Validation of a method for direct determination of glyphosate and AMPA in sugar beet root using hydrophilic interaction liquid chromatography and tandem mass spectrometry

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Annex 1 : Reported acid dissociation constants (pKa) of PMG in literature.

Ionic strength [M]	Temperature [°C]	pK_{a0}	pK_{a1}	pK_{a2}	pK_{a3}	Source
n.m.	n.m.	< 2	2.6	5.6	10.6	Sprankle *et al.*, 1975
0.1	20	< 2	2.25	5.5	10.34	Wollerton and Husband (1997) cited by FAO, 2005
0	n.m.	0.78	2.29	5.96	10.98	Chamberlain (1996)
1	n.m.	n.m.	1.77	5.08	9.76	Barja and Dos Santos Afonso (1998)
0.5	n.m.	n.m.	1.96	5.28	9.98	Barja and Dos Santos Afonso (1998)
0.1	n.m.	n.m.	2.09	5.52	10.28	Barja and Dos Santos Afonso (1998)
0.1	n.m.	n.m.	2.23	5.46	10.14	Subramaniam and Hoggard, cited by Barja and Dos Santos Afonso (1998)

n.m. : not mentioned

Annex 2 : Reported solubility of PMG in various solvents in literature.

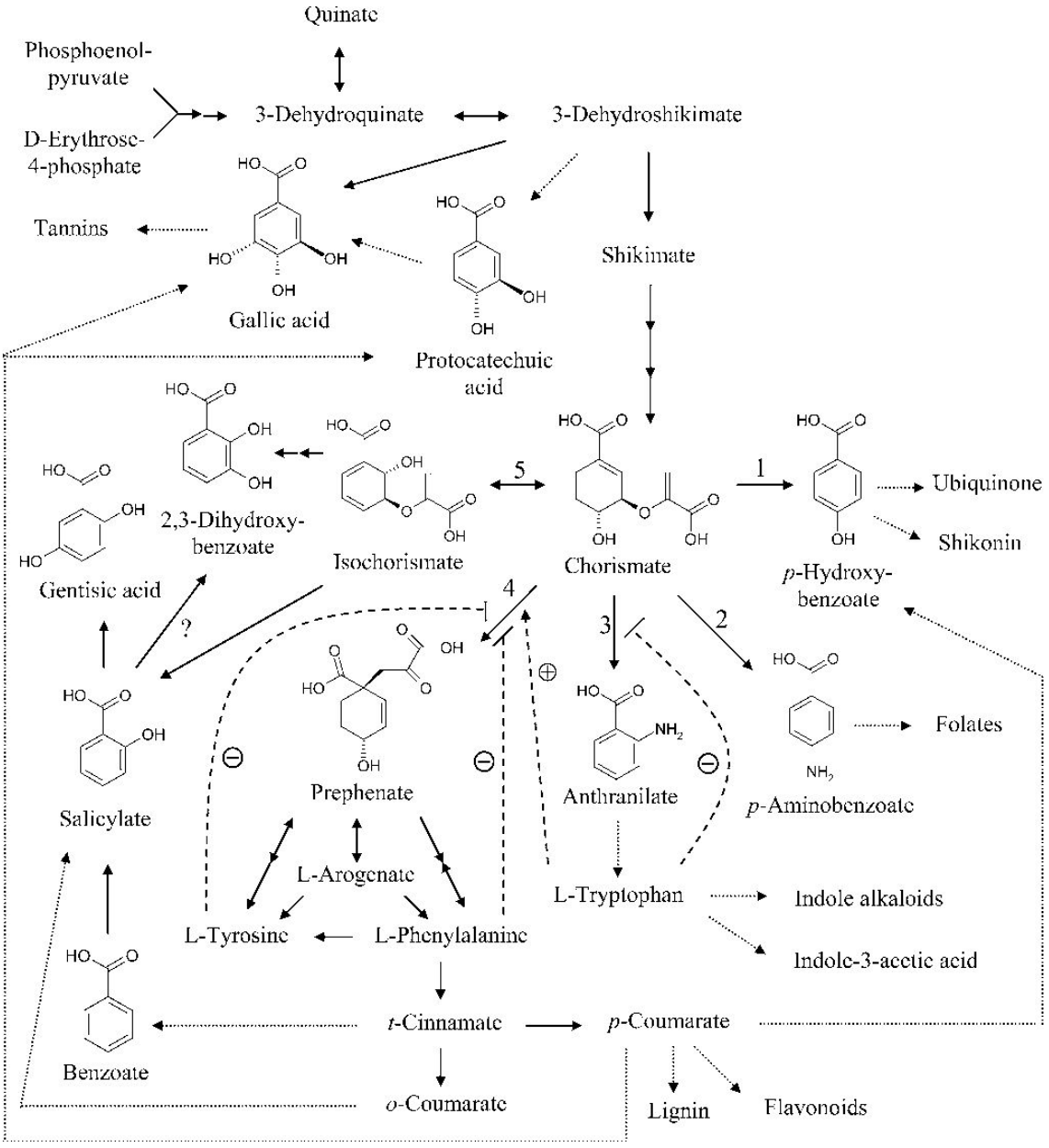
Solvents	Solubility (mg_{PMG}/L)	Sources
Water (pH 1.9)	10,500	Tomlin (2006)[1] ; MacBean (2009)[2]
Water (pH 7)	157,000	
Dichloromethane and methanol	231	Robson (1991) cited by FAO (2005)
Acetone	78	Robson (1991) cited by FAO (2005)
Toluene	36	Robson (1991) cited by FAO (2005)
Hexane	26	Robson (1991) cited by FAO (2005)
Propan-2-ol	20	Robson (1991) cited by FAO (2005)
Ethyl acetate	12	Robson (1991) cited by FAO (2005)
Methanol	10	Wollerton and Husband (1997) cited by FAO, 2005
Acetonitrile	0.8	Wollerton and Husband (1997) cited by FAO, 2005
Methanol, heptane, octan-1-ol, xylenes, ethyl acetate, acetone, 1,2-dichloroethane	< 0.6	Wollerton and Husband (1997) cited by FAO, 2005

Annex 3 : Conversion table for common PMG salts included in formulations.

Compound	Molecular formula	Molar mass (g/mol)	acid equivalent ($g_{a.e.}/g_{salt}$)
PMG	$C_3H_8NO_5P$ *	169.07	1
PMG-isopropylamine (1:1)	$(C_3H_7NO_5P)^-$ $(C_3NH_{10}^+)$	228.18	0.74
PMG-dimethylamine (1:1)	$(C_3H_7NO_5P)^-$ $(C_2NH_8^+)$	214.16	0.79
Trimethylsulfonium PMG salt (1:1)	$(C_3H_7NO_5P)^-$ $(S(CH_3)_3^+)$	245.24	0.69
Potassium PMG salt (1:1)	$(C_3H_7NO_5P)^-$ (K^+)	207.16	0.81
Sodium PMG salt (1:1)	$(C_3H_7NO_5P)^-$ (Na^+)	191.06	0.88
Ammonium PMG salt (1:1)	$(C_3H_7NO_5P)^-$ (NH_4^+)	186.10	0.90
Diammonium PMG salt (1:2)	$(C_3H_6NO_5P)^-$ $(NH_4^+)_2$	203.13	0.83

* This formula relates to the non-ionized molecule $(COOH)CH_2NHCH_2(H_2PO_3)$

[1] cited by Puchem https://pubchem.ncbi.nlm.nih.gov/compound/glyphosate
[2] cited by Puchem https://pubchem.ncbi.nlm.nih.gov/compound/glyphosate



Annex 4 : Biosynthesis pathways from chorismate.
Source : Mustafa & Verpoorte (2005)

Annex 5 : dates of approval for food use, feed use and cultivation of the GM sugar beets worldwide.
Source : International Service for the Acquisition of Agro-Biotech Applications (ISAAA)[a,b,c]

Event name : H7-1 (KM-ØØØH71-4, Roundup Ready™ sugar beet).

Countries	Date of approval for food use	Date of approval for feed use	Date of approval for cultivation
Australia	2005		
Canada	2005	2005	2005
China	2009	2009	
Colombia	2005	2010	
European Union	2007	2007	
Japan	2003	2005	2007
Mexico	2006		
New Zealand	2005		
Philippines	2005	2005	
Russia	2006		
Singapore	2014	2014	
South Korea	2006		
Taiwan	2015		
United States	2004	2004	2005

Event name : GTSB77 (SY-GTSB77-8, InVigor™ sugar beet)

Countries	Date of approval for food	Date of approval for feed	Date of approval for cultivation
Australia	2002		
Japan	2003	2003	
New Zealand	2002		
United States	1998	1998	1998

Event name : T120-7 (ACS-BVØØ1-3, Liberty Link™ sugar beet)

Countries	Date of approval for food	Date of approval for feed	Date of approval for cultivation
Canada	2000	2001	2001
Japan	2001	2003	
United States	1998	1998	1998

a : https://www.isaaa.org/gmapprovaldatabase/event/default.asp?EventID=224&Event=H7-1
b : https://www.isaaa.org/gmapprovaldatabase/event/default.asp?EventID=226&Event=GTSB77%20(T9100152)
c : https://www.isaaa.org/gmapprovaldatabase/event/default.asp?EventID=225&Event=T120-7

Annex 6 : Freundlich adsorption constants for PMG and AMPA in various soils.
Source : Vereecken *et al*. (2005)

Soil type	C (%)	Sand (%)	Silt (%)	Clay (%)	pH	n^a	K_f^b	Ref.
Clay loam	1.56	9.9	37.5	52.6	7.5	0.67	76	26
Silt loam	1.64	28	55	17	5.8	0.51	56	26
Sandy loam	1.24	75.9	17	7.1	5.6	0.46	33	26
Sandy loam	9.2	82	13	5	4.6	0.58	51.1	31
Sandy silt	3.7	46	37	17	5.8	0.77	40.64	31
Silt loam	0	13	64	23	8	0.77	13.8	31
Loamy silt	0.5	2	82	16	8.3	0.44	152.9	31
Sand	0.9	—	—	—	6.25	1	3060	37
Sand	0.1	—	—	—	6.65	1	5.0×10^5	37
Sand	3	—	—	—	4.32	1	25.5	37
Sand	0.3	—	—	—	4.2	1	48	37
Sand	0.4	—	—	—	4.1	1	72	37
Silty loam	2.42	21	62	17	5.4	—	—	29
Silty loam	0.47	17	66	17	6.1	0.97	166	29
Clay	2.88	39	15	46	5.8	0.92	55	29
Clay	0.54	29	13	58	5.6	0.91	249	29
Sandy loam	2.57	79	8	13	5.8	0.9	44	29
Sandy loam	0.72	93	3	4	5.7	1	55	29
Clay	7.06	35	24	41	6	1.03	97	29
Clay	2.96	25	28	47	6	1.02	41	29
Sandy loam	5.93	81	15	4	6.4	0.85	97	29
Sandy loam	1.77	83	13	4	5.9	0.86	51	29
Clay	2.67	—	—	41	8.1	0.93	58	29
Clay	2.5	—	—	>30	7.9	0.87	113	29
Sandy loam	2.35	—	—	21	7.1	0.9	93	29
Sandy loam	0.75	—	—	8	6.8	0.86	90	29
Clay	7.05	35	24	41	6	1.26	179	29
Sandy loam	5.93	81	15	4	6.3	0.98	121	29
Silt loam	7.9	79	16	5	5.4	0.93	159	29
Silt loam	4.5	76	20	4	5.6	1.05	102	29
Silt loam	1.3	62	30	8	5.4	0.76	37	29
Muddy clay	12.6	—	—	57	6.9	0.91	84	29
Organic soil	26	78	13	9	5.2	1.14	303	29
Coarse sandy loam	1.7	65	17	15	7.67	0.778	30.9	74
Coarse sandy loam	1.7	65	17	15	7.67	0.261	180	74
Coarse sandy loam	1.7	65	17	15	6.13	0.749	78.5	74
Coarse sandy loam	1.7	65	17	15	6.3	0.75	78.4	74
Coarse sandy loam	1.7	65	17	15	6.53	0.77	48.4	74
Coarse sandy loam	1.7	65	17	15	6.57	0.757	53.3	74
Coarse sandy loam	1.7	65	17	15	7.07	0.76	45.7	74
Coarse sandy loam	1.7	65	17	15	7.3	0.79	37.9	74
Coarse sandy loam	1.7	65	17	15	6.43	0.794	49.8	74
Coarse sandy loam	1.7	65	17	15	6.8	0.779	42.2	74
Coarse sandy loam	1.7	65	17	15	6.97	0.8	31.3	74
Coarse sandy loam	1.7	65	17	15	10.4	0.92	0.6	74
Coarse sandy loam	1.7	65	17	15	9.7	0.962	0.7	74
Coarse sandy loam	1.7	65	17	15	8.77	0.954	2.2	74
Coarse sandy loam	1.7	65	17	15	4.83	0.965	1.9	74
Coarse sandy loam	1.7	65	17	15	5.57	0.95	5.8	74
Coarse sandy loam	1.7	65	17	15	6.3	0.928	10.4	74
Coarse sandy loam	1.7	65	17	15	9.8	1	1.4	74
Coarse sandy loam	1.7	65	17	15	9.17	1.027	0.6	74
Coarse sandy loam	1.7	65	17	15	8.1	0.944	3	74
Sandy loam	3	65	17	15	6,3	0.753	78.4	50
Sandy soil	3.1	90	2.9	4	5.32	0.787	59	50

[a] n = Freundlich exponent.
[b] K_f = Freundlich distribution coefficient.

