

# EURL FOR PESTICIDE RESIDUES IN FRUITS AND VEGETABLES (EURL-FV)

# Activity Programme 2015

**EURL-FV 2015 Working Programme** 

12<sup>th</sup> September 2014



#### TITLE III

#### REFERENCE LABORATORIES

#### Article 32

#### Community reference laboratories

 The Community reference laboratories for feed and food referred to in Annex VII shall be responsible for:

- (a) providing national reference laboratories with details of analytical methods, including reference methods;
- (b) coordinating application by the national reference laboratories of the methods referred to in

   (a), in particular by organising comparative testing and by ensuring an appropriate follow-up
   of such comparative testing in accordance with internationally accepted protocols, when
   available;
- (c) coordinating, within their area of competence, practical arrangements needed to apply new analytical methods and informing national reference laboratories of advances in this field;
- (d) conducting initial and further training courses for the benefit of staff from national reference laboratories and of experts from developing countries;

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#### September 2014



- (e) providing scientific and technical assistance to the Commission, especially in cases where Member States contest the results of analyses;
- (f) collaborating with laboratories responsible for analysing feed and food in third countries.





- Four groups of activities:
  - A) General tasks
  - B) Development and validation of analytical methods
  - C) Quality assurance and quality control programme; including the organisation of Proficiency Tests and Intercomparative Studies

D) Technical and scientific support to DG SANCO, EU Member States and Third Countries including the organisation of Courses and Workshops



### A - GENERAL TASKS.

### A1. Management of administrative duties

A2. EURL-FV web page.

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

Fruits and Vegetables





You are here: Home : Pesticides in Fruits and Vegetables

EURL EURL for Portal Fruits and Veget				
Topics	Latest News			
EURL-FV Network	19-06-2014   EURL-FV			
April-2014 Updated	European Union Ring Test Certified Standard Solutions FV16			
Proficiency Tests	EU-RT-FV16			
EU-RT-FV16	10-06-2014   EURL-FV			
EUPT-FV-T02	European Union Proficiency Test for Pesticide Residues in tea EUPT-FV-T02			
EUPT-FV-16	Application form available!!!			
EUPT-FV-SM06	23-05-2014   EURL-FV			
EUPT-FV-15	EURL/NRLs-FV Workshop 2014 for Pesticide Residues in fruits and vegetables			
EUPT-FV-SM05	Almeria, Spain, on 11th and 12th of September 2014			
EUPT-FV-T01	02-05-2014   EURL-FV			
EUPT-Panel Meeting	EU Proficiency Test for Pesticide Residues in Fruit and Vegetables 16 (EUPT-FV-16)			
EUPT-FV Archive	European Proficiency Test FV 16 PRELIMINARY REPORT AVAILABLE!!			
Workshops 2014 FV-Workshop Workshop Overview EURL Webinars	23-04-2014   EURL-FV EUPT-FV16 Preliminary Results WEBINAR EUPT-FV16 Preliminary Results, PDF Available!!!			



### Workshops 2014 FV-Workshop Workshop Overview EURL Webinars Trainings

Standard Solutions

# AQC Panel NEW EU Procedures EU Procedures Help AQC Documents AG-Workshop Conversion Factors

Library
 LIST OF METHODS
 Last Publications
 CODEX Contributions
 Conference Contributions
 News Archive

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### A - GENERAL TASKS.

A1. Management of administrative duties

A2. EURL-FV web page.

### A3. Development of bilateral cooperation with other organisations:

- Special collaboration with EFSA by agreement with DG SANCO through Ms. Paula Medina Pastor, second national expert in EFSA and former member of the staff at EURL-FV.

New activities:

-Cooperation with the IRMM-JRC (Institute for Reference Materials and Measurements-Joint Research Centre) in a study of the viability of preparing certified reference material.

- Collaboration with the Dutch Institute of Food Safety (RIKILT) in the study of the variability of ion ratios by GC-MS/MS.



### A - GENERAL TASKS.

A1. Management of administrative duties

A2. EURL-FV web page.

A3. Development of bilateral cooperation with other organisations:

A4. Collaboration with the other pesticide residue EURLs.



