

**DISCUSSION PAPER
ON THE EVALUATION OF DISTRIBUTION
OF PESTICIDE RESIDUES
AFTER PRIMARY PROCESS
IN CITRUS FRUIT, POME FRUIT, OILSEEDS
AND WINE GRAPES**

**EU Community Reference Laboratory for Pesticide
Residues in Fruits and Vegetables**

OECD/OCDE

508

Adopted: 3 October 2008

OECD GUIDELINE FOR THE TESTING OF CHEMICALS

Magnitude of the Pesticide Residues in Processed Commodities

PURPOSE

2. Studies on the magnitude of residues in processed commodities provide data on the transfer of residues to different processed commodities from the raw agricultural commodity (RAC). Studies on the magnitude of residues are conducted in order to quantify levels of residues in processed commodities and to provide the distribution of residues (active ingredient, and/or metabolites, degradation products) in various processed products resulting from the processing of a commodity. This information about dilution and concentration of residues and the estimation of processing factors (the ratio of residue levels in processed commodities to those in the raw agricultural commodity) is used to:

- conduct refined dietary exposure assessments with primary processed products to assess consumer safety;
- provide results on residues in commodities that may be used as animal feedstuffs and thus to allow a more realistic calculation of the dietary burden of livestock;
- establish MRLs for processed commodities; and
- monitor compliance with the RAC MRL.

8. The processing factor (P_f) that originates only from residues of the same single compound in the RAC is calculated as follows:

$$P_f = \frac{\text{residue level in processed commodity}}{\text{residue level in the RAC or commodity to be processed}}$$

14. Important conclusions concerning the behaviour of the active ingredient and/or metabolites during processing can be drawn from the distribution coefficients for n-octanol/water, hydrolysis stability, heat stability and solubility behaviour. For example, when the log Pow is greater than three, it can be assumed that the residue will likely be concentrated in oil or solids like meal, whereas high water solubility indicates that residues may be expected in juices. For example: the extremely high potential concentration factors for citrus oil ($P_f = 1000$) and mint oil ($P_f = 330$) should be considered.

ENV/JM/MONO(2008)23
29 Jul 2008

**OECD GUIDANCE DOCUMENT ON MAGNITUDE
OF PESTICIDE RESIDUES IN PROCESSED COMMODITIES**

Study Number	Commodity	Thiacloprid (mg/kg)	Pf
1	Tomato (RAC)	0.24	-
	Paste	0.48	2.0
2	Tomato (RAC)	0.07	-
	Paste	0.22	3.1

The mean processing factor for tomato paste, 2.6, may be used for both monitoring (enforcement) and dietary exposure assessment considerations.

Thiacloprid: Pkow = 1.26; water solubility= 185 mg/L

Further Consideration of Processing as Related to the Establishment of MRLs for Processed Foods: Recommendations on Principles and Practices

Prepared by the USA and the EC

1. Processing studies should be mandatory for a short list of commodities (about 16 at that time). Proposed MRLs for the relevant raw agricultural commodities may not advance to Step 8 without processing studies deemed acceptable by the JMPR.
2. CXLs or processing factors should be established for those processed commodities where a significant increase (>1.3 times) of residue of concern occurs from the RAC to the processed commodity. It should be decided in advance for which commodities and for which processing factors, they will be established.
3. CXLs or processing factors should be established for processed commodities where a significant decrease in residue occurs from RAC to processed commodity *and* the processing factor must be considered in order to achieve a satisfactory dietary exposure assessment. It should be decided in advance for which commodities *and* for which processing factors they will be established.
4. A limited number of generic default processing factors should be established for some predefined common processes, such as dehydration. These would be useful for risk assessment purposes.

1. Mandatory Processing Studies

The need for a processing study is based on a consideration of the 5 factors above. A series of large industrial scale processes are listed, and studies are usually considered essential for these processes. These processes include the preparation of:

- • fruit juice (and byproducts)
- • alcoholic beverages (such as fermentation, malting, brewing, distillation)
- • vegetable juices
- • oils
- • milling fractions
- • silage (livestock only)
- • sugar

**Further Consideration of Processing as Related to the
Establishment of MRLs for Processed Foods: Recommendations
on Principles and Practices**

Prepared by the EC and the USA

Commodities with Established Dehydration Factors

Raw agricultural commodity	Processed product	Dry matter content in RAC	Dry matter in dried product	Theoretical processing factor
FT 0297 Figs	Fruit, dried	22%	74%	3.4
FB 0269 Grapes	Fruit, dried	18%	85%	4.7
Grass	Hay	20%	86%	4.3
FS 0014 Plums	Prunes	20%	70%	3.5
FP 226 Apple	Fruit, dried	17%	68%	4.0
FS 240 Apricot	Fruit, dried	14%	69%	4.9
FP 230 Pear	Fruit, dried	16%	73%	4.6
VO 448 Tomato	Tomato, sun dried	6.1%	85%	14
VO 445 Peppers Sweet	Sweet pepper, dry	9%	92.9%	10
VO 444 Peppers chili	Chili pepper, dry	13%	92.9%	7



20-25/04/2009

**APPLE JUICE**Pesticid
Community

ON

ORANGE JUICEPe
Comm

ADRIA

ADRIA

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ON





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**OLIVE OIL**

Con


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Thiabendazole Distribution



1000 g Orange
1 mg pesticide
1 mg/kg



330 g Juice (33%)
0.03 mg pesticide (3%)
0.09 mg/kg



670 g Wet Pomace (67%)
0.96 mg pesticide (96%)
1.43 mg/kg



420 g Pulp (42%)
0.13 mg pesticide (13%)
0.31 mg/kg

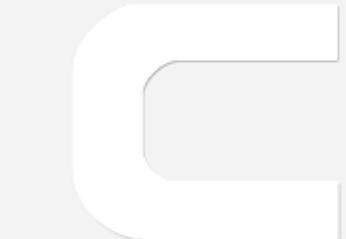
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250 g Peel (25%)
0.81 mg pesticide (81%)
3.24 mg/kg

Thiabendazole pKow=2.4		Pesticide Distribution %	Mass (g)		Pesticide Mass (mg)	Pesticide Concentration (mg/kg)	
Orange		100	1000		1	1	
Juice		3	330		0.03	0.09	
Wet Pomace	Pulp	96	13	420	0.13	1.43	0.31
	Peel	670	81	250	0.81		3.24

EXPERIMENTAL



Pesticides in Fruits And Vegetables

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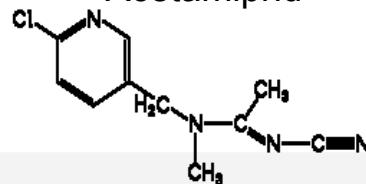
PESTICIDES USED

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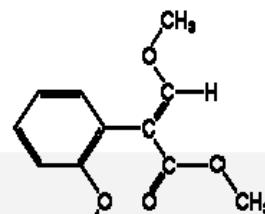
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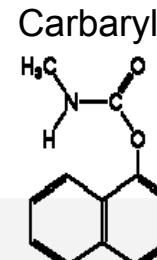
Acetamiprid



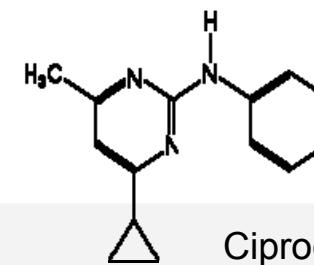
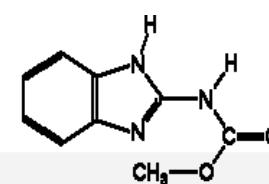
Azoxystrobin



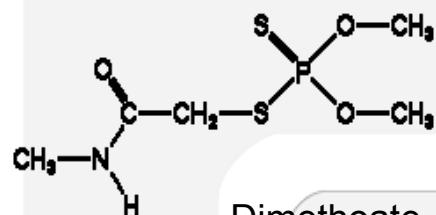
Carbaryl



Carbendazim

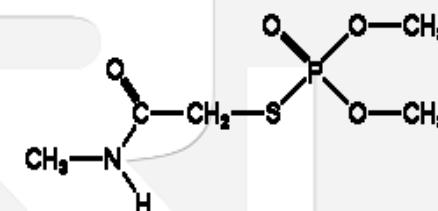


Ciprodinil

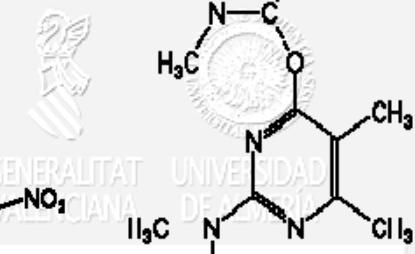


Dimethoate

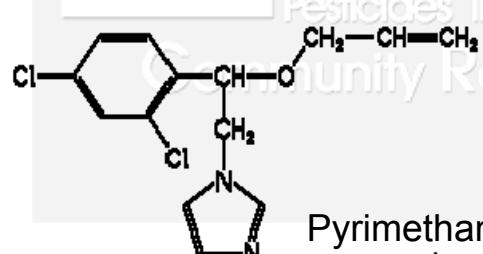
Omethoate



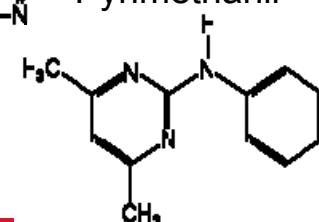
Pirimicarb



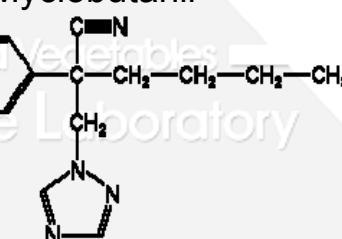
Imazalil



Pyrimethanil



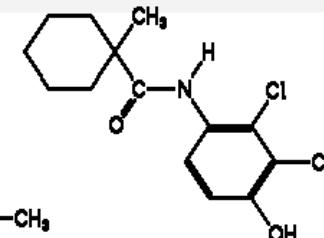
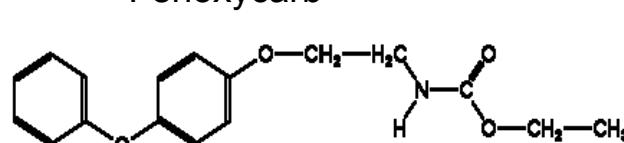
Myclobutanil



Imidacloprid

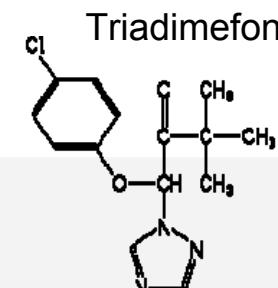
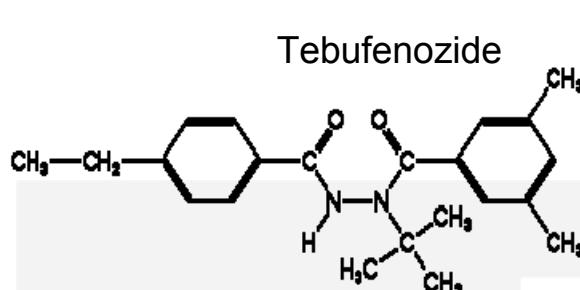



Fenoxycarb

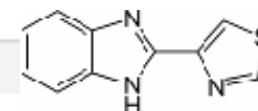
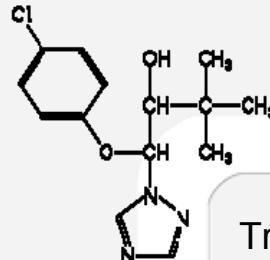
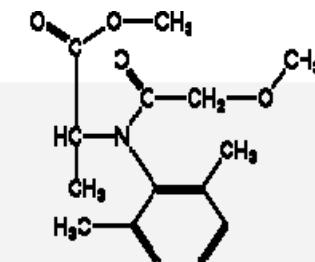
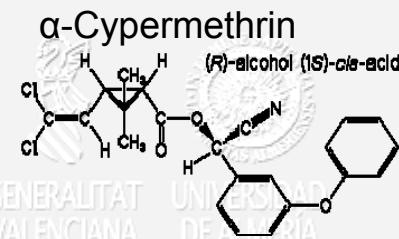


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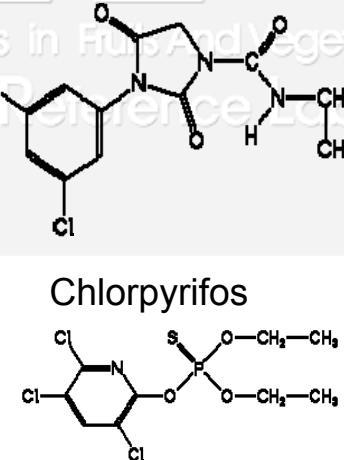
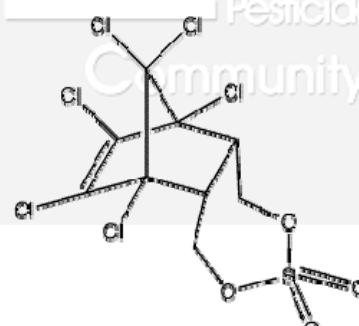


Metalaxyl

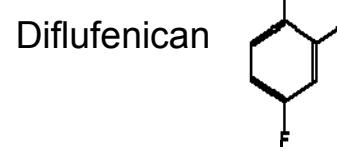
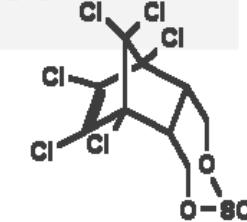
 β -Cyfluthrin

Endosulfan sulfate

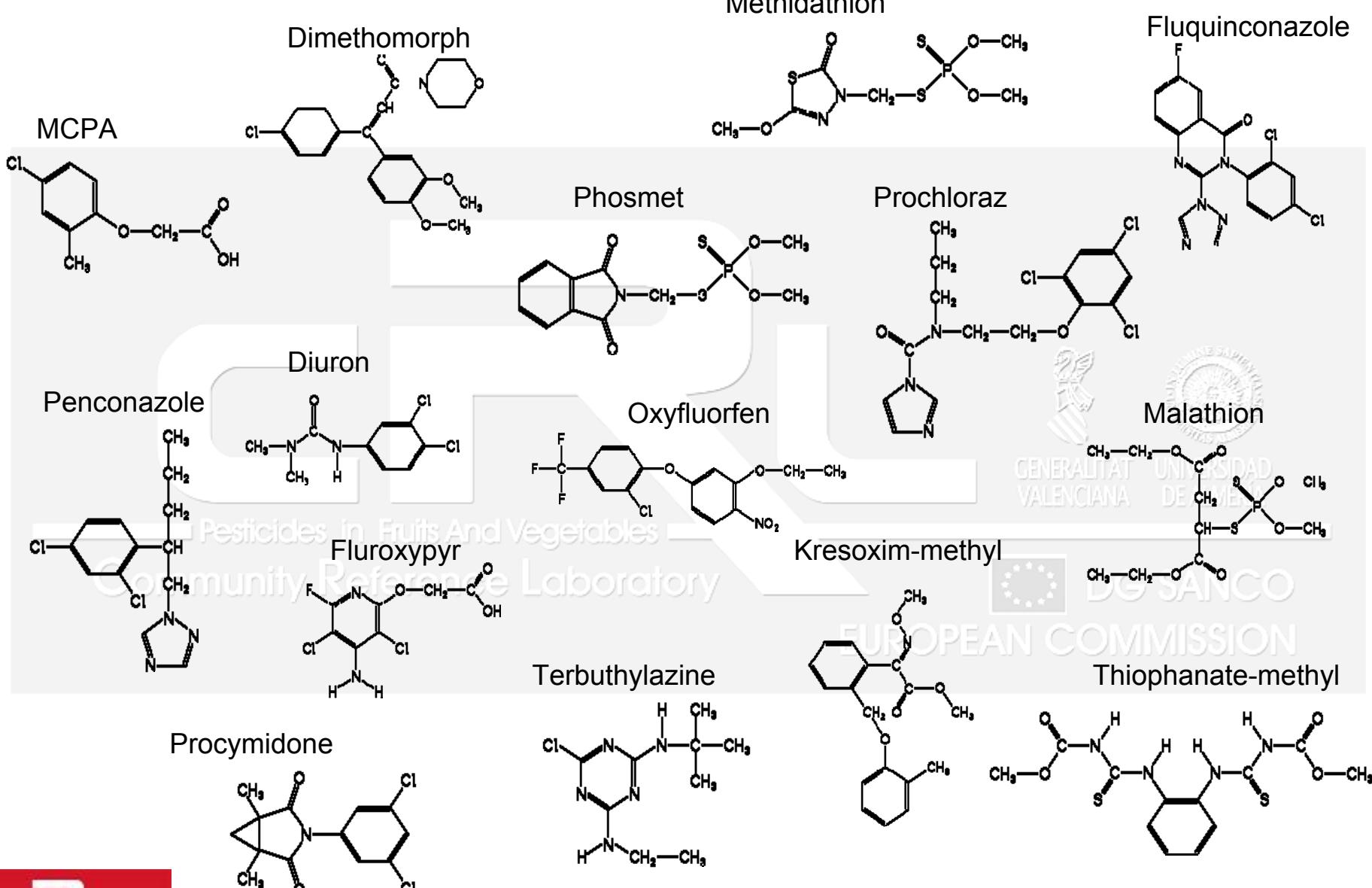
Iprodione



Difenoconazole



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Grapes	
Pesticide	Log Kow
Imidacloprid	0,57
Dimethoate	0,7
Acetamiprid	0,8
Carbendazima	1,51
Carbaril	1,85
Thiabendazol	2,39
Azoxystrobin	2,5
Dimethomorf	2,7
Procymidone	3,14
Kresoxim methyl	3,4
Penconazole	3,72
Imazalil	3,82
Ciprodynil	4

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Olives

Pesticide	Log Kow
MCPA	-0.71
Methidathion	2.2
Malathion	2.75
Diuron	2.85
Phosmet	2.95
Terbutylazine	3.21
Difenconazole	4.2
Oxyfluorfen	4.47
Chlorpyriphos	4.70
α -Endosulfan	4.74
β -Endosulfan	4.79
Diflufenican	4.9
β -Cyfluthrin	5.9
α -Cypermethrin	6.6