Annex 7 : Half-lives of PMG in water

Concentration (µg a.e./mL)	Matrix	Sterility	Light exposure	pH	Temperature (°C)	Reported half-life* (days)	Source
8	water	n.m.	Crossby reactor	n.m.	n.m.	no degradation	Rueppel et al. (1977)
1	deionized water	n.m.	550-650 nm[a]	n.m.	20	no degradation	Lund-Hoie and Friestad (1986)
1	deionized water	n.m.	400 - 600 nm[a]	n.m.	20	no degradation	Lund-Hoie and Friestad (1986)
1	deionized water	n.m.	254 nm[a]	n.m.	20	4	Lund-Hoie and Friestad (1986)
2,000	deionized water	n.m.	darkness	n.m.	20	no degradation	Lund-Hoie and Friestad (1986)
2,000	deionized water	n.m.	254 nm[a]	n.m.	20	21	Lund-Hoie and Friestad (1986)
100.000	deionized water	n.m.	sunlight[b]	n.m.	-5–20	14	Lund-Hoie and Friestad (1986)
100.000	deionized water + silty clay loam (30-40% clay) (5% w/w)	n.m.	sunlight[b]	n.m.	-5–20	12	Lund-Hoie and Friestad (1986)
100.000	polluted lake water	n.m.	sunlight[b]	n.m.	-5–20	> 63	Lund-Hoie and Friestad (1986)
100.000	polluted lake water + silty clay loam (30-40% clay) (5% w/w)	n.m.	sunlight[b]	n.m.	-5–20	7	Lund-Hoie and Friestad (1986)
n.r.	Lake water	no	n.r.	n.r.	n.r.	> 63	Mackay et al. (2006) cited by Mercurio et al. (2014)
n.r.	Pond water	no	n.r.	n.r.	n.r.	70	Mackay et al. (2006) cited by Mercurio et al. (2014)
n.r.	Swamp water	no	n.r.	n.r.	n.r.	63	Mackay et al. (2006) cited by Mercurio et al. (2014)
n.r.	*Sphagnum* bog water	no	n.r.	4.2	n.r.	49	Brightwed and Malik (undated)[c] cited by Ghassemi et al. (1981)
n.r.	cattail swamp water	no	n.r.	6.2	n.r.	63	Brightwed and Malik (undated)[c] cited by Ghassemi et al. (1981)
n.r.	pond water	no	n.r.	7.3	n.r.	70	Brightwed and Malik (undated)[c] cited by Ghassemi et al. (1981)
0.01	coastal sea water	no	low	n.m.	25	47	Mercurio et al. (2014)
0.01	coastal sea water	no	darkness	n.m.	25	267	Mercurio et al. (2014)
0.01	coastal sea water	no	darkness	n.m.	31	315	Mercurio et al. (2014)

n.m. : not mentioned n.r. : not recovered

a : 30 W/m^2

b : mean : 295–385 nm, 0.6–0.2 $MJ/m^2/d$, covered by a thin film of polyethylene.

c : Brightwed, B.B., and J.M. Malik. Monsanto Agricultural Research Departments, St. Louis, Missouri. Data provided by Monsanto Company. In: Environmental Fate File, Glyphosate, U.S. ENvironmental Protection Agency. Undated

*It is very important to notice the fact that the reported half-lives either refer to degradation or dissipation of the compound, depending on the methodology used. In water, dissipation is the sum of degradation and adsorption on solid surfaces and is measured as the quantity of PMG that is not recovered in the water after a given period of incubation. Degradation is equal to dissipation only when adsorption on surfaces is negligible, i.e. in a plastic container that do not contain any sediment or mineral.

Annex 8 : Half lives of PMG in various soils and conditions

Concentration (µg a.e./g)	Soil type[a]	Sterility	Light exposure	Humidity	Oxygen condition	Temperature (°C)	Reported half-life* (days)	Source
1.18	Loam	no	n.m.	50 % of WHC	aerobic	14.3	4	Simonsen et al. (2008)
n.m.	Rendzic leptosol clay loam[b]	no	darkness	80 % of WHC	n.m.	20	14	Al-Rajab et al. (2008)
n.m.	Stagnic luvisol silt clay loam[b]	no	darkness	80 % of WHC	n.m.	20	19	Al-Rajab et al. (2008)
n.m.	Fluvic cambisol sandy loam[b]	no	darkness	80 % of WHC	n.m.	20	14.5	Al-Rajab et al. (2008)
11.21 ± 0.33	Sand topsoil	no	darkness	60 % of WHC	aerobic	20	16.9	Bergström et al. (2011)
12.27 ± 0.19	Sand subsoil	no	darkness	60 % of WHC	aerobic	20	36.5	Bergström et al. (2011)
11.99 ± 0.15	Clay topsoil	no	darkness	60 % of WHC	aerobic	20	110	Bergström et al. (2011)
11.61 ± 0.41	Clay subsoil	no	darkness	60 % of WHC	aerobic	20	151	Bergström et al. (2011)
2	Catlin silt loam (oxic)	yes	darkness	100 % of WHC	aerobic	25	209	Kanissery et al. (2015)
2	Catlin silt loam (anoxic)	yes	darkness	100 % of WHC	anaerobic	25	154	Kanissery et al. (2015)
2	Flanagan silt loam (oxic)	yes	darkness	100 % of WHC	aerobic	25	228	Kanissery et al. (2015)
2	Flanagan silt loam (anoxic)	yes	darkness	100 % of WHC	anaerobic	25	140	Kanissery et al. (2015)
2	Drummer silty clay loam (oxic)	yes	darkness	100 % of WHC	aerobic	25	210	Kanissery et al. (2015)
2	Drummer silty clay loam (anoxic)	yes	darkness	100 % of WHC	anaerobic	25	200	Kanissery et al. (2015)
2	Catlin silt loam (oxic)	no	darkness	100 % of WHC	aerobic	25	18	Kanissery et al. (2015)
2	Catlin silt loam (anoxic)	no	darkness	100 % of WHC	anaerobic	25	42	Kanissery et al. (2015)
2	Flanagan silt loam (oxic)	no	darkness	100 % of WHC	aerobic	25	15	Kanissery et al. (2015)
2	Flanagan silt loam (anoxic)	no	darkness	100 % of WHC	anaerobic	25	51	Kanissery et al. (2015)
2	Drummer silty clay loam (oxic)	no	darkness	100 % of WHC	aerobic	25	18	Kanissery et al. (2015)
2	Drummer silty clay loam (anoxic)	no	darkness	100 % of WHC	anaerobic	25	45	Kanissery et al. (2015)
169 and 845	Ultisol silt loam	yes	darkness	60 % of WHC	aerobic	20	not detected	Sun et al. (2019)
169 and 845	Ultisol silt loam	no	darkness	60 % (w/w)	aerobic	5 and 35 °C	29 and 32	Sun et al. (2019)

a : all soils are topsoils

b : only the extractable PMG with 0.1 M KH_2PO_4 was considered, that is to say 18 % to 35 % of the applied dose.

WHC : water-holding capacity

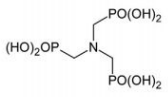
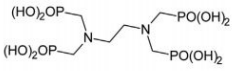
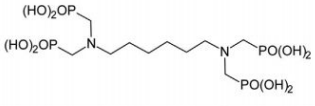
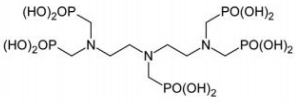
n.m. : not mentioned or measured

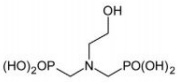
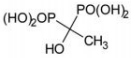
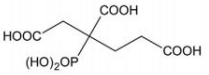
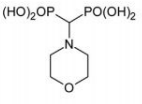
*It is very important to notice the fact that the reported half-lives either refer to degradation or dissipation of the compound, depending on the methodology used. In water, dissipation is the sum of degradation and adsorption on solid surfaces and is measured as the quantity of PMG that is not recovered in the water after a given period of incubation. Degradation is equal to dissipation only when adsorption on surfaces is negligible, i.e. in a plastic container that do not contain any sediment or mineral.

Annex 9 : Data regarding PMG contamination in hydrologic settings in the US. Source : Battaglin *et al.* (2014)

Hydrologic setting	Number of samples	Samples with PMG detected (%)	Median of PMG content (µg L^{-1} or µg kg^{-1})	Maximum of PMG content (µg L^{-1} or µg kg^{-1})	Samples with AMPA detected (%)	Median of AMPA content (µg L^{-1} or µg kg^{-1})	Maximum of AMPA content (µg L^{-1} or µg kg^{-1})
All sites	3,732	39.4	< 0.02	476	55.0	0.04	397
Streams	1,508	52.5	0.03	73	71.6	0.20	28
Groundwater	1,171	5.8	< 0.02	2.03	14.3	> 0.02	4.88
Ditches and drains	374	70.9	0.20	427	80.7	0.43	397
Large rivers	318	53.1	0.03	3.08	89.3	0.22	4.43
Soil water	116	34.5	< 0.02	1.00	65.5	0.06	1.91
Lakes, ponds and wetlands	104	33.7	< 0.02	301	29.8	< 0.02	41
Precipitation	85	70.6	0.11	2.50	71.8	0.04	0.48
Soil and sediment	45	91.1	9.6	476	93.3	18.0	341
Wastewater treatment plant	11	9.09	< 0.02	0.30	81.8	0.45	2.54

Annex 10 : Examples of phosphonates used as antiscalants approved for drinking water production according to the German drinking water ordinance. Source : Armbruster *et al.* (2019)

Phosphonate	Structure
ATMP, NTMP Aminotris(methylenephosphonic acid), Nitrilotris(methylenephosphonic acid) CAS 6419-19-8 Formula $C_3H_{12}NO_9P_3$ MW 299.1 g/mol	
EDTMP, EDATMP Ethylendiaminetetra(methylene-phosphonic acid) CAS 1429-50-1 Formula $C_6H_{20}N_2O_{12}P_4$ MW 436.1 g/mol	
HDTMP Hexamethylenediamine-tetra(methylenephosphonic acid) CAS 23605-74-5 Formula $C_{10}H_{28}N_2O_{12}P_4$ MW 492.2 g/mol	
DTPMP, DETAPMP Diethylenetriaminepenta(methylene-phosphonic acid) CAS 15827-60-8 Formula $C_9H_{28}N_3O_{15}P_5$ MW 573.2 g/mol	

Phosphonate	Structure
HEMPA, EABMP Hydroxyethylaminodi(methylene-phosphonic acid) CAS 5995-42-6 Formula $C_4H_{13}NO_7P_2$ MW 249.1 g/mol	
HEDP 1-Hydroxyethanediphosphonic acid, 1-Hydroxyethylidene-1,1-diphosphonic acid, Etidronic acid CAS 2809-21-4 Formula $C_2H_8O_7P_2$ MW 206.0 g/mol	
PBTC 2-Phosphonobutane-1,2,4-tricarboxylic acid CAS 37971-36-1 Formula $C_7H_{11}O_9P$ MW 270.1 g/mol	
MOMP (Morpholine-4-ylmethylene)bisphosphonic acid CAS 32545-75-8 Formula $C_5H_{13}NO_7P_2$ MW 261.1 g/mol	

Annex 11 : MRLs in the EU for PMG per product and per year.

