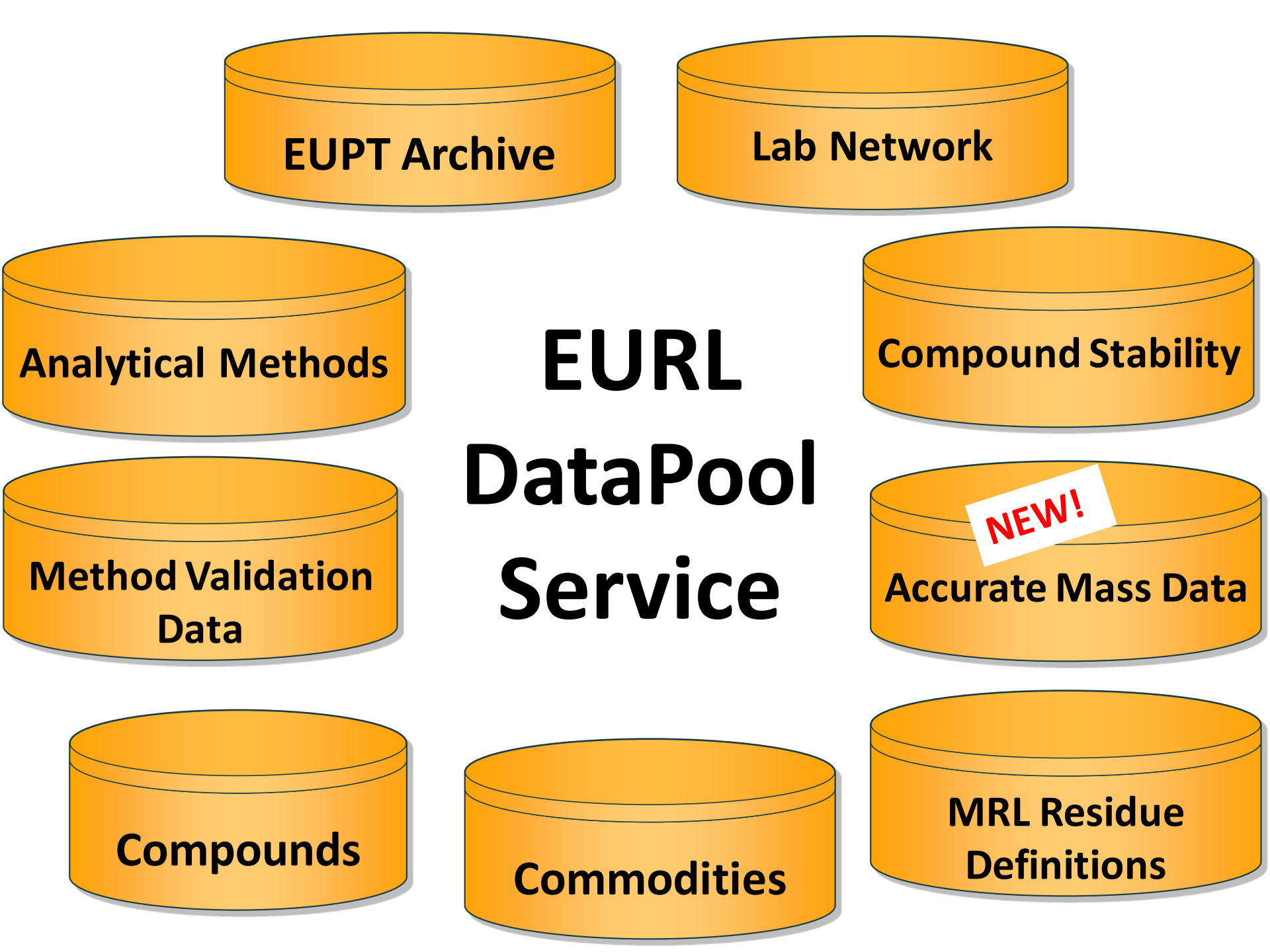
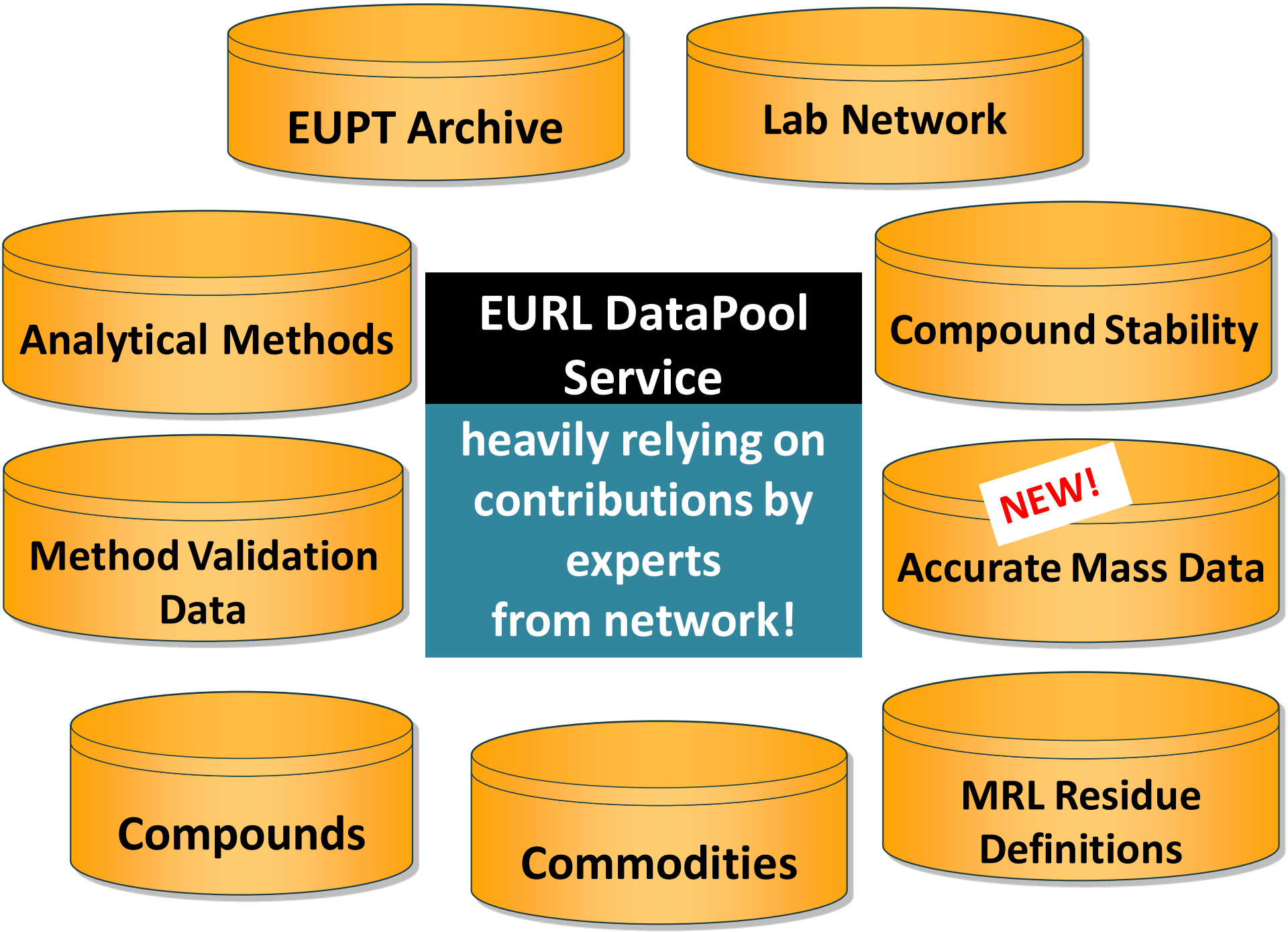
News on EURL DataPool Service

Outline

- **Accurate Mass Data DB**
- **PestiPedia**
- **Analytical Capabilities on Pesticides** (WD SANCO/12745/2013 rev. 9(1))
- **Update of official sample scope**

- CS2 Data Collection Project
- **Outlier-Test in Method Validation DB**


EUPT Archive
Lab Network
Analytical Methods
EURL DataPool Service
Compound Stability
Method Validation Data
NEW!
Accurate Mass Data
Compounds
Commodities
MRL Residue Definitions


EUPT Archive
Lab Network
Analytical Methods
EURL DataPool Service
heavily relying on contributions by experts from network!
Compound Stability
Method Validation Data
NEW!
Accurate Mass Data
Compounds
Commodities
MRL Residue Definitions

Tools offered

Lab Networking

Surveys

Search Tool for Accurate Mass Data

PestiPedia

Check myScope

EUPT Registration

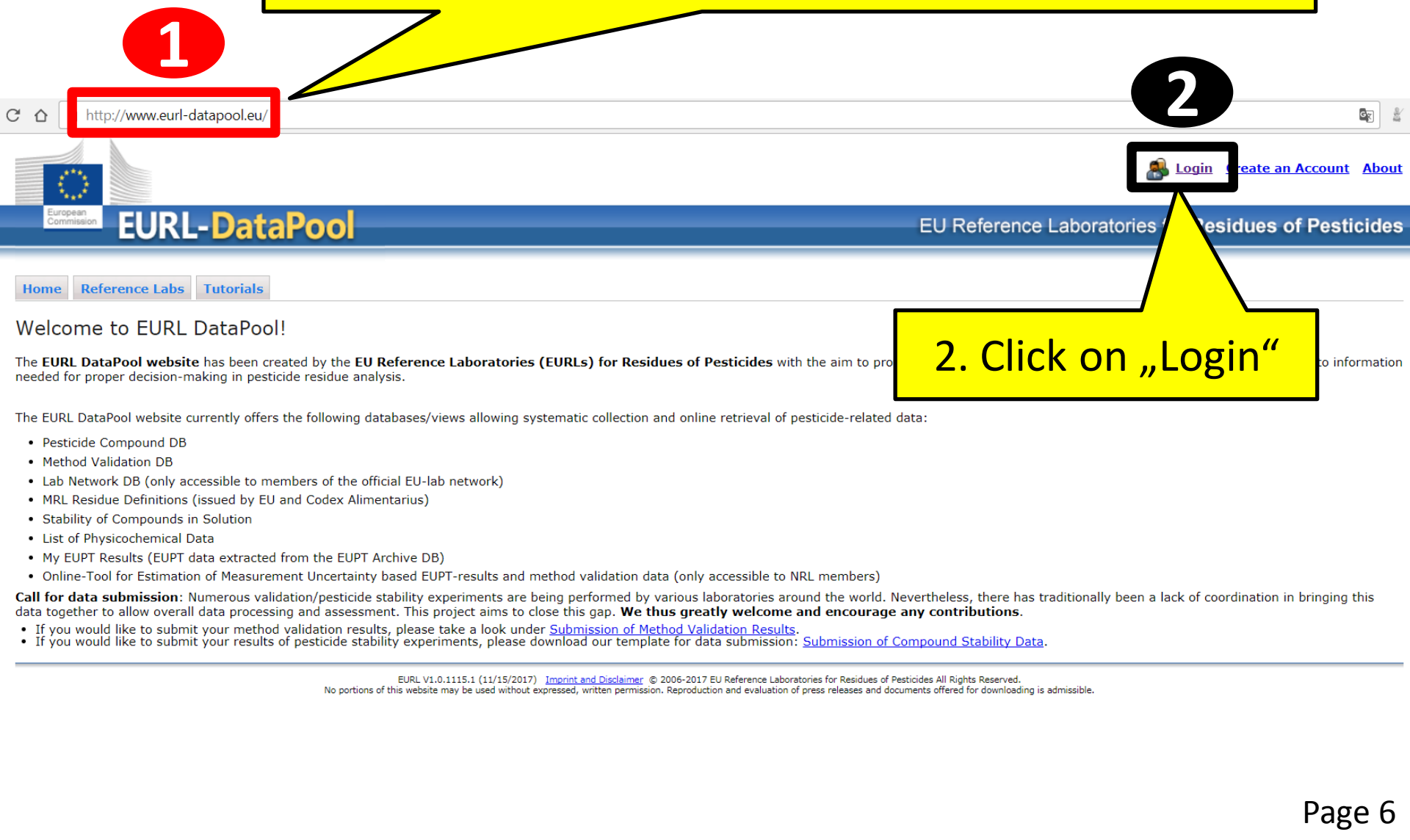
MU Estimation
(based on PT-results & validation data)