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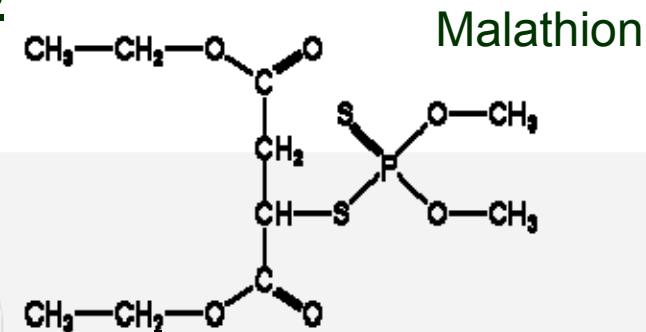
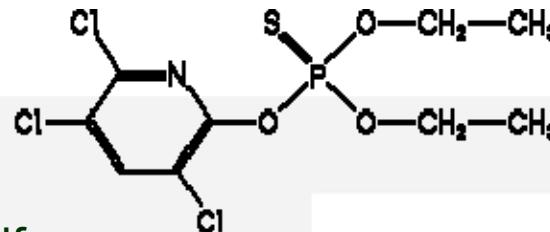

Apple and Orange	
Pesticide	Log Kow
Dimethoate	0.7
Omethoate	-0.74
Acetamiprid	0.8
Thiacloprid	1.26
Carbendazim	1.5
Pirimicarb	1.7
Metalaxyl	1.75
Carbaril	1.85
Thiabendazole	2.4
Azoxystrobin	2.5
Pyrimethanil	2.84
Myclobutanil	2.94
Iprodione	3.0
Triadimenol	3.08, 3.28
Triadimefon	3.11
Fluquinconazole	3.24
Fenhexamid	3.51
Fenoxicarb	4.07
Tebufenozide	4.25
Imazalil	4.6



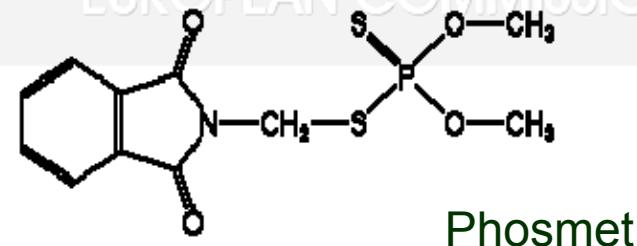
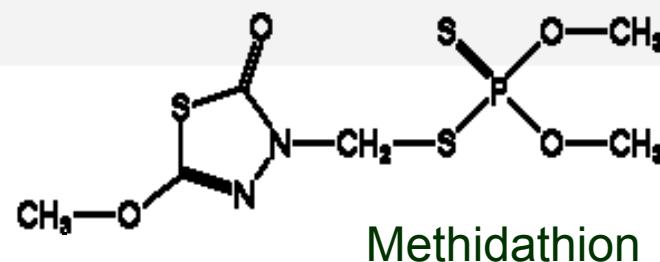
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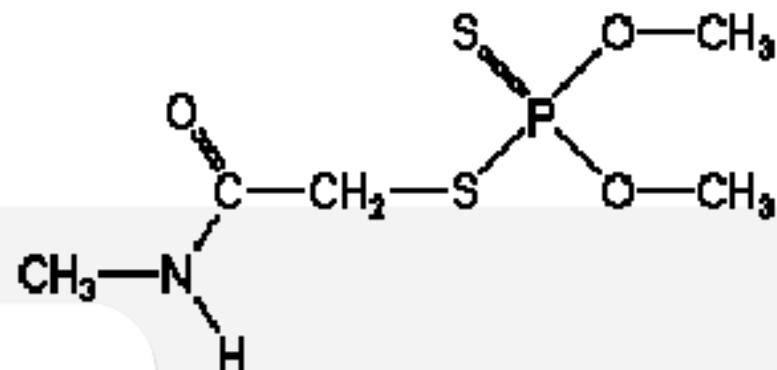
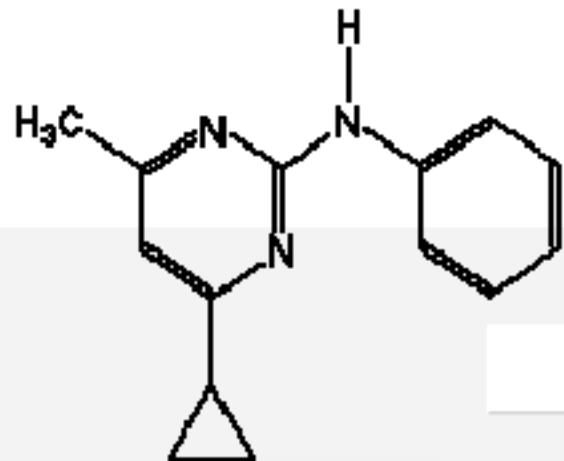
Olives

Pesticide	pK _{ow}	S _w (mg/L)
Chlorpyrifos	4.70	1.40
Malathion	2.75	145
Methidathion	2.20	200
Phosmet	2.95	25





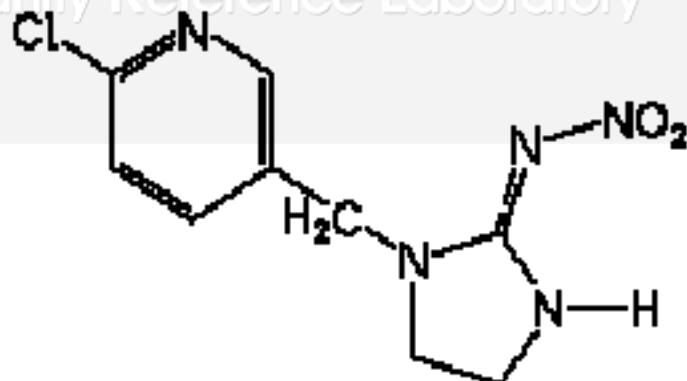
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Pesticida	Log Kow	S_w (mg/L)
Ciprodinil	4	13

Pesticida	Log Kow	S_w (mg/L)
Dimetoato	0.7	23800

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Pesticida	Log Kow	S_w (mg/L)
Imidacloprid	0.57	610

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Apple

	Recommended Dose	Elapsing Time (days)	MRL (mg/kg) Reg. (EC) No 396/2005	Status under Directive 91/414/EEC	Dose in fruit (mg/kg) estimation
Acetamiprid	25-35% (20% p/p)	14	0.1	Included	7
Azoxystrobin			0.05	Included	
Carbaryl	100-200g/hl (85% p/v)	7	0.05	Not included	1.7
Carbendazim	60g/hl (50% p/v)	15	0.2	Included	3
Dimethoate			0.02	Included	
Fenhexamid			0.05	Included	
Fenoxy carb	0.2-0.3% (25% p/p)	21	1	Not included	15
Imazalil	125 cc/hl (34% p/v)	30	2	Included	4
Iprodione	0.15-0.23% (50% p/v)	21	5	Included	75
Metalaxil			1	Not included	
Myclobutanil	0.04-0.05% (12.5% p/v)	28	0.5	Not included	6
Omethoate			0.02	Not included	
Pirimicarb	0.1% (50% p/p)	7	2	Included	50
Pyrimethanil	0.75-1 l/ha	28	5	Included	8
Tebunofenozide	0.075% (24% p/v)	Not applicable	1	Not included	2
Thiabendazol	0.15-0.2% (60% p/v)	Not applicable	5	Included	2
Thiacloprid	0.025-0.03% (48% p/v)	14	0.3	Included	3
Triadimefon	50cc/hl (25% p/v)	15	0.2	Not included	0.5
Triadimenol	50cc/hl (25% p/v)	15	0.2	Included	0.5

Juice processing



Vegetables
Laboratory

Imazalil
Thiophanate methyl
Thiabendazole
Prochloraz



332 g (33%)

675 g (67%)



Solid Residue

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Postharvest treatment

aprox.1 ppm

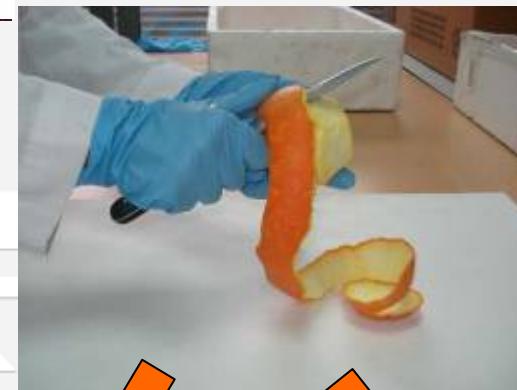


1630 g



Vegetables
Laboratory

Imazalil
Thiophanate methyl
Thiabendazol
Prochloraz



389 g (24%)



1241 g (76%)

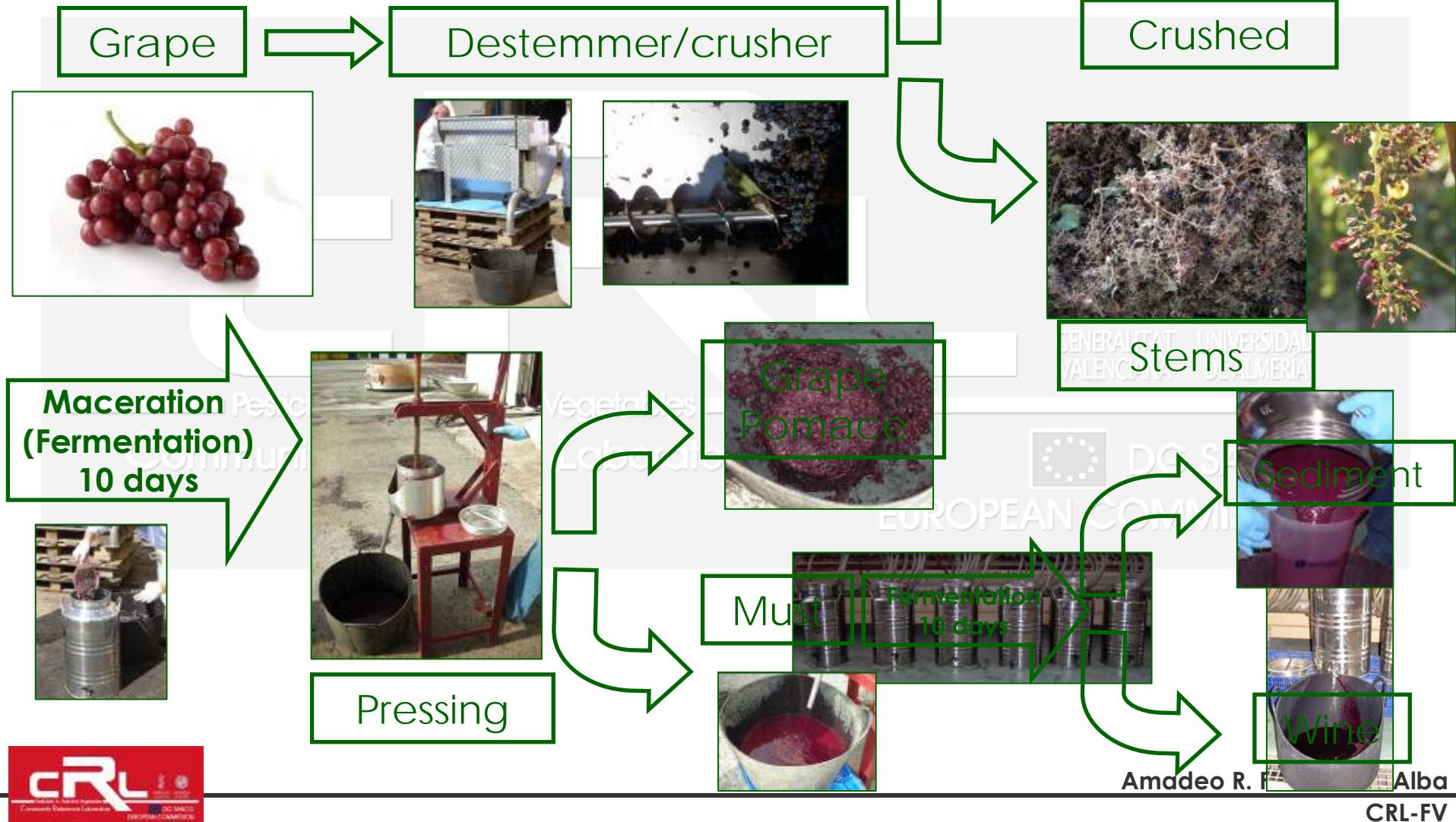


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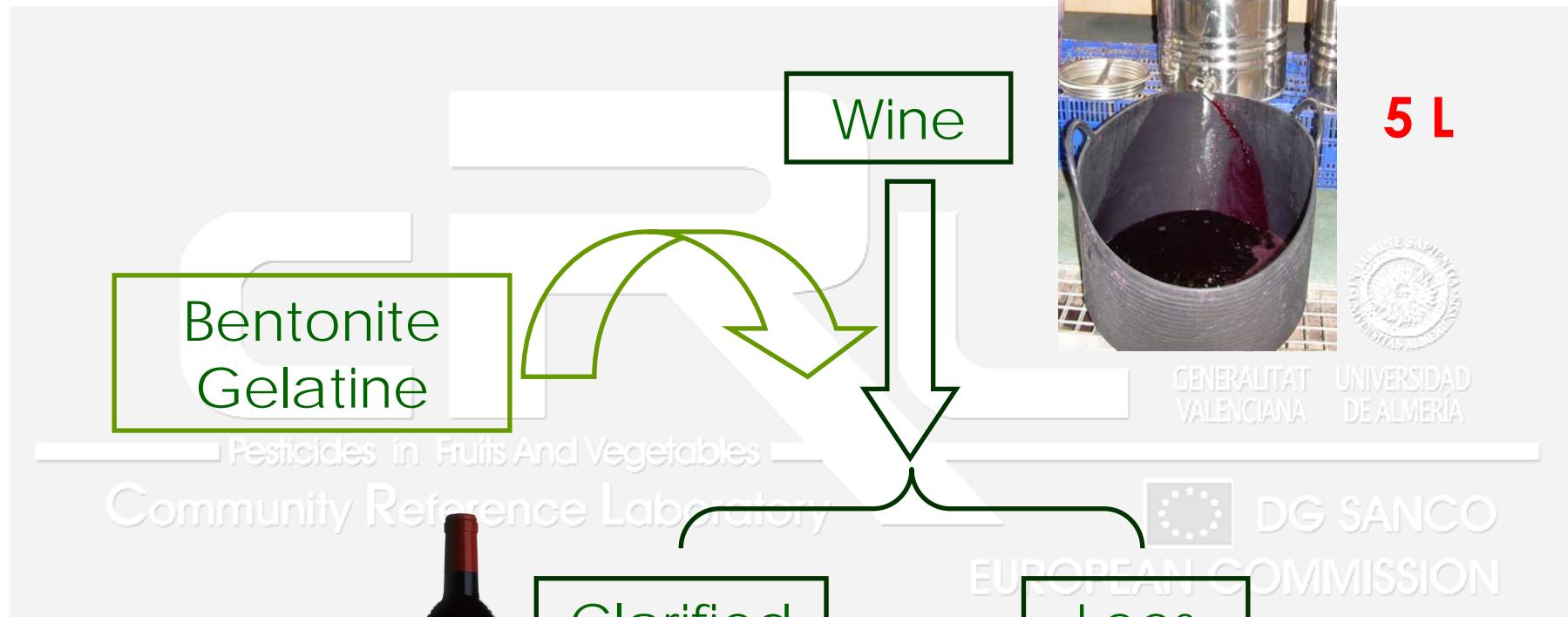
Introduction

