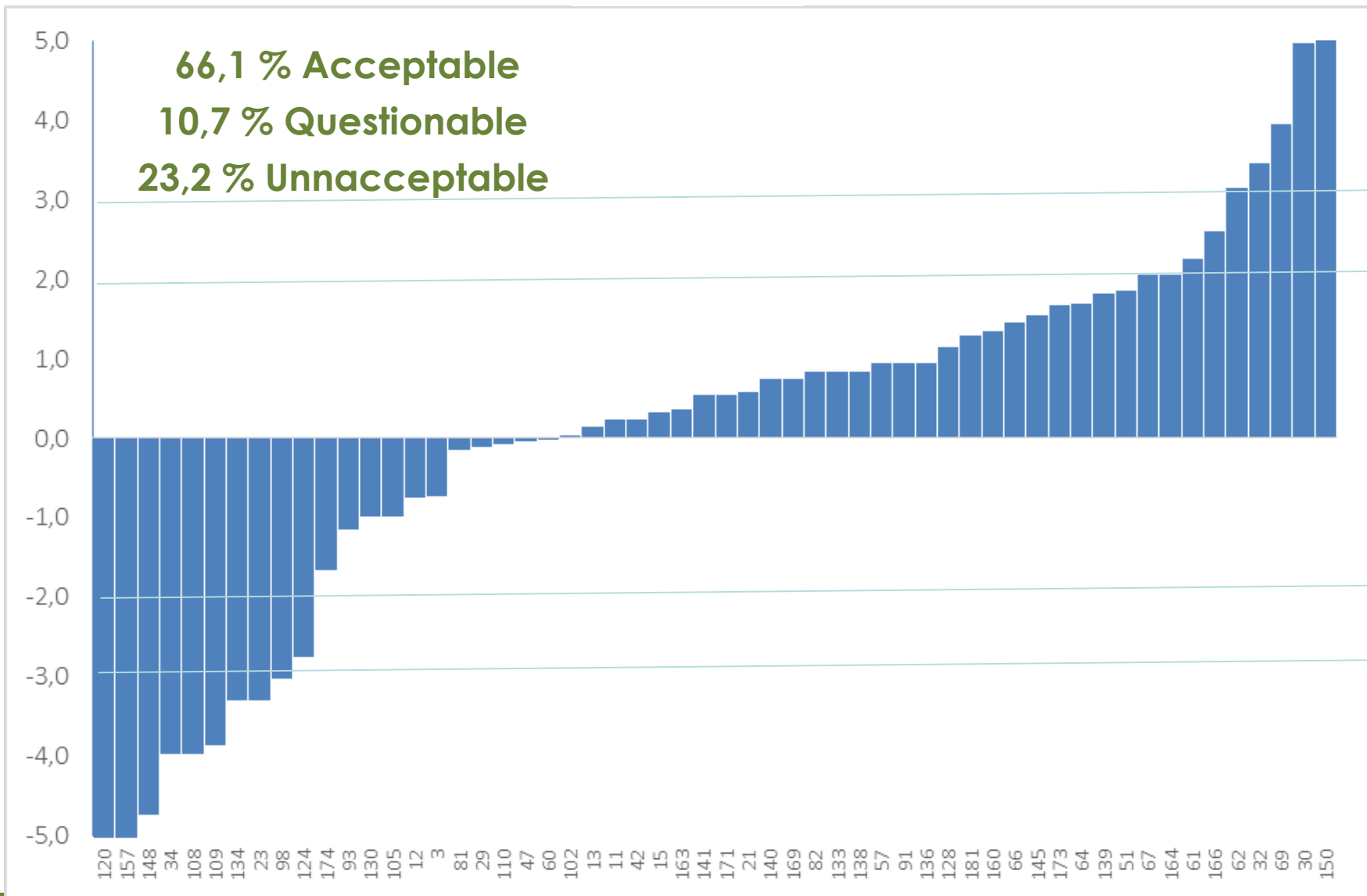


# Methoxyfenozide



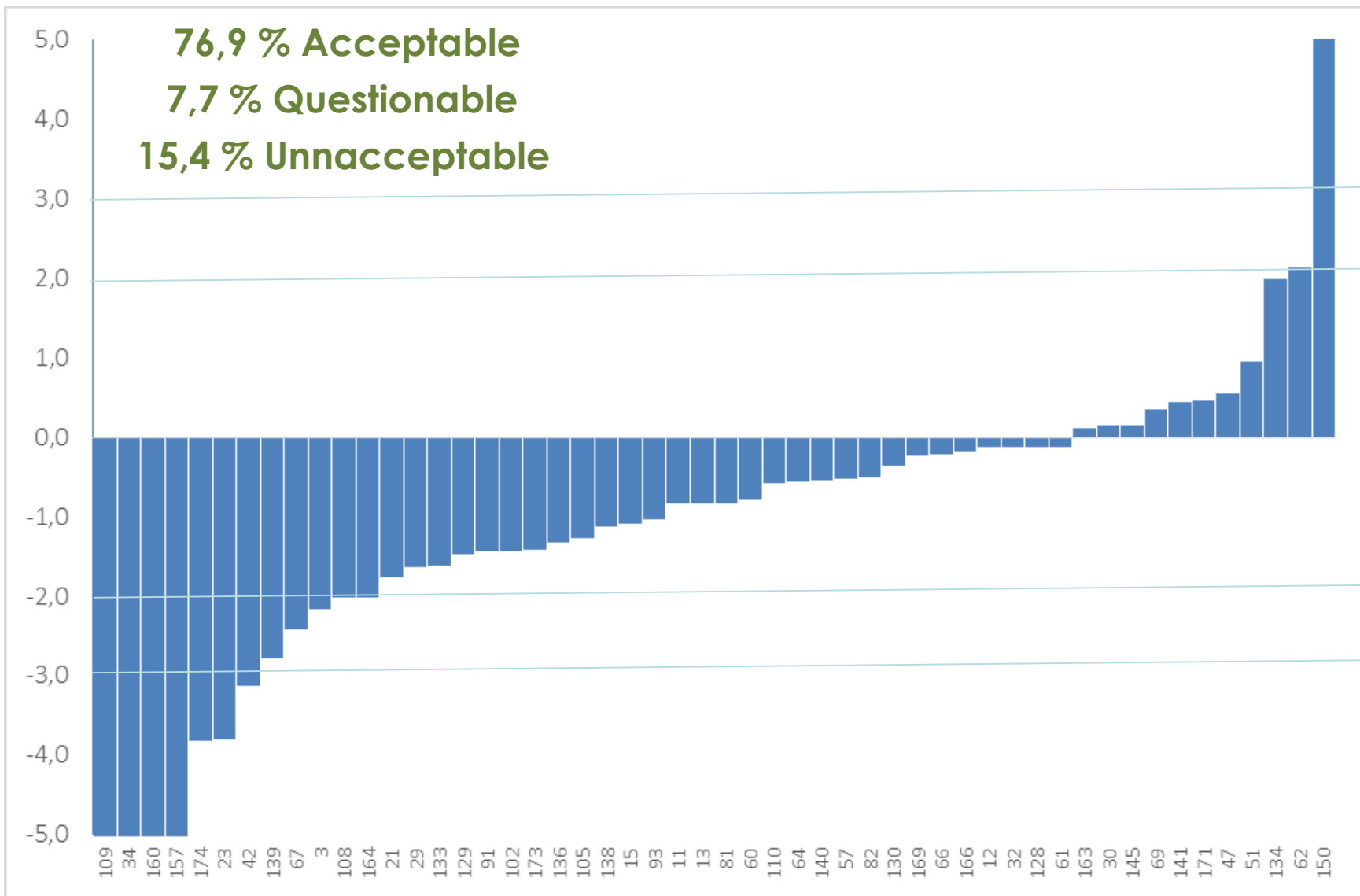