Groups and examples of individual products to which the MRLs apply	Reg. (EU) No 293/2013 (mg/kg)	Reg. (EU) No 441/2012 (mg/kg)	Reg. (EC) No 839/2008 (mg/kg)	Reg. (EC) No 149/2008 (mg/kg)
. FRUITS, FRESH or FROZEN; TREE NUTS				
. Citrus fruits				
. Grapefruits	0.1*	0.1*	0.1*	0.1*
. Oranges	0.5	0.5	0.5	0.5
. Lemons	0.1*	0.1*	0.1*	0.1*
. Limes	0.1*	0.1*	0.1*	0.1*
. Mandarins	0.5	0.5	0.5	0.5
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Tree nuts	0.1*	0.1*	0.1*	0.1*
. Almonds	0.1*	0.1*	0.1*	0.1*
. Brazil nuts	0.1*	0.1*	0.1*	0.1*
. Cashew nuts	0.1*	0.1*	0.1*	0.1*
. Chestnuts	0.1*	0.1*	0.1*	0.1*
. Coconuts	0.1*	0.1*	0.1*	0.1*
. Hazelnuts/cobnuts	0.1*	0.1*	0.1*	0.1*
. Macadamias	0.1*	0.1*	0.1*	0.1*
. Pecans	0.1*	0.1*	0.1*	0.1*
. Pine nut kernels	0.1*	0.1*	0.1*	0.1*
. Pistachios	0.1*	0.1*	0.1*	0.1*
. Walnuts	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Pome fruits	0.1*	0.1*	0.1*	0.1*
. Apples	0.1*	0.1*	0.1*	0.1*
. Pears	0.1*	0.1*	0.1*	0.1*
. Quinces	0.1*	0.1*	0.1*	0.1*
. Medlars	0.1*	0.1*	0.1*	0.1*
. Loquats/Japanese medlars	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Stone fruits	0.1*	0.1*	0.1*	0.1*
. Apricots	0.1*	0.1*	0.1*	0.1*
. Cherries (sweet)	0.1*	0.1*	0.1*	0.1*
. Peaches	0.1*	0.1*	0.1*	0.1*
. Plums	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Berries and small fruits				0.1*
. (a) grapes	0.5	0.5	0.5	0.1*
. Table grapes	0.5	0.5	0.5	0.1*
. Wine grapes	0.5	0.5	0.5	0.1*
. (b) strawberries	0.1*	0.1*	0.1*	0.1*
. (c) cane fruits	0.1*	0.1*	0.1*	0.1*
. Blackberries	0.1*	0.1*	0.1*	0.1*
. Dewberries	0.1*	0.1*	0.1*	0.1*
. Raspberries (red and yellow)	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (d) other small fruits and berries	0.1*	0.1*	0.1*	0.1*
. Blueberries	0.1*	0.1*	0.1*	0.1*
. Cranberries	0.1*	0.1*	0.1*	0.1*
. Currants (black, red and white)	0.1*	0.1*	0.1*	0.1*
. Gooseberries (green, red and yellow)	0.1*	0.1*	0.1*	0.1*
. Rose hips	0.1*	0.1*	0.1*	0.1*
. Mulberries (black and white)	0.1*	0.1*	0.1*	0.1*
. Azaroles/Mediterranean medlars	0.1*	0.1*	0.1*	0.1*
. Elderberries	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Miscellaneous fruitswith				
. (a) edible peel				
. Dates	0.1*	0.1*	0.1*	0.1*
. Figs	0.1*	0.1*	0.1*	0.1*
. Table olives	1	1	1	1
. Kumquats	0.1*	0.1*	0.1*	0.1*
. Carambolas	0.1*	0.1*	0.1*	0.1*
. Kaki/Japanese persimmons	0.1*	0.1*	0.1*	0.1*
. Jambuls/jambolans	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (b) inedible peel, small	0.1*	0.1*	0.1*	0.1*
. Kiwi fruits (green, red, yellow)	0.1*	0.1*	0.1*	0.1*
. Litchis/lychees	0.1*	0.1*	0.1*	0.1*
. Passionfruits/maracujas	0.1*	0.1*	0.1*	0.1*
. Prickly pears/cactus fruits	0.1*	0.1*	0.1*	0.1*
. Star apples/cainitos	0.1*	0.1*	0.1*	0.1*
. American persimmons/Virginia kaki	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (c) inedible peel, large	0.1*	0.1*	0.1*	0.1*
. Avocados	0.1*	0.1*	0.1*	0.1*
. Bananas	0.1*	0.1*	0.1*	0.1*
. Mangoes	0.1*	0.1*	0.1*	0.1*
. Papayas	0.1*	0.1*	0.1*	0.1*
. Granate apples/pomegranates	0.1*	0.1*	0.1*	0.1*
. Cherimoyas	0.1*	0.1*	0.1*	0.1*
. Guavas	0.1*	0.1*	0.1*	0.1*
. Pineapples	0.1*	0.1*	0.1*	0.1*

* indicates lower limit of analytical determination (LOQ)

Annex 11 : MRLs in the EU for PMG per product and per year.

. Breadfruits	0.1*	0.1*	0.1*	0.1*
. Durians	0.1*	0.1*	0.1*	0.1*
. Soursops/guanabanas	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. VEGETABLES, FRESH or FROZEN				
. Root and tuber vegetables				
. (a) potatoes	0.5	0.5	0.5	0.5
. (b) tropical root and tuber vegetables	0.1*	0.1*	0.1*	0.1*
. Cassava roots/manioc	0.1*	0.1*	0.1*	0.1*
. Sweet potatoes	0.1*	0.1*	0.1*	0.1*
. Yams	0.1*	0.1*	0.1*	0.1*
. Arrowroots	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (c) other root and tuber vegetables except sugar beets	0.1*	0.1*	0.1*	0.1*
. Beetroots	0.1*	0.1*	0.1*	0.1*
. Carrots	0.1*	0.1*	0.1*	0.1*
. Celeriacs/turnip rooted celeries	0.1*	0.1*	0.1*	0.1*
. Horseradishes	0.1*	0.1*	0.1*	0.1*
. Jerusalem artichokes	0.1*	0.1*	0.1*	0.1*
. Parsnips	0.1*	0.1*	0.1*	0.1*
. Parsley roots/Hamburg roots parsley	0.1*	0.1*	0.1*	0.1*
. Radishes	0.1*	0.1*	0.1*	0.1*
. Salsifies	0.1*	0.1*	0.1*	0.1*
. Swedes/rutabagas	0.1*	0.1*	0.1*	0.1*
. Turnips	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Bulb vegetables	0.1*	0.1*	0.1*	0.1*
. Garlic	0.1*	0.1*	0.1*	0.1*
. Onions	0.1*	0.1*	0.1*	0.1*
. Shallots	0.1*	0.1*	0.1*	0.1*
. Spring onions/green onions and Welsh onions	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Fruiting vegetables		0.1*	0.1*	0.1*
. (a) Solanaceae and Malvaceae	0.1*	0.1*	0.1*	0.1*
. Tomatoes	0.1*	0.1*	0.1*	0.1*
. Sweet peppers/bell peppers	0.1*	0.1*	0.1*	0.1*
. Aubergines/eggplants	0.1*	0.1*	0.1*	0.1*
. Okra/lady's fingers	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (b) cucurbits with edible peel	0.1*	0.1*	0.1*	0.1*
. Cucumbers	0.1*	0.1*	0.1*	0.1*
. Gherkins	0.1*	0.1*	0.1*	0.1*
. Courgettes	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (c) cucurbits with inedible peel	0.1*	0.1*	0.1*	0.1*
. Melons	0.1*	0.1*	0.1*	0.1*
. Pumpkins	0.1*	0.1*	0.1*	0.1*
. Watermelons	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (d) sweet corn	3	0.1*	0.1*	0.1*
. (e) other fruiting vegetables	0.1*	0.1*	0.1*	0.1*
. Brassica vegetables (excluding brassica roots and brassica baby leaf crops)	0.1*	0.1*	0.1*	0.1*
. (a) flowering brassica	0.1*	0.1*	0.1*	0.1*
. Broccoli	0.1*	0.1*	0.1*	0.1*
. Cauliflowers	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (b) head brassica	0.1*	0.1*	0.1*	0.1*
. Brussels sprouts	0.1*	0.1*	0.1*	0.1*
. Head cabbages	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (c) leafy brassica	0.1*	0.1*	0.1*	0.1*
. Chinese cabbages/pe-tsai	0.1*	0.1*	0.1*	0.1*
. Kales	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (d) kohlrabies	0.1*	0.1*	0.1*	0.1*
. Leaf vegetables, herbs and edible flowers	0.1*	0.1*	0.1*	0.1*
. (a) lettuces and salad plants	0.1*	0.1*	0.1*	0.1*
. Lamb's lettuces/corn salads	0.1*	0.1*	0.1*	0.1*
. Lettuces	0.1*	0.1*	0.1*	0.1*
. Escaroles/broad-leaved endives	0.1*	0.1*	0.1*	0.1*
. Cresses and other sprouts and shoots	0.1*	0.1*	0.1*	0.1*
. Land cresses	0.1*	0.1*	0.1*	0.1*
. Roman rocket/rucola	0.1*	0.1*	0.1*	0.1*
. Red mustards	0.1*	0.1*	0.1*	0.1*
. Baby leaf crops (including brassica species)	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (b) spinaches and similar leaves	0.1*	0.1*	0.1*	0.1*
. Spinaches	0.1*	0.1*	0.1*	0.1*
. Purslanes	0.1*	0.1*	0.1*	0.1*
. Chards/beet leaves	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. (c) grape leaves and similar species	0.1*	0.1*	0.1*	0.1*

* indicates lower limit of analytical determination (LOQ)

Annex 11 : MRLs in the EU for PMG per product and per year.