- Wine process



Introduction

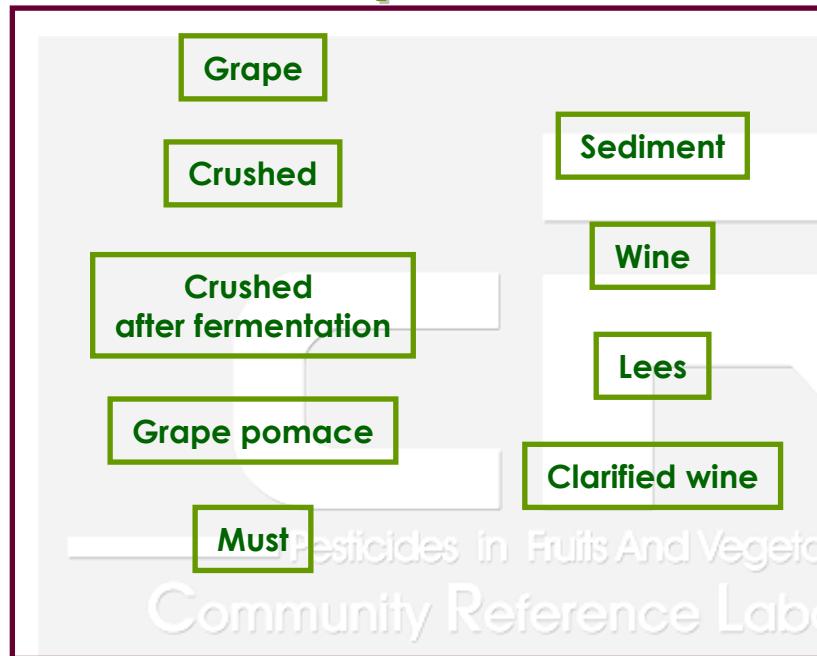
- Wine process



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Introduction

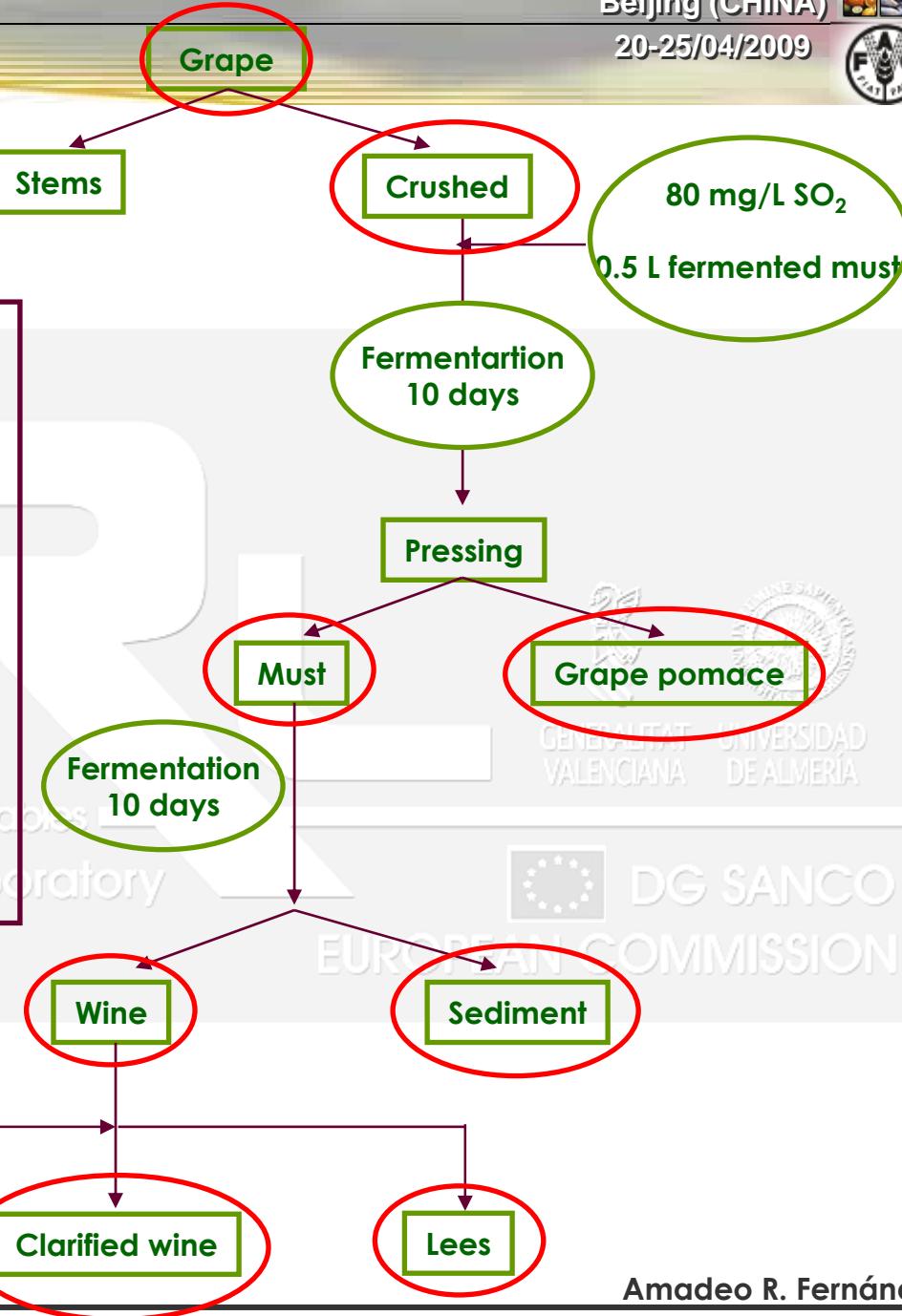
- Wine process



5 replicates and a blank

3 analysis of each

Bentonite and gelatin



Introducción

- **Pesticidas**

Azoxystrobin
 Carbendazima
 Ciprodynil
 Dimethomorf
 Imazalil
Kresoxim methyl
 Penconazole
 Procymidone
 Thiabendazol

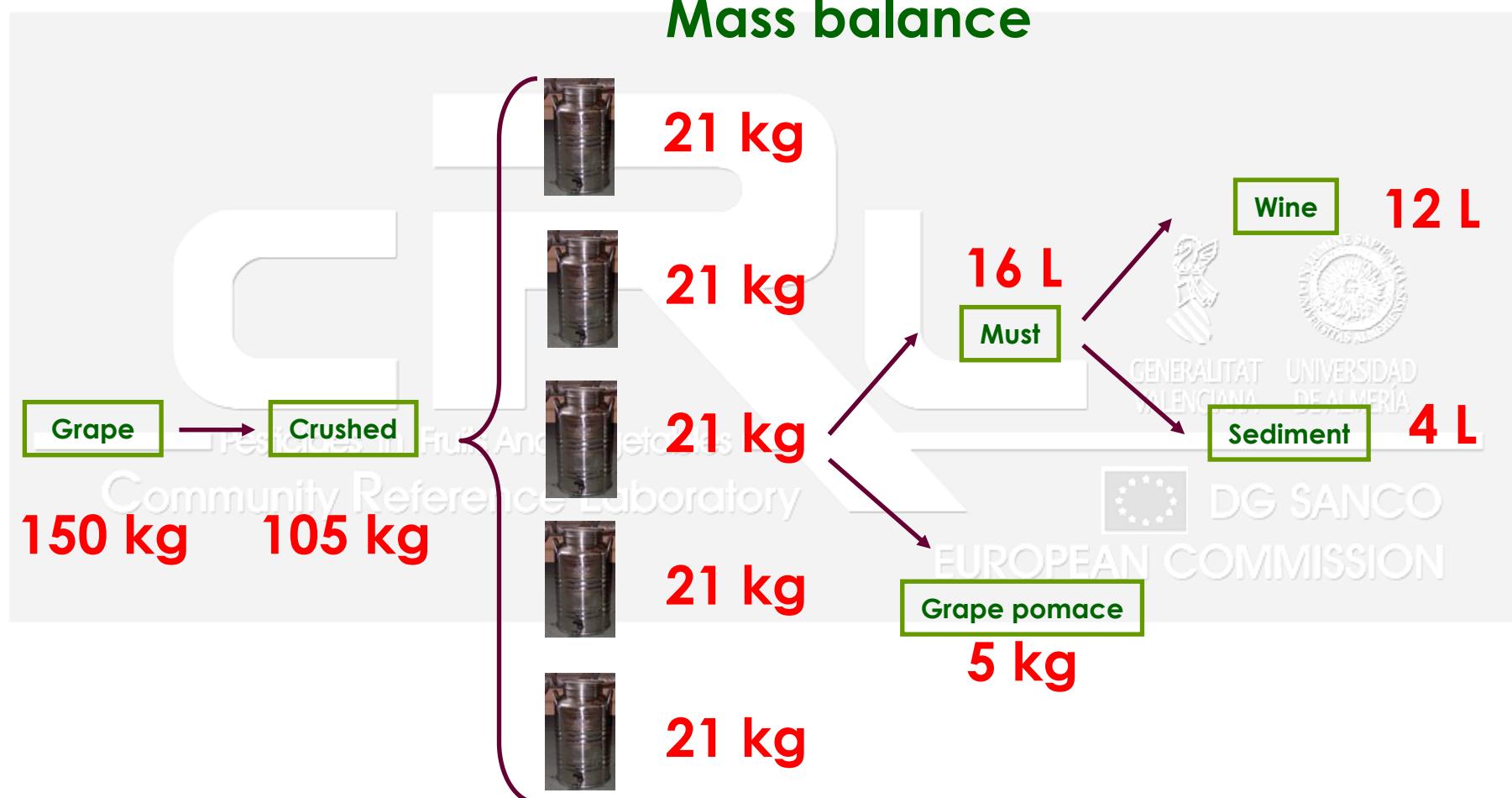
Fungicidas

Acetamiprid
Carbaril
 Dimethoate
 Imidacloprid

Insecticidas

Introduction

- Wine process



**Olive Paste**

100mL of
water are
added

Olive Oil Process



30 min. stirring



The rest is the Alpechin



Centrifuge it for 1 min



Supernatant
Olive Oil



15 min.
centrifuge
at 3500rpm



Olive Pomace

Gravity decantation

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RESULTS

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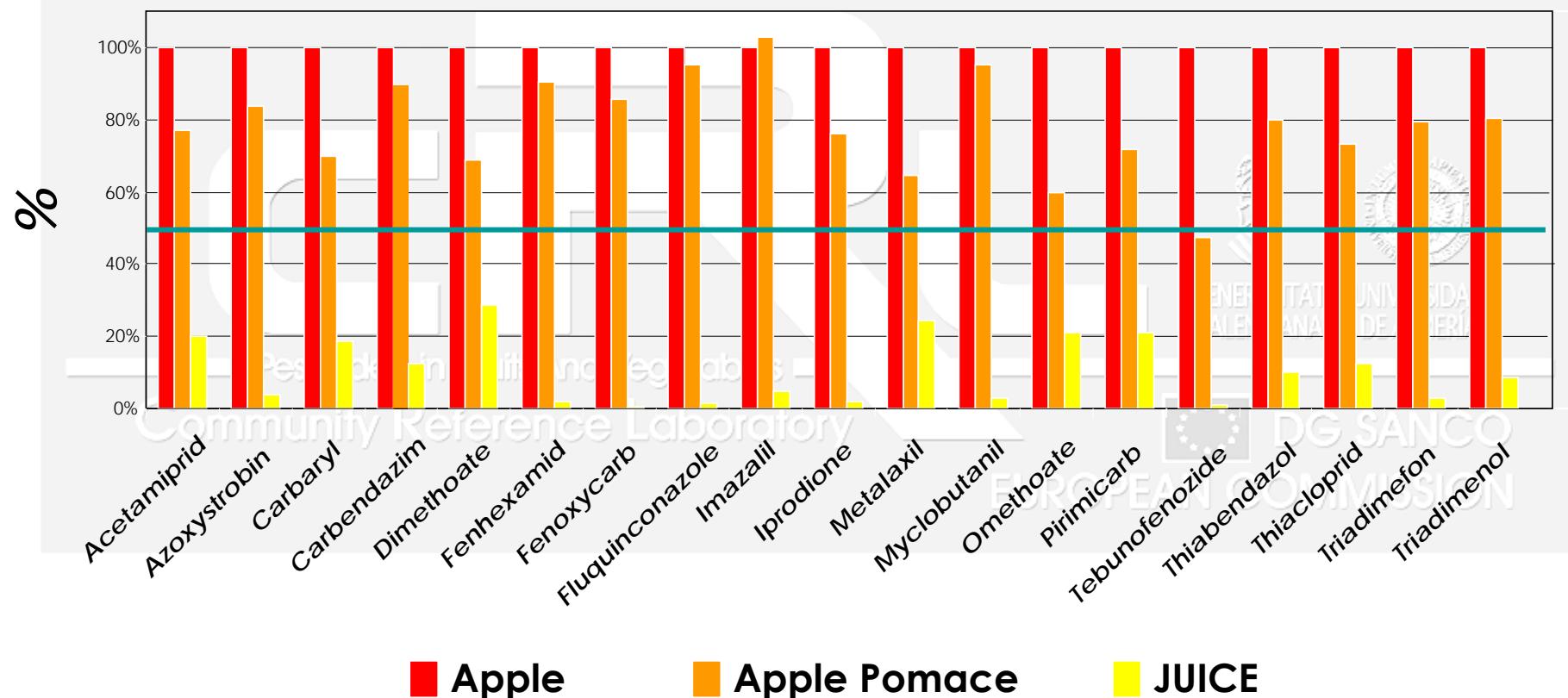
Yields obtained

$$\% \text{ of Juice(Oil/Wine)} = \frac{\text{Volume obtained (mL)} \times \text{density (g/mL)}}{\text{Initial Weight (g)}}$$

RAC	% Juice	%
Olive oil	15-20%	Pomace (65-70%)
Orange	35-63%	Peel(12%-25%) Pulp(30%-42%)
Apple	40-50%	Pomace (50%)
Wine	55-70%	Pomace (30%)