### **B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS**

B1. Development of validated procedures (including the use of new instrumentation)

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

### Formula	<b>Retention Time</b>	Mass	Compound name
# Formula	RT	Mass	Cpd
C17H16Br2O3	28,12	425,9466	Bromopropylate
C13H9Br2O	28,12	338,9020	Bromopropylate F1
C7H6BrO	28,12	184,9602	Bromopropylate F2
C6H4Br	28,12	154,9496	Bromopropylate F3
C13H12BrCl2N3O	27,92	374,9541	Bromuconazole
C10H8BrCl2O	27,92	292,9136	Bromuconazole F1
C13H12CI2N3O	27,92	296,0357	Bromuconazole F2
C10H9CI2O	27,92	215,0030	Bromuconazole F3
C7H3CI2O	27,92	172,9561	Bromuconazole F4
C8H7CI2	27,92	172,9925	Bromuconazole F5
C13H24N4O3S	24,01	316,1569	Bupirimate
C11H18N3O3S	24,01	272,1069	Bupirimate F1
C11H18N3O	24,01	208,1450	Bupirimate F2
C5H4N2O	24,01	108,0324	Bupirimate F3
C16H23N3OS	23,85	305,1562	Buprofezin
C9H9N2S	23,85	177,0486	Buprofezin F1
C7H5NO	23,85	119,0371	Buprofezin F2
C7H8N	23,85	106,0657	Buprofezin F3
C7H11N2OS	23,85	171,0592	Buprofezin F4
C8H16N2S	23,85	172,1034	Buprofezin F5
Continue			••••

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B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

### Formula	<b>Retention Time</b>	Mass	Compound name	
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C13H12BrCl2N3O	27,92	374,9541	Bromuconazole	
C10H8BrCl2O	27 92	292 9136	Bromuconazole F1	
<b>C</b> 100 PESTIC	IDES			
C 2 FRAGMEN	ITS AT LEAST F	OR EACH P	ESTICIDE	
<b>C</b> RETENTION	<b>TIMES OF EAC</b>	<b>H PESTICID</b>	E _	
Стэпz41140ээ	24,VI	510,1303	Dupinniale	
C11H18N3O3S	24,01	272,1069	Bupirimate F1	
C11H18N3O	24,01	208,1450	Bupirimate F2	
C5H4N2O	24,01	108,0324	Bupirimate F3	
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Continue			••••	

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in EI mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

### # Compound

### 1 Trifluralin

- 2 Dicloran Lindane (HCH-
- 3 Gamma)
- 4 Fonofos
- 5 Propyzamide
- 6 Chlorothalonil
- 7 Tefluthrin
- 8 Parathion-Methyl
- 9 Chlorpyriphos-Methyl
- 10 Vinclozolin
- 11 Tolclofos-Methyl
- 12 Heptachlor
- 13 Malaoxon
- 14 Fenitrothion
- 15 Dichlofluanid
- 16 Malathion
- 17 Chlorpyriphos
- 18 Parathion
- 19 Tetraconazole
- 20 Pendimethalin
- 21 Pyrifenox I
- 22 Tolylfluanid

### Compound

- 23 Chlozolinate
- 24 Chlorfenvinphos
- 25 Fipronil
- 26 Chinomethionat
- 27 Methidathion
- 28 Pyrifenox II
- 29 Endosulfan alpha
- 30 Tetrachlorvinphos
- 31 Hexaconazole
- 32 Prothiofos
- 33 Dieldrin
- 34 Myclobutanil
- 35 Bupirimate
- 36 Chlorfenapyr
- 37 Endosulfan beta
- 38 Ethion
- 39 Ofurace
- 40 Carbophenothion
- 41 Quinoxyfen
- 42 Endosulfan Sulfate
- 43 Fenhexamid

#### # Compound

- 44 Propiconazole
- 45 Trifloxystrobin
- 46 Nuarimol
- 47 Iprodione
- 48 Phosmet
- 49 Bifenthrin
- 50 Fenpropathrin
- 51 Bifenox
- 52 Tetradifon
- 53 Phosalone Lambda-
- 54 Cyhalothrin
- 55 Fenarimol
- 56 Pyrazophos
- 57 Acrinathrin
- 58 Pyridaben
- 59 Fluquinconazole
- 60 Cypermethrin
- 61 Flucythrinate I
- 62 Flucythrinate II
- 63 Fluvalinate-tau
- 64 Azoxystrobin