Calculation of MRL Residue Definition's Sum

EURL DataPool Service

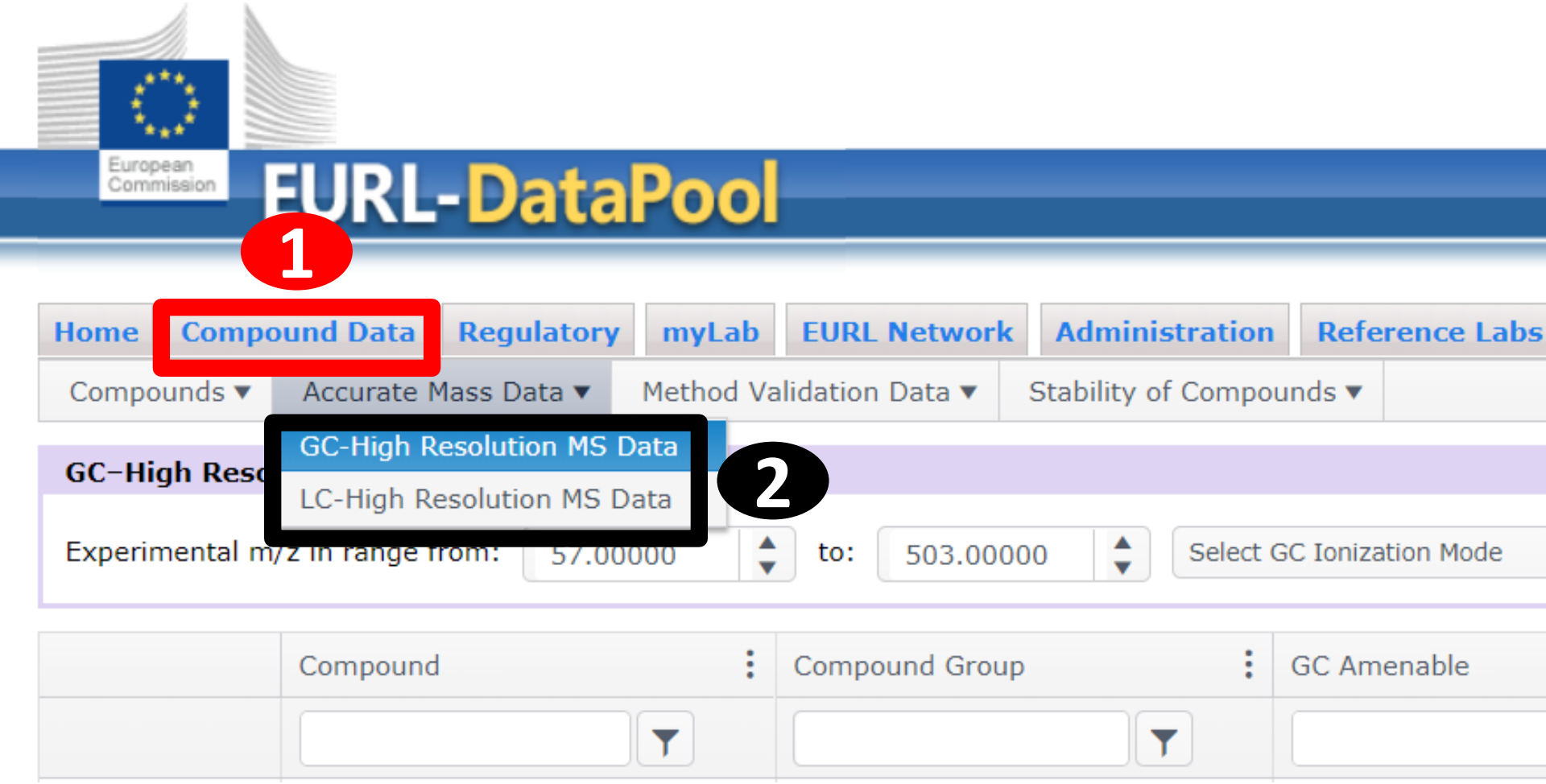
EURL DataPool | Login required

1. Go to „**https://www.eurl-pesticides-datapool.eu**“

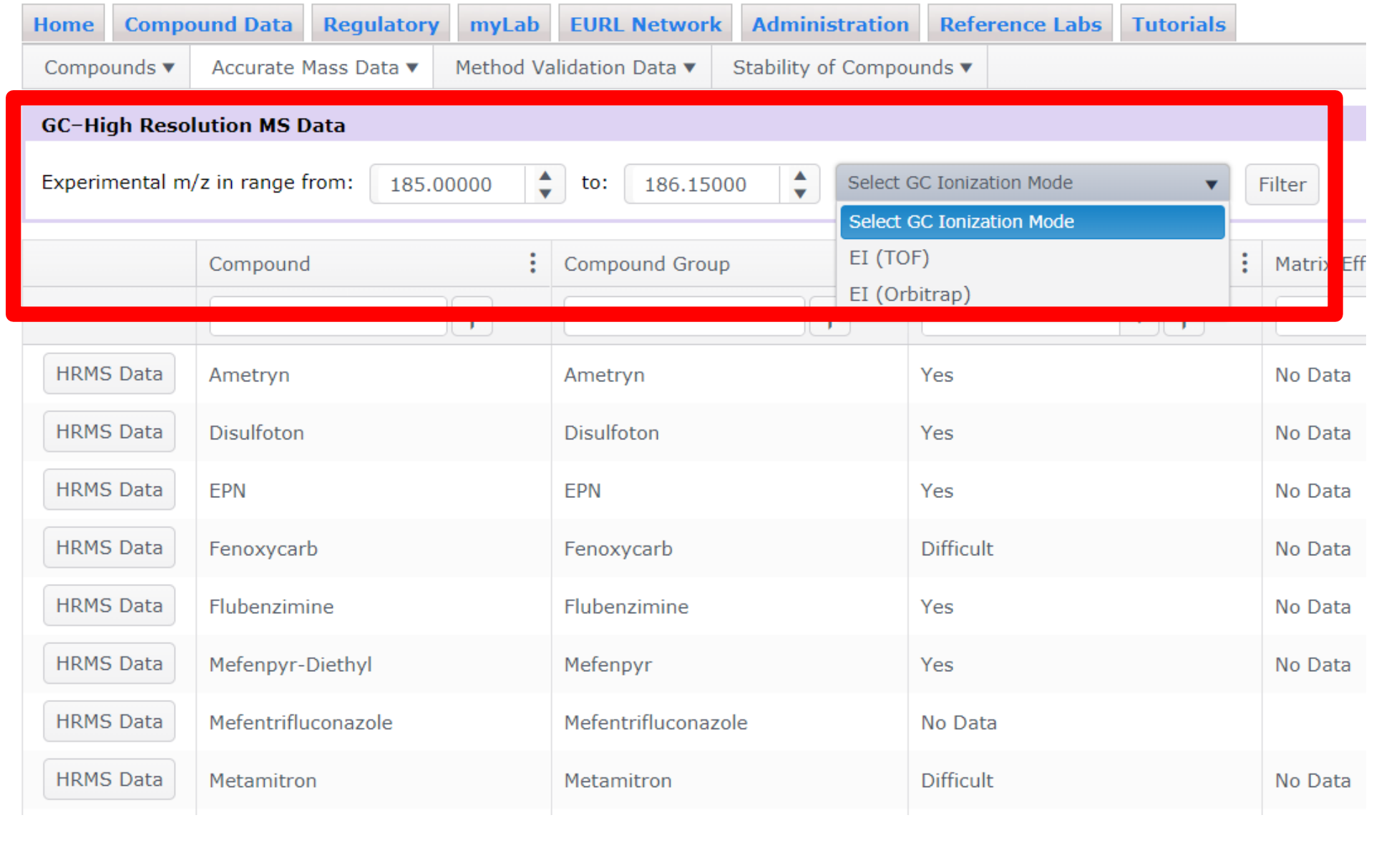


2. Click on „Login“

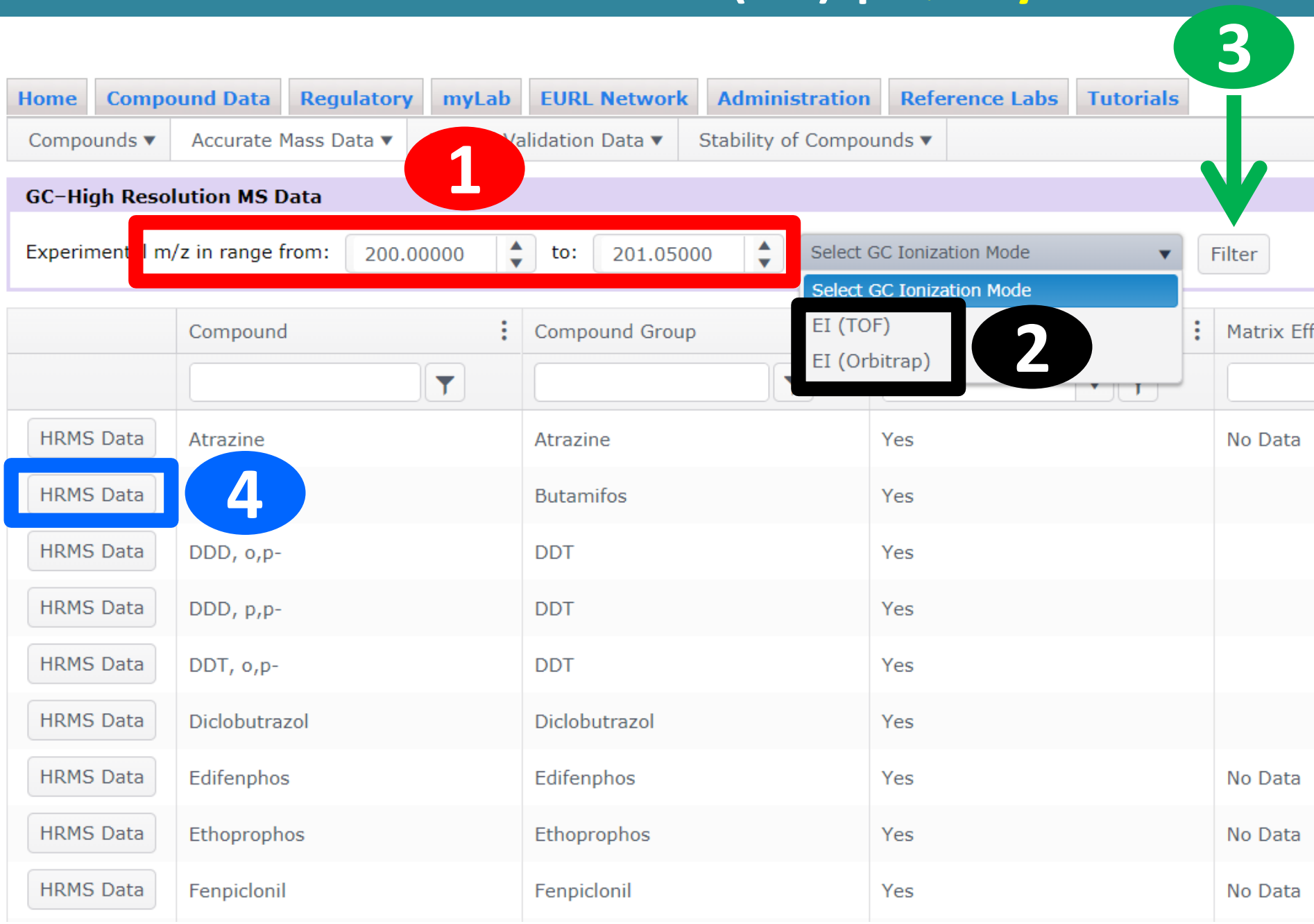
Accurate Mass DB | Search Tool



Accurate Mass DB (GC) | Query



Accurate Mass DB (GC) | Query

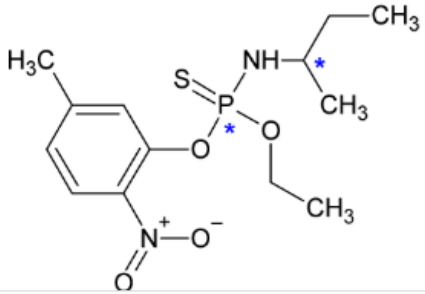


Accurate Mass DB (GC-HRMS) | Result of Query

Butamifos

GC-High Resolution MS

Molecular Formula	C13H21N2O4PS
Monoisotopic Mass [g/mol]	332.09596
GC-Amenable	Yes
Matrix Effects	
Tailing	
GC Decomposition	



Theoretical Ion Mass	Fragment Ion	Ionization Mode	Ion Formula	Remark	Reference
286.102513	Fragment 1	EI (TOF)	C13H21NO2PS		EURL-CF