Results on Apples

- Pesticide distribution %**

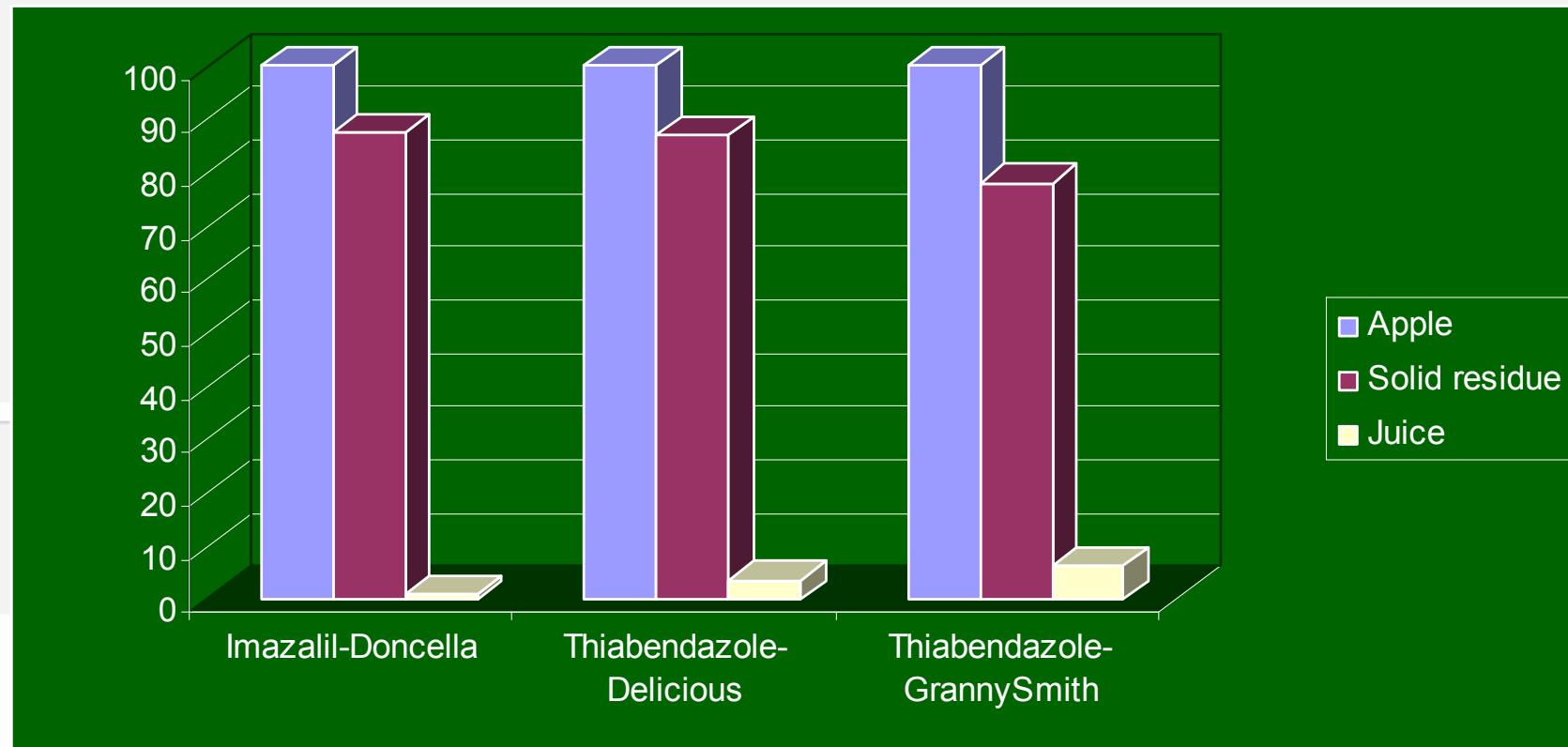

■ Apple

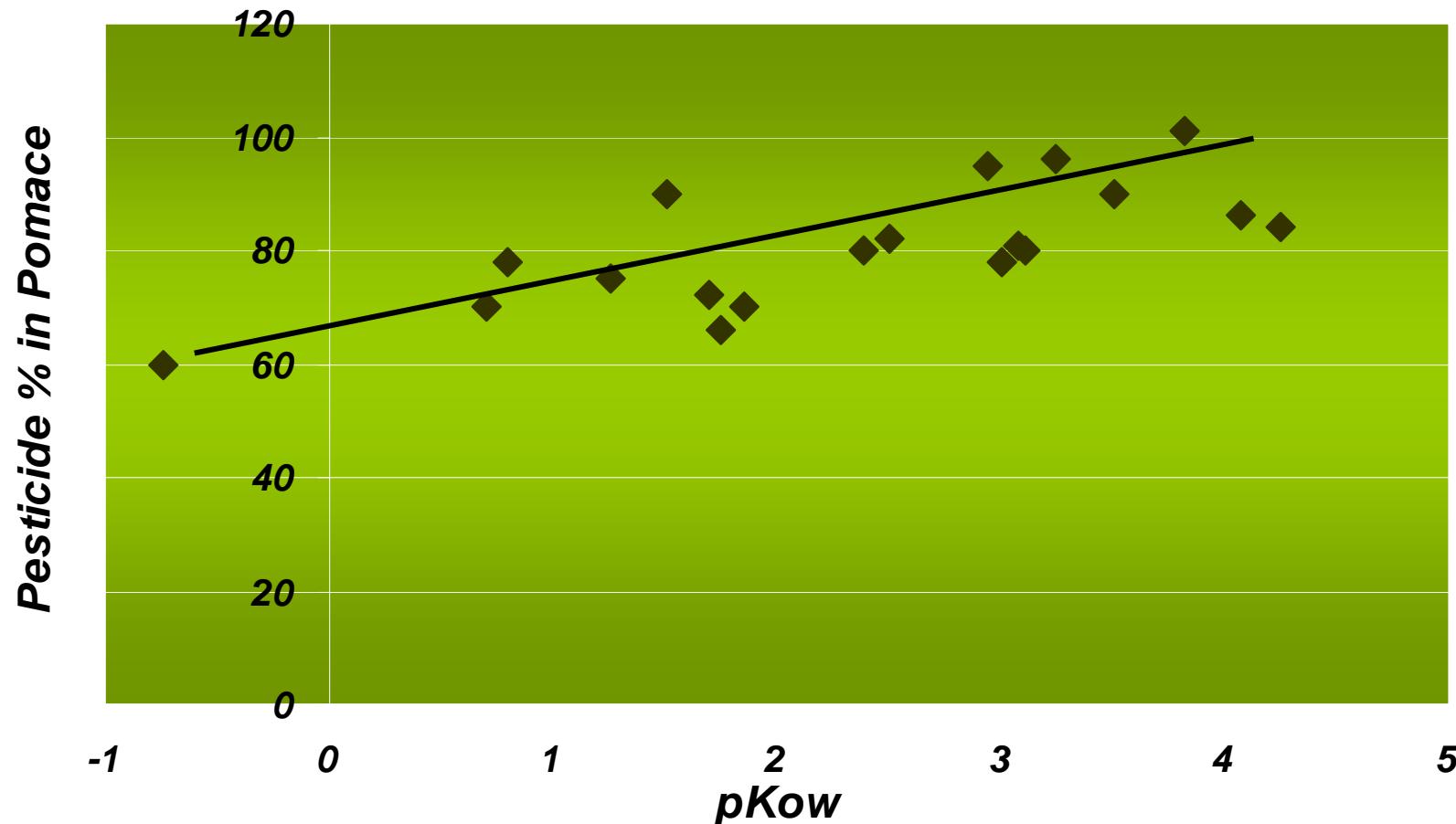
■ Apple Pomace

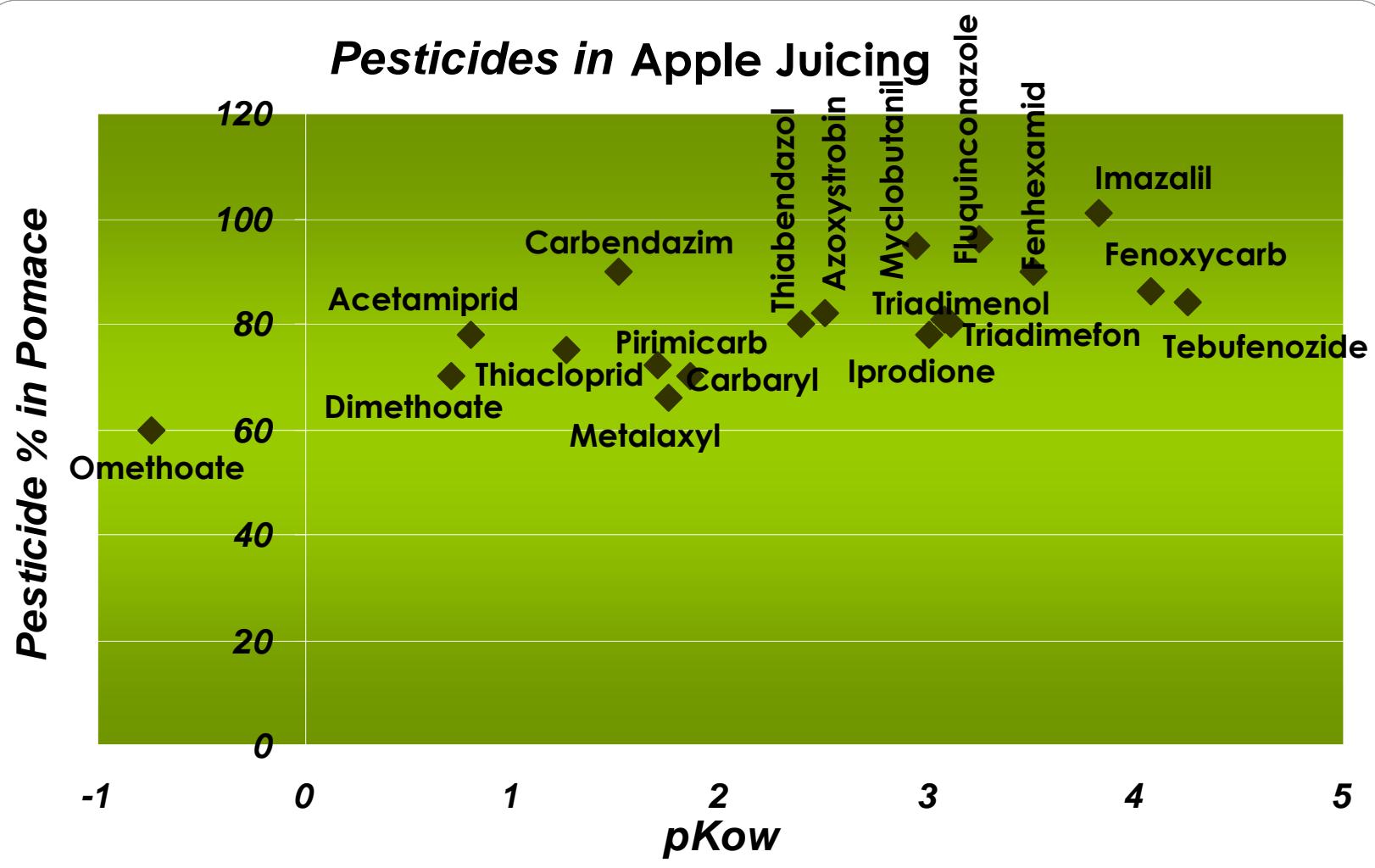
■ JUICE

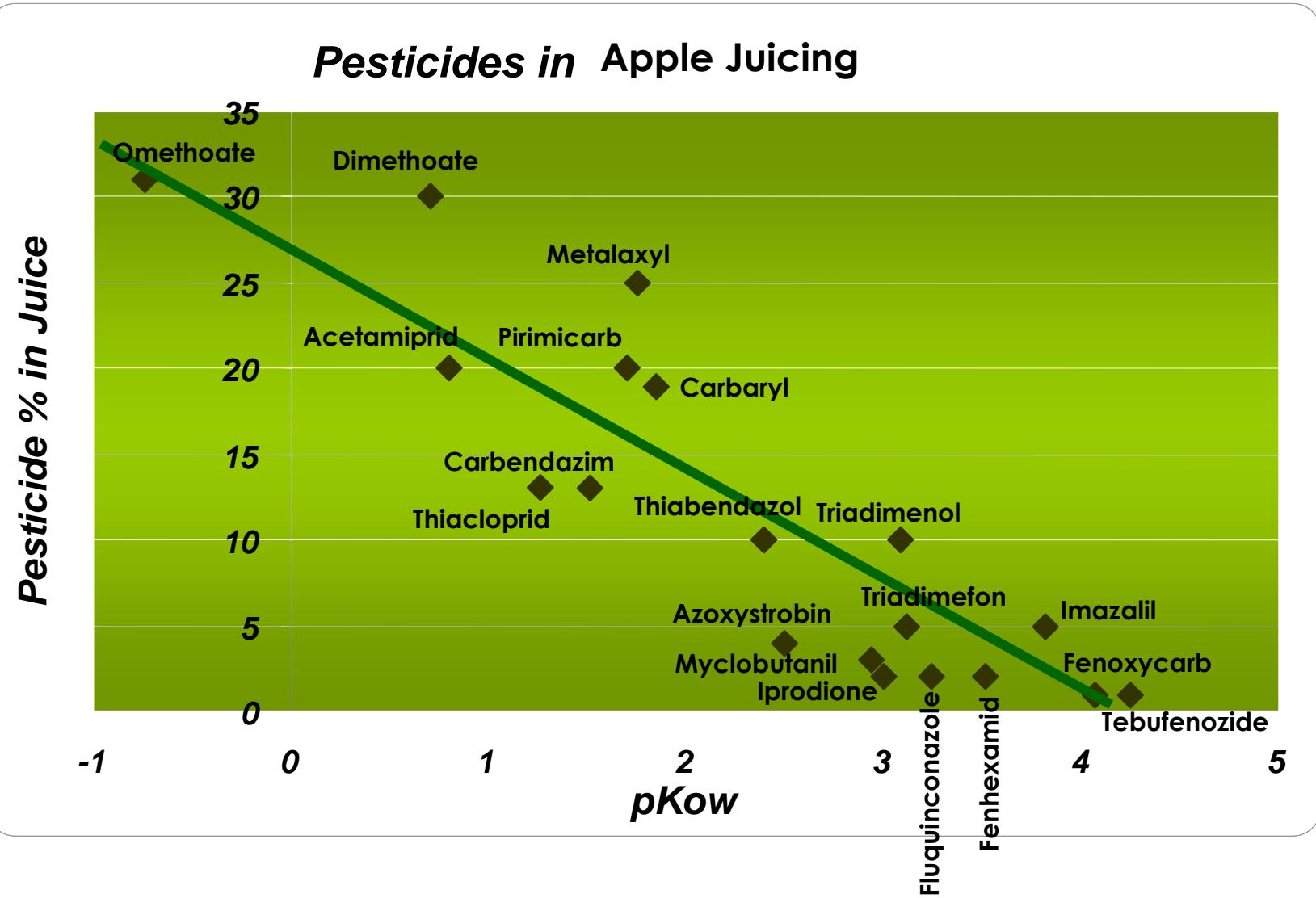
Results

- Real samples analysis

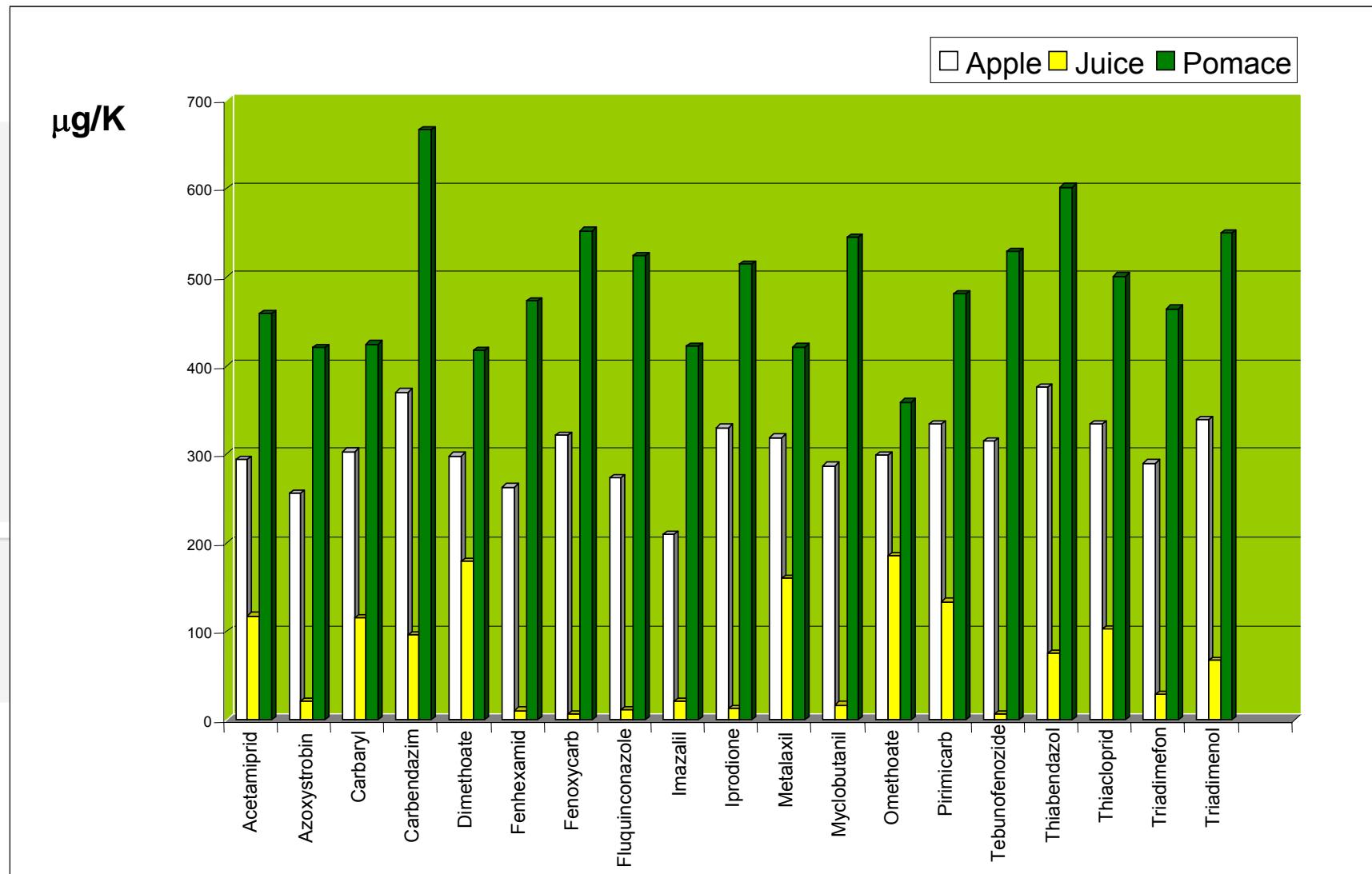


Pesticides in Apple Juicing





Results • Pesticide distribution-concentration



Pesticide	Log Kow	Water solubility	
Thiacloprid	1.26	184 mg/L	
Carbendazim	1.50	8 mg/L	
Pirimicarb	1.70	2700 mg/L	
Metalaxil	1.75	8400 mg/L	

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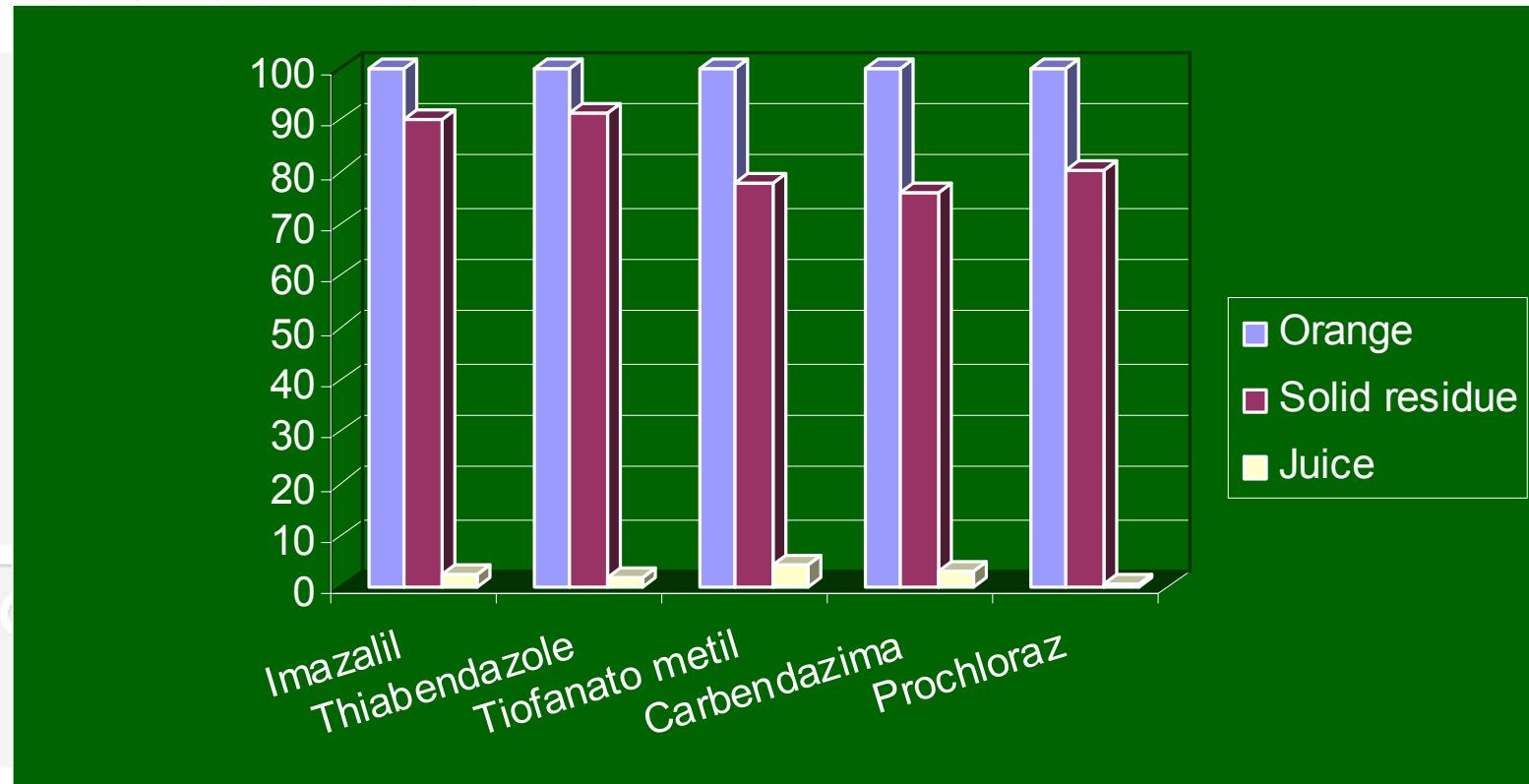
Pesticides in Fruits And Vegetables
Community Reference Laboratory

