

Towards Single System for Total Water Analysis. Screening of 300 pesticides in drinking Water

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1. Overview

A very promising start for a total water analysis platform with automatic, rapid and smooth switching of methods.

2. Introduction

The objective of this development is to set up a method of pesticides analysis which combines directly (without human intervention) with the method of PPCPs analysis in water. This method follows the European directive on the quality of water intended for human consumption. It also meets the WHO guideline values for chemicals from agricultural activities having an impact on health in drinking water.

In order to meet these regulations, this quantification method for pesticides need to reach a minimum limit of quantification (LOQ) of 30 ng/L with a maximum of 30% accuracy in order to target the waters with more than 100ng/L of pesticides. .

3. Methods

3-1. Calibration standards and controls

Commercially mix of pesticides were used for this experiment. Some standards were present in 1 commercial solution, 2, 3 or 4. Standards were diluted in (80/20) water/acetonitrile and added in distilled water to setting up the calibration curve. The range was between 0.5 and 500ng/L for standards present in only 1 solution. One sample of bottle water and one of tap water were spiked at 10, 40 and 200ng/L. 3 of each were prepared.

3-2. Analytical conditions



UHPLC conditions: Nexera X3 (LC40) system

Column	Shim-pack Velox Biphenyl 2.7µm 2.1 x 100mm
Mobile phases	Water/methanol + ammonium fluoride 0.15 mM + acetic acid 0.025%
Flow rate	0.4 mL/min
Column temperature	30°C
Injection vol	100 µL

MS conditions: LCMS-8060NX

Ionization: DIUS source Positive/Negative MRM mode

Nebulizing gas	3 L/min
Heating Gas	17 L/min
Interface temperature	400°C
Desolvation temperature	650°C
Heat block temperature	250°C
Drying gas flow	3 L/min

4. Results

4-1. Limit of quantification and ion ratio

Limit of quantification: 1ng/L					
Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio
5-Hydroxythiabendazole	QT 217.75>193.00; RT 217.75>147.00; IR 173.17	Cloransulfam	QT 430.00>369.80; RT 430.00>152.90; IR 64.11	Halosulfuron-methyl	QT 435.00>181.95; RT 435.00>83.05; IR 18.25
Acetoph	QT 184.00>143.00; RT 184.00>49.15; IR 25.3	Dimethametryn	QT 256.20>186.00; RT 256.20>68.05; IR 16.62	Iodosulfuron-methyl-sodium	QT 508.00>167.00; RT 508.00>56.05; IR 9.64
Aldicarb sulfone	QT 240.10>86.20; RT 240.10>148.15; IR 82.22	Dimethimrol	QT 210.20>140.10; RT 210.20>98.05; IR 88.24	Isouron	QT 212.05>167.05; RT 212.05>72.15; IR 157.26
Aminocarb	QT 209.00>152.05; RT 209.00>137.05; IR 125.9	Dimethomorph	QT 388.10>301.00; RT 388.10>165.10; IR 53.39	Metominostrobin	QT 285.10>193.95; RT 285.10>195.95; IR 109.78
Amirtraz	QT 293.90>211.05; RT ND; IR ND	Endosulfan	QT 405.80>251.00; RT 407.80>253.00; IR 65.03	Metosulam	QT 418.00>174.90; RT 420.00>177.00; IR 78.73
Azamethiphos	QT 325.00>112.10; RT 325.00>182.90; IR 160.11	Ethametsulfuron-methyl	QT 411.10>196.10; RT 411.10>168.05; IR 78.59	Metsulfuron-methyl	QT 382.00>166.90; RT 382.00>77.10; IR 9.38
Azimsulfuron	QT 425.10>182.10; RT 425.10>139.15; IR 21.04	Etovazole	QT 360.10>141.10; RT 360.10>113.05; IR 37.05	Omethoate	QT 214.10>125.00; RT 214.10>183.00; IR 85.73
Benalaxyl	QT 326.20>148.10; RT 326.20>208.10; IR 49.47	Fenamiphos	QT 304.10>216.95; RT 304.10>201.95; IR 44.98	Oxyacboxin	QT 268.10>175.00; RT 268.10>43.00; IR 21.66
Butafenacil	QT 492.10>330.85; RT 492.10>179.90; IR 90.4	Flamprop-methyl	QT 336.10>105.15; RT 336.10>77.05; IR 55.21	Oxyfluorfen	QT 362.15>330.10; RT 362.15>256.00; IR 25.04
Butocarbinoxim sulfioxide	QT 207.10>75.10; RT 207.10>132.10; IR 85.56	Flufenpyr-ethyl	QT 408.85>186.00; RT 408.85>206.05; IR 44.46	Pencycuron	QT 329.10>325.00; RT 331.10>127.05; IR 31.56
Carbetamide	QT 236.90>192.10; RT 236.90>118.15; IR 82.78	Flumetsulam	QT 325.90>129.00; RT 325.90>109.05; IR 15.42	Penoxsulam	QT 484.00>195.10; RT 484.00>194.05; IR 26.66
Chloridazon	QT 222.10>104.10; RT 222.10>92.15; IR 72.68	Flumioxazin	QT 371.90>158.90; RT 371.90>70.15; IR 49.16	Picolinafen	QT 377.00>238.00; RT 377.00>145.05; IR 34.43
Chlorimuron-ethyl	QT 415.10>185.90; RT 415.10>185.00; IR 40.43	Foramsulfuron	QT 453.10>182.00; RT 453.10>139.00; IR 31.51	Piperophos	QT 354.10>171.00; RT 354.10>255.00; IR 61.14
Chlorsulfuron	QT 358.00>141.15; RT 358.00>167.00; IR 104.16	Forchlorfenuron	QT 248.10>129.15; RT 248.10>137.15; IR 38.88	Pretilachlor	QT 312.20>252.05; RT 312.20>176.00; IR 29.38