258.071212	Fragment 2	EI (TOF)	C11H17NO2PS		EURL-CF
231.982791	Fragment 3	EI (TOF)	C7H7NO4PS		EURL-CF
200.010721	Fragment 4	EI (TOF)	C7H7NO4P		EURL-CF
286.102514	Target	EI (Orbitrap)	[C13H21NO2PS]+	Is NOT a molecular ion	EURL-SRM; RIKILT
258.071214	Fragment 1	EI (Orbitrap)	[C11H1		EURL-SRM; RIKILT
231.982794	Fragment 2	EI (Orbitrap)	[C7H7		EURL-SRM; RIKILT
200.010722	Fragment 3	EI (Orbitrap)	[C7H7		EURL-SRM; RIKILT

Reference

Title: Compilation of GC-Orbitrap MS Data (2018)

Data Type: Submitted Lab Data

Author: EURL-SRM

Email Address: eurl-srm@cvuas.bwl.de

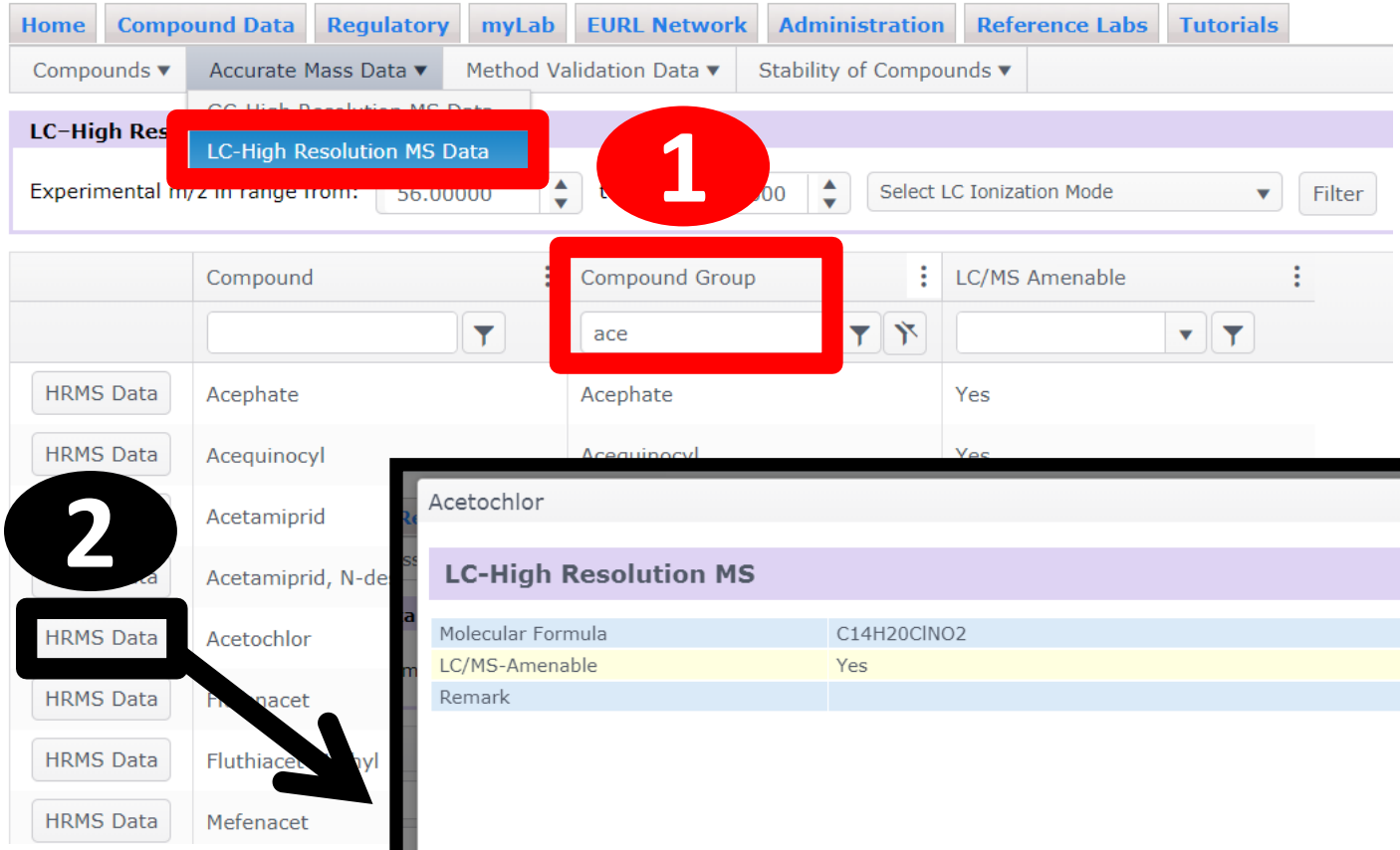
Website: http://www.eurl-pesticides.eu

Author: Dr. J.G.J (Hans) Mol

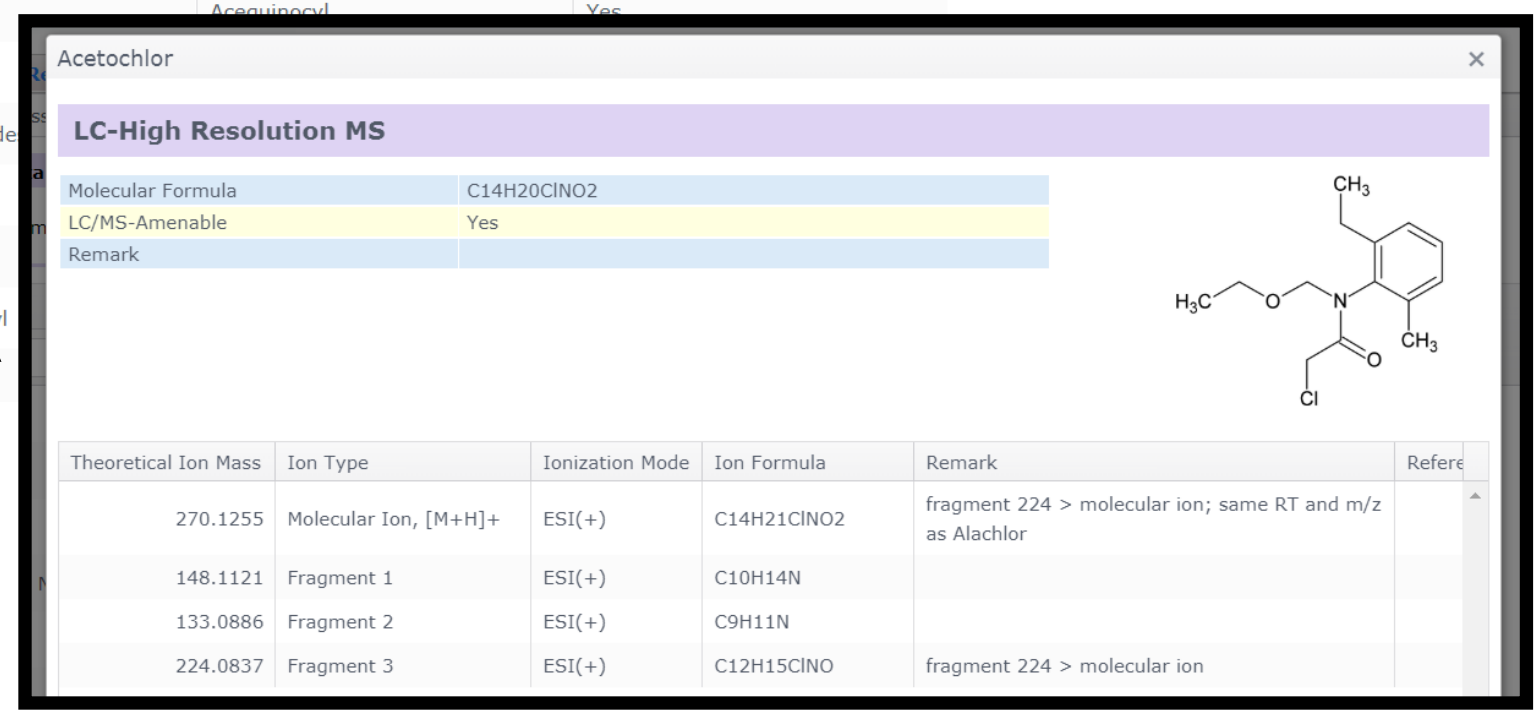
Institute: RIKILT Wageningen University & Research