DG SANCO
EUROPEAN COMMISSION

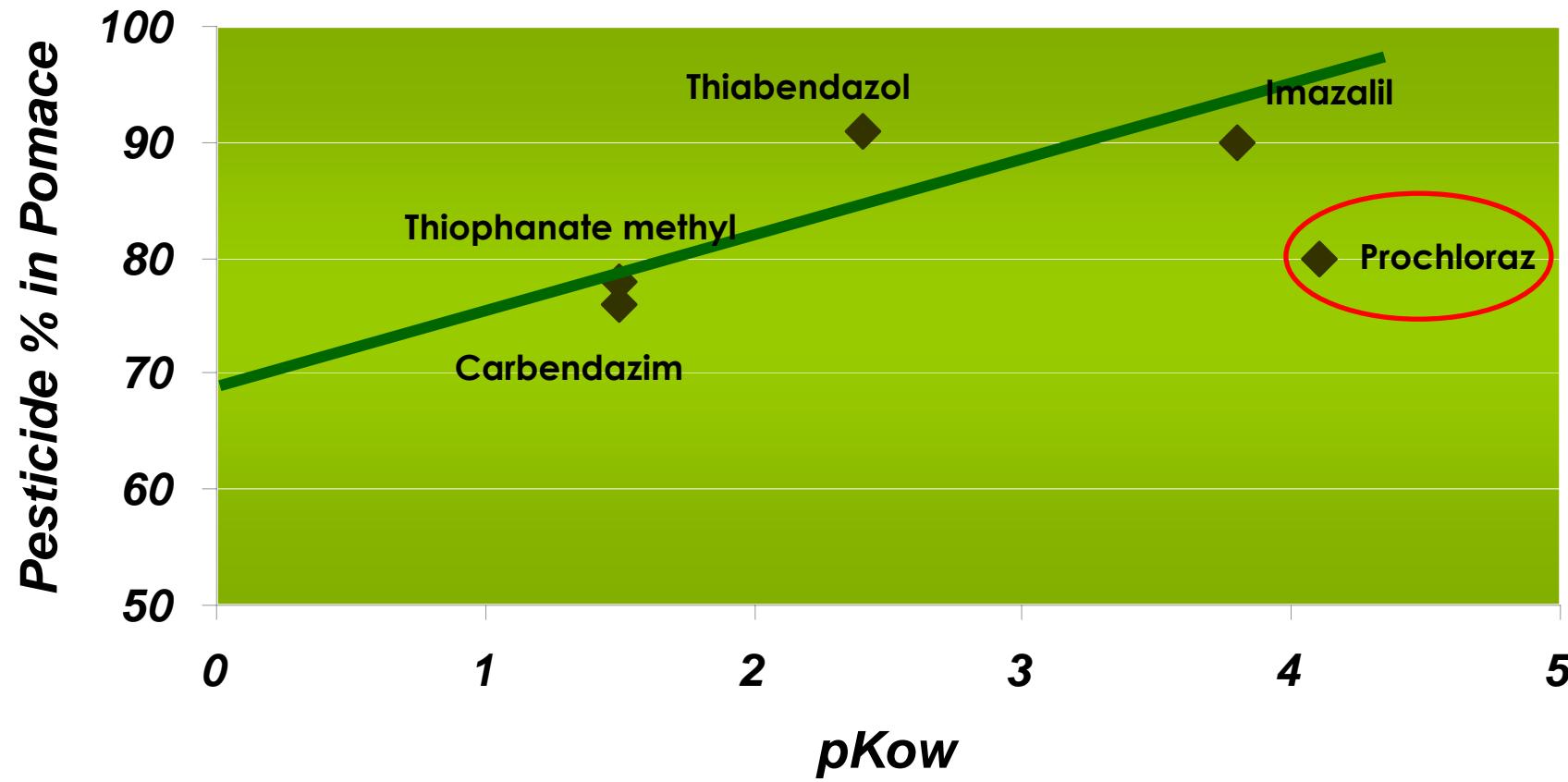
Results on Oranges

Juice

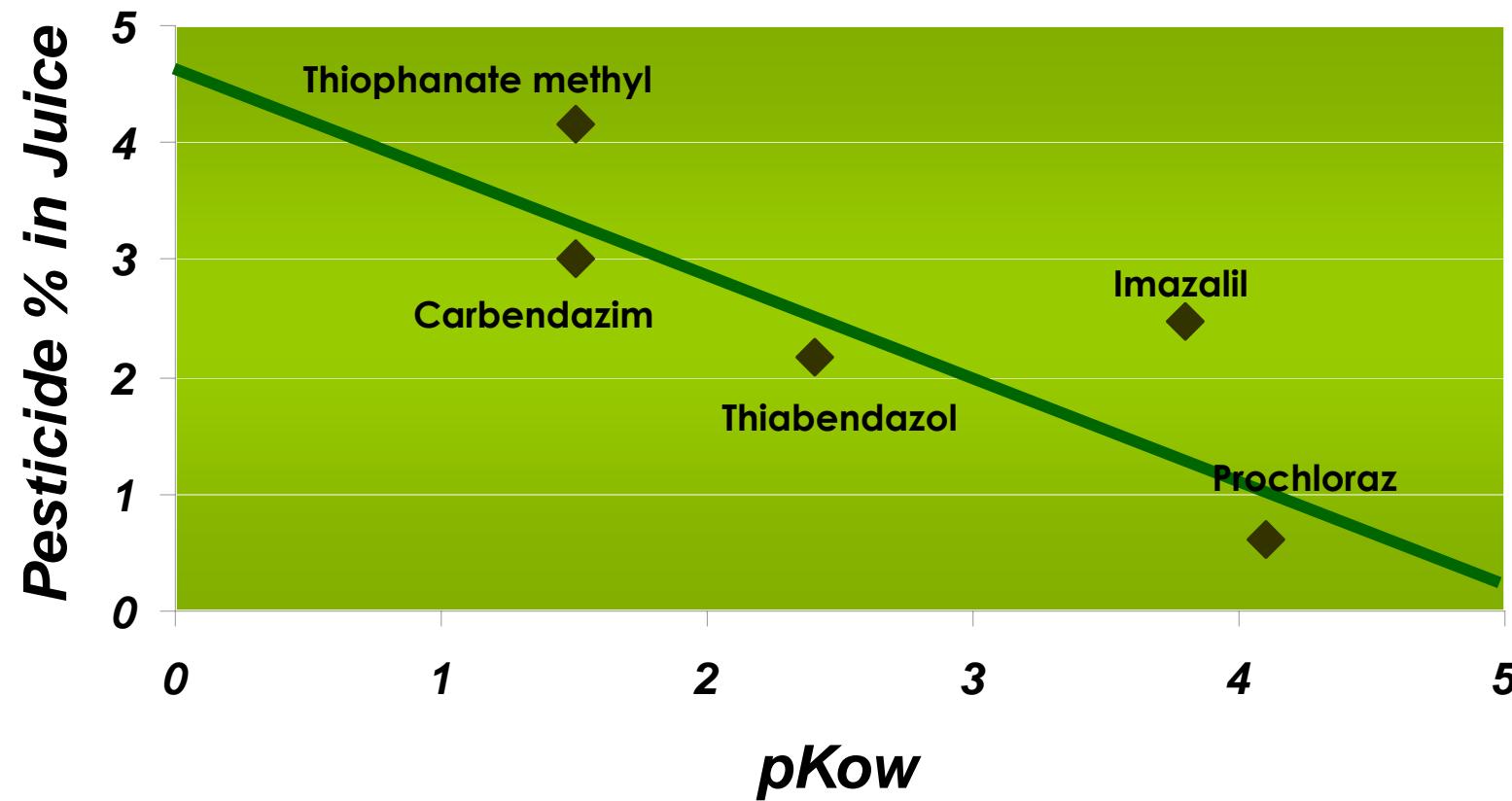
- Pesticide distribution



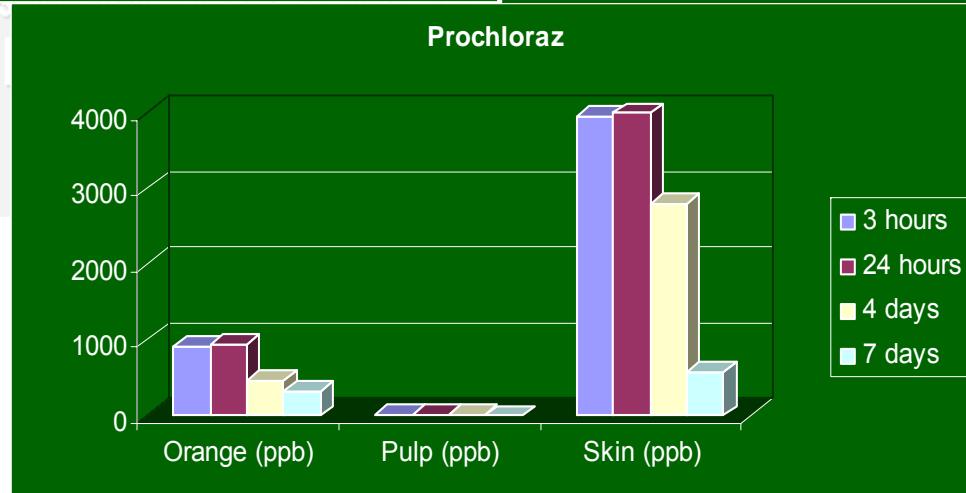
Pesticides in Orange Juicing



Pesticides in Orange Juicing

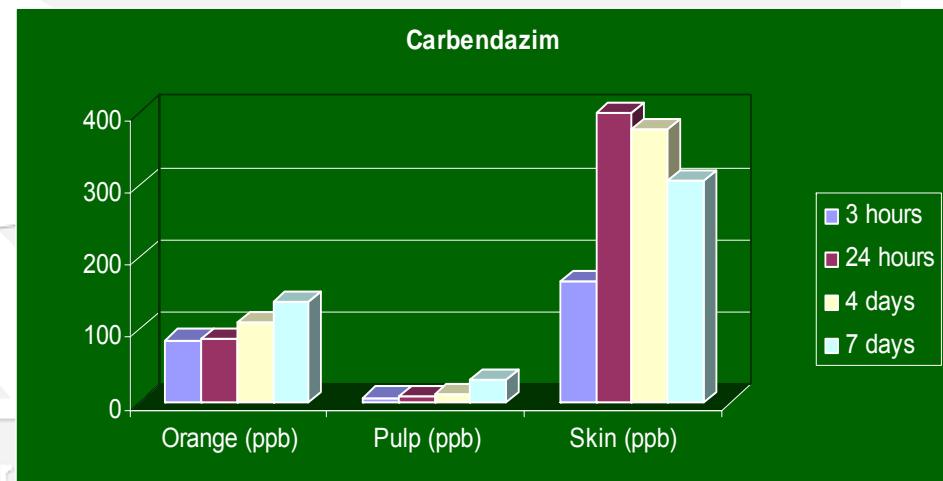
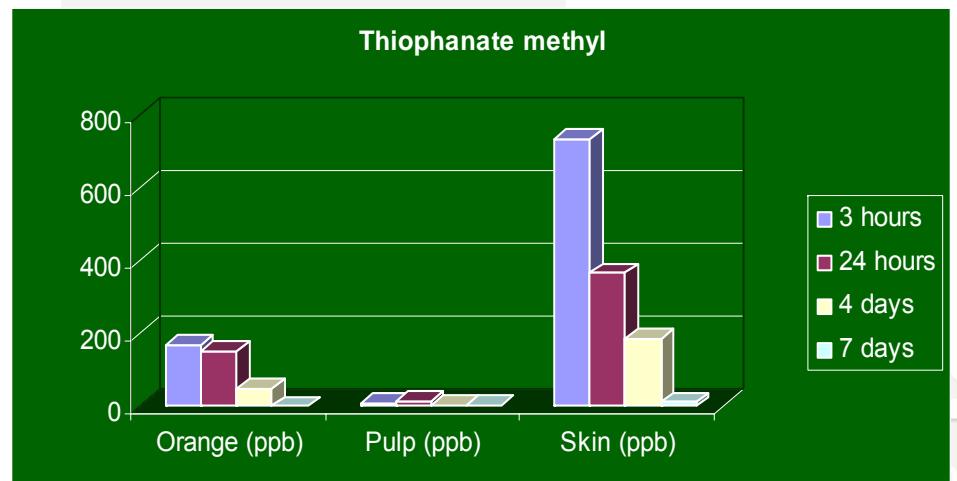


Orange - Concentration ($\mu\text{g}/\text{kg}$)



Results

Orange - Concentration ($\mu\text{g}/\text{kg}$)



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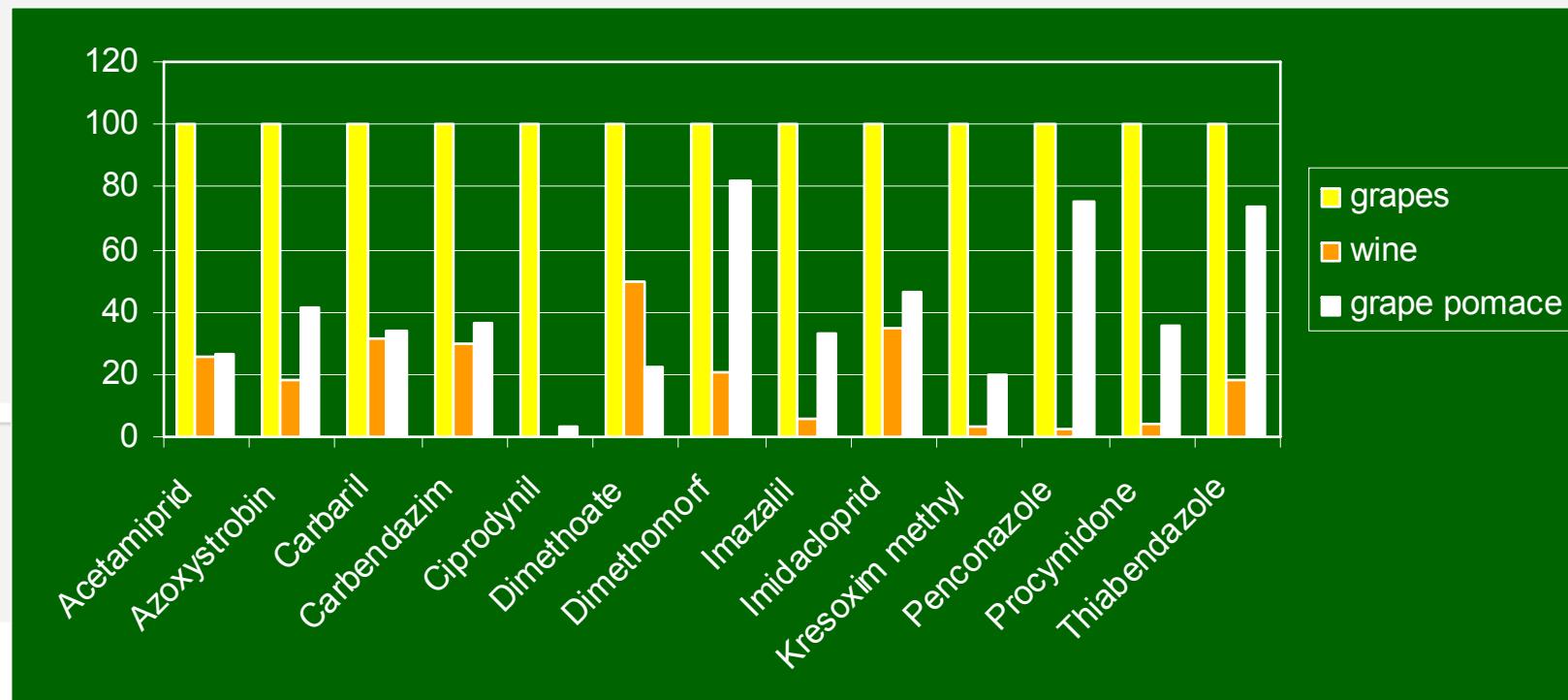
Results

- Real samples analysys

Pesticide	Orange (µg/kg)	Peel (µg/kg)	Pomace (µg/kg)
Carbendazim	9	18	-
Imazalil	955	3510	37