Limit of quantification: 5ng/L					
Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio
(2)-Metominostrobin	QT 285.10>193.95; RT 285.10>195.95; IR 91.75	Diphenamide	QT 239.90>134.15; RT 239.90>165.10; IR 12.85	Isoxathion	QT 314.00>105.15; RT 314.00>97.05; IR 48.9
1-Naphthaleneacetamide	QT 186.10>141.15; RT 186.10>115.15; IR 45.99	Disulfoton sulfone	QT 307.00>97.00; RT 307.00>125.05; IR 54.91	Lactofen	QT 479.10>343.90; RT 479.10>222.90; IR 38.71
2,6-Dichlorobenzamide	QT 190.00>173.00; RT 190.00>145.00; IR 44.7	Diuron	QT 233.00>72.10; RT 234.80>72.10; IR 79.29	Mecarbam	QT 330.00>227.00; RT 330.00>198.90; IR 55.77
3,5-Dimethylphenyl Methylcarbamate	QT 180.10>123.10; RT 180.10>108.00; IR 29.31	Edifenphos	QT 311.00>282.95; RT 311.00>111.05; IR 101.07	Mefenacet	QT 299.00>148.15; RT 299.00>120.15; IR 95.37
Acetochlor	QT 270.05>119.05; RT 270.05>228.05; IR 45.96	Esprocarb	QT 266.20>91.05; RT 266.20>71.10; IR 43.17	Mefenpyr-diethyl	QT 373.10>326.90; RT 373.10>159.95; RT 27.35
Azaconazole	QT 300.00>159.00; RT 300.00>230.85; IR 41.8	Ethion	QT 385.00>143.00; RT 385.00>198.90; IR 101.34	Mepanipirim metaboli	QT 244.20>200.05; RT ND; IR
Azoxystrobin	QT 404.00>328.95; RT 404.00>343.95; IR 87.07	Ethoxysulfuron	QT 399.10>261.00; RT 399.10>217.90; IR 73.74	Mepronil	QT 270.20>119.05; RT 270.20>228.00; IR 54.92
Bensulfuron-methyl	QT 411.10>149.15; RT 411.10>181.90; IR 52.95	Etobenzazid	QT 339.95>149.00; RT 339.95>121.10; IR 128.24	Mesosulfuron-methyl	QT 503.80>182.10; RT 503.80>139.00; IR 26.58
Bupirimate	QT 317.20>166.00; RT 317.20>108.00; IR 52.14	Etofenprox	QT 394.20>177.05; RT 394.20>359.05; IR 71.36	Metalaxyl	QT 280.10>220.00; RT 280.10>192.05; IR 74.7
Carbendazim	QT 192.10>160.15; RT 192.10>105.15; IR 11.58	Fenarimol	QT 331.00>268.00; RT 331.00>139.10; IR 48.09	Methomyl	QT 163.00>87.90; RT 163.00>106.15; IR 62.49
Carbosulfan	QT 222.10>123.15; RT 222.10>165.00; IR 101.11	Fenhexamid	QT 302.10>97.10; RT 302.10>55.05; IR 63.42	Metolachlor	QT 284.10>252.05; RT 284.10>176.10; IR 38.14
Chlorfeniphos	QT 359.00>155.15; RT 359.00>169.95; IR 90.14	Fenoxycarb	QT 302.10>88.00; RT 302.10>116.15; IR 74.77	MPMC	QT 180.10>123.05; RT 180.10>108.05; IR 52.48
Chlormephos	QT 235.00>153.05; RT 235.00>136.00; IR 17.38	Ferimzone E	QT 255.20>132.15; RT 255.20>91.05; IR 97.09	Napraanilide	QT 292.00>171.10; RT 292.00>120.15; IR 67.62
Chlorobenzilate	QT 326.00>148.05; RT 326.00>208.15; IR 40.44	Ferimzone Z	QT 255.20>132.15; RT 255.20>124.00; IR 72.89	Nitenpyram	QT 271.10>237.00; RT 271.10>189.20; IR 109.21
Chloroxuron	QT 291.10>72.15; RT 291.10>46.15; IR 62.81	Flazasulfuron	QT 408.00>182.05; RT 408.00>139.00; IR 26.47	Nitralin	QT 363.10>288.90; RT 363.10>121.15; IR 15.59
Chlorthai-dimethyl	QT 330.90>180.95; RT 330.90>190.10; IR 55.58	Florasulam	QT 360.00>129.10; RT 360.00>109.05; IR 11.69	Oxadiazon	QT 345.10>302.90; RT 345.10>176.90; IR 15.15