Website: http://www.wur.nl/rikilt

Accurate Mass DB (LC-HRMS) | Result of Query



2

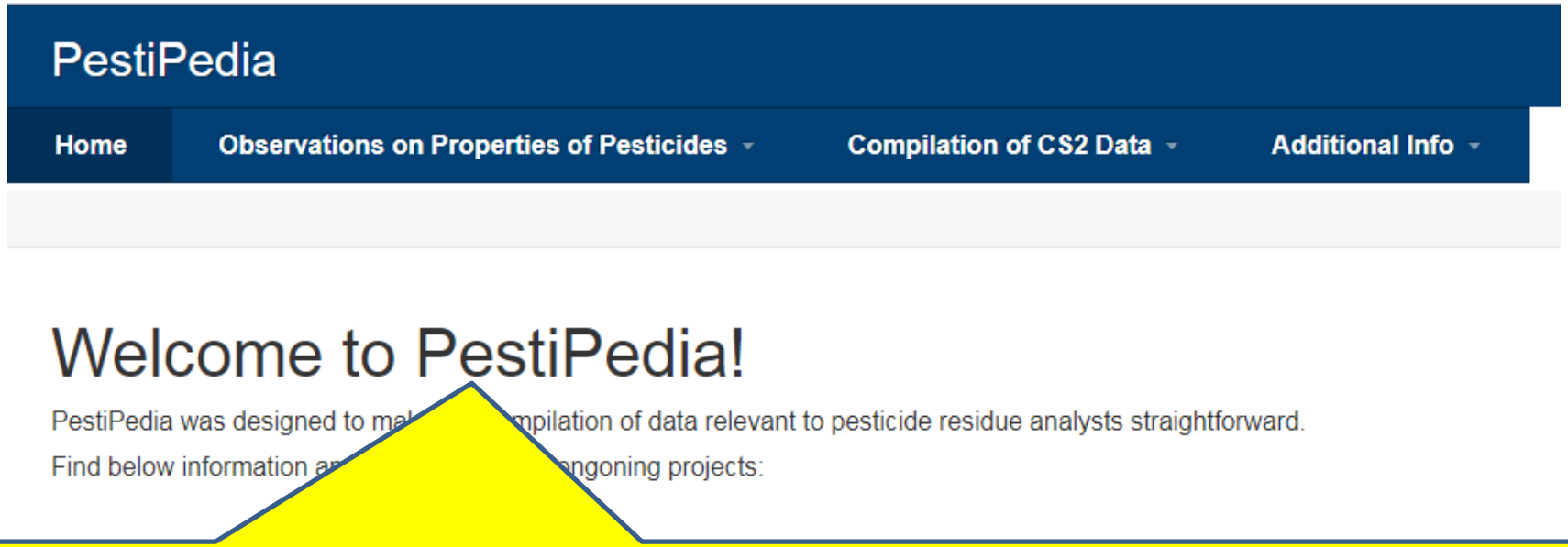


(Cooperation with Swedish NRL (National Food Agency, Sweden) is ongoing.)

Take Home Message

Online-Search for HR-MS data is available.
Submission of HR-MS data is highly welcome!

PestiPedia | Tool to Compile Pesticide Expert Knowledge



- Cooperation with **WG on Pesticides of the German Chemical Society ("AG Pestizide")**
- **pesticide-residue-experts** can make **contributions** to aspects that are relevant in pesticide residue analysis.
- individual contributions will be discussed by registered experts and the outcome will be imported into the EURL DataPool

PestiPedia | Tool to Compile Pesticide Expert Knowledge

PestiPedia

Home | Observations on Properties of Pesticides | ...pilation of CS2 Data | Additional Info

List of Compounds

1

List of Compounds

To search for a specific compound, click on the "Advanced Search"-feature (see below), choose the column that should be searched through, add the filter text and click on the „Add“ button.

Click on the link "Open" in the column "Datasheet" in order to make your contributions to this compound.

folpet search

2

Showing 1-100 of 1423 Advanced Search

100 per page | Page 1 of 15

Datasheet	Compound	Compound Group	Pesticide Property	Status
Open	1,2-Benzisothiazol-3(2H)-one	1,2-Benzisothiazol-3(2H)-one	No Data	
Open	1-MCP	1-MCP	Neutral interm. polar (pKow 0.5-4.5)	
Open	2,4,5-T	2,4,5-T	Potentially anionic	
Open	2,4,5-T-Methylester	2,4,5-T	Neutral interm. polar (pKow 0.5-4.5)	
Open	2,4,5-TP	2,4,5-TP	Potentially anionic	
Open	2,4-D-Methylester	2,4-D	No Data	Prio I
Open	2,4-D	2,4-D	Potentially anionic	Prio I

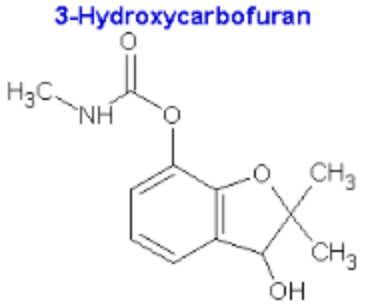
PestiPedia | Tool to Compile Pesticide Expert Knowledge

PestiPedia

Home | Observations on Properties of Pesticides | Compilation of CS2 Data | Additional Info

List of Compounds → Compound Details

3-Hydroxycarbofuran



Choose one of the buttons below to make your contribution to a specific topic:

Stability of Pure/Neat Standards | Stability of Standard Solutions | Stability in Matrix-Extracts | LC-MS Behaviour | GC Behaviour

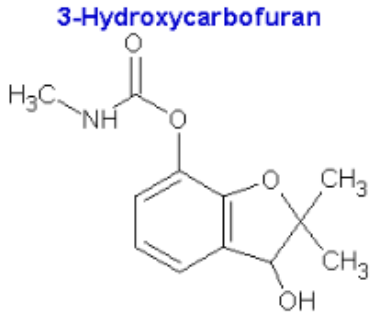
Losses during Extraction/Cleanup | Practical/Legal Aspects

PestiPedia | Contributions to Specific Topics

Stability of Pure/Neat Standards | Stability of Standard Solutions | Stability in Matrix-Extracts | LC-MS Behaviour | 1

3-Hydroxycarbofuran

More Details from DataPool



Existing Da

LC-MS Ame

Remark

To add NEW or ADDITIONAL data sets on **LC-MS Behaviour**, click on the buttons below:

General Info | Ionization, Fragmentation & Identification | Compromized Identification and Interferences | Sensitivity

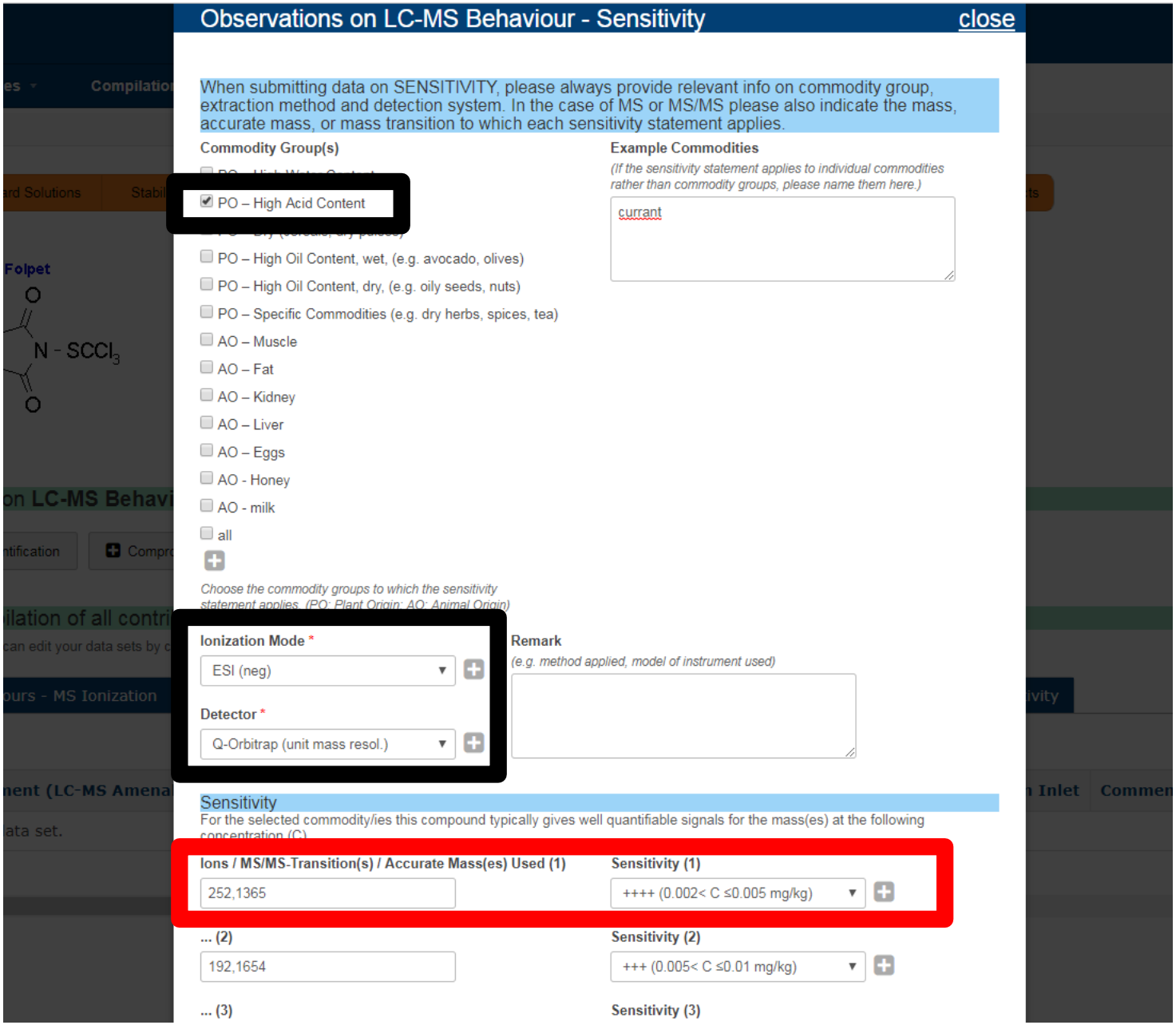
To see Your Contribution(s) or the Compilation of all contributions collec "LC-MS Behaviou

Tab **UR contributions** show the prefix "**Yours**". You contributions ha

Open here the form to make comments on e.g. “Sensitivy”.

2

PestiPedia | Form to Enter Observations



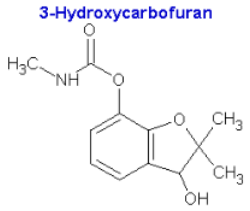
PestiPedia | Contributions to Specific Topics

Stability of Pure/Neat Standards | Stability of Standard Solutions | Stability in Matrix-Extracts | LC-MS Behaviour | Losses during Extraction/Cleanup | Practical/Legal Aspects

1

3-Hydroxycarbofuran

More Details from DataPool



Existing Data from EURL DataPool:

LC-MS Amenable	Yes
Remark	

To add NEW or ADDITIONAL data sets on **LC-MS Behaviour**, click on the buttons below:

General Info | Ionization, Fragmentation & Identification | Compromized Identification and Interferences | Sensitivity

2

To see Your Contribution(s) or the Compilation of all contributions collected so far on "LC-MS Behaviour", click on the TABS below:

Tabs with **YOUR contributions** show the prefix "**Yours**". You can edit your data sets by clicking on "Edit". Tabs with the compilation of **ALL contributions** have the prefix "**All**".