. (d) watercresses	0.1*	0.1*	0.1*	0.1*
. (e) witloofs/Belgian endives	0.1*	0.1*	0.1*	0.1*
. (f) herbs and edible flowers	0.1*	0.1*	0.1*	0.1*
. Chervil	0.1*	0.1*	0.1*	0.1*
. Chives	0.1*	0.1*	0.1*	0.1*
. Celery leaves	0.1*	0.1*	0.1*	0.1*
. Parsley	0.1*	0.1*	0.1*	0.1*
. Sage	0.1*	0.1*	0.1*	0.1*
. Rosemary	0.1*	0.1*	0.1*	0.1*
. Thyme	0.1*	0.1*	0.1*	0.1*
. Basil and edible flowers	0.1*	0.1*	0.1*	0.1*
. Laurel/bay leaves	0.1*	0.1*	0.1*	0.1*
. Tarragon	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Legume vegetables	0.1*	0.1*	0.1*	0.1*
. Beans (with pods)	0.1*	0.1*	0.1*	0.1*
. Beans (without pods)	0.1*	0.1*	0.1*	0.1*
. Peas (with pods)	0.1*	0.1*	0.1*	0.1*
. Peas (without pods)	0.1*	0.1*	0.1*	0.1*
. Lentils	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Stem vegetables	0.1*	0.1*	0.1*	0.1*
. Asparagus	0.1*	0.1*	0.1*	0.1*
. Cardoons	0.1*	0.1*	0.1*	0.1*
. Celeries	0.1*	0.1*	0.1*	0.1*
. Florence fennels	0.1*	0.1*	0.1*	0.1*
. Globe artichokes	0.1*	0.1*	0.1*	0.1*
. Leeks	0.1*	0.1*	0.1*	0.1*
. Rhubarbs	0.1*	0.1*	0.1*	0.1*
. Bamboo shoots	0.1*	0.1*	0.1*	0.1*
. Palm hearts	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Fungi, mosses and lichens				
. Cultivated fungi	0.1*	0.1*	0.1*	0.1*
. Wild fungi	50	50	50	50
. Mosses and lichens	0.1*	0.1*	0.1*	0.1*
. Algae and prokaryotes organisms				
. PULSES				
. Beans	2	2	2	2
. Lentils	10	10	0.1*	0.1*
. Peas	10	10	10	10
. Lupins/lupini beans	10	10	10	10
. Others (2)	0.1*	0.1*	0.1*	0.1*
. OILSEEDS AND OIL FRUITS				
. Oilseeds				
. Linseeds	10	10	10	10
. Peanuts/groundnuts	0.1*	0.1*	0.1*	0.1*
. Poppy seeds	0.1*	0.1*	0.1*	0.1*
. Sesame seeds	0.1*	0.1*	0.1*	0.1*
. Sunflower seeds	20	20	20	20
. Rapeseeds/canola seeds	10	10	10	10
. Soyabeans	20	20	20	20
. Mustard seeds	10	10	10	10
. Cotton seeds	10	10	10	10
. Pumpkin seeds	0.1*	0.1*	0.1*	0.1*
. Safflower seeds	0.1*	0.1*	0.1*	0.1*
. Borage seeds	0.1	0.1	0.1	0.1
. Gold of pleasure seeds	0.1	0.1	0.1	0.1
. Hemp seeds	0.1*	0.1*	0.1*	0.1*
. Castor beans	0.1	0.1	0.1	0.1
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Oil fruits				
. Olives for oil production	1	1	1	1
. Oil palms kernels	0.1	0.1	0.1	0.1
. Oil palms fruits	0.1	0.1	0.1	0.1
. Kapok	0.1	0.1	0.1	0.1
. Others (2)	0.1*	0.1*	0.1*	0.1*
. CEREALS				
. Barley	20	20	20	20
. Buckwheat and other pseudocereals	0.1*	0.1*	0.1*	0.1*
. Maize/corn	1	1	1	1
. Common millet/proso millet	0.1*	0.1*	0.1*	0.1*
. Oat	20	20	20	20
. Rice	0.1*	0.1*	0.1*	0.1*
. Rye	10	10	10	10
. Sorghum	20	20	20	20
. Wheat	10	10	10	10
. Others (2)	0.1*	0.1*	0.1*	0.1*
. TEAS, COFFEE, HERBAL INFUSIONS, COCOA AND CAROBS				
. Teas	2	2	2	2
. Coffee beans	0.1	0.1	0.1	0.1
. Herbal infusions from	2.0*	2.0*	2	2
. (a) flowers	2.0*	2.0*	2	2

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. Chamomile	2.0*	2.0*	2	2
. Hibiscus/roselle	2.0*	2.0*	2	2
. Rose	2.0*	2.0*	2	2
. Jasmine	2.0*	2.0*	2	2
. Lime/linden	2.0*	2.0*	2	2
. Others (2)	2.0*	2.0*	2	2
. (b) leaves and herbs	2.0*	2.0*	2	2
. Strawberry	2.0*	2.0*	2	2
. Rooibos	2.0*	2.0*	2	2
. Mate/maté	2.0*	2.0*	2.0*	2.0*
. Others (2)	2.0*	2.0*	2.0*	2.0*
. (c) roots	2.0*	2.0*	2.0*	2.0*
. Valerian	2.0*	2.0*	2.0*	2.0*
. Ginseng	2.0*	2.0*	2.0*	2.0*
. Others (2)	2.0*	2.0*	2.0*	2.0*
. (d) any other parts of the plant	2.0*	2.0*	2.0*	2.0*
. Cocoa beans	0.1*	0.1*	0.1*	0.1*
. Carobs/Saint John's breads	0.1*	0.1*	0.1*	0.1*
. HOPS	0.1*	0.1*	0.1*	0.1*
. SPICES	0.1*	0.1*	0.1*	0.1*
. Seed spices	0.1*	0.1*	0.1*	0.1*
. Anise/aniseed	0.1*	0.1*	0.1*	0.1*
. Black caraway/black cumin	0.1*	0.1*	0.1*	0.1*
. Celery	0.1*	0.1*	0.1*	0.1*
. Coriander	0.1*	0.1*	0.1*	0.1*
. Cumin	0.1*	0.1*	0.1*	0.1*
. Dill	0.1*	0.1*	0.1*	0.1*
. Fennel	0.1*	0.1*	0.1*	0.1*
. Fenugreek	0.1*	0.1*	0.1*	0.1*
. Nutmeg	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Fruit spices	0.1*	0.1*	0.1*	0.1*
. Allspice/pimento	0.1*	0.1*	0.1*	0.1*
. Sichuan pepper	0.1*	0.1*	0.1*	0.1*
. Caraway	0.1*	0.1*	0.1*	0.1*
. Cardamom	0.1*	0.1*	0.1*	0.1*
. Juniper berry	0.1*	0.1*	0.1*	0.1*
. Peppercorn (black, green and white)	0.1*	0.1*	0.1*	0.1*
. Vanilla	0.1*	0.1*	0.1*	0.1*
. Tamarind	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Bark spices	0.1*	0.1*	0.1*	0.1*
. Cinnamon	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Root and rhizome spices	0.1*	0.1*	0.1*	0.1*
. Liquorice	0.1*	0.1*	0.1*	0.1*
. Ginger (10)	0.1*	0.1*	0.1*	0.1*
. Turmeric/curcuma	0.1*	0.1*	0.1*	0.1*
. Horseradish (11)	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Bud spices	0.1*	0.1*	0.1*	0.1*
. Cloves	0.1*	0.1*	0.1*	0.1*
. Capers	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1	0.1
. Flower pistil spices	0.1*	0.1*	0.1	0.1
. Saffron	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. Aril spices	0.1*	0.1*	0.1*	0.1*
. Mace	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. SUGAR PLANTS				
. Sugar beet roots	15	1.0*	1.0*	1.0*
. Sugar canes	0.1*	0.1*	0.1*	0.1*
. Chicory roots	0.1*	0.1*	0.1*	0.1*
. Others (2)	0.1*	0.1*	0.1*	0.1*
. PRODUCTS OF ANIMAL ORIGIN -TERRESTRIAL ANIMALS				
. Commodities from				
. (a) swine				
. Muscle	0.05*	0.05*	0.05*	0.05*
. Fat	0.05*	0.05*	0.05*	0.05*
. Liver	0.05*	0.05*	0.05*	0.05*
. Kidney	0.5	0.5	0.5	0.5
. Edible offals (other than liver and kidney)	0.05*	0.05*	0.05*	0.05*
. Others (2)	0.05*	0.05*	0.05*	0.05*
. (b) bovine				
. Muscle	0.05*	0.05*	0.05*	0.05*
. Fat	0.05*	0.05*	0.05*	0.05*
. Liver	0.2	0.2	0.2	0.2
. Kidney	2	2	2	2
. Edible offals (other than liver and kidney)	0.05*	0.05*	0.05*	0.05*
. Others (2)	0.05*	0.05*	0.05*	0.05*
. (c) sheep	0.05*	0.05*	0.05*	0.05*

* indicates lower limit of analytical determination (LOQ)

Annex 11 : MRLs in the EU for PMG per product and per year.

. Muscle	0.05*	0.05*		
. Fat	0.05*	0.05*		
. Liver	0.05*	0.05*		
. Kidney	0.05*	0.05*		
. Edible offals (other than liver and kidney)	0.05*	0.05*		
. Others (2)	0.05*	0.05*		
. d) goat	0.05*	0.05*	0.05*	0.05*
. Muscle	0.05*	0.05*		
. Fat	0.05*	0.05*		
. Liver	0.05*	0.05*		
. Kidney	0.05*	0.05*		
. Edible offals (other than liver and kidney)	0.05*	0.05*		
. Others (2)	0.05*	0.05*		
. (e) equine	0.05*	0.05*	0.05*	0.05*
. Muscle	0.05*	0.05*	0.05*	0.05*
. Fat	0.05*	0.05*	0.05*	0.05*
. Liver	0.05*	0.05*	0.05*	0.05*
. Kidney	0.05*	0.05*	0.05*	0.05*
. Edible offals (other than liver and kidney)	0.05*	0.05*	0.05*	0.05*
. Others (2)	0.05*	0.05*	0.05*	0.05*
. (f) poultry				
. Muscle	0.05*	0.05*	0.05*	0.05*
. Fat	0.05*	0.05*	0.05*	0.05*
. Liver	0.05*	0.05*	0.05*	0.05*
. Kidney	0.1*	0.1*	0.1*	0.1*
. Edible offals (other than liver and kidney)	0.05*	0.05*	0.05*	0.05*
. Others (2)	0.05*	0.05*	0.05*	0.05*
. (g) other farmed terrestrial animals	0.05*	0.05*	0.05*	0.05*
. Muscle	0.05*	0.05*	0.05*	0.05*
. Fat	0.05*	0.05*	0.05*	0.05*
. Liver	0.05*	0.05*	0.05*	0.05*
. Kidney	0.05*	0.05*	0.05	0.05
. Edible offals (other than liver and kidney)	0.05*	0.05*	0.05	0.05
. Others (2)	0.05*	0.05*	0.05	0.05
. Milk	0.05*	0.05*	0.05*	0.01*
. Cattle	0.05*	0.05*	0.05*	
. Sheep	0.05*	0.05*	0.05*	
. Goat	0.05*	0.05*	0.05*	
. Horse	0.05*	0.05*	0.05*	
. Others (2)	0.05*	0.05*	0.05*	
. Birds eggs	0.05*	0.05*	0.05*	0.01*
. Chicken	0.05*	0.05*		
. Duck	0.05*	0.05*	0.05*	0.01
. Geese	0.05*	0.05*	0.05*	0.01
. Quail	0.05*	0.05*	0.05*	0.01
. Others (2)	0.05*	0.05*	0.05*	0.01
. Honey and other apiculture products (7)	0.05*	0.05*		
. Amphibians and Reptiles	0.05*	0.05*		
. Terrestrial invertebrate animals	0.05*	0.05*		
. Wild terrestrial vertebrate animals	0.05*	0.05*		

* indicates lower limit of analytical determination (LOQ)

Annex 12 : List of other european regulations and directives relevant for PMG.

"A regulation shall have general application. It shall be binding in its entirety and directly applicable in all Member States. A directive shall be binding, as to the result to be achieved, upon each Member State to which it is addressed, but shall leave to the national authorities the choice of form and methods. A decision shall be binding in its entirety upon those to whom it is addressed. Recommendations and opinions shall have no binding force."[3]

- **Regulation 2017/2324** of 12 December 2017 renewing the approval of the active substance glyphosate in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market, and amending the Annex to Commission Implementing Regulation (EU) No 540/2011.

- **Regulation (EU) 2019/724** of 10 May 2019 amending Implementing Regulation (EU) No 686/2012 as regards the nomination of rapporteur Member States and co-rapporteur Member States for the active substances glyphosate, lambda-cyhalothrin, imazamox and pendimethalin and amending Implementing Regulation (EU) No 844/2012 as regards the possibility that a group of Member States assumes jointly the role of the rapporteur Member State (Text with EEA relevance.).

- **Regulation (EU) No 540/2011** of 25 May 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the list of approved active substances.

- **Directive 2002/63/EC** of 11 July 2002 establishing Community methods of sampling for the official control of pesticide residues in and on products of plant and animal origin and repealing Directive 79/700/EEC.

- **Directive (EU) 2015/1787/EU,** of 6 October 2015 amending Annexes II and III to Council Directive 98/83/EC on the quality of water intended for human consumption**.**

- **Directive 2000/60/EC** of the European Parliament and of the Council of 23 October 2000 establishing a framework for Community action in the field of water policy (Water Framework Directive). It settles a general and integrated prescriptions for water and aquatic environment management, including monitoring and control of the levels of particular chemicals, including organophosphorus compounds such as PMG.