Clomeprop	QT 324.10>120.05; RT 324.10>202.95; IR 74.61	Flufenacet	QT 364.10>152.05; RT 364.10>194.00; IR 74.22	Oxamyl	QT 237.10>72.10; RT 237.10>90.00; IR 33.39
Cloquintocet-mexyl	QT 335.90>191.85; RT 335.90>237.90; IR 159.59	Flusilazole	QT 316.10>247.00; RT 316.10>165.10; IR 70.66	Paclotrazol	QT 294.10>70.10; RT 296.10>70.10; IR 24.54
Clothianidin	QT 249.80>169.10; RT 249.80>132.05; IR 74.06	Fluthiacet-methyl	QT 404.00>274.00; RT 404.00>344.00; IR 78.91	Penconazole	QT 284.10>70.05; RT 376.00>158.95; IR 95.72
Cymuluron	QT 303.30>185.05; RT 303.30>125.00; IR 74.4	Flutolanil	QT 324.10>261.90; RT 324.10>281.90; IR 40.95	Phosalone	QT 356.20>174.05; RT 356.20>132.00; IR 32.78
Cyanazine	QT 240.80>214.00; RT 240.80>104.00; IR 18.34	Fosthiazate	QT 334.10>157.10; RT 334.10>290.00; IR 52.36	Phosphamidon	QT 376.00>307.95; RT 376.00>70.00; IR 32.79
Cyanofenphos	QT 304.10>275.95; RT 304.10>157.00; IR 111.09	Furametryp	QT 362.10>316.00; RT 362.10>91.20; IR 34.42	Piperonyl butoxide	QT 283.80>70.05; RT 283.80>158.95; IR 74.89
Cyclusulfamuron	QT 422.10>260.95; RT 422.10>217.90; IR 83.52	Haloxypol	QT 253.20>171.15; RT 253.20>71.05; IR 31.91	Prochloraz	QT 375.00>304.70; RT 375.00>346.75; IR 34.48
Daimuron	QT 269.20>151.00; RT 269.20>108.10; IR 23.25	Hexazinone	QT 289.20>144.00; RT 289.20>229.00; IR 57.11	Propamocarb	QT 189.20>102.15; RT 189.20>74.15; IR 32.9
Diclobutrazol	QT 328.00>70.10; RT 330.00>70.10; IR 58.58	Imazamethabenz-methyl	QT 312.10>199.00; RT 312.10>181.10; IR 71.81	Propaquizafop	QT 443.80>100.15; RT 443.80>371.00; IR 39.89
Diclosulam	QT 406.00>160.90; RT 406.00>377.90; IR 19.05	Imazaquin	QT 413.00>156.10; RT 413.00>257.80; IR 51.76	Propazine	QT 230.20>146.15; RT 230.20>188.10; IR 78.22
Dicrotophos	QT 237.90>72.00; RT 237.90>127.00; IR 87.86	Imazosulfuron	QT 256.10>209.00; RT 256.10>174.90; IR 79.44	Propiconazole	QT 342.00>165.10; RT 342.00>69.10; IR 49.25
Diflofencanarb	QT 268.20>226.05; RT 268.20>152.00; IR 68.04	Imidacloprid	QT 286.10>250.90; RT 286.10>188.00; IR 18.2	Propoxur	QT 209.90>93.10; RT 209.90>168.15; IR 82.07
Difenconazole	QT 406.10>250.90; RT 406.10>188.00; IR 18.2	Iprobofenaz	QT 395.10>265.90; RT 395.10>246.00; IR 15.32	Proflumuron	QT 419.90>141.00; RT 419.90>167.10; IR 87.7
Diflufenican	QT 395.10>265.90; RT 395.10>246.00; IR 15.32	Iprovalicarb	QT 275.80>244.00; RT 275.80>168.10; IR 27.42	Pymetrozine	QT 218.10>105.00; RT 218.10>78.10; IR 84.9
Dimethenamid	QT 275.80>244.00; RT 275.80>168.10; IR 27.42	Isazofos	QT 230.25>228.95; RT 230.25>201.00; IR 94.89	Pyraclofos	QT 361.10>256.90; RT 361.10>138.00; IR 61.33
Dimethoate	QT 230.00>198.90; RT 230.00>171.00; IR 37.69	Isofenphos Oxon	QT 326.10>70.05; RT 328.00>70.00; IR 46.13		
Diniconazole	QT 326.10>70.05; RT 328.00>70.00; IR 46.13	Isoxadifen-ethyl	QT 224.10>167.00; RT 224.10>123.00; IR 142.1		
Dioxacarb	QT 224.10>167.00; RT 224.10>123.00; IR 142.1				