Yours - General Info | ALL - General Info | Yours - MS Ionization | ALL - MS Ionizati... | ...urs - Interferences | ALL - Interferences | Yours - Sensitivity | ALL - Sensitivity

3

ALL - Sensitivity

Advanced Search export

User	Compound	Ionization Mode	Detector	Remark	Ions / MS/MS-Transition(s) / Accurate Mass(es) Used (1)	Sens. (1)	Ions/... (2)
Norbert	3-Hydroxycarbofuran	ESI (neg)	Triple Quad.	bad reproducability of Peak area in ESI (neg) mode			
Sabine	3-Hydroxycarbofuran	ESI (pos)	(Q-)Orbitrap (High Resol.)	§64 L00.00 115/1 Q-Exactive	260,08933	+++ (0.005< C ≤0.01 mg/kg)	

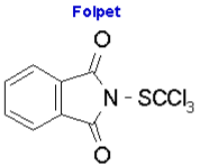
PestiPedia | Practical/Legal Aspects

Stability of Pure/Neat Standards | Stability of Standard Solutions | Stability in Matrix-Extracts | LC-MS Behaviour | GC Behaviour | Losses during Extraction/Cleanup | Practical/Legal Aspects

1

Folpet

More Details from DataPool



To add NEW or ADDITIONAL data sets on **Practical/Legal Aspects**, click on the button below:

Add New Data on Practical/Legal Aspects

To see Your Contribution(s) or the Compilation of all contributions collected so far on "Practical/Legal Aspects", click on the TABS below:

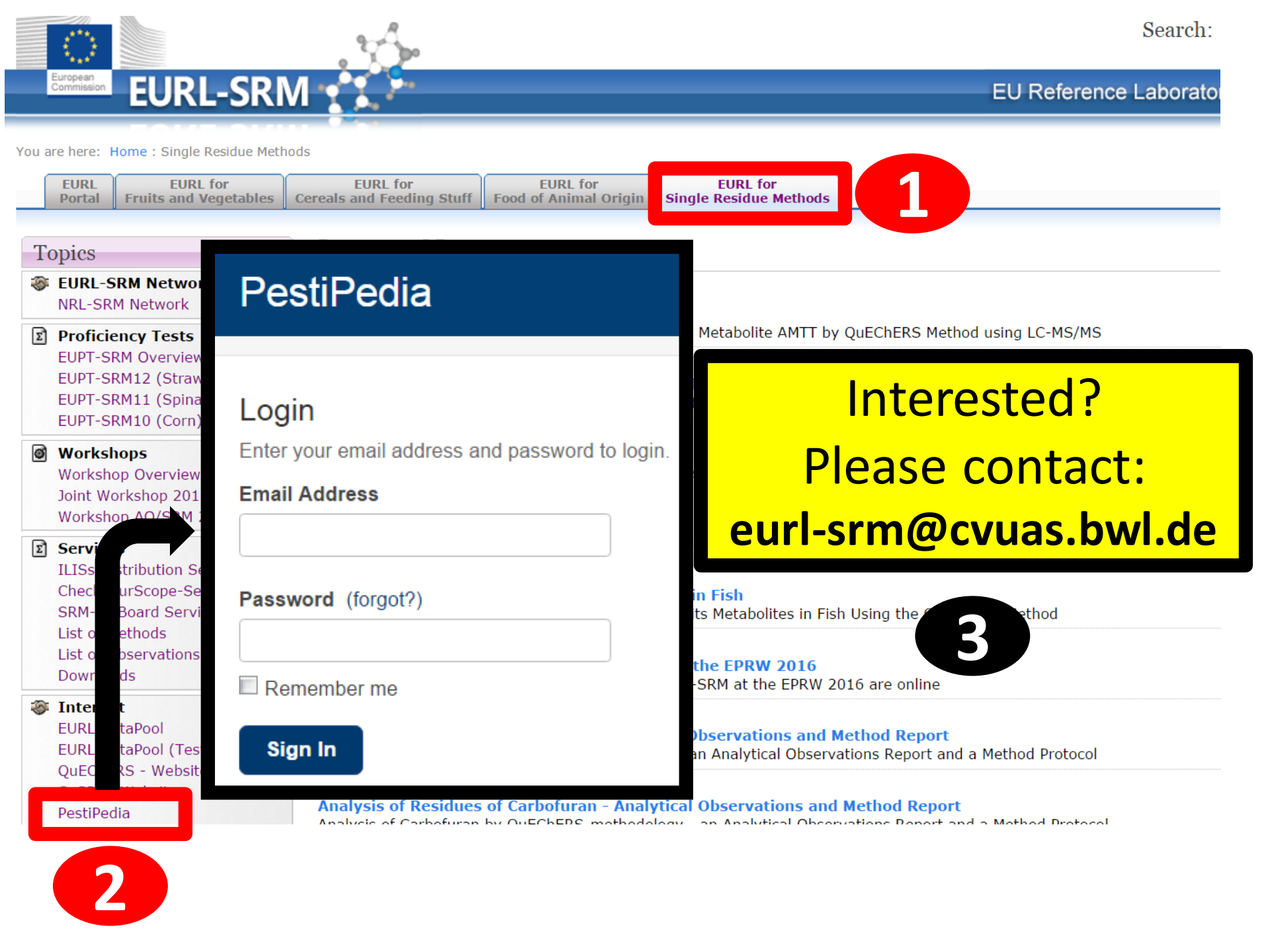
Tabs with **YOUR contributions** show the prefix "**Yours**". You can edit your data sets by clicking on "Edit". Tabs with the compilation of **ALL contributions** have the prefix "**All**".

Yours - Practical/Legal Aspects | ALL - Practical/Legal Aspects

2

ALL - Practical/Legal Aspects

User	Compound	Name Relevant Commodities for this Compound	Compound Originates from Other Sources	Remark (Other ...)	Compound or Degradation Products thereof may Originate from a
Anna	Folpet	hops, wine	Generated from the degradation of other pesticides, From a non-pesticide related contamination, As a natural product in food, Formed during food processing, Formed as an artefact in GC inlet, Formed as an artefact during Extraction, Formed as an artefact during Cleanup	under "Compound Originates from Other Sources" phthalimide, the metabolite of folpet is meaning:	drifts of phthalic acid from fruit plantations (Captan, Folpet applications) the prevalent presents of phthalic acid during drying is possible
Andreas	Folpet	processed dried food product			
Günter	Folpet	Herbs and spices, tea, fruit and herbal teas, processed (dried) food products		Phthalimid (PI) as the major metabolite might derive from environmental impact (Phthalic anhydrid or Phthalic acid) and might be formed during processing of food products and during injection process in GC.	


Search:
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EU Reference Laborato
You are here: Home : Single Residue Methods
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EURL for Fruits and Vegetables
EURL for Cereals and Feeding Stuff
EURL for Food of Animal Origin
EURL for Single Residue Methods
1
Topics
EURL-SRM Networ
NRL-SRM Network
Proficiency Tests
EUPT-SRM Overview
EUPT-SRM12 (Straw
EUPT-SRM11 (Spina
EUPT-SRM10 (Corn)
Workshops
Workshop Overview
Joint Workshop 201
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ILISs tribution Se
Chec urScope-Se
SRM- Board Servi
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List o bservations
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in Fish
ts Metabolites in Fish Using the ethod
3
the EPRW 2016
-SRM at the EPRW 2016 are online
bservations and Method Report
an Analytical Observations Report and a Method Protocol
Analysis of Residues of Carbofuran - Analytical Observations and Method Report

Take Home Message

PestiPedia = website to share experiences/observations/… with other pesticide experts.
Give/Take-Concept!

Collection of CS_2-Findings non-Originating from DTC-Pesticides

- Re-evaluation of dithiocarbamate-MRLs is planned for 2019
- DTCs are usually analyzed by using **common moiety methods involving the release of carbon disulfide (CS_2)** via an acid digestion/hydrolysis step (using a mixture of tin(II)-chloride and hydrochloric acid)
- Some crops **naturally contain compounds** (e.g. mustard oil glycosides) **that do release CS_2** (either spontaneously or) during the digestion/hydrolysis step of DTC analysis (=> background CS_2-levels):
  - crops belonging to *Brassicaceae* + *Alliaceae* family
  - Papaya

CS_2-Background Levels | Some Results

Common Name	Genus	MRL (CS_2) [mg/kg]	CS_2 Findings by the EURL-SRM [mg/kg]			
			n	Homogenized and analyzed frozen	Homogenized at room temp. and analyzed after …	
					15 min	2 h
Arugula (rocket/rucola)	Eruca	5	3	0,37±0,06 - 0,82±0,08	2,6±0,07 - 8,33±0,3	2,2±0,2 - 7,24±0,9
Caper	Capparis	25				
Caper Berries (canned)			1		1,40	
Brine of Caper Berries (canned)			1		0,35	
Capers (edible flower buds; canned)			1		5,24	
Brine of Capers (…)			1		0,20	
Cabbage	Brassica	Head cabbages: 3	1	0,18±0,004	0,23±0,02	0,20±0,05
Cauliflower	Brassica	1	1	0,08±0,01	0,12±0,02	0,07±0,01
Champignon mushroom	Agaricus	0.05*	1	≤0,01	≤0,01	≤0,01
Chinese cabbage, napa cabbage	Brassica	Chinese cabbages/pe-tsai: 0,5	1	0,08±0,01	0,15±0,04	0,11±0,02
Collard greens	Brassica	Kales: 0,5				
Garden cress	Lepidium	Cresses and other sprouts and shoots: 5,0	1	(≈1,1)	(≈5,9)	(≈3,6)
Moringa	Moringa	0.1*Processing Factor 0.1 (from 2018 onwards)	7		0,70±0,08 – 11,2±0,09	

CS_2-Background Levels | Some Results

Common Name	Genus	MRL (CS_2) [mg/kg]	CS_2 Findings by the EURL-SRM [mg/kg]			
			n	Homogenized and analyzed frozen	Homogenized at room temp. and analyzed after …	
					15 min	2 h
Mustard	Brassica	seeds: 0,1*				
Black mustard seeds	Brassica	seeds: 0,1*	1		0,93	
Table Mustard (medium-strength)	Brassica	seeds: 0,1*	1		30	
Table Mustard (hot)	Brassica	seeds: 0,1*	1		263	
Radish (black)	Raphanus	2	1	0,17±0,02	1,22±0,05	1,1±0,03
Radish (white)		2	1	0,04±0,01	0,22±0,09	0,22±0,07
Turnip cabbage	Brassica	1	1	0,012	0,09±0,1	0,05±0,03
May turnip	Brassica		1	0,38±0,03	0,59±0,04	0,7±0,1
Onions	Allium	1	2	0,09±0,02	0,055±0,003	0,055±0,006
Shallots	Allium	1	1	0,24±0,03	0,24±0,01	0,25±0,03
Spring onions/green onions and Welsh onions	Allium	1	1	0,15±0,01	0,07±0,006	0,09±0,01

=> Need to collect data on non-pesticide related CS2 levels of ORGANIC products

CS_2-Background Levels | Priority List (by EFSA & EURL-SRM)

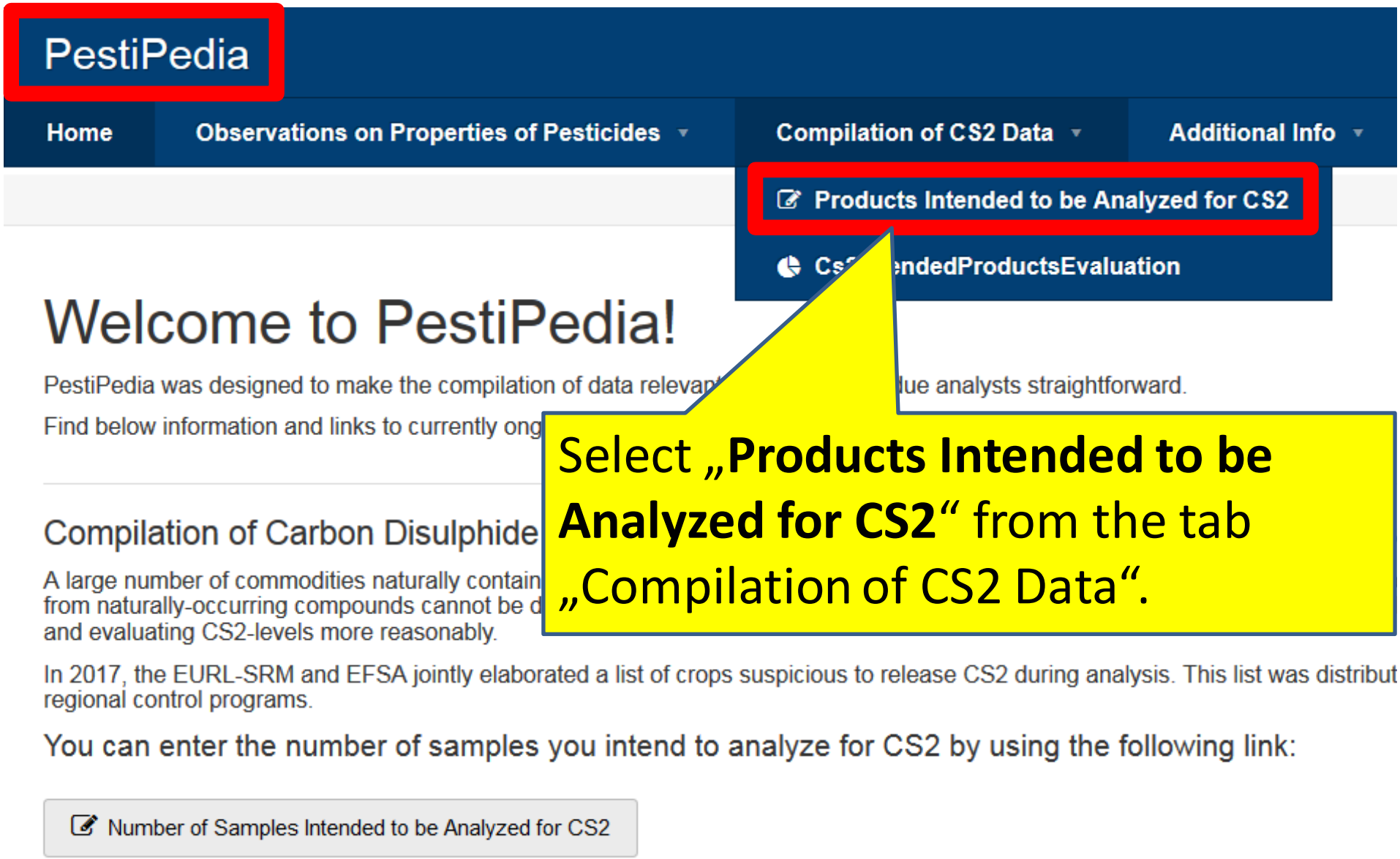
Final Decision: Keep Product	Priority	Confirmation by GLP-experiments	EFSA conclusion whether further data need to be generated to derive MRL proposal	Crop suspicious to CS2-release from matrix during: Based on EFSA data collection	Based on EURL-SRM Data Call	Based on other Info	Product Name	SUMMARY of CS2-Results submitted by EFSA, EURL-SRM and other sources: CS2-Commodity Group	N° of samples to be analyzed (group OR individual	N° of samples still to be analyzed	% positive CS2-samples (only calc. for n>5)	Total-N° of CS2-Results >LOQ	Total-N° of organic-samples analyzed for CS2	EU monitoring 2013/2014/2015 (EFSA): # of CS2 results MACP13-16	# of CS2 results >0.01 MACP13-16	Max. Conc. MACP13-16	MRLs: Dithiocarbamates (DTC expr. as CS2, including maneb, mancozeb, metiram, propineb, thir	CS2-Findings by EURL-SRM [mg/l: # of samples analyzed	# of pos. samples (>LOQ)	Homogenized and analyzed frozen	Homogenized at RT and analyzed after 15 min	Homoge d at RT analyz afte 2 h
							Flowering brassica	Brassica (flower)									1 (ft)					
x	high		Y (2)		Yes	Strong	Broccoli	Brassica (flower)	59	36	65,2	15	23	23	15	0,52	1 (ft)					
x	high					Strong	Calabrese	Brassica (flower)	59	59		0	0									
x	high					Strong	Chinese broccoli/kai-lan	Brassica (flower)	59	59		0	0									
x	high					Strong	Choi sum/tsoi sam	Brassica (flower)	59	59		0	0									
x	high					Strong	Rapini/broccoletti/broccol	Brassica (flower)	59	59		0	0									
x	high		Yes	Y (2)		Yes	Strong Cauliflowers	Brassica (flower)	59	56		1	3	2	0		1 (ft)	1	1	0,08±0,01	0,12±0,02	0,07±0,01
x	high					Strong	Romanesco cauliflowers/f	Brassica (flower)	59	59		0	0									
						Strong		Brassica (flower)	59	59		0	0									
x	high			Y (2)		Strong	Other flowering brassica	Brassica (flower)									1 (ft)					
						Strong		Brassica (leaf)	59	59		0	0									
x	high					Strong	Head brassica	Brassica (leaf)	59	59		0	0									
x	high			Y (2)		Strong	Brussels sprouts	Brassica (leaf)	59	58		1	1	1	1	5,39	2 (ft)					
x	high					Strong	Flower sprouts	Brassica (leaf)	59	59		0	0									
x	high		Yes	Y (2)		Yes	Strong Head cabbages	Brassica (leaf)	59	32	40,7	11	27	26	10	0,41	3 (ft)	1	1	0,18±0,004	0,23±0,02	0,20±0,05
x	high					Strong	Pointed head cabbages	Brassica (leaf)	59	59		0	0									
x	high					Strong	Red cabbages	Brassica (leaf)	59	59		0	0									
x	high					Strong	Savoy cabbages	Brassica (leaf)	59	59		0	0									
x	high					Strong	White cabbage	Brassica (leaf)	59	59		0	0									

Priority	# of crops	Exemplary crops
High	91	Radish, turnips, broccoli, cauliflower, caper, papaya, guava
Medium	46	Figs, sweet potatoes, pomegranate, mango, maracuja, chamomile, ginger
Low	186	Almonds, berries & small fruits, avocado, lentil

>5300 organically grown crops have theoretically to be analyzed to quantify CS_2-background levels (*)

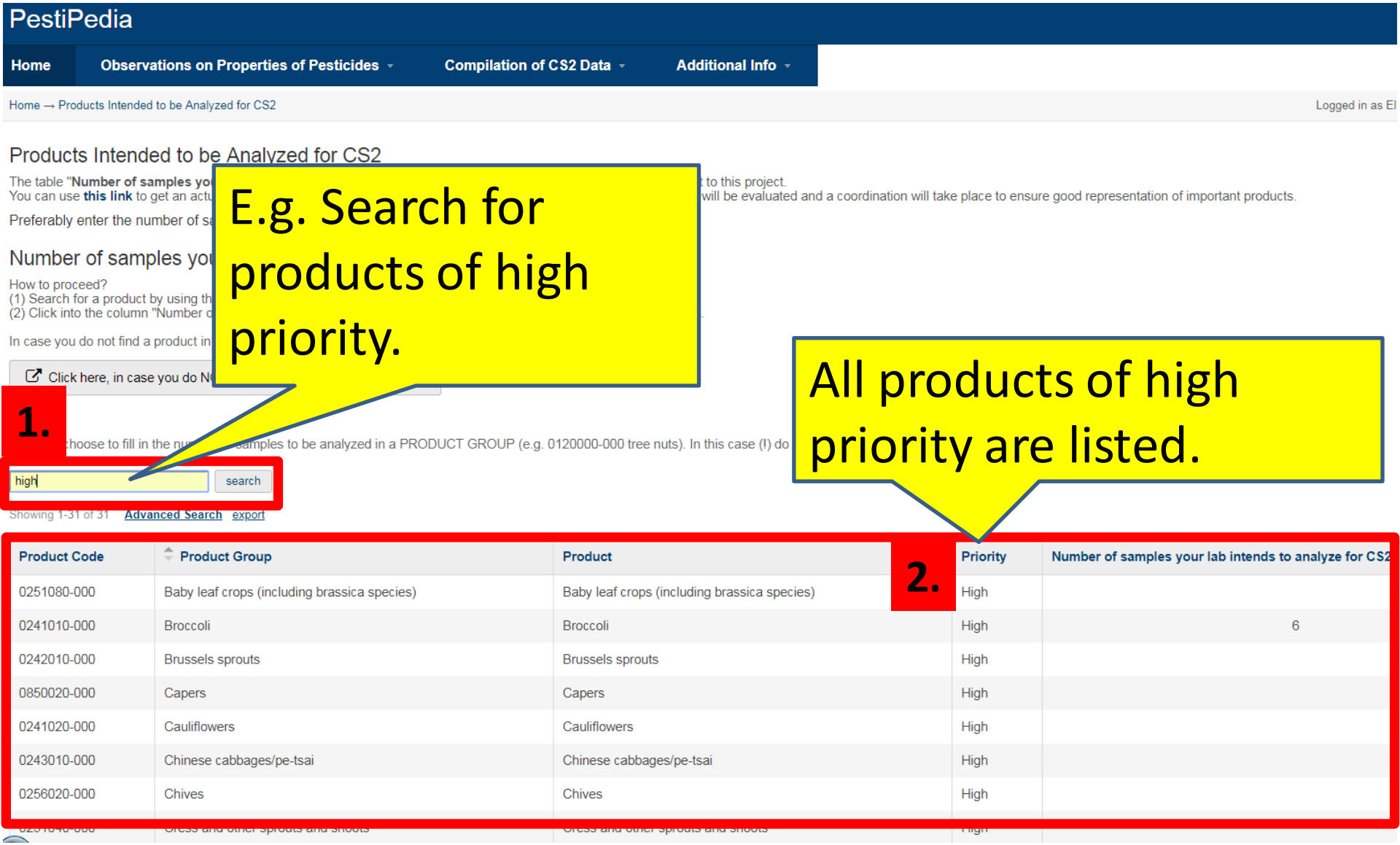
(*) For relevant commodities data from 59 samples is needed

CS_2-Background Levels | Coordination Required!



Select „**Products Intended to be Analyzed for CS2**“ from the tab „Compilation of CS2 Data“.

CS_2-Background Levels | Coordination via PestiPedia



Product Code	Product Group	Product	Priority	Number of samples your lab intends to analyze for CS2
0251080-000	Baby leaf crops (including brassica species)	Baby leaf crops (including brassica species)	High	
0241010-000	Broccoli	Broccoli	High	6
0242010-000	Brussels sprouts	Brussels sprouts	High	
0850020-000	Capers	Capers	High	
0241020-000	Cauliflowers	Cauliflowers	High	
0243010-000	Chinese cabbages/pe-tsai	Chinese cabbages/pe-tsai	High	
0256020-000	Chives	Chives	High	

CS_2-Background Levels | Coordination via PestiPedia

PestiPedia

Home | Observations on Properties of Pesticides | Compilation of CS2 Data | Additional Info

Home → Products Intended to be Analyzed for CS2

Products Intended to be Analyzed for CS2

The table "**Number of samples your lab intends to analyze for CS2 in 2018**" lists the most important pr
You can use **this link** to get an actual overview of the data submitted by the entire EU-lab-network. Period

Preferably enter the number of samples of each product you intend to analyze for CS2 into this tab

Number of samples your lab intends to analyze for CS2 in 2018

How to proceed?
(1) Search for a product by using the "search by keyword"-field or the "Advanced Search"-feature.
(2) Click into the column "Number of samples ..." and enter the number of samples you intend to analyze f

In case you do not find a product in the table below, please use this link:

Click here, in case you do NOT find the product in the table below

Remark:
You may choose to fill in the number of samples to be analyzed in a PRODUCT GROUP (e.g. 0120000-00

high search

Showing 1-31 of 31 Advanced Search export

Product Code	Product Group	Product	Priority	Number of samples your lab intends to analyze for CS2
0251080-000	Baby leaf crops (including brassica species)	Baby leaf crops (including brassica species)	High	
0241010-000	Broccoli	Broccoli	High	6
0242010-000	Brussels sprouts	Brussels sprouts	High	
0850020-000	Capers	Capers	High	
0241020-000	Cauliflowers	Cauliflowers	High	
0243010-000	Chinese cabbages/pe-tsai	Chinese cabbages/pe-tsai	High	
0256020-000	Chives	Chives	High	

Enter the number of samples you intend to analyzed for CS2 in the pop-up window.

Then, click on „Submit“.

Edit NumberOfIntendedSamples

7

Submit

Background CS_2-Levels | Results

N° of participating labs	N° of different crops intended to be analyzed by Member State	N° of samples intended to be analyzed per crop-priority		
		High	Medium	Low
11	34	194	16	39

Could be shifted to crops of higher priority!

Background CS_2-Levels | Results

Crop	Number of samples to be analyzed					N° of intended samples to be analyzed per Member State
	Required	Done	Missing	Intended to be analyzed	Still missing	
Broccoli	59	23	36	28	8	DE (23), GR (4), PT (1)
Brussels sprouts	59	1	58	1	57	PT (1)
Cauliflowers	59	3	56	27	29	DE (22), GR (4), PT (1)
Chinese cabbages	59	2	57	13	44	DE (12), PT (1)
Chives	59	0	59	2	57	DE (2)
Cress & similar	59	2	57	1	56	PT (1)
Garlic	59	1	58	5	53	DE (5)
Head cabbages	59	27	32	7	25	DE (3), GR (4)
Horseradishes	59	0	59	1	58	PT (1)
Kales	59	1	58	6	52	DE (6)
Kohlrabies	59	2	57	9	48	DE (9)
Land cress	59	0	59	5	53	DE (5)
Leeks	59	62	/	23	-	DE (21), PT (2)
Onions	59	10	49	10	39	DE (4), GR (4), PT (2)
Papayas	59	1	58	21		DE (20), PT (1)
Parsnips	59	0	59	5	5	DE (5)
Radishes	59					
Rucola	59					
Spring onions	59					
Turnips	59					

Enough CS2-background data for leek!

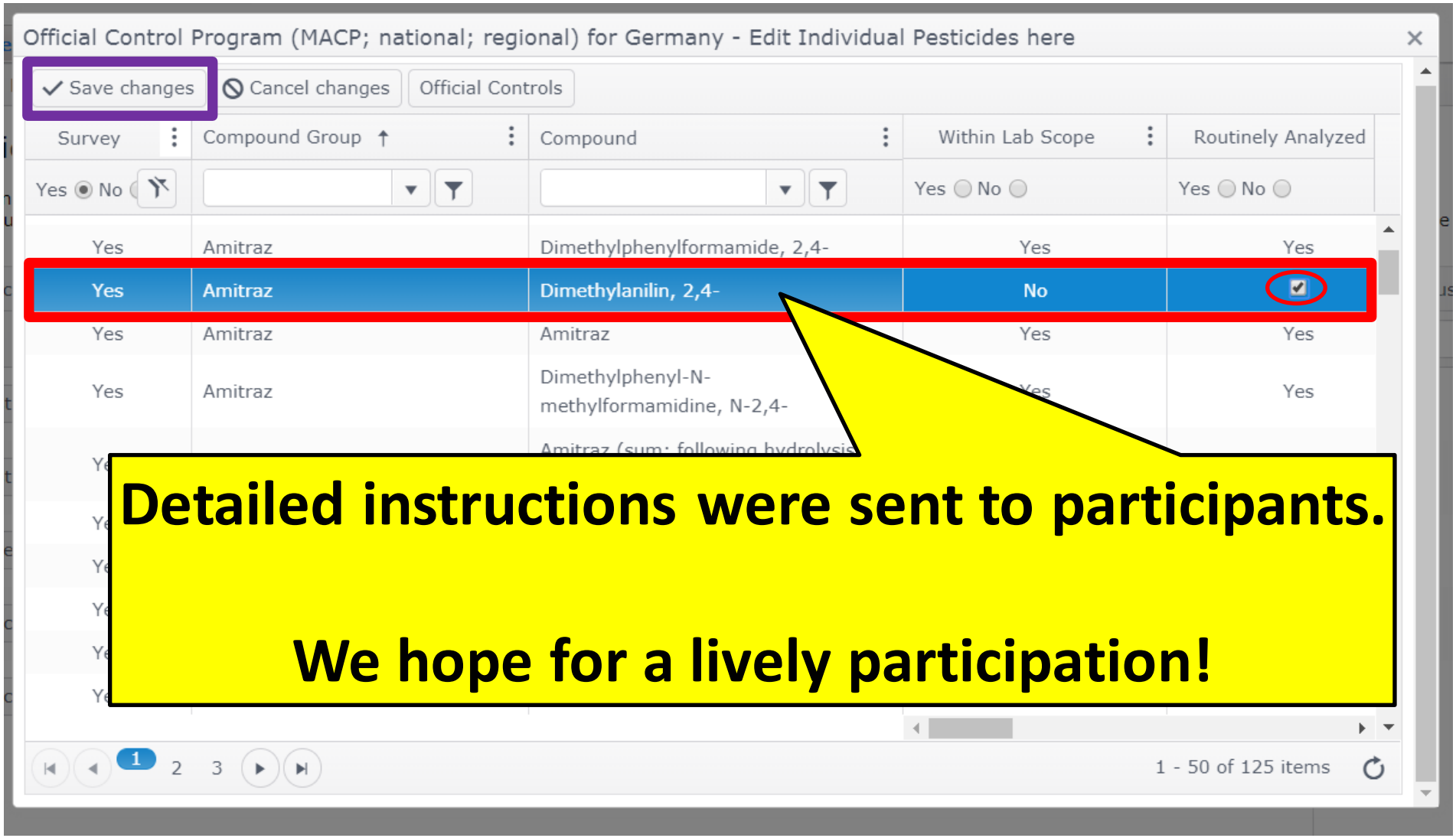
EURL DataPool is used to run surveys:

- **Update of Sample Scope** by OFLs
  -> invitation will be sent at the end of October
- **Survey on Analytical Capabilities** (currently ongoing)

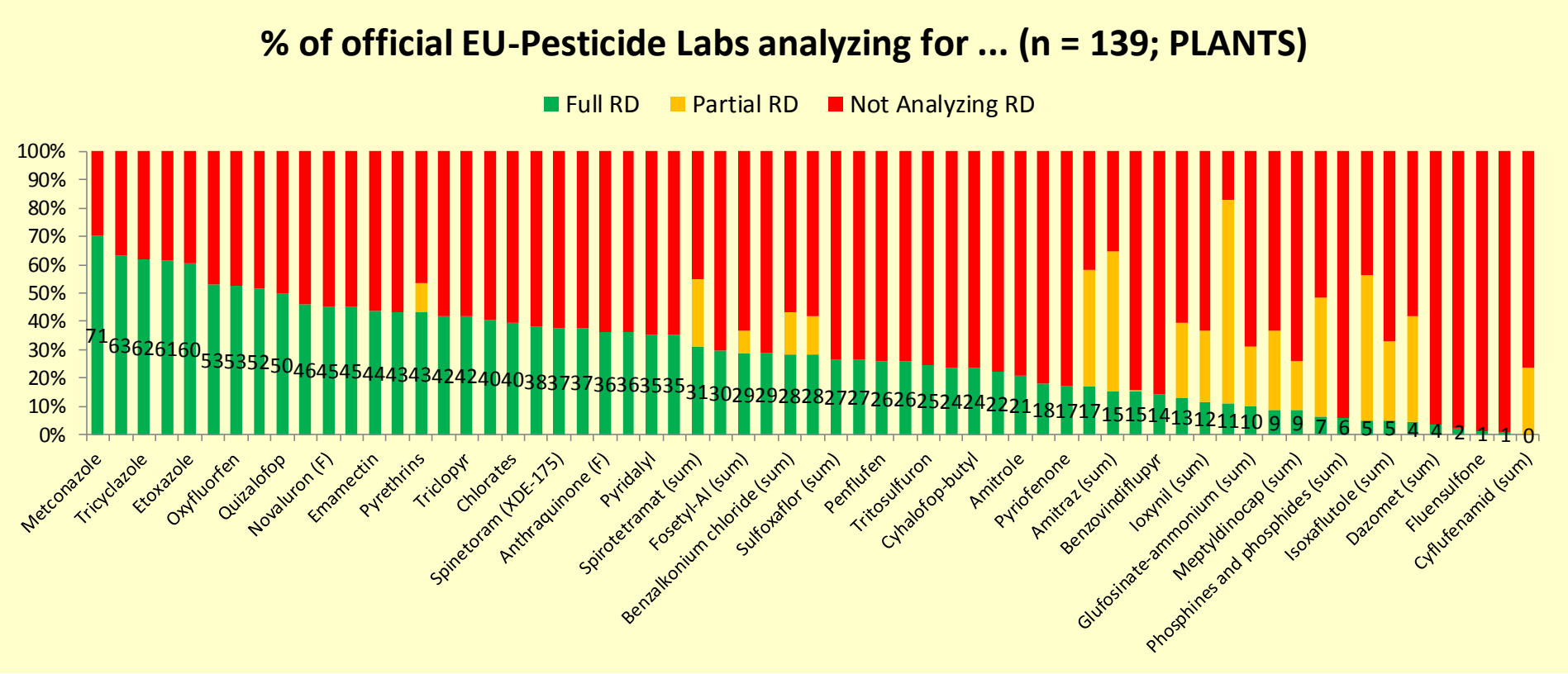
Surveys on Analytical Capabilities

- Surveys conducted on behalf of COM (2016, 2017, 2018 (ongoing))
- **Goal of Surveys**:
  how many EU-OFLs **routinely analyze for full residue definition of pesticides** that are included in MACP Working Document (chapter 4 of SANCO/12745/2013 Rev)?
- **Participation (FV-OFLs)**: 69 % in 2017
  51 % in 2016

Survey on Analytical Capabilities | Survey 2018 is ongoing



Survey on Analytical Capabilities: Results from 2017-Survey



Survey on Analytical Capabilities: Results from 2017-Survey

- **FULL RD** is covered by **≥ 70%** of participating OFLs
  - Metconazole

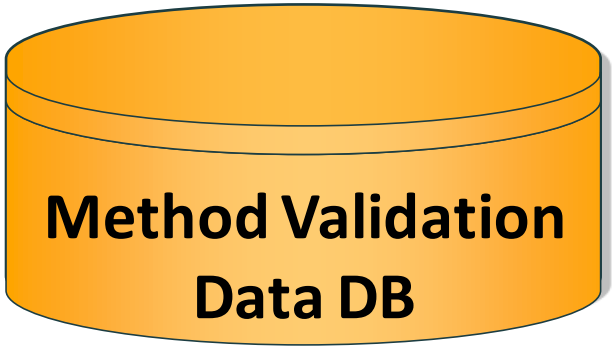
- **FULL RD** is covered by **≥ 50% and < 70%** of participating OFLs
  - Metrafenone (F)
  - Tricyclazole
  - Cyazofamid
  - Etoxazole
  - Glyphosate
  - Oxyfluorfen
  - Proquinazid (R)

Survey on Analytical Capabilities: Results from 2017-Survey

- FULL RD is covered by **less than 20%** of participating OFLs: in total: 22 compounds; excerpt from the list:

RDs	# of OfLs intending to introduce RD/compound in 2018
Oxasulfuron	18
Pyriofenone	10
Amitraz (not approved)	DMF: 6; Amitraz: 5; DMPF: 6
MCPA and MCPB	Various MCPA esters: 4 MCPA: 2 MCPB: 3
Benzovindiflupyr	15
Ioxynil (sum of Ioxynil, its salts and its esters, expressed as ioxynil (F))	Ioxynil: 5 Ioxynil-heptanoate: 4 Ioxynil-octanoate: 5
Bifenazate	Bifenazate: 4 Bifenazate-Diazene: 15
Sulfuryl fluoride	6
Fluensulfone	11
Cyflufenamid: sum of cyflufenamid (Z-isomer) and its E-isomer	0

Method Validation DB



- ≈500.000 recoveries
- submitted by 46 labs

EURL-DataPool

EU Reference Laboratories for Residues

Home | Compound Data | Regulatory | myLab | EURL Network | Administration | Reference Labs | Tutorials

Compounds ▾ | Method Validation Data ▾ | Stability of Compounds ▾

Detailed Query

Commodity Information

Commodity	Search Commodity (min. 3 chars)
Commodity Group	Select...
Water Content	Select...
Fat Content	Select...
Sugar Content	Select...
pH Value	Select...

Method Information

Extraction Method	Select...
Spiking Step	Select...
Extraction pH	Select...
ISTD	Search ISTD (min. 3 chars)
ISTD Addition Step	Select...
Cleanup	Select...
Cleanup Details	Select...
Post Cleanup Details	Select...
Chromatography	Select...
Interface	Select...
Calibration	Select...
Calibration Details	Select...

Compound Information

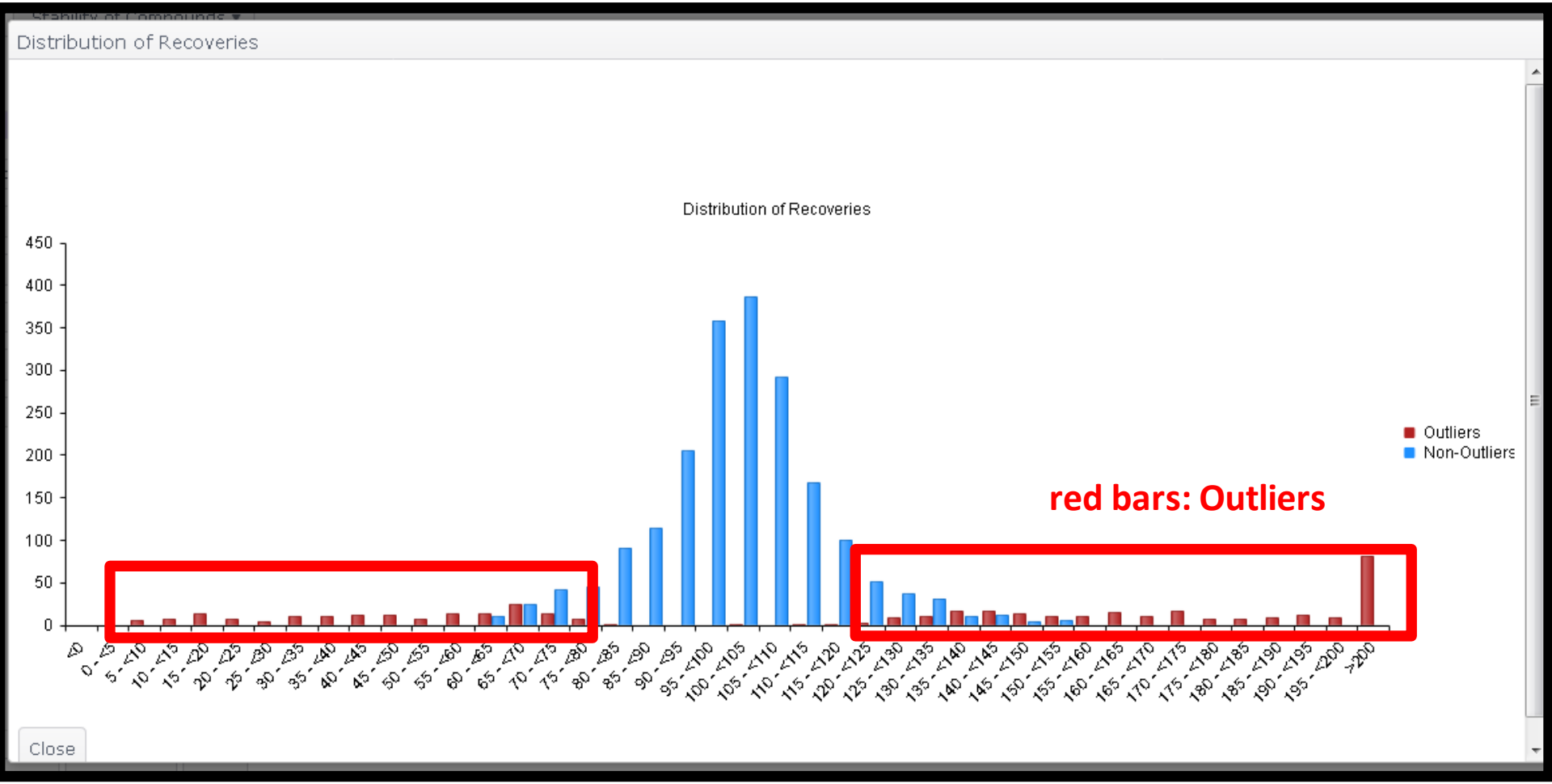
Compound (spiked)	1,8-Diaminooctane
Spiking Level From	Select...
Spiking Level To	Select...

General Information

Lab	Search Lab (min. 3 chars)
Date From	
Date To	
Validation Context	Select...
Context Details	
Exclude Outliers	
Experiment No.	

Detailed Result List | Aggregate by Spiking Level | Recoveries | Clear

Method Validation DB | Distribution of Recoveries



Query:

- **Extraction Method: QuPPe**
- **Water Content: 30-80%**
- **Exclude Outliers: No**

Method Validation DB | Outlier Calculation

Outlier – How do we calculate the Outliers?

- Grubbs Outlier Test:
  - Recoveries: 95% | 98% | 96% | 97% | 90% (=> outlier)

    -> too strict for pesticide residue analysis!

Method Validation DB | „Self-Made“ Outlier-Test

1. Group the data by
   - Compound
   - Extraction Method
   - Commodity Group (according to AQC Guideline)
   - Extraction pH (of method)
   - Cleanup Principle
2. Exclude groups with less than 5 recoveries
3. Calculate the median for each group: “**Global Median**”

   (only for groups with n(labs) > 2)

Method Validation DB | „Self-Made“ Outlier-Test

4. Compare “Global Median” with Experiment-Median:
   - <u>N(recoveries) > 2:</u>
     if Experiment-Median > 1 + **0.4*** “Global Median”
     OR
     if Experiment-Median > 1 - **0.4*** “Global Median”
     then whole Procedure => outlier

   - <u>N(recoveries) < 3:</u>
     if Experiment-Median > 1 + **0.6*** “Global Median”
     OR
     if Experiment-Median > 1 - **0.6*** “Global Median”
     then whole Procedure => outlier

   - All recoveries < 1 => outliers

Method Validation DB | „Self-Made“ Outlier-Test

5. Calculate the “Global Rel. Standarddeviation” (**Global RSD**) for each group:
   - Group the data by
     - Compound
     - Extraction Method
     - Commodity Group (according to AQC Guideline)
     - Extraction pH (of method)
     - Cleanup Principle

Method Validation DB | „Self-Made“ Outlier-Test

6. Compare the “**Global RSD”** with recovery-data set belonging to each group:

- For **Global RSD ≤ 10%**
  - If recovery < Global Median * (1 - **3** * Global RSD/100) => outlier
  - If recovery > Global Median * (1 + **3** * Global RSD/100) => outlier

- For **Global 10% < RSD ≤ 25 %**
  - If recovery < Global Median * (1 - **2** * Global RSD/100) => outlier
  - If recovery > Global Median * (1 + **2** * Global RSD/100) => outlier

- For **Global RSD > 25 %**
  - If recovery < Global Median * (1 - **1** * Global RSD/100) => outlier
  - If recovery > Global Median * (1 + **1** * Global RSD/100) => outlier

EURL DataPool | Take Home Message

- Search Tool for **Accurate Mass Data**
- **PestiPedia** = Tool to share experiences with other pesticide experts
- EURL DataPool is used to **run surveys**:
  - Analytical Capabilities on Pesticides
  - Update of official sample scope

- **Outlier-Test** in Method Validation DB:
  -> open for discussion/contributions

Thank You
for Your Attention

www.eurl-pesticides-datapool.eu