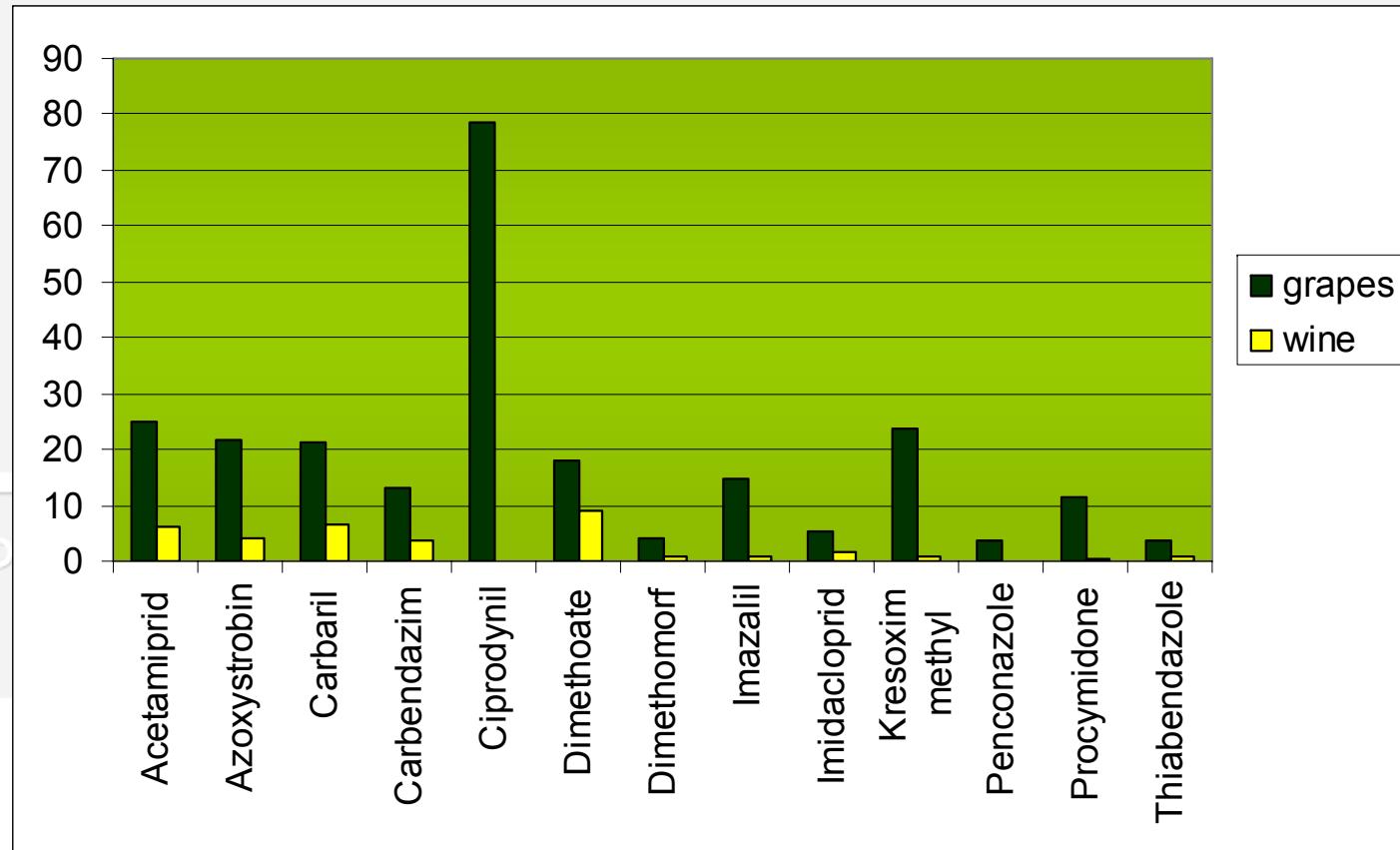
Results on Wine

- Distribution of pesticides (% mg)**



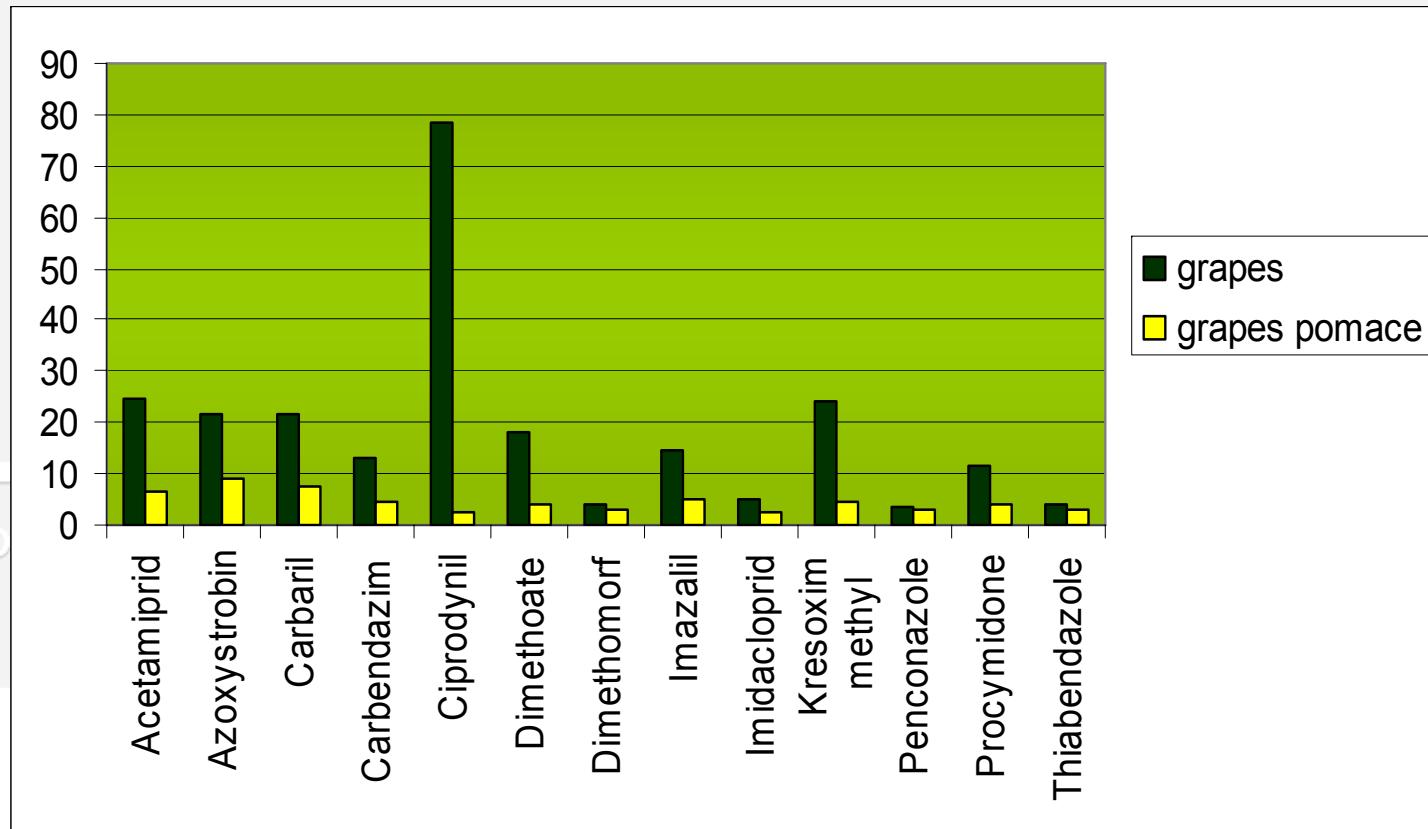
Results

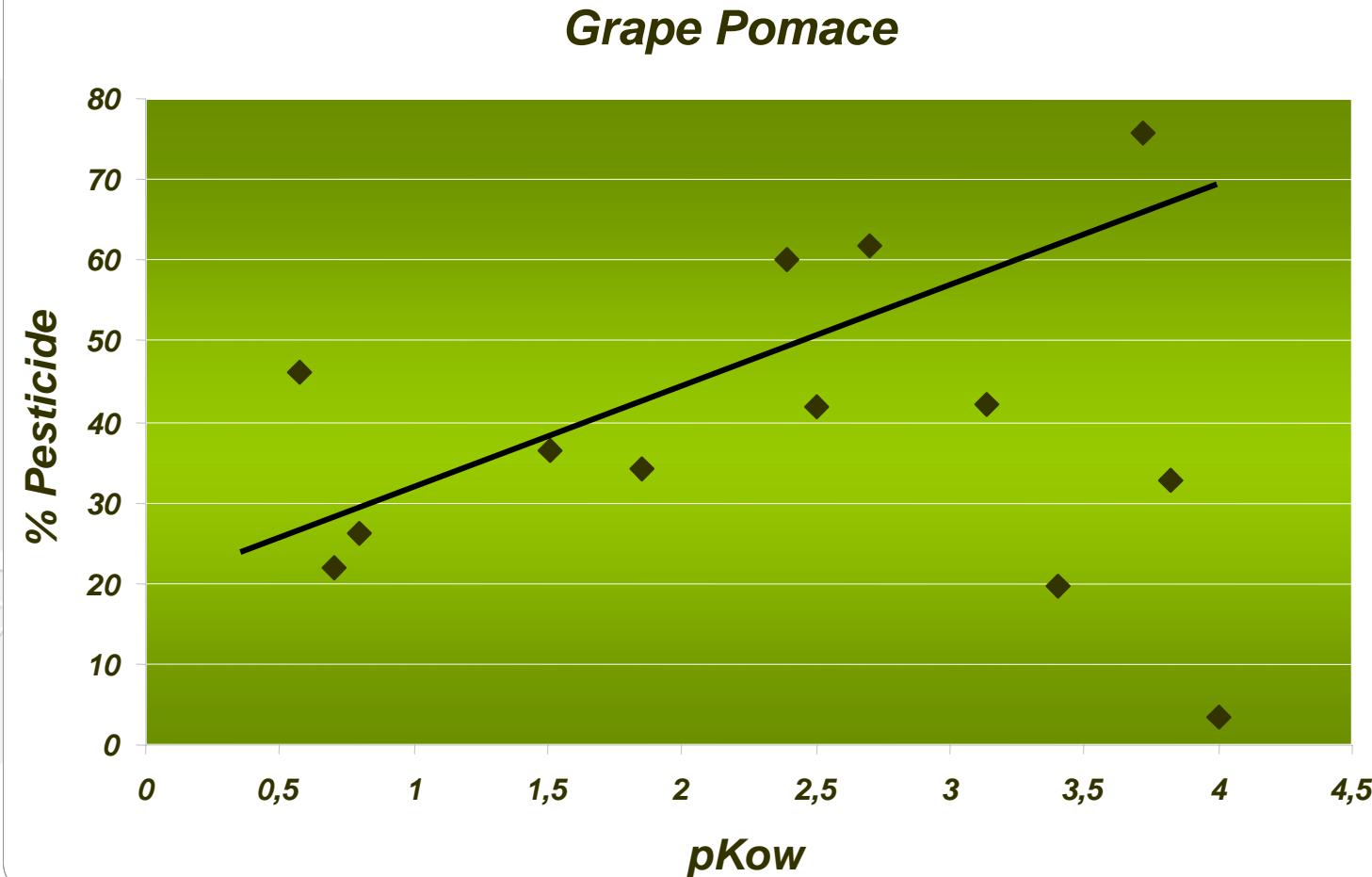
- **Distribution of pesticides (mg)**



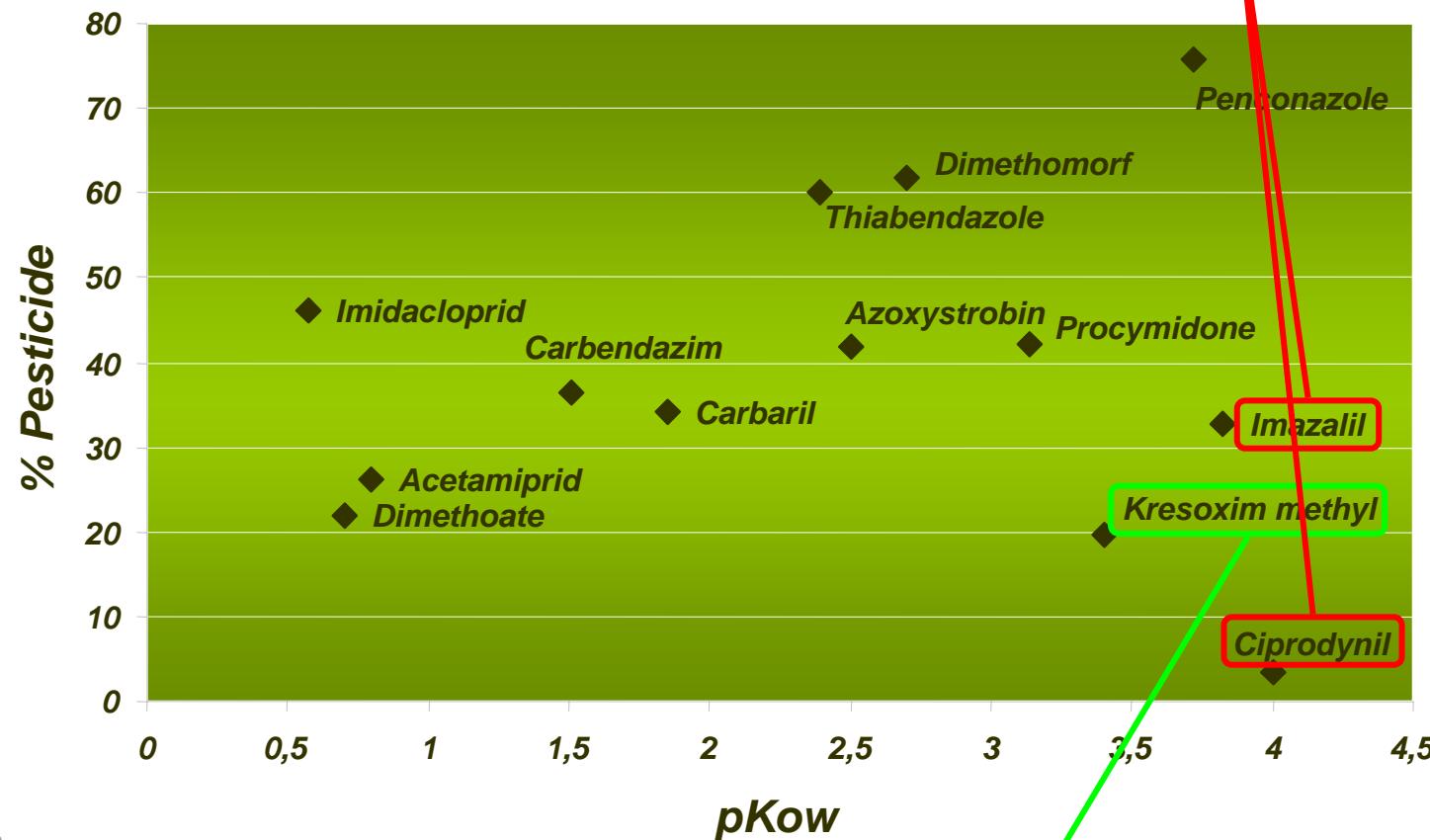
Results

- Distribution of pesticides (mg)**



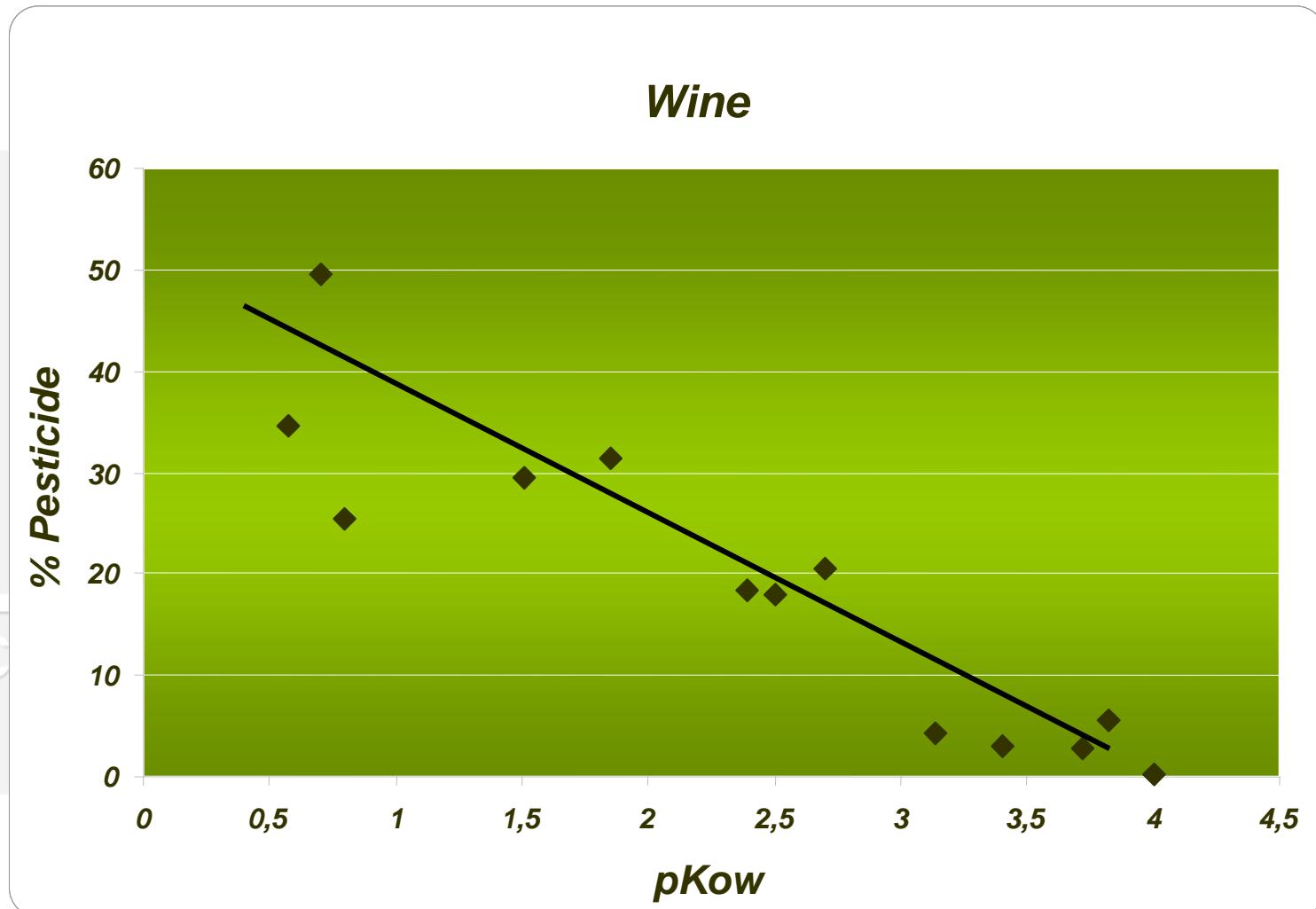


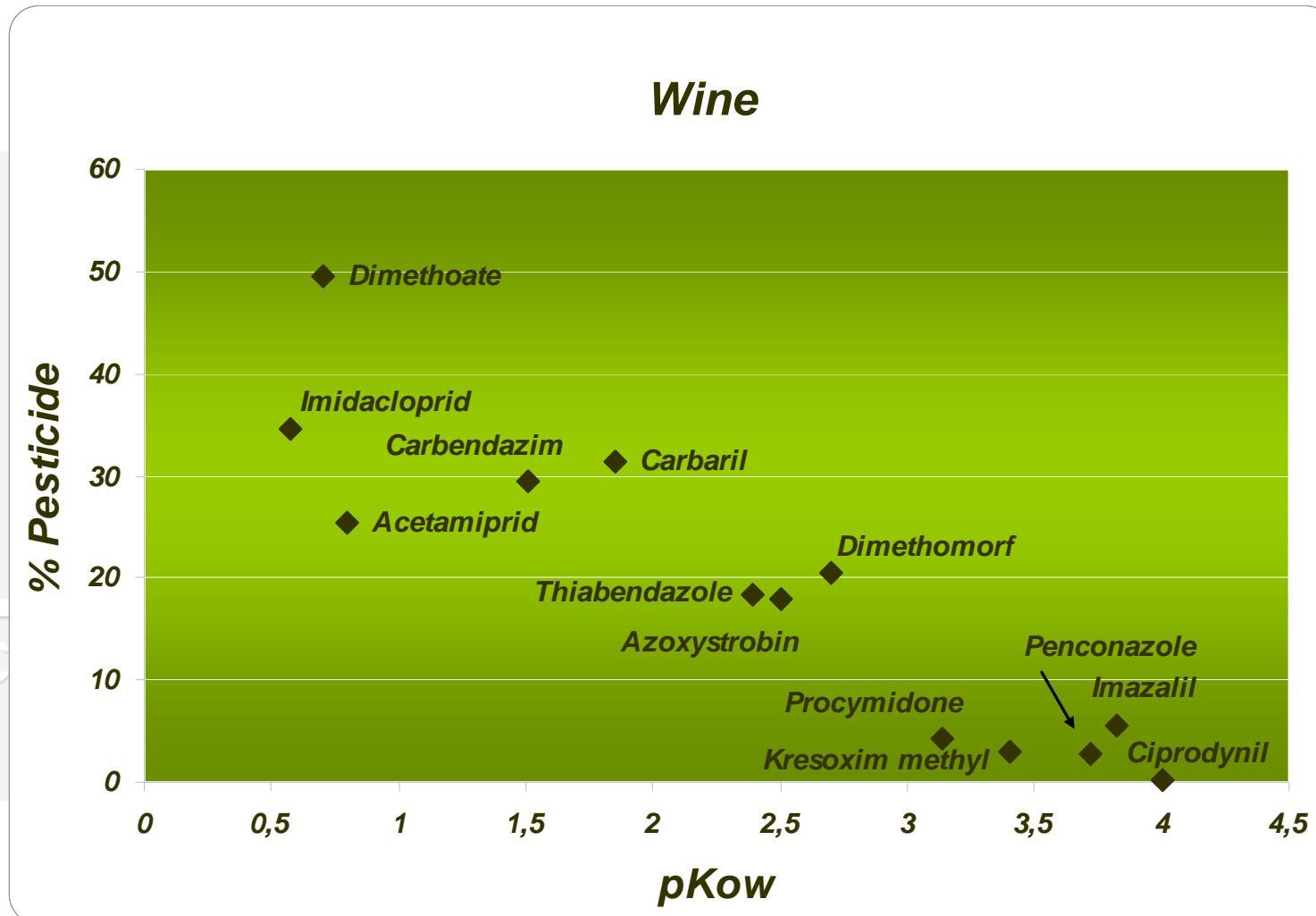
Losses in the Maceration Process

Grape Pomace

Losses in the Crushed Process

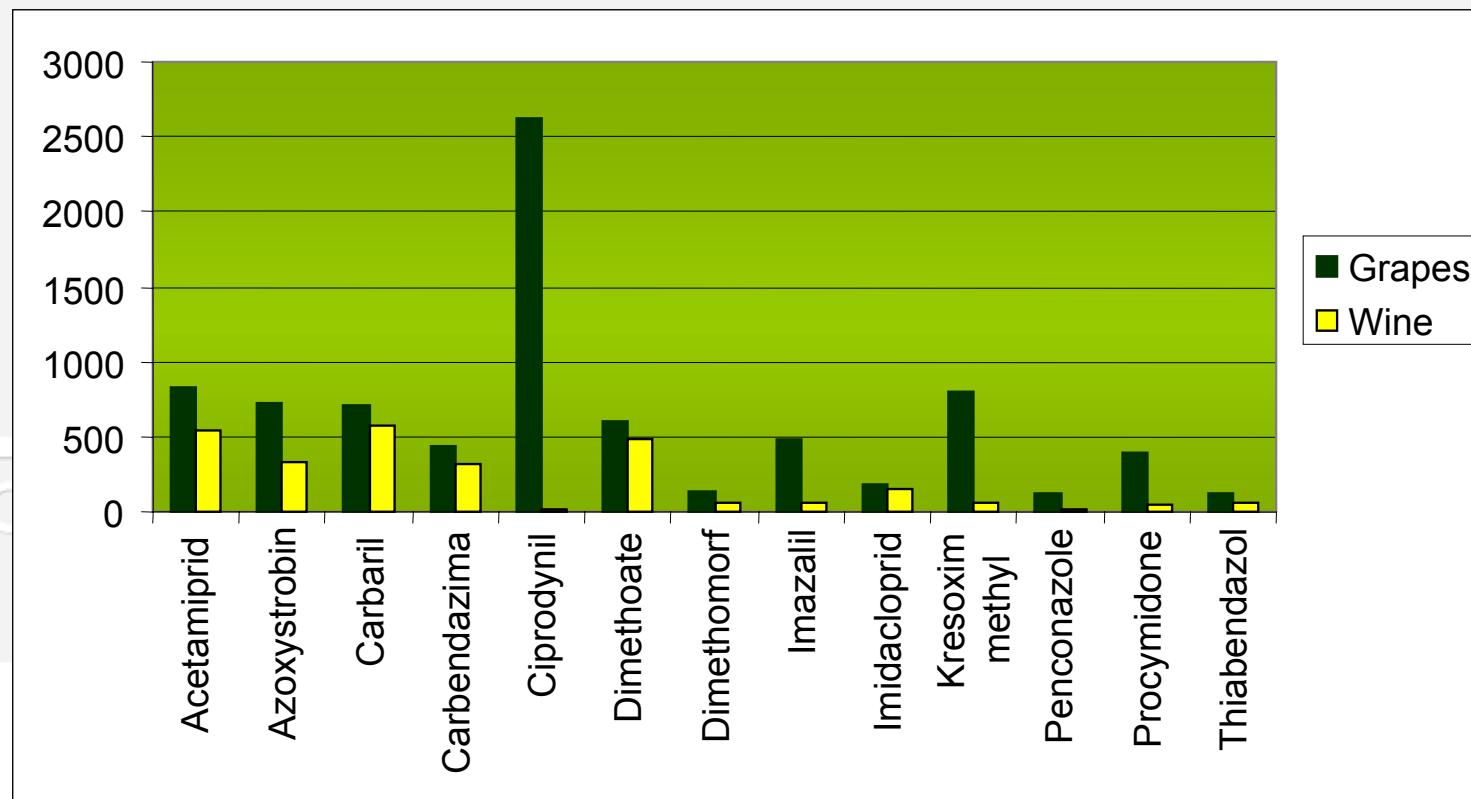
 Amadeo R. Fernández-Alba
 CRL-FV





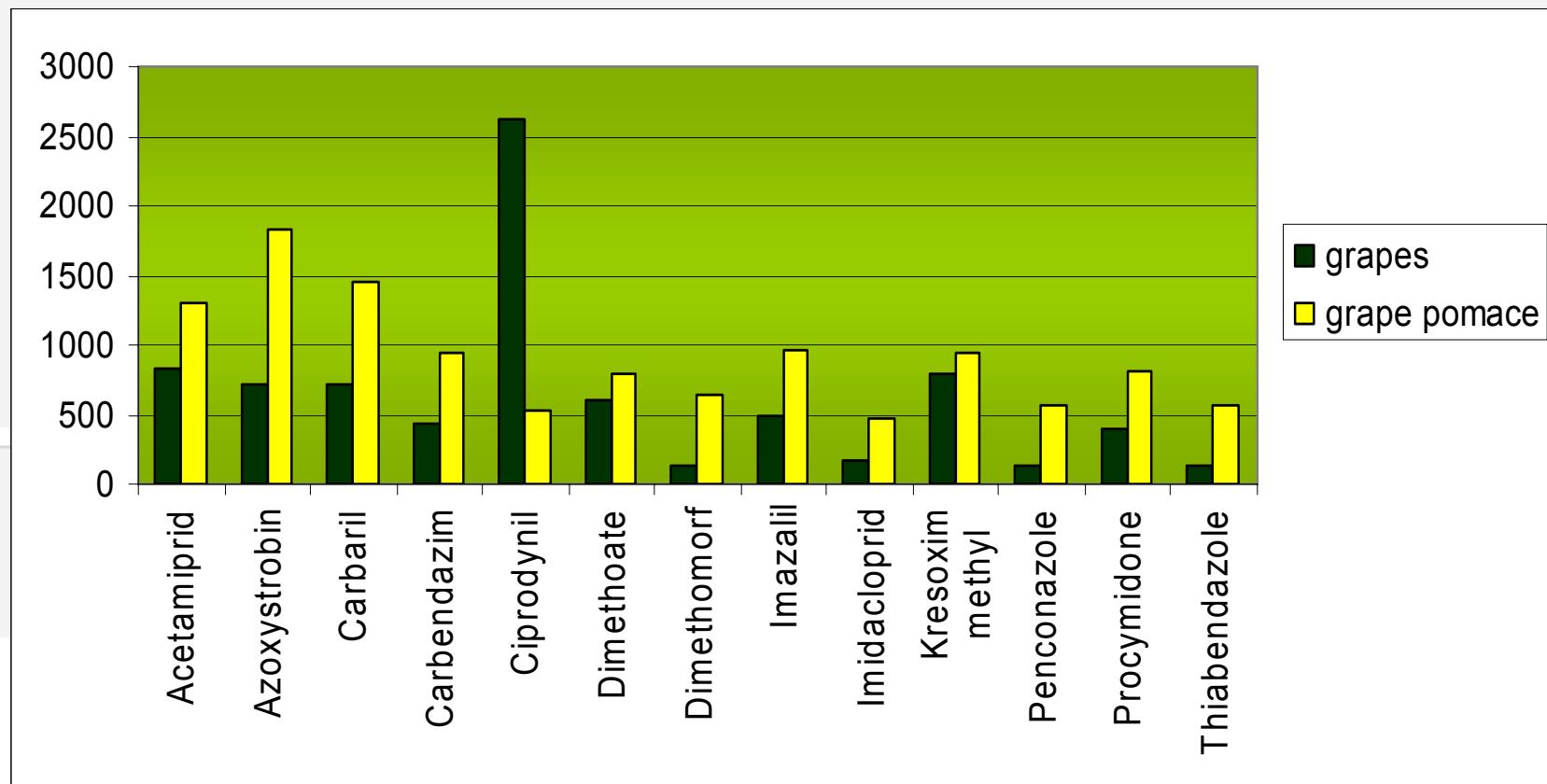
Results

- Distribution of pesticides ($\mu\text{g/Kg}$)**



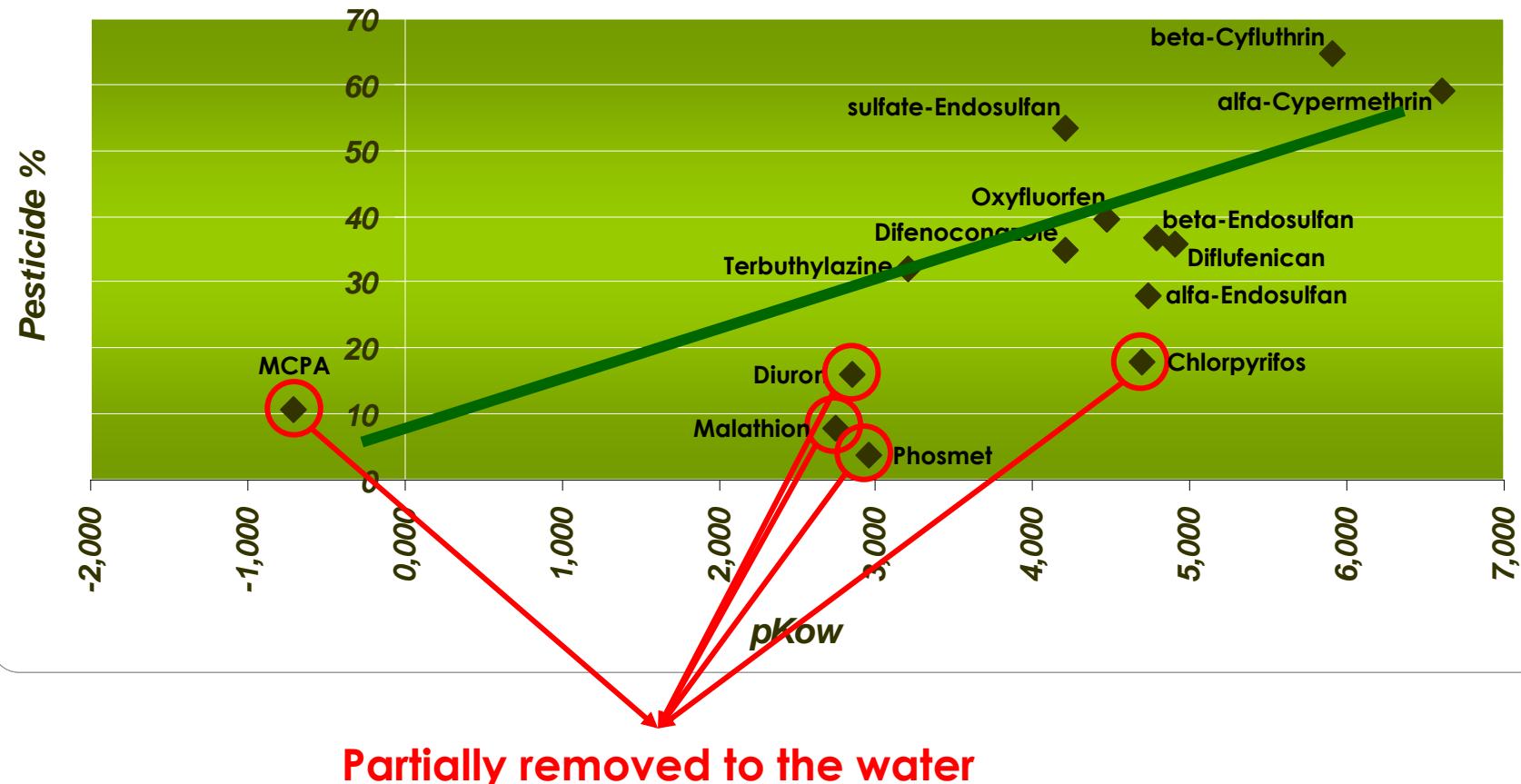
Results

- Distribution of pesticides ($\mu\text{g/Kg}$)**

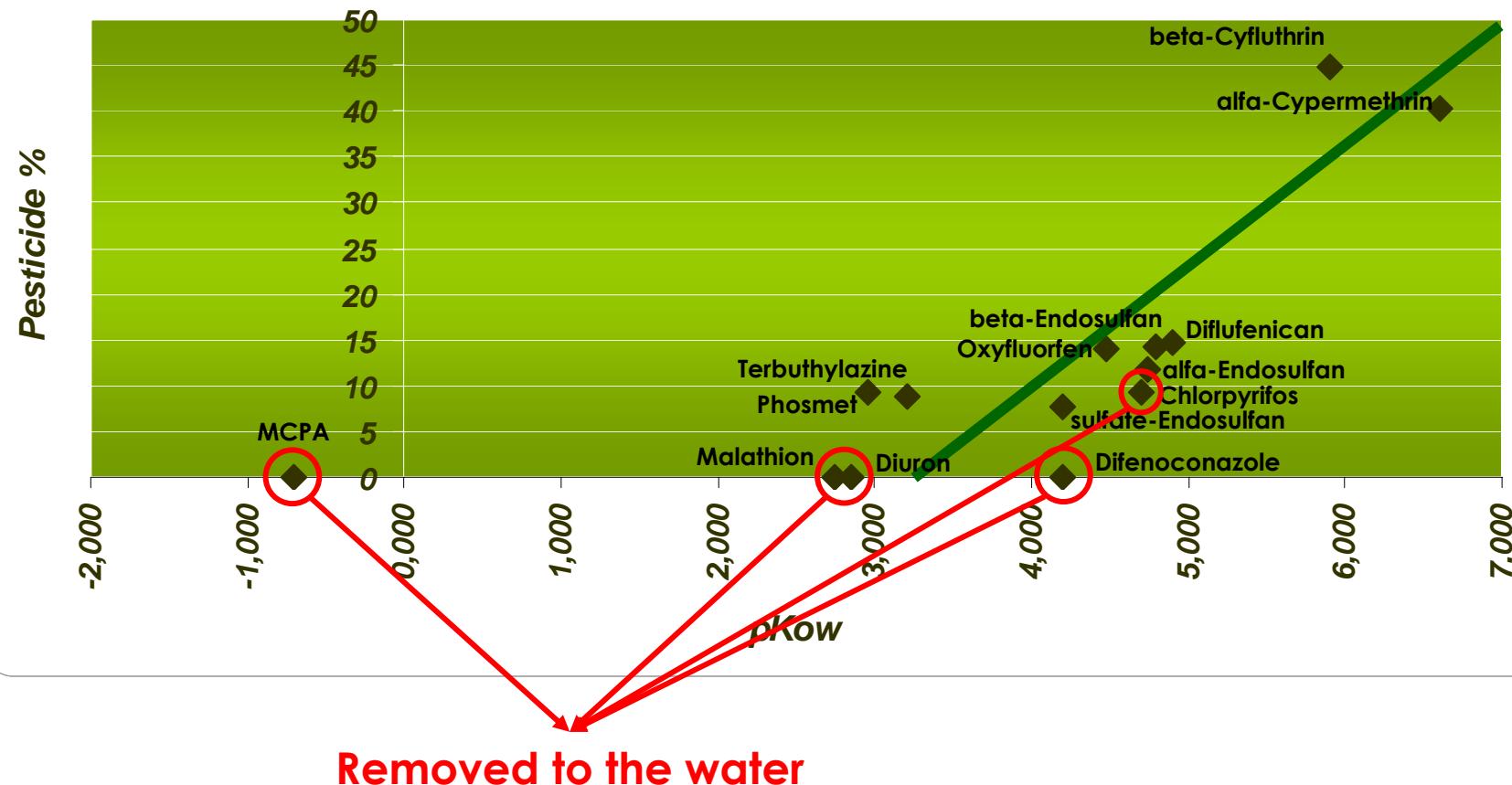


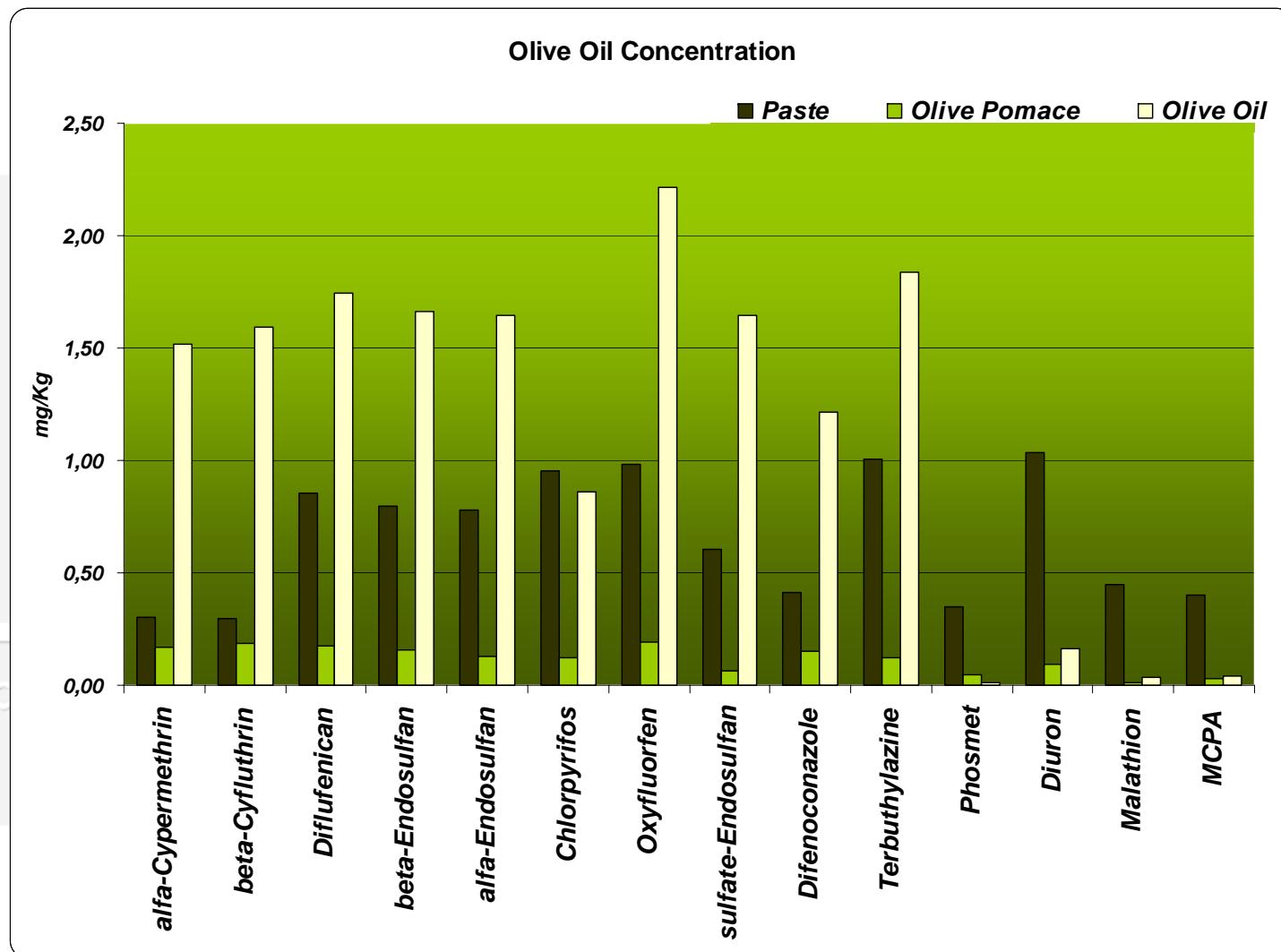
Results on Olive Oil

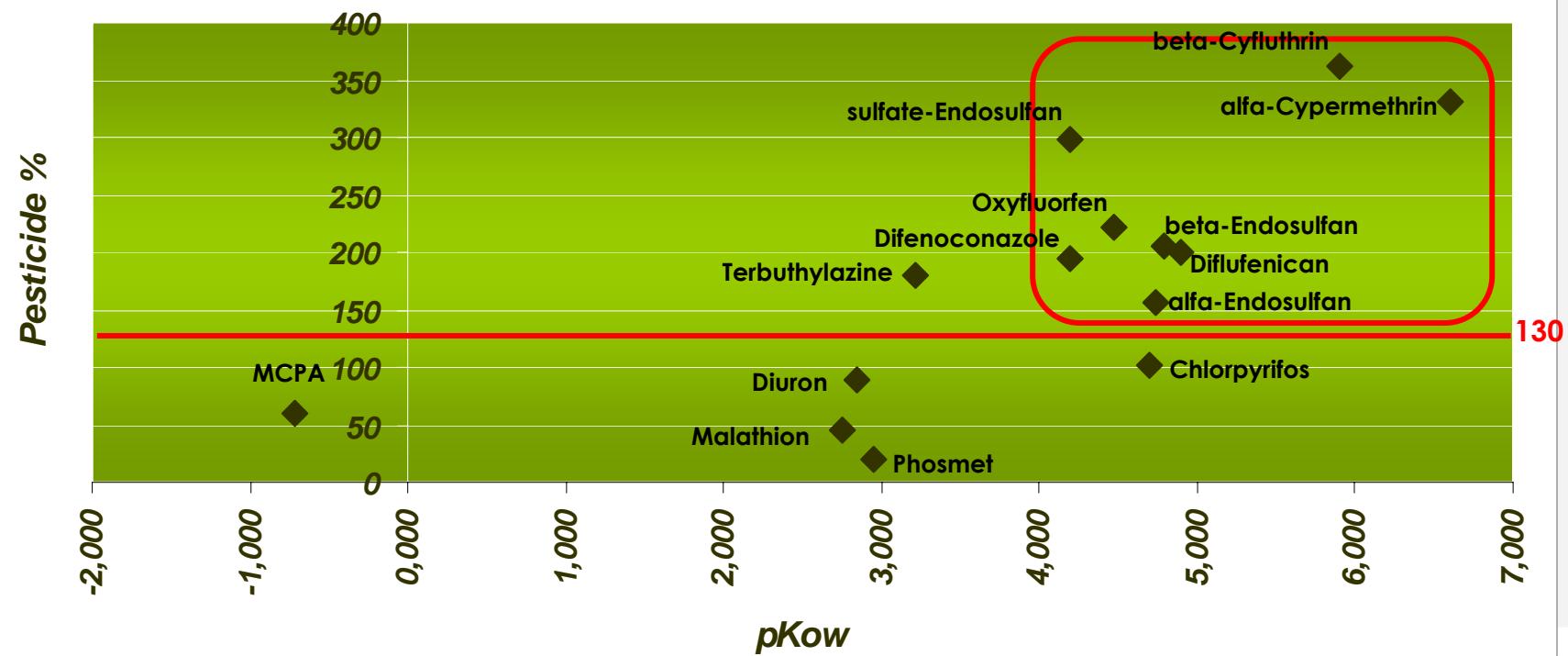
Pesticide % in Olive Oil

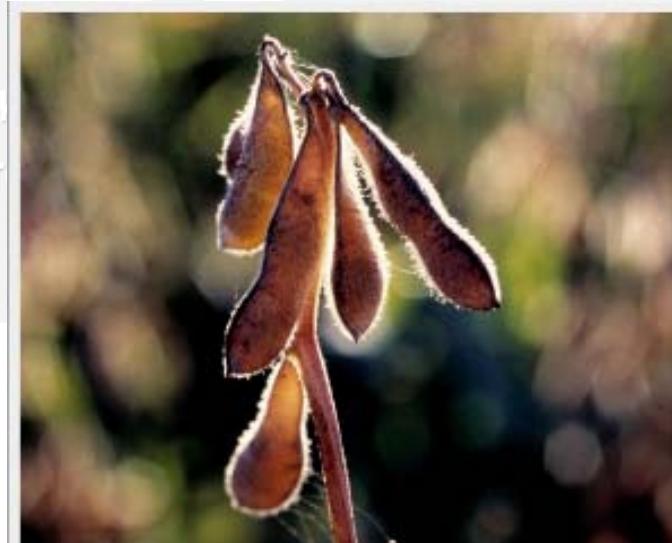


Pesticide	Solubility
alfa-Cypermethrin	0.004
beta-Cyfluthrin	0.0012
Diflufenican	0.05
beta-Endosulfan	0.33
alfa-Endosulfan	0.32
Chlorpyrifos	1.4
Oxyfluorfen	0.116
sulfo-Endosulfan	>0.33
Difenoconazole	15
Terbutylazine	8.5
Phosmet	25
Diuron	36.4
Malation	145
MCPA	273.9

Pesticide % in Olive Pomace



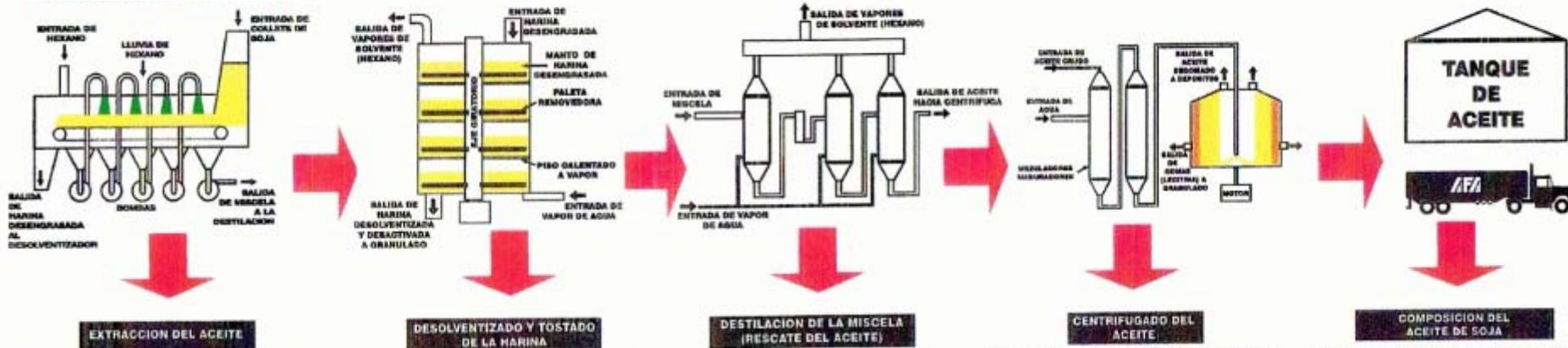
Pesticide % * 5,5

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Pesticides in Soya

Insecticides		Fungicides	
Alfametrina	Meticarb	Captan	Flusilazol
Cartap	Metomil	Carbendazim	Kresoxim metil
Cipermetrina	Metxiofenocide	Metalaxil	Myclobutanil
Cialotrina	Novaluron	Carboxin	Pyraclostrobin
Clorpirifos	Pirimifos metil	Difeconazol	Tebuconazol
Clorpirifosmetil	Propenofos	Flutriafol	
Deltametrina	Spinosad	Fludioxinil	
Diazinon	Teflubenzuron	Tiodicarb	
Diflubenzuron	Thiametoxam	Iprodione	
Endosulfan	Tiodicarb	Azoxystrobin	
Fipronil	Imidacloprid	Ciproconazol	
Flufenuxuron	Tefubonizide	Epoxiconazol	
Lufenuron	Triflumuron	Tetraconazol	
Malathion	Zetametrina	Propiconazol	

PROCESO DE EXTRACCION DE ACEITE DE SOJA, DEGOMADO DEL ACEITE