Limit of quantification: 10ng/L					
Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio	Compounds	Transitions and Ion ratio
2,3,5-Trimethacarb	QT 193.75>122.10; RT 193.75>137.10; IR 116.94	Diazinon	QT 305.10>169.10; RT 305.10>96.95; IR 38.36	Inabenfide	QT 339.30>80.10; RT 339.30>321.00; IR 476.01
3,4,5-Trimethacarb	QT 194.10>137.10; RT 194.10>107.05; IR 30.8	Dichlorvos	QT 238.00>109.10; RT ND; IR	Indoxacarb	QT 527.70>203.00; RT 527.70>150.10; IR 69.69
Acetamiprid	QT 223.10>126.10; RT 223.10>55.95; IR 41.38	Diffubenzuron	QT 311.00>158.10; RT 311.00>141.05; IR 88.72	Isoprocarb	QT 194.10>95.00; RT 194.10>137.00; IR 16.22
Ametryn	QT 228.10>186.00; RT 228.10>68.00; IR 38.97	Epn	QT 324.10>156.95; RT 324.10>295.85; IR 122.39	Isoprothiolane	QT 290.80>231.00; RT 290.80>145.00; IR 41.38
Anilofos	QT 368.00>199.05; RT 368.00>170.90; IR 64.2	Epoxiconazol	QT 330.00>121.10; RT 330.00>101.10; IR 46.65	Lenacil	QT 234.90>153.15; RT 234.90>136.00; IR 81.33
Atrazine	QT 216.10>174.10; RT 216.10>104.05; IR 30.84	Ethylchlorazate	QT 238.85>165.00; RT 238.85>138.05; IR 11.3	Mepanipirim	QT 224.10>106.05; RT 224.10>104.10; IR 46.07
Benodiocarb	QT 224.10>109.00; RT 224.10>167.00; IR 77.17	Etrifimos	QT 293.10>265.00; RT 293.10>125.00; IR 116.37	Methabenzthiazuron	QT 222.10>165.10; RT 222.10>150.10; IR 40.43
Benzenefap	QT 430.80>105.10; RT 430.80>119.05; IR 41.48	Fenamidon	QT 312.10>236.00; RT 312.10>92.10; IR 89.46	Metrubuzin	QT 215.10>49.10; RT 215.10>187.10; IR 192.84
Bifenazate	QT 301.10>170.00; RT 301.10>152.10; IR 39.01	Fenbuconazole	QT 337.10>125.05; RT 337.10>70.10; IR 63.72	Mevinphos	QT 225.10>127.00; RT 225.10>193.00; IR 77.89
Boscalid	QT 343.00>306.95; RT 343.00>270.95; IR 37.66	Fenbuocarb	QT 208.10>95.00; RT 208.10>77.00; IR 22.56	Monocrotophos	QT 240.90>127.10; RT 240.90>193.00; IR 96.46
Bromaeol	QT 261.00>204.90; RT 261.00>188.00; IR 13.2	Fenoxypor-ethyl	QT 362.10>287.90; RT 362.10>91.05; IR 15.23	Momiduron	QT 215.10>148.80; RT 215.10>99.10; IR 95.68
Butamifos	QT 333.10>95.05; RT 333.10>179.95; IR 91.12	Fensulfuthion	QT 309.00>281.00; RT 309.00>157.05; IR 93.24	Myclobutanil	QT 289.10>126.00; RT 289.10>70.05; IR 52.72
Cadusafos	QT 270.80>131.00; RT				