- **Directive 2008/105/EC** of the European Parliament and of the Council of 16 December 2008 on environmental quality standards in the field of water policy, amending and subsequently repealing Council Directives 82/176/EEC, 83/513/EEC, 84/156/EEC, 84/491/EEC, 86/280/EEC and amending Directive 2000/60/EC of the European Parliament and of the Council. This directive defines precise quality criteria for surface water in [µg/L] wherein PMG is classified in the category "substances subject to review for possible identification as priority substances or priority hazardous substances";

- **Directive 2013/39/EU** of the European Parliament and of the Council of 12 August 2013 amending Directives 2000/60/EC and 2008/105/EC as regards priority substances in the field of water policy.

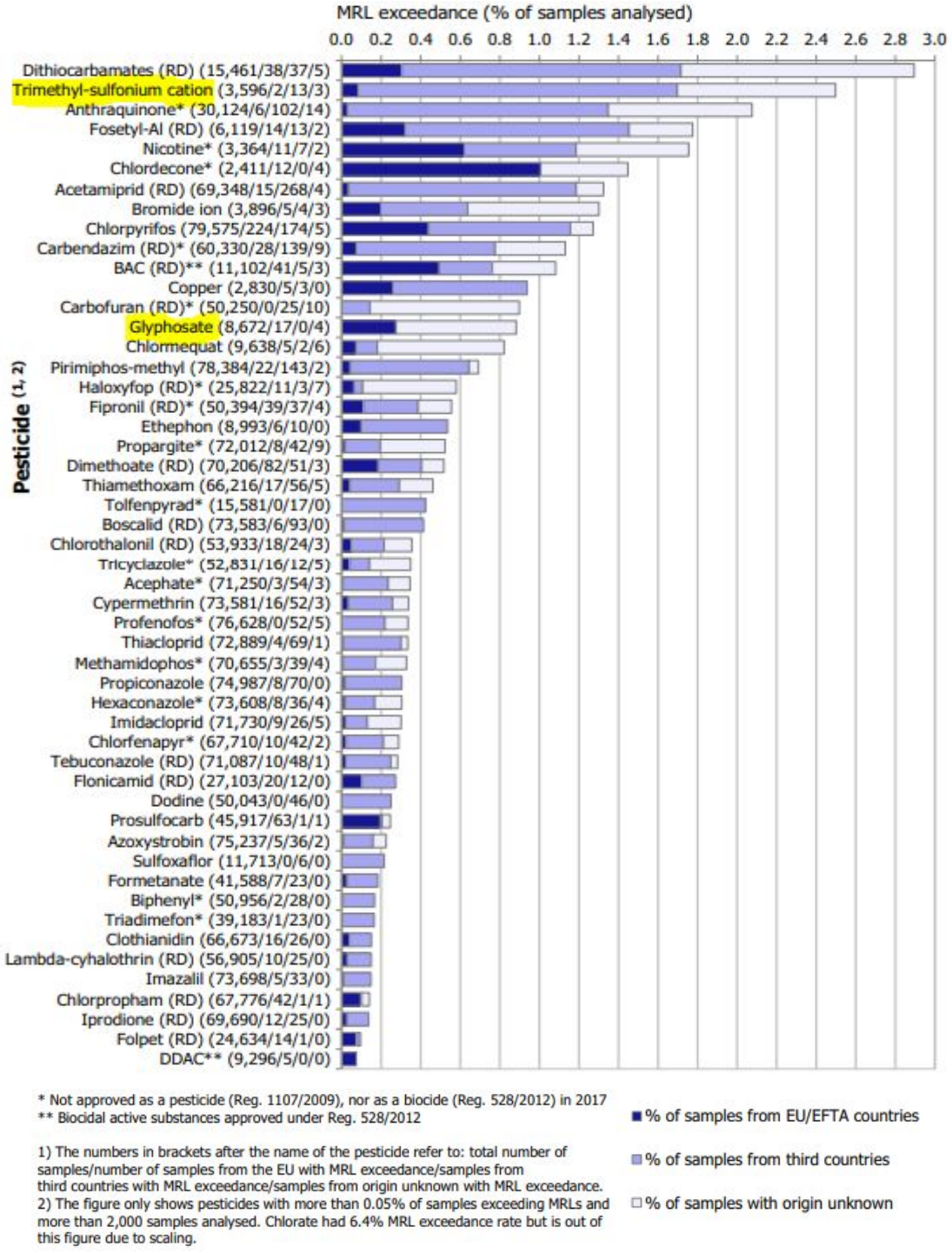
- **Directive 2009/128/EC** of the European Parliament and of the Council of 21 October 2009 establishing a framework for Community action to achieve the sustainable use of pesticides.

- **Regulation No 1272/2008** of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006

All the mentioned regulations are available online on the EUR-Lex website[4]

[3] https://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CELEX:12002E249:EN:HTML (consultation date : 22/03/2020)
[4] https://eur-lex.europa.eu/homepage.html

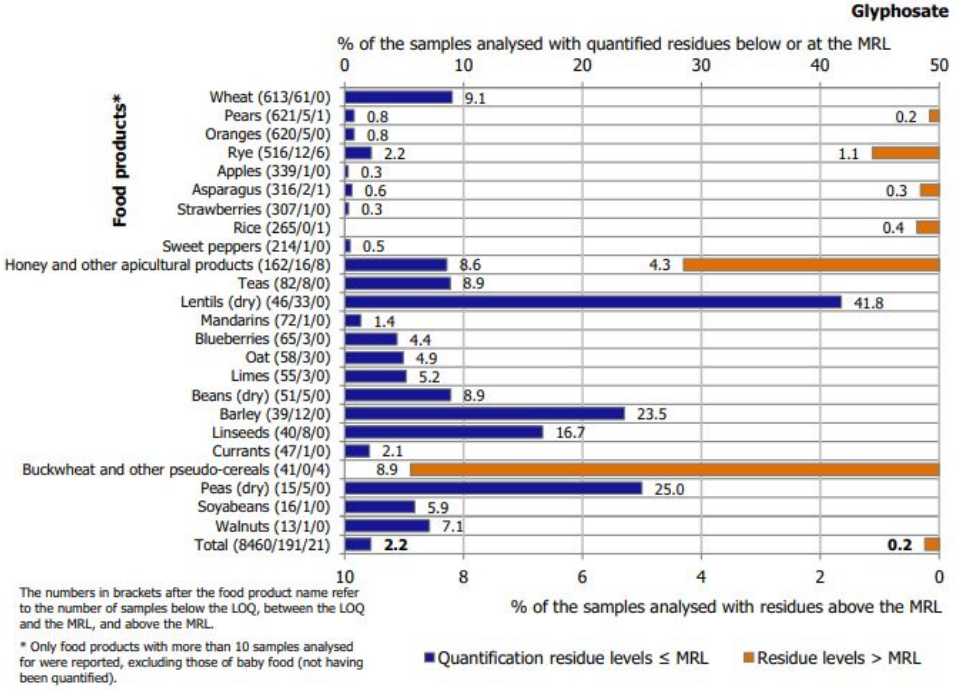
Annex 13 : MRL exceedance rates per pesticide in EU in 2017 - Source : EFSA (2019).



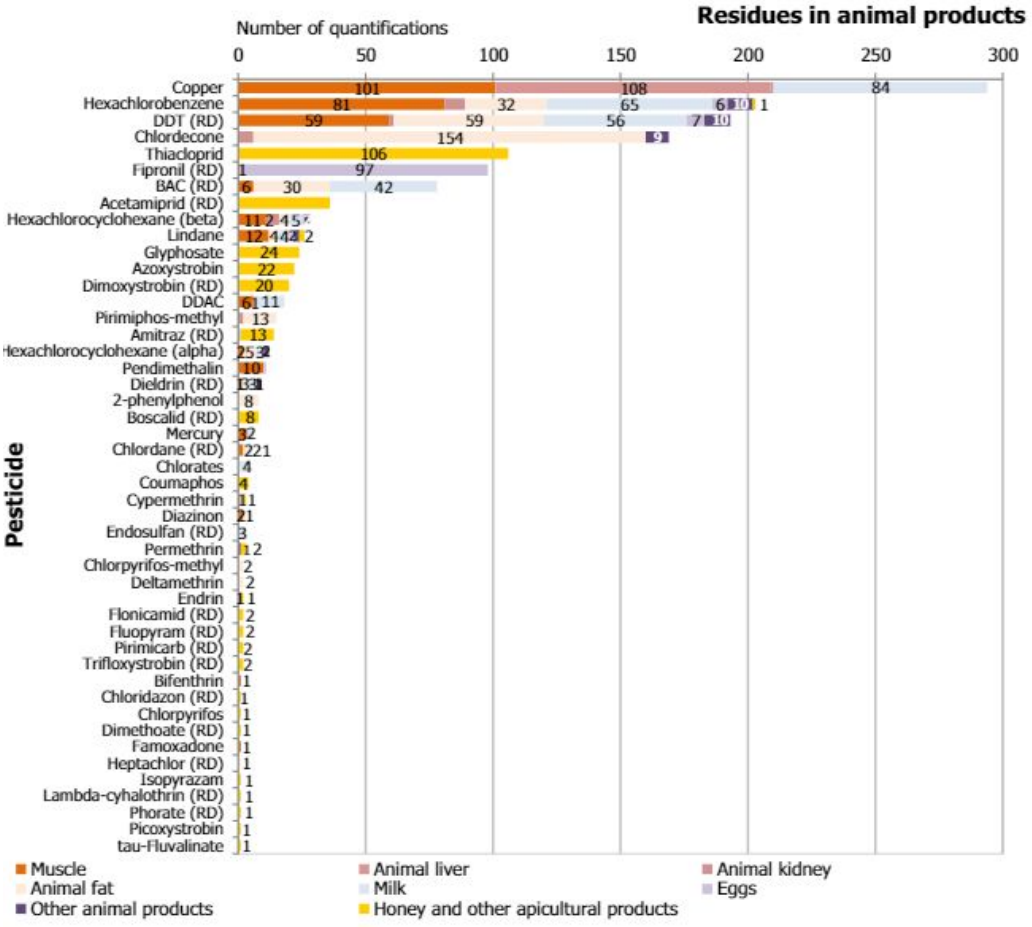
* Not approved as a pesticide (Reg. 1107/2009), nor as a biocide (Reg. 528/2012) in 2017
** Biocidal active substances approved under Reg. 528/2012

1) The numbers in brackets after the name of the pesticide refer to: total number of samples/number of samples from the EU with MRL exceedance/samples from third countries with MRL exceedance/samples from origin unknown with MRL exceedance.
2) The figure only shows pesticides with more than 0.05% of samples exceeding MRLs and more than 2,000 samples analysed. Chlorate had 6.4% MRL exceedance rate but is out of this figure due to scaling.

