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# Quinolone and Organophosphorus Insecticide Residues in Bivalves and Their Associated Risks in Taiwan

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## Supplementary Materials

**Table S1.** Recovery, repeatability, and limit of quantification of quinolones spiked into bivalve samples

| Analyte | Hard clam | | | Freshwater clam | | |
|---|---|---|---|---|---|---|
| | Spikedlevel (ng/g) | Recovery (%) | RSD (%) | Recovery (%) | RSD (%) | LOQ (ng/g) |
| Ciprofloxacin | 5 | 98.52 | 5.27 | 97.36 | 3.71 | 5 |
| | 25 | 99.89 | 6.72 | 98.59 | 5.32 | |
| Danofloxacin | 5 | 98.15 | 6.17 | 99.36 | 5.42 | 5 |
| | 25 | 99.47 | 7.96 | 99.58 | 8.35 | |
| Difloxacin | 5 | 96.38 | 8.32 | 97.21 | 7.41 | 5 |
| | 25 | 98.71 | 8.95 | 98.56 | 8.23 | |
| Enrofloxacin | 5 | 100.52 | 2.83 | 99.17 | 10.15 | 5 |
| | 25 | 102.57 | 3.58 | 100.46 | 9.81 | |
| Fleroxacin | 5 | 99.52 | 7.83 | 98.62 | 6.72 | 5 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | 25 | 100.65 | 6.56 | 99.86 | 8.29 | |
| Flumequine | 5 | 98.56 | 3.82 | 99.23 | 9.21 | 5 |
| | 25 | 99.16 | 5.61 | 100.72 | 8.53 | |
| Marbofloxacin | 5 | 96.35 | 8.57 | 95.47 | 9.36 | 5 |
| | 25 | 97.68 | 9.13 | 98.72 | 11.35 | |
| Sarafloxacin | 5 | 99.62 | 5.73 | 98.48 | 7.73 | 5 |
| | 25 | 100.76 | 9.25 | 99.39 | 8.39 | |

**Table S2.** Recovery, repeatability, and limit of