# Pirimicarb





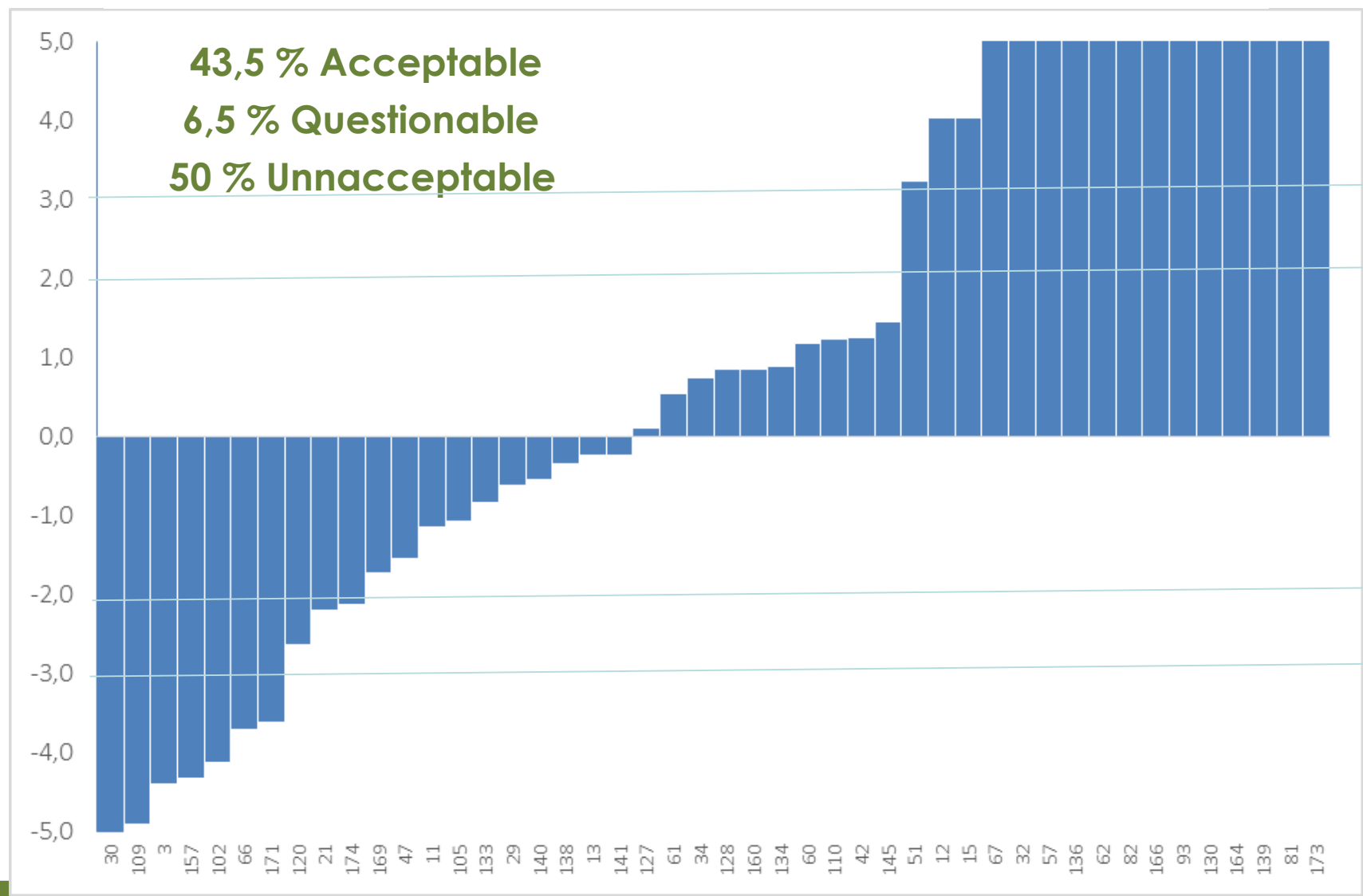