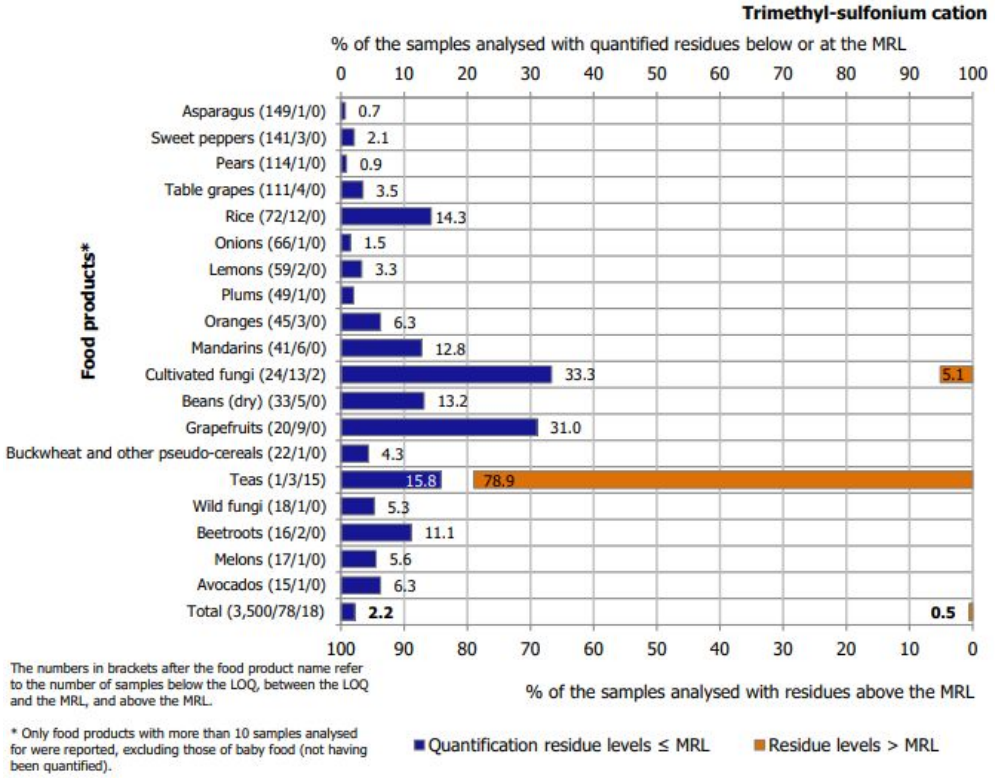
Annex 14 : Samples of plant origin tested with PMG residues above LOD.



Annex 15 : Pesticides found in animal products at or above the LOQ.



Annex 16 : Samples of plant origin tested with trimethyl sulfonium cation above LOD.



Annex 17 : Main impurities in raw juice extracted from sugar beet.

Impurities neither removed or decomposed by carbonation (g/100g sugar)		**Impurities partly removed by carbonation (g/100g sugar)**		**Impurities totally removed by carbonation (g/100g sugar)**	
Betaine	1.6	Citrate	0.8	Saponin	0.5
Potassium	1.2	Malate	0.2	Proteins	0.5
Glutamine (D)	1.2	Sulphate	0.1	Oxalate	0.4
Invert sugar (D)	0.6			Pectin	0.3
Amino acids*	0.4			Phosphate	0.2
Raffinose	0.4			Magnesium	0.1
Nitrate	0.3			Calcium	0.05
Sodium	0.2				
Lactate	0.1				
Inositol	0.1				
Galactinol	0.1				
Chloride	0.1				
Araban	0.05				
Nucleosides	0.05				
Total	**6.4**		**1.1**		**2.05**

Annex 18 : Comparison of relevant sample preparation methods for PMG and AMPA determination in water, plant material and soil.

Method	Sample	Extraction	Centrifugation	Intermediary clean-up / concentration	Derivatization	Final Clean-up / concentration
USGS method 5-A10	10 mL water	none	none	none	500 µL $Na_2B_4O_7$ 50 g/L + 1,500 µL FMOC-Cl 2.5 mM in ACN at 40°C for 24h, stopped with 600 µL H_3PO_4 2.5 mM	2x dilution (total 11 mL) SPE (Oasis HLB)
Guo et al. (2019)	150 µL water	none	none	filtration on 0.45 µm syringe filter	none	addition of 100 µL 0.1% FA
LIB 4596 (FDA)	2 g soybean or corn	10 min agitation with 10 mL 10 mM Na_2EDTA and 50 mM HAc	3,000 g, 5 min	none	none	SPE (Oasis HLB)
QuPPE-PO (EURL-SRM)	10 g fruit or vegetable	15 min agitation with 10 mL formic acid 1 % (v/v) in MeOH, then cooling to -18 °C	≥ 3,000 g, temperature as cold as possible	filtration of 2–3 mL in 0.2 µm or 0.45 + 0.2 µm syringe filter	none	none
ISP-AGES	1.00 g fruit or vegetable[a]	1 min Ultra Turrax with 10 mL water + 10 mL MeOH + 5 mL DCM	4,000 rpm, 20 min, 4°C	evaporation of 10 mL aqueous extract to dryness at 55 °C (3h), redissolve in 1 mL water and sonication 5 min	1 mL H_3BO_3 31 mg/mL, 37.5 mg/mL KCl and adjusted to pH 9 with NaOH 1M + 1 mL FMOC-Cl 20 mg/mL in ACN for 4 h and stopped with 2 mL DCM.	Centrifugation 10 min at 4,000 rpm and filtration of 500 µL supernatant optional : SPE (STRATA X, wash with 3 ml MeOH 5% and elute with 10 ml de MeOH, evaporation to dryness and re-dissolution in 1 mL water.
Liao et al. (2018)	5 g fruit or vegetable[b]	20 min shaking with 20 mL 0.1 % FA + 20 mL DCM	3,000 rpm, 20 min, 4°C	none	4 mL of aqueous extract + 4 mL $Na_2B_4O_7$ 0.05 mM+ 4 mL FMOC-Cl 20 mg/mL in ACN, left overnight and stopped with 3 drops HCl 37 % and pH adjustment to 1.5.	SPE (C18[c]) conditioned with 3 mL methanol, then 0.1 % FA and washed with 3 mL FA 0.1 % and 3 mL DCM. Evaporation of the eluate to dryness at 50°C and re-dissolution in 200 µL of a mixture of phases A and B (90/10 v/v).
EPA 43265-06-S	20 g soil	90 min shaking with 80 mL NH_4OH 0.25 M and KH_2PO_4	2,000 rpm, 20 min	filtration on 0.45 µm syringe filter	1 mL of trifluoroacetic anhydride/ heptafluorobutanol 2:1	none
Ibanez et al. (2005), Botero-Coy et al. (2013) and Sun et al. (2019)	1–5–10 g dried soil	30–60 min shaking with 5–10 mL 0.6 M KOH	2755–3500 g 10–30 min	adjustment to pH 9 with HCl 6M and 0.6 M, SPE (Oasis HLB)	120 µl of $Na_2B_4O_7$ 50 g/L + 120 µL FMOC-Cl agitated overnight	filtration 0.45 µm nylon syringe filters
Sun et al. (2017)	10 g fresh soil	30 min sonication with 50 mL 0.03 M Na_3PO_4 and 0.01 M Na_3CITR	10,000 rpm, 5 min	adjustment to pH 9, 10 min, filtration, 2x washing with 50 mL n-hexane	120 µl $Na_2B_4O_7$ 0.05 M + 200 µl FMOC-Cl in ACN 1.0 mg/mL for 4 h.	filtration 0.22 µm syringe filter

Ac : acétate; ACN : acetonitrile; CITR : citrate; DCM : dichloromethane; FA : formic acid; FLD : fluorescence detector; HAc : acetic acid; MeOH : methanol
Water is the default solvent if not mentioned otherwise, room temperature is the default temperature if not mentioned otherwise.
a : high water content (> 80 %)
b : high water content (> 60 %)
c : All the SPE considered are performed on 60 mg cartidges.

Annex 19 : Comparison of relevant LC separation methods for PMG and AMPA determination in water, plant material and soil.

Method	System	Column	Temperature (°C)	Injection volume (µL)	Flow rate (mL min^{-1})	mobile phases	Gradients (%B)	Detector
USGS method 5-A10	Agilent model 1100 Series 2	Luna C18 (3 µm, 150 × 3.0 mm)	45	not mentioned	0.350	A : NH_4Ac 5 mM B : ACN	0–5 min, 20 %; 11.5 min, 60 %; 11.51–14 min, 100 %; 14.01–17 min, 15 %	ESI-MS/MS
Guo et al. (2019)	Prominence™ 20A HPLC	Acclaim Trinity Q1 (3 µm, 100 × 3 mm)	40	20	0.5	A : 0.1 % FA B : 0.1%FA in ACN	0–2.0 min, 20%; 2.0–2.5 min, 20%–0% B; 2.5–7.5 min,0%; 7.5–8.0 min, 0%–20%; followed by a 3.5 min washing step at20% B; then a 2 min equilibrium step with the initial conditions	MS/MS
LIB 4596 (FDA)	Shimadzu HPLC	Acclaim™ Trinity™ Q1 (3 µm, 100 × 3 mm)	35	10	0.5	50 mM AF, pH 2.9	isocratic	ESI-MS/MS
QuPPE-PO method 1.5 (EURL-SRM)	not mentioned	Acclaim™ Trinity™ Q1 (3 µm, 100 × 2.1 mm)	30	10	0.5	A : 50 mM AF (pH 2.9) in H_2O:ACN 6:4 (v/v); B : ACN	0–10 min, 0 %; 10.1–13 min, 90 %; 13.1–18, 0 %.	ESI-MS/MS
QuPPE-PO method 1.6 (EURL-SRM)	not mentioned	Torus™DEA (1.7 µm, 2.1 × 100 mm)	50	10	0.5	A : 1.2 % FA B : 0.5 % FA in ACN	0–0.5 min, 90 %; 1.5 min, 20 %; 4.5–17.5 min, 10 %; 17.6–23 min, 90 %	ESI-MS/MS
ISP-AGES	Acquity UPLC	Acquity UPLC BEH C18 (1.7µm, 2.1 × 100mm)	45	5	0.45	A : MeOH 10 % (v/v) B : MeOH 90 % (v/v)	0 min, 0.1 %; 8 min, 99.9 %; 8.1–10 min, 0.1 %	ESI-MS/MS
Liao et al. (2018)	Dionex UltiMate 3000	Atlantic C18 (2.6 µm, 150 × 2.1 mm)	not mentioned	5	0.2	A : ACN B : NH_4Ac 0.05 mM, pH 5	0–2.5 min, 95 %; 8.0 min, 95 %; 10.0 min, 95 %; and 10.1 min, 5 %	ESI-MS/MS
Ibanez et al. (2005)	Waters Alliance 2695	Discovery C18 (5 µm, 50 × 2.0 mm)	not mentioned	50	not mentioned	A : 5 mM HAc/NH_4Ac, pH 4.8 B : Water with FA (pH 2.5)	0–5 min, 10 %; 5.1 min, 90 %; 9 min, 90 %; 9.1 min, 10 %	ESI-MS/MS
Botero-Coy et al. (2013)	Acquity UPLC	Discovery C18 (5 µm, 50 × 2.0 mm)	40	20	0.3	A : 5 mM HAc/NH_4Ac, pH 4.8 B : ACN	0–2.5 min, 10 %; 2.6 min, 90 %; 4.6 min, 90 %; 4.7 min, 10%; 10 min, 10 %	ESI-MS/MS
Sun et al. (2019)	not mentioned	Acclaim 120 C18 (2.1 × 250 mm)	not mentioned	not mentioned	0.35	A : ACN B : 5 mM HAc/NH_4Ac	0 min, 80 %; 6 min, 60 %; 9 min, 25 %; 10.2 min, 0 %; 12 min, 0%; 12.1 min, 80 %; 14 min, 80%	ESI-MS
Sun et al. (2017)	HPLC, Waters	AR-C18	35	20	1.0	A : 5 mM HAc/NH_4Ac, pH 4.8 B : Water with FA (pH 2.5)	0 min, 35 %; 10 min, 25 %; 15 min, 80 %; 20 min, 35 %	FLD
Waters	ACQUITY UPLC I-Class PLUS	Anionic Polar Pesticide (5 µm, 2.1 × 100 mm)	50	10	0.5	A : 0.9 % FA B : 0.9 % FA in ACN	0 min, 90 %; 4–12 min, 15%; 18 min, 90 %	ESI-MS/MS

Ac : acetate; ACN : acetonitrile; AF : ammonium formate; CITR : citrate; DCM : dichloromethane; FA : formic acid; FLD : fluorescence detector; HAc : acetic acid; MeOH : methanol; ESI : electro-spray ionization; MS : mass spectrometry

Water is the default solvent if not mentioned otherwise, room temperature is the default temperature if not mentioned otherwise.

Annex 20 : Fundamental theory of chromatography.

Performances of a chromatographic systems are usually indicated by its **efficiency** (i.e. the number of theoretical plates (N)) and **resolution** (R) (i.e. the capacity of the system to separate -or resolve- two given compounds).