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

				Moleculai
#	Compound	Rt (min)	Exact Mass	Formula
1	Trifluralin* <sup>,</sup> **	13.888	335.1093	C13H16F3N3O4
2	Dicloran*	14.706	205.9650	C6H4Cl2N2O2
	Dicloran cluster		207.9622	C6H4Cl2N2O2
3	Lindane (HCH-Gamma)*	15.523	252.8912	C6H6CI5
	Lindane (HCH-Gamma)			
	cluster		254.8883	C6H6CI5
4	Fonofos*	15.894	168.9911	C4H10OPS2
	Fonofos F1		109.0112	C6H5S
5	Propyzamide*	15.934	255.0218	C12H11Cl2NO
	Propyzamide F1		187.9670	C7H4Cl2NO
6	Chlorothalonil*	16.645	263.8816	C8CI4N2
	Chlorothalonil cluster		265.8787	C8CI4N2
	Chlorothalonil F1		229.9205	C8CI3N2
7	Tefluthrin*	16.831	241.0243	C9H9O2CIF3
	Tefluthrin F1		205.0476	C9H8F3O2
8	Parathion-Methyl*	18.039	153.9963	C6H4NO2S
	Parathion-Methyl F1		263.0017	C8H10NO5PS
9	Chlorpyriphos-Methyl*	18.053	211.8895	C5HCI3NS
	Chlorpyriphos-Methyl F1		140.9775	C2H6O3PS
* •				

\* Quantifier ion

\*\* Compounds fully identified with MSMS experiment

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### **B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS**

B1. Development of validated procedures (including the use of new instrumentation)

B1.1 Update of the GC-Q-TOF-MS database of exact masses of pesticide fragments in El mode and development of a GC-Q-TOF-MS database in NCI mode. Validation of the corresponding screening methods.

B1.2 Validation of a LC-QTOF HRMS method (request of CEN standardised method).





# B1. Development of validated procedures (including the use of new instrumentation)

B1.3. Study of the efficiency of extensive clean-ups using high speed centrifugation or specific sorbents based on Zr or others.



Particle size: 22 µm= 22000 nm Relative surface area: 310 m<sup>2</sup>/g

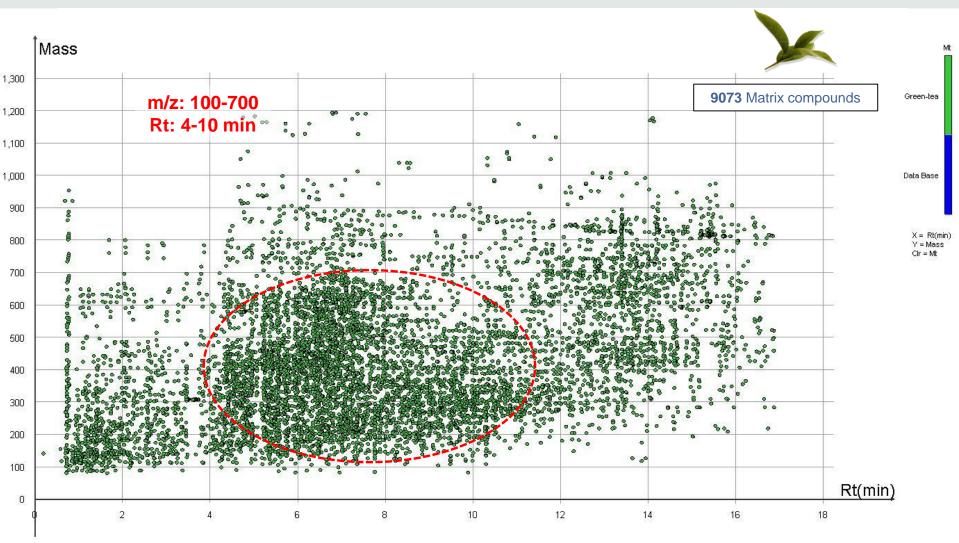


# B1. Development of validated procedures (including the use of new instrumentation)

B1.3. Study of the efficiency of extensive clean-ups using high speed centrifugation or specific sorbents based on Zr or others.

B1.4. Development of a "components map" for all the commodity groups in Document SANCO 12571/2013.

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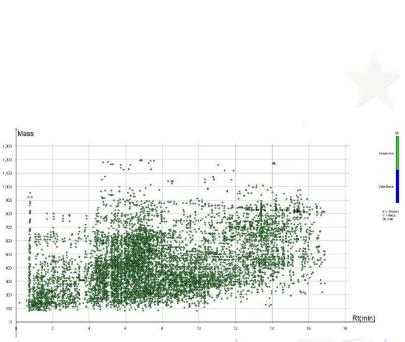


Miner 3D Enterprise

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# B1.4. Development of a "components map" for all the commodity groups in Document SANCO 12571/2013. Annex A Commodity groups and representative commodities<sup>5</sup>