EL MATERIAL PREPARADO INGRESA AL EXTRACTOR CON LAS SIGUIENTES CARACTERISTICAS: HUMEDAD 10%, MATERIA GRASA 20%, TEMPERATURA INFERIOR A LOS 60 °C. EN LA EXTRACCION SE REALIZA UN LAVADO DEL MATERIAL PREPARADO COMO YA VIMOS, CON UN SOLVENTE QUE DILUYE EL ACEITE ARRASTRANDOLE Y DEJANDO LA HARINA DESENGRASADA. LOS COLLETS PROVENIENTES DE LA EXTRACCION SON DISPUESTOS EN UN MANTO DE 2 METROS DE ESPESOR A UNA LLUVIA DE SOLVENTE QUE SE RECIRCULA MEDIANTE BOMBAS. EL EXTRACTOR SALENT ENTONCES MATERIALES EN DOS FORMAS: 1/ SOLIDA, QUE ES LA HARINA DESENGRASADA PERO HUMEDA Y MOJADA EN SOLVENTE; 2/ LIQUIDA, QUE ES UNA MEZCLA DE ACEITE Y SOLVENTE QUE ES LLAMADA MISCELA.

*LA HARINA TIENE MEZCLADO UN 25% DE SOLVENTE Y UN RESTO DE ACEITE MENOR AL 1%. LA MISCELA TIENE UNA PROPORCIÓN DE 25% DE ACEITE Y UN 75% DE SOLVENTE.

EN ESTE PROCESO SE EFECTUAN BÁSICAMENTE DOS OPERACIONES: 1/ LA PRIMERA ES DESOLVENTIZAR O SECAR EL SOLVENTE DE LA HARINA; 2/ LA SEGUNDA ES EL TOSTADO QUE ES UNA COCCIÓN HUMEDA QUE ELIMINA LA ENZIMA URIBASA PERMITIENDO ASÍ QUE ESTA HARINA SEA DIGESTIBLE POR LOS ANIMALES A QUIEN SERÁ DADA EN ALIMENTO.

ESTAS OPERACIONES SE REALIZAN EN UN EQUIPO LLAMADO "DT" QUE ES EL PRINCIPAL CONSUMIDOR DE ENERGÍA DE LA PLANTA DE EXTRACCION. LA HARINA SALE CON UNA TEMPERATURA DE 100 °C Y UNA HUMEDAD DE 17-18% DEBIDO A LA UTILIZACIÓN DEL VAPOR. EL SOLVENTE AQUÍ ELIMINADO EN FORMA DE VAPOR SE RECICLA Y UTILIZA NUEVAMENTE EN LA PLANTA PARA OTRO CICLO DE EXTRACCION.

COMO YA HEMOS EXPLICADO ANTES EL ACEITE SE SEPARA DE LA SEMILLA PREPARADA USANDO UN SOLVENTE QUE QUEDA MEZCLADO CON ESTE FORMANDO UNA MEZCLA LLAMADA MISCELA. MEDIANTE EL PROCESO DE DESTILACION SE SEPARA EL ACEITE DEL SOLVENTE. EN ESTE PROCESO SE CALIENTA LA MISCELA A TEMPERATURAS SUPERIORES AL PUNTO DE EVAPORACION DEL SOLVENTE EN PRESENZA DE VACÍO, ENTONCES EL SOLVENTE SE EVAPORA DEJANDO AL ACEITE LIBRE DE ESTE. EL SOLVENTE EN ESTADO DE VAPOR SE ENVÍA LUEGO VOLVIENDO AL ESTADO LIQUIDO, FORMANDOSE ENTONCES UN CIRCUITO CERRADO EN EL QUE EL SOLVENTE ES USADO NUEVAMENTE PARA OTRO CICLO DE EXTRACCION.

EL ACEITE OBTENIDO DEL PROCESO DE EXTRACCION- DESTILACION INCORPORA EN SU MASA ALGUNOS ELEMENTOS QUE SON INDESEABLES Y QUE DIFICULTAN SU ALMACENAJE Y POSTERIOR UTILIZACION. ESTOS ELEMENTOS SON ALGUNOS DERIVADOS DEL FOSFORO LLAMADOS FOSFATIDOS QUE TIENEN LA PARTICULARIDAD DE COMBINARSE CON AGUA FORMANDO COMPUESTOS DE GRUESA VISCOSIDAD. ESTA PROPIEDAD SE USA PARA EFECTUAR SU SEPARACION: COMO PRIMERA ETAPA SE AGREGA AL ACEITE UN 5% DE AGUA, LUEGO ESTA MEZCLA SE HACE PASAR POR UNA CLARIFICADORA CENTRIFUGA QUE SEPARA LOS COMPUESTOS DE ALTA DENSIDAD DEL ACEITE DEJANDO A ESTE PERFECTAMENTE CLARO. LOS COMPUESTOS AQUÍ SEPARADOS PUEDEN SER UTILIZADOS PARA LA PRODUCCION DE LECITINA Y OTROS COMPUESTOS, O AGREGADOS YA SECOS A LA HARINA GRANULADA.