The number of theoretical plates (N) is usually calculated by measuring retention time (t_r) and either base width (w) or half-height width ($w_{1/2}$) of the signal of standard compounds in isothermal conditions (equation a). A solution of 5% (v/v) acetone measured at 280 nm or a solution of 0.5 M NaCl measured with a conductometry detector are commonly used.

$$\text{(a)} \qquad N = 16\,(t_r / w)^2 = 5.545\,(t_r / w_{1/2})^2 = (t_r / \sigma)^2$$

t_r : retention time
w : width at the base of the peak (in time dimension)
σ : standard deviation of the distribution formed by the peak (in time dimension)

Width at half-height is equal to 2.354 standard deviations (σ) while base width is equal to 4 standard deviations (σ). The relation between base width (w) and half-height width ($w_{1/2}$) is given in equation b. **Base width** is defined as the segment (in time dimension) comprised between the two tangents of the curve at the two inflection points, which are at a height of 0.607 times the peak height from the baseline.

$$\text{(b)} \qquad w = 4\,\sigma = w_{1/2} \times 4 / 2.354$$

Resolution between two signals can be calculated from the retention times (t_r) and base width (w) of the signals (equation c). A resolution of 1.5 is conventionally required for quantitative analysis using peak areas.

$$\text{(c)} \qquad R = \frac{t_{rb} - t_{ra}}{(w_a + w_b) / 2}$$

Alternatively to equation c, resolution can be expressed as a function of efficiency (N), retention factor (or capacity factor) of the most retained of the two considered compounds (k_b) and relative retention factor (α) (equation d).

$$\text{(d)} \qquad R = \frac{\sqrt{N}}{4} + \frac{\alpha - 1}{\alpha} + \frac{k_b}{1 + k_b}$$

t_{ra} : retention time of the first eluted compound (a)
t_{rb} : retention time of the last eluted compound (b)
w_a : width at the base of the peak (in time dimension) of the less retained compound (a)
w_b : width at the base of the peak (in time dimension) of the most retained compound (b)
N : number of theoretical plates
α : relative retention factor between the two compounds a and b
k_b : retention factor (or capacity factor) of the last eluted compound (b)

Resolution is optimised through three main parameters :

- **Retention factor** or **capacity factor** (k) (equation e) describes how retained by the stationary phase a compound is. It is related It is equal to the adjusted retention time of the analyte divided by the elution time of an unretained compound. It is calculated from **adjusted retention time** (t_r'), which is equal to retention time (t_r) minus dead time (t_0) (equation f).

(e) $k = t_r' / t_m$

(f) $t_r' = (t_r - t_0) / t_0$

t_0 : dead time or holdup time

Dead time is defined as the time required for an unretained compound to go through the column. It can be measured by measuring retention time of unretained compounds such as uracil for reversed phase or hexane for normal phase, or by observing the time at which a baseline irregularity appears when injection solvent reaches some detectors such as absorbance or refractive index.

The retention factor mainly depends on temperature, pH and composition of the mobile phase, affecting its "strength" relatively to the stationary phase : the stronger the mobile phase, the lower is the retention factor.

> "When retention factors are very high or very low, the quality of the separation is reduced. Retention factors below 1, for any of the analytes, generally indicate that the separation will be poor. The largest gain in resolution is achieved when the k value is between 1 and 5. [...] [Retention factors] less than 1 are unreliable as analytes may be eluting with other sample components or solvent. Above a k value of approximately 5, increasing retention only provides minimal increases in resolution. Too much retention wastes valuable analysis time and the chromatographic peak height will decrease as the bandwidth of the peaks increases" (CHROMacademy[5]).

- **Selectivity**, **relative retention** or **separation factor** (α) (equation g) describes the distance between the modes of two signals. However, it does not consider the broadening of the peaks. Just like the retention factor, relative retention depends on the composition of the mobile phase and stationary phase, temperature and pH of the mobile phase. This last parameter can affect drastically the relative retention of ionised compounds such as PMG.

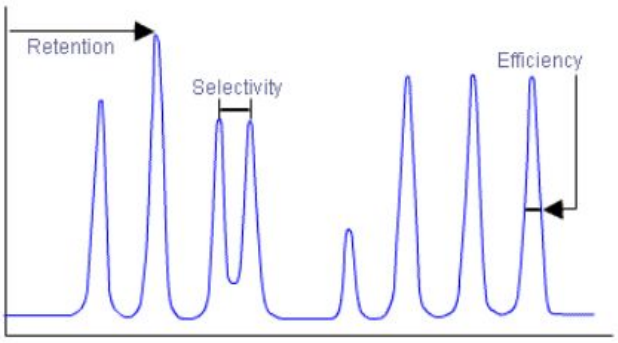
(g) $\alpha = t_{rb}' / t_{ra}' = k_b / k_a$

- **Efficiency (number of theoretical plates)** (N) (equation h) is directly related to the height of a theoretical plate (H) and the length of the column (L). The height of a theoretical plate is defined as the distance a compound has to travel in the column to reach equilibrium between stationary and mobile phase. More plates means better separation as more theoretical partitions occured. Due to pressure loss, the column length is limited by the maximum operating pressure of the system. The use of longer columns with higher pressure loss and thus lower linear velocity also results in significantly extended elution time. When length is doubled, analysis time is doubled but resolution only increases by $\sqrt{2}$ (≈ 1,414).

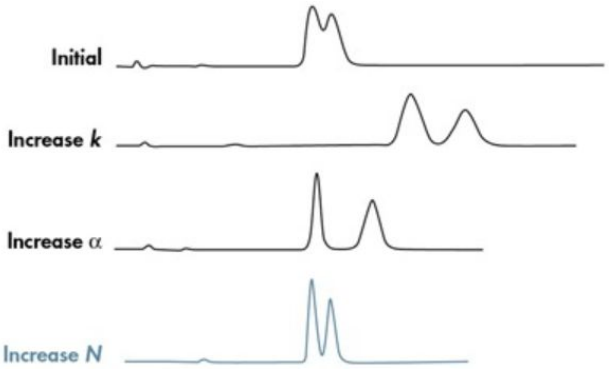
(h) $N = L / H$

Increasing any of the three parameters improves resolution differently (Annex 19a and 19b).

[5] https://www.chromacademy.com/lms/sco2/Theory_Of_HPLC_Chromatographic_Parameters.pdf (Consultation date : 03/02/20)



Annex 19a : Representation of retention, selectivity and efficiency on a chromatogram. Source : CHROMAcademy[6]



Annex 19b : Effect on a chromatogram of the individual modification of retention, selectivity and efficiency. Source : CHROMAcademy[7]

Increasing the retention factor (k) will also increases analysis time and peak broadening, leading to a loss of sensibility. Increasing the separation factor (α) may also increase or reduce analysis time and peak broadening for one or both considered compounds, as it is equal to the ratio of the retention factors of the considered compounds. Increasing efficiency (N) by reducing theoretical plate height (H) will reduce broadening of the peaks without changing mode-to-mode peak distance, while increasing efficiency (N) only by switching to a longer column will.

The height of a theoretical plate is a direct indicator of broadening per unit of time (or volume) required to elute a compound through the column. The **Van Deemter equation** (equation i) expresses the height of a theoretical plate as a function of linear velocity and three phenomena affecting broadening listed hereunder :

$$(i) \qquad H = A + B/u + u\,(C_s + C_m)$$

- **Linear velocity** (u) [m s^{-1}], which is equal to volumetric flow rate [m^3 s^{-1}] divided by column cross section area [m^2] (equation j).

$$(j) \qquad u = \frac{volumetric\ flow\ rate}{column\ cross\ section\ area} = \frac{volumetric\ flow\ rate}{\pi \times d_c^{\,2}/\,4}$$

d_c : internal column diameter

From equation 9, optimal linear velocity is then expressed in equation k.

$$(k) \qquad u_{opt} = \sqrt{B\,/\,C}$$

- **Eddy diffusion** (A) [m], describing the broadening of the peaks due to turbulence and eluent flowing through different pathways of variable lengths between the particles of a non-ideal packed bed. The more homogenous is the packing of the column, the lower is Eddy diffusion.

- **Longitudinal diffusion** (B) [m^2 s^{-1}], describing diffusion of the analytes in the longitudinal direction, outward from the center of its band. This broadening is alleviated as the flow rate increases.

- **Resistance to mass transfer** (C) [s], which can be split in two terms : stationary phase resistance to mass transfer (C_s) and mobile phase resistance to mass transfer (C_m). The first is related to the delay appearing between molecules diffusing inside the porous particles and molecules that keep flowing outside particles. The second term is related to the convective dispersion through the mobile phase.

[6] https://www.chromacademy.com/lms/sco2/Theory_Of_HPLC_Chromatographic_Parameters.pdf (Consultation date : 03/02/20)
[7] https://www.chromacademy.com/lms/sco2/Theory_Of_HPLC_Chromatographic_Parameters.pdf (Consultation date : 03/02/20)

Katz *et al.* (1983) proposed an adapted equation for liquid chromatography (equation l) :

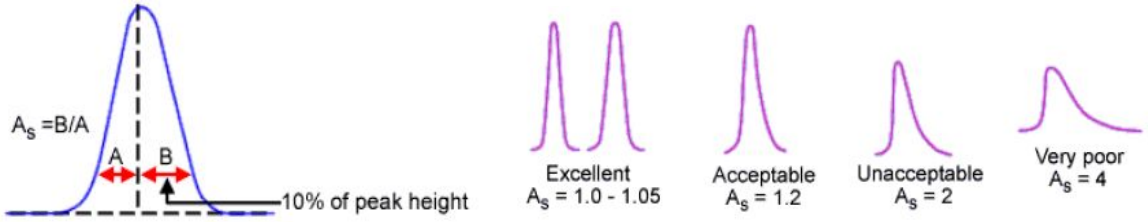
$$(l) \qquad H = 2\,\lambda\,d_p + \frac{2\gamma\,D_m}{u} + \frac{(0.37 + 4.69\,k + 4.04\,k^2)}{24\,(1+k)^2}\,\frac{u\,d_p^2}{D_m}$$

λ : tortuosity coefficient (around 0.5 (Katz *et al.* (1983)) [dimensionless] ;
D_m : mass diffusion coefficient (or mass diffusivity) [$m^2\ s^{-1}$] ;
u : linear velocity [$m\ s^{-1}$] ;
d_p : particles diameter [m] ;
k : capacity factor [dimensionless];
γ : obstructive coefficient related to diffusion restriction by column packing (around 0.8 (Katz *et al.* (1983)) [dimensionless].

Aside from flow rate and the column itself, several factors affect resolution :

- **Extra-column volume** : the sum of the volumes comprised between the injector and the column and between the column and the detector. This volumes includes the needle seat, tubing connectors, detector flow cells, preheaters, etc.
- **Peak asymmetry** (A_s) is measured as the ratio between the right and left segments (in time dimension) intercepted by the retention time of the mode of the peak and the signal curve at 10 % of the height of the peak (equation m) (Annex 19c).

$$(m) \qquad A_s = B / A$$



Annex 19c : asymmetry in chromatography
Source : CHROMacademy[8]

Asymmetry between 0.8 and 1.2 is acceptable. The peak is said **frontting** when asymmetry is under 0.8, and said **tailing** while above 1.2. Asymmetry can be caused by insufficient packing, overloading due to inappropriate sample concentration or volume, interaction of the solvent with the stationary phase etc.

Pore size should also be adapted to the compound analyzed.

[8] https://www.chromacademy.com/lms/sco2/Theory_Of_HPLC_Chromatographic_Parameters.pdf (Consultation date : 03/02/20)

Annex 21 : Definitions of Mass spectrometry.

Full scan MS : scan mode used for identification (qualitative analysis) in which the fragments of a selected m/z range are recorded.

Single ion monitoring (or Selected ion monitoring) (SIM) : scan mode used for quantification. Only the ions in a narrow m/z range around the theoretical m/z are recorded in order to include isotopes, allowing a greater sensitivity than full scan. Several analytes or internal standards are recorded in sequential SIM.