# Pyridaben



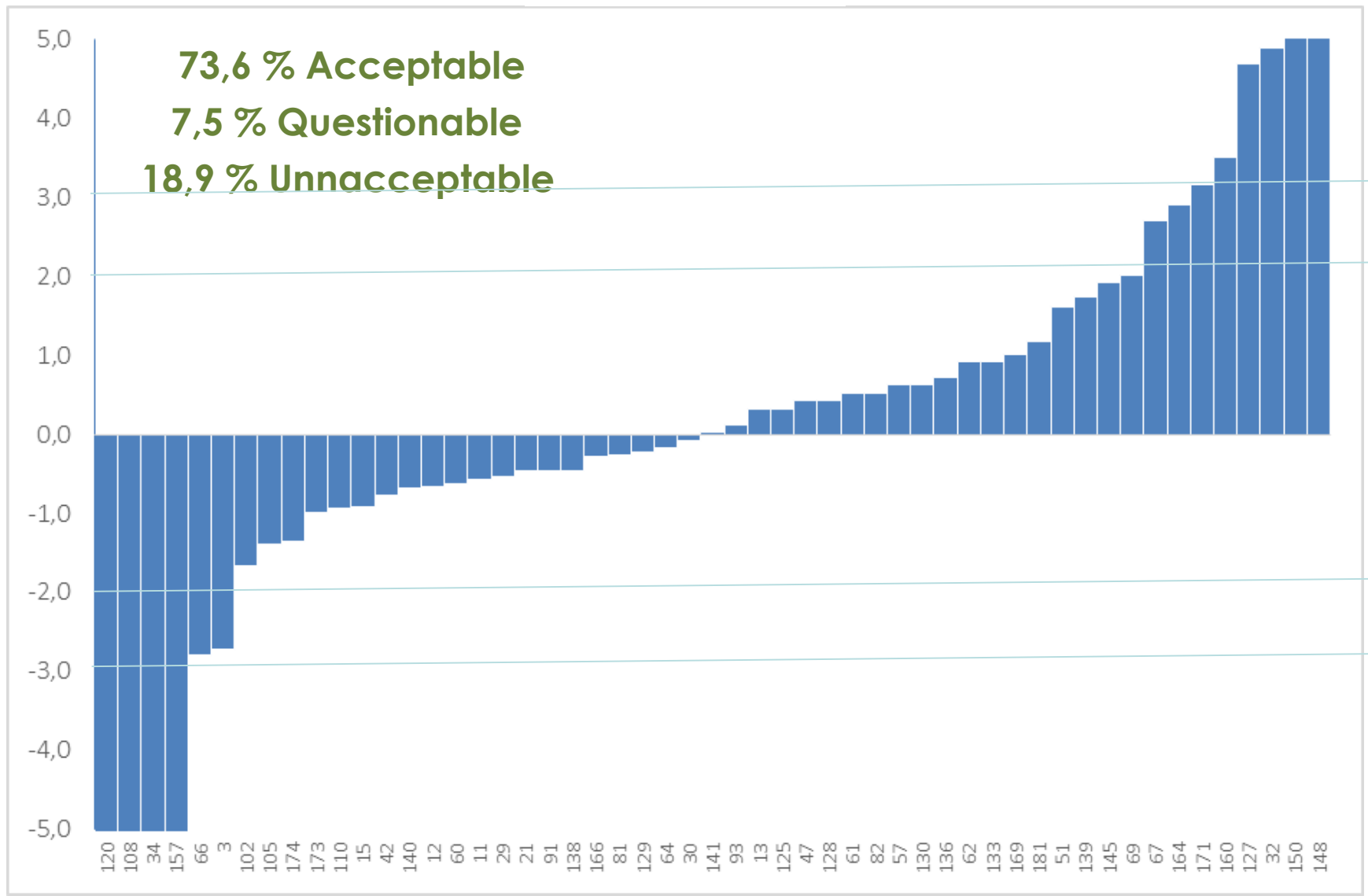


# Spinosad

(sum of spinosyn A and spinosyn D, expr. as spinosad)



# Tetraconazole





**THANK YOU  
FOR YOUR ATTENTION**