ACEITE VEGETAL: PRODUCTO CONSTITUIDO POR ESTERES GLICERIDOS DE ACIDOS GRASOS Y SUS FOSFATIDOS ASOCIADOS, ESTEROLAS, ALCOHOLAS, HIDROCARBUROS Y PIGMENTOS.

COMPOSICIÓN DEL ACEITE	COMPOSICIÓN DE A. GRASOS
TRIGLICERIDOS 95-97%	UNDECENO 5%
FOSFATIDOS 1,5-2,5%	OLEICO 19%
HIERNA 1-3%	PALMITICO 11%
M. INSAPONIFICABLE 1,6%	LINOLENICO 10%
	ESTEARICO 4%

UTILIZACION DEL ACEITE DE SOJA CRUDO: SE LO UTILIZA PARA LA ELABORACION DE: ESTEROLAS, ACIDOS GRASOS GLICEROL, DEBIDO A QUE ES COMESTIBLE LUEGO DE SER REFINADO SE LO UTILIZA PARA: ACEITES PARA COCINA, MAYONESAS, MARGARINAS, ACEITES PARA ENSALADAS, ELEMENTOS PARA REPETIRIA, ETC.

CODEX alimentarius

- The factors affecting the concentration or dilution of pesticide residues in the PPF are various depending on; (i) pesticide (ii) process applied, (ii) commodity and (iv) degradation processes involved.
- In the apple and orange juicing processes studied a dilution was the general effect. It means PF lower than 1. In general PF of 20-30% are common but lower values were also detected (in especial in the case of orange juice).
- For wine production, with yields of around 70%, the PF are in general close to 0.5. Higher values up to 1 are obtained with pesticides with low Kow and high water solubility. Only in a few cases, fermentation processes have a significant effect in pesticide degradation.
- The low yield obtained for olive oil production lead to an important concentration of the pesticide residues with PF from 2 to almost 6.
- An acceptable linear trend of the Log Kow versus pesticide distribution is noticed. It can be used as a rough estimation of the residue distribution. That behavior could be used for risk assessment purposes.



20-25/04/2009

- Further evaluations should be considered for setting MRLs on processed commodities in specific cases.
- Furthermore studies on the most frequent transformation products are needed in setting those MRLs.

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