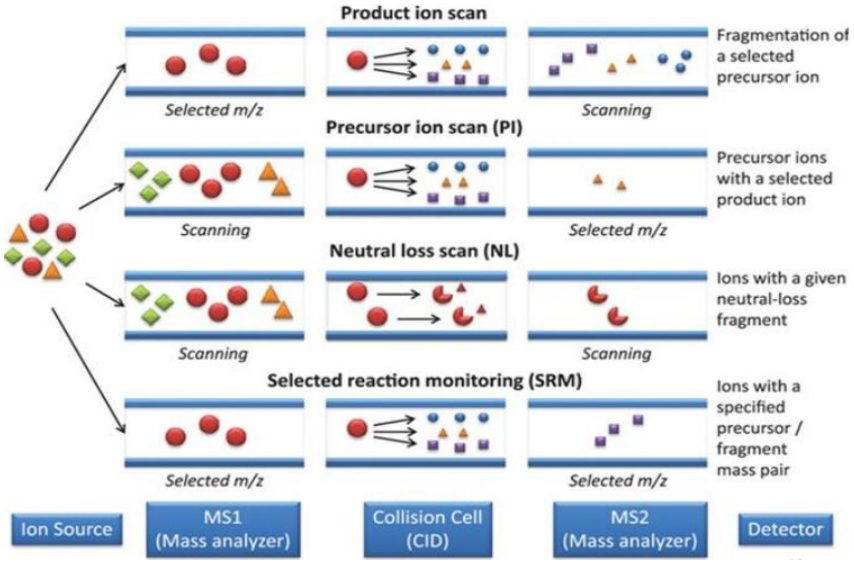
Product ion scan : acquisition mode in which the first mass analyzer is set on SIM mode and the second mass analyze is set on scan mode.

Precursor ion scan : acquisition mode in which the first mass analyzer is set on full scan and the second mass analyzer is set on SIM mode.

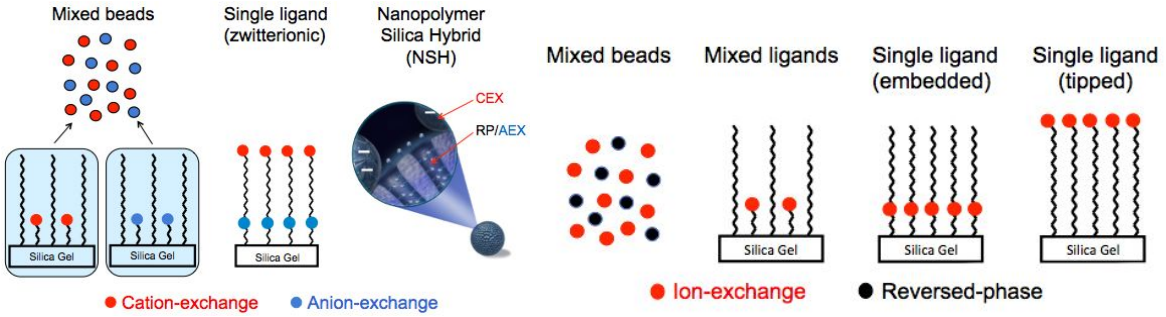
Neutral loss scan : acquisition mode in which both mass analyzers are set on full scan mode. Neutral loss scan is used for the screening of samples containing compounds with the same fragment loss. The second mass analyzer has an offset scan range compared to the first mass analyzer. This offset corresponds to the neutral fragment loss shared by the compounds of the same chemical class.

Selected (or Single) reaction monitoring (SRM) : acquisition mode in which both mass analyzer are set on SIM mode. A first fragment (precursor ion) is selected, fragmented anew in the collision cell, and one product ion is selected and monitored. This acquisition mode has the lowest background noise.

Multiple reaction monitoring : multiple simultaneous SRM.



Annex 20.a : Different MS/MS scan modes



Annex 22 : Examples of fixed mode stationary phase.
Source : CHROMacademy[9]

Annex 23 : Technical characteristics of the columns and precolumn used.

Characteristics	Anionic Polar Pesticide	Pre-column used with APP	BEH Phenyl
Name	Anionic Polar Pesticide (APP) Column	Anionic Polar Pesticide VanGuard Cartridge	ACQUITY UPLC BEH Phenyl Column
Reference number (SKU)	186009287	186009285	186002885
Chemistry	tri-functionnaly bonded DEA	tri-functionnaly bonded DEA	phenyl
Inner Diameter	2.1 mm	2.1 mm	2.1 mm
Length	100 mm	5 mm	100 mm
Separation Mode	HILIC	HILIC	Reversed phase
Particle Shape	Spherical	Spherical	Spherical
Particle Size	5 µm	5 µm	1.7 µm
Pore Size	130 Å	130 Å	130 Å
Surface area	185 m^2/g	185 m^2/g	185 m^2/g
Particle technology	BEH	BEH	BEH
pH Range	2–7	2–7	1–12

Annex 24 : Optimized transitions for PMG and AMPA detection in MS/MS.

Compound	Ionisation mode	Transition (m/z)	Collision energy optimum (V)	Capillary voltage optimum (kV)	Cone voltage optimum (V)
PMG	ESI negative	168 → 150	7–8	3.5 - 3.6	30–35
PMG	ESI negative	168 → 124	16	3.6	40–42
PMG	ESI negative	168 → 81	12–15	3.4 - 3.7	40
PMG	ESI negative	168 → 63	18–20	3.4–3.7	30
AMPA	ESI negative	110 → 81	11–13	3.6	30–31
AMPA	ESI negative	110 → 63	12–14	3.6	40
PMG	ESI positive	168 → 88	7–8	0.8–2	20–25
PMG	ESI positive	168 → 60	17–19	1.3	20–23
PMG	ESI positive	168 → 42	26	1.4	23
AMPA	ESI positive	111 → 30	6–8	1	20–26

[9] https://www.chromacademy.com/Mixed-Mode-Chromatography.html?tpm=1_1

Annex 25 : Calculated confidence intervals of the inverse prediction of PMG and AMPA concentrations in frozen sugar beet extract from peak area using various models.

Model	$CI_{\hat{x}}(\hat{x})$	
	PMG (mg/L)	**AMPA(mg/L)**
OLS, 0.1–10 mg/L PMG, untransformed data	$4.42 \times 10^{-3}\ \hat{x} + 5.13 \times 10^{-1}$	$4.48 \times 10^{-3}\ \hat{x} + 5.33 \times 10^{-1}$
WLS (w = 1/x), 0.1–10 mg/L PMG, untransformed data	$7.59 \times 10^{-2}\ \hat{x} + 1.81 \times 10^{-1}$	$7.31 \times 10^{-2}\ \hat{x} + 2.08 \times 10^{-1}$
WLS (w = $1/y^2$), 0.1–10 mg/L PMG, untransformed data	$3.21 \times 10^{-1}\ \hat{x} + 1.01 \times 10^{-2}$	$3.18 \times 10^{-1}\ \hat{x} + 5.77 \times 10^{-3}$
WLS (w = $1/s_y^2$), 0.1–10 mg/L PMG, untransformed data	$2.24 \times 10^{-1}\ \hat{x} + 5.27 \times 10^{-2}$	$6.63 \times 10^{-2}\ \hat{x} + 1.03 \times 10^{-2}$
OLS, 0.08–1 mg/L PMG, untransformed data	$2.25 \times 10^{-3}\ \hat{x} + 4.80 \times 10^{-2}$	$1.55 \times 10^{-3}\ \hat{x} + 3.00 \times 10^{-2}$

b_{upr} : slope of the upper limit of the confidence interval as a function of y
a_{upr} : intercept of the upper limit of the confidence interval as a function of y ;
b_{lwr} : slope of the lower limit of the confidence interval as a function of y ;
a_{lwr} : intercept of the lower limit of the confidence interval as a function of y .

Annex 26 : Intermediary values for the calculation of the confidence intervals of the inverse prediction.

Model	Compound	a_{reg}	b_{reg}	a_{upr}	b_{upr}	a_{lwr}	b_{lwr}
OLS, 0.1–10 mg/L PMG, untransformed data	PMG	9.495×10^{2}	2.828×10^{4}	8.207×10^{3}	2.834×10^{4}	-6.308×10^{3}	2.821×10^{4}
WLS (w = 1/x), 0.1–10 mg/L PMG, untransformed data	PMG	4.299×10^{2}	2.849×10^{4}	$2{,}471 \times 10^{3}$	2.975×10^{4}	-1.612×10^{3}	2.723×10^{4}
WLS (w = $1/y^2$), 0.1–10 mg/L PMG, untransformed data	PMG	3.437×10^{4}	3.011×10^{4}	1.478×10^{2}	3.482×10^{4}	-1.478×10^{2}	2.540×10^{4}
WLS (w = $1/s_y^2$), 0.1–10 mg/L PMG, untransformed data	PMG	3.093×10^{2}	2.952×10^{4}	1.290×10^{3}	3.252×10^{4}	-6.715×10^{2}	2.651×10^{4}
OLS, 0.08–1 mg/L PMG, untransformed data	PMG	2.054×10^{2}	1.150×10^{5}	2.963×10^{3}	1.151×10^{5}	-2.552×10^{3}	1.149×10^{5}
OLS, 0.1–10 mg/L PMG, untransformed data	AMPA	$5{,}537 \times 10^{1}$	$2{,}876 \times 10^{3}$	$8{,}215 \times 10^{2}$	$2{,}883 \times 10^{3}$	$-7{,}108 \times 10^{2}$	$2{,}870 \times 10^{3}$
WLS (w = 1/x), 0.1–10 mg/L PMG, untransformed data	AMPA	$3{,}789 \times 10^{1}$	$2{,}886 \times 10^{3}$	$2{,}372 \times 10^{2}$	$3{,}020 \times 10^{3}$	$-1{,}615 \times 10^{2}$	$2{,}751 \times 10^{3}$
WLS (w = $1/y^2$), 0.1–10 mg/L PMG, untransformed data	AMPA	$3{,}735 \times 10^{4}$	$3{,}042 \times 10^{3}$	$8{,}562 \times 10^{0}$	$3{,}514 \times 10^{3}$	$-8{,}561 \times 10^{0}$	$2{,}570 \times 10^{3}$
WLS (w = $1/s_y^2$), 0.1–10 mg/L PMG, untransformed data	AMPA	$2{,}317 \times 10^{1}$	$2{,}992 \times 10^{3}$	$1{,}465 \times 10^{2}$	$3{,}286 \times 10^{3}$	$-1{,}000 \times 10^{2}$	$2{,}698 \times 10^{3}$
OLS, 0.08–1 mg/L PMG, untransformed data	AMPA	$-1{,}320 \times 10^{1}$	$3{,}497 \times 10^{3}$	$3{,}934 \times 10^{1}$	$3{,}500 \times 10^{3}$	$-6{,}574 \times 10^{1}$	$3{,}494 \times 10^{3}$

b_{upr} : slope of the upper limit of the confidence interval as a function of y
a_{upr} : intercept of the upper limit of the confidence interval as a function of y ;
b_{lwr} : slope of the lower limit of the confidence interval as a function of y ;
a_{lwr} : intercept of the lower limit of the confidence interval as a function of y .

Annex 27 : Calculated LODs and LOQ for PMG and AMPA in Frozen sugar beet (OLS, untransformed data only) (mg/L extract)

Data origin	PMG (n = 3)			AMPA (n = 3)		
	Slope (0.08–1 mg/L)	LOD (mg/L)	LOQ (mg/L)	Slope (0.073–0.91 mg/L)	LOD (mg/L)	LOQ (mg/L)
Linearity assessment (0.1–10 mg/L)	2.83×10^4	0.03	0.08	2.88×10^3	0.02	0.05
Reproducibility (day 1)	1.15×10^5	0.003	0.009	3.49×10^3	0.006	0.02
Reproducibility (day 2)	9.91×10^4	0.01	0.03	3.86×10^3	0.01	0.03
Reproducibility (day 3)	1.01×10^5	0.006	0.02	2.67×10^3	0.2	0.6
Reproducibility (day 4)	1.09×10^5	0.01	0.03	2.70×10^3	0.02	005
Reproducibility (day 5)	1.17×10^5	0.01	0.03	2.62×10^3	0.01	0.03