Commodity groups	Typical commodity categories	Typical representative commodities
1. High water content	Pome fruit	Apples, pears
	Stone fruit	Apricots, cherries, peaches,
	Other fruit	Bananas
	Alliums	Onions, leeks
	Fruiting vegetables/cucurbits	Tomatoes, peppers, cucumber, melon
	Brassica vegetables	Cauliflower, Brussels-sprouts, cabbage, broccoli
	Leafy vegetables and fresh herbs	Lettuce, spinach, basil
	Stem and stalk vegetables	Celery, asparagus
	Forage/fodder crops	Fresh alfalfa, fodder vetch, fresh sugar beets
	Fresh legume vegetables	Fresh peas with pods, peas, mange tout,
		broad beans, runner beans, French beans
	Leaves of root and tuber vegetables	Sugar beet and fodder beet tops
	Fresh Fungi	Champignons, canterelles
	Root and tuber vegetables or feed	Sugar beet and fodder beet roots, carrots, potatoes, sweet potatoes
2. High acid content	Citrus fruit	Lemons, mandarins, tangerines, oranges
and high water	Small fruit and berries	Strawberry, blueberry, raspberry, black
content <sup>6</sup>	-	currant, red currant, white currant, grapes
	Other	Kiwifruit, pineapple, rhubarb
<ol> <li>High sugar and low water content<sup>7</sup></li> </ol>	Honey, dried fruit	Honey, raisins, dried apricots, dried plums, fruit jams
4a. High oil content and	Tree nuts	Walnuts, hazelnuts, chestnuts
very low water content	Oil seeds	Oilseed rape, sunflower, cotton-seed, soybeans, peanuts, sesame etc.
	Pastes of tree nuts and	Peanut butter, tahina, hazelnut paste
	oil seeds	
	Oils from tree nuts, oil	Olive oil, rapeseed oil, sunflower oil, pumpkin
	seeds and oily fruits	seed oil
4b. High oil content and intermediate water	Oily fruits and products	Olives, avocados and pastes thereof
content 5. High starch and/or	Dry legume	Field bean, dried broad bean, dried haricot
protein content and low water and fat	vegetables/pulses	bean (yellow, white/navy, brown, speckled), lentils
content	Cereal grain and products thereof	Wheat, rye, barley and oat grain; maize, rice Wholemeal bread, white bread, crackers, breakfast cereals, pasta
6. "Difficult or unique commodities"		Hops Cocoa beans and products thereof, coffee, tea Spices

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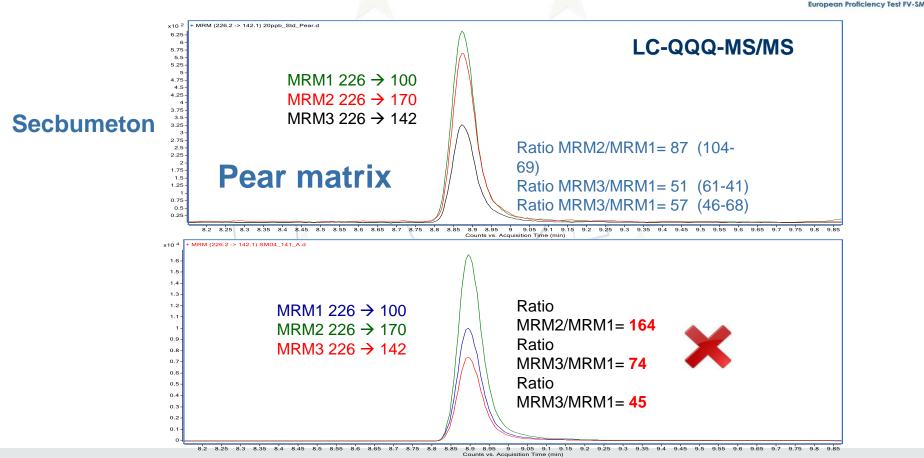
# B1. Development of validated procedures (including the use of new instrumentation)

B1.3. Study of the efficiency of extensive clean-ups using high speed centrifugation or specific sorbents based on Zr or others.

B1.4. Development of a "components map" for all the commodity groups in Document SANCO 12571/2013.

B1.5 Evaluation of interferences between matrix-analyte for the correct identification of the pesticides by GC-QQQ-MS/MS and LC-QQQ-MS/MS

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EUPT-FV-



# B1. Development of validated procedures (including the use of new instrumentation)

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B1.5 Evaluation of interferences between matrix-analyte for the correct identification of the pesticides by GC-QQQ-MS/MS and LC-QQQ-MS/MS

# B1.6 Development of an analytical method by LC-microflow coupled to HRMS (QTOF).

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B1.6 Development of an analytical method by LC-microflow coupled to HRMS (QTOF).

	I.D. Column (mm)	Flow (µL/min)	Nebulizer	I.D. column (mm)	Flow (µL/min)	Sensitivity Theoretically Gain
Standard LC	8.0 – 1.6	200 - 5000	Standard	4.6	400	1
				2.1	200	5
micro LC	0.3 – 1.0	5 - 200	Micro	1	40	20
				0.8	20	30
				0.5	35	≈ 30

Most used

# Standard-LC vs micro-LC

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### **B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS**

B1. Development of validated procedures (including the use of new instrumentation)

B2. Follow-up of the implementation of the developed methods by the Official Laboratories.

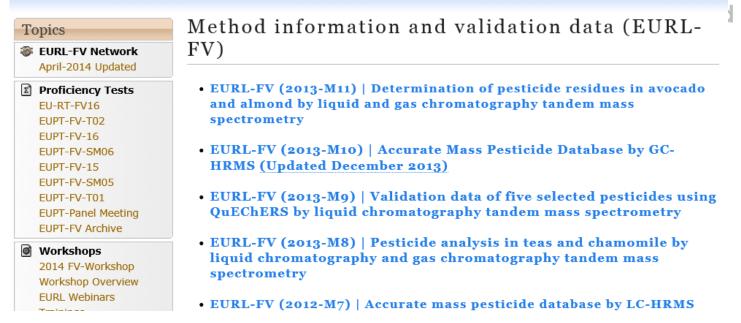


### **B - DEVELOPMENT AND VALIDATION OF ANALYTICAL METHODS**



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### <u>C -QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMME, INCLUDING</u> <u>THE ORGANISATION OF PROFICIENCY TESTS AND INTERCOMPARATIVE</u> <u>STUDIES.</u>

C1. Update of EU Guidelines on Quality Control Procedures.

### ANALYTICAL QUALITY CONTROL AND METHOD VALIDATION PROCEDURES FOR PESTICIDE RESIDUES ANALYSIS IN FOOD AND FEED

Document N° SANCO/12571/2013 Supersedes Document No. SANCO/12495/2011 Implemented by 01/01/2014

### SANCO/xxxx/2015

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### <u>C -QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMME, INCLUDING</u> <u>THE ORGANISATION OF PROFICIENCY TESTS AND INTERCOMPARATIVE</u> <u>STUDIES.</u>

- C1. Update of EU Guidelines on Quality Control Procedures.
- C2. Organisation and Development of Proficiency Tests and Intercomparative studies.



C2.1 Development and conduction of EUPT-FV17.

- C2.2 EU Intercomparative Study on Mass Spectrometry Screening Methods 07 (EUPT-FV-SM07).
- C2.3 EU Intercomparative Study on incurred fresh herbs 01 (EUPT-FV-FH01).

C2.4 Ring Test of certified standard solution of EUPT-FV17 (EU-RT-FV17).



### <u>C -QUALITY ASSURANCE AND QUALITY CONTROL PROGRAMME, INCLUDING</u> <u>THE ORGANISATION OF PROFICIENCY TESTS AND INTERCOMPARATIVE</u> <u>STUDIES.</u>

C1. Update of EU Guidelines on Quality Control Procedures.

C2. Organisation and Development of Proficiency Tests and Intercomparative studies.

C3. Establishment of criteria for defining underperformance in multiresidue methods.



# D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.

D1. Joint EURL-SRM/FV/CF/AO Workshop for Pesticide Residues.





# D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.

D1. Joint EURL-SRM/FV/CF/AO Workshop for Pesticide Residues.

**D2. Advisory Group Expert Meeting** 



# D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.

D1. Joint EURL-SRM/FV/CF/AO Workshop for Pesticide Residues.

**D2. Advisory Group Expert Meeting** 

D3. Technical assistance to DG SANCO.

D3.1. Support to COM and EFSA

D3.2. Assistance to COM in drawing up the coordinated multiannual control programme of the Union.



# D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.

- D1. Joint EURL-SRM/FV/CF/A
- D2. Advisory Group Expert N
- D3. Technical assistance to I D3.1. Support to CO D3.2. Assistance to multiannual control

### D4. Training for the NRLs.



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### D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.





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### D - TECHNICAL AND SCIENTIFIC SUPPORT TO DG SANCO, EU MEMBER STATES AND THIRD COUNTRIES INCLUDING THE ORGANISATION OF COURSES AND WORKSHOPS.

D5. Assistance to Third Countries.

D6. Webinars

D7. Arbitration in the event of litigation



# Thank You for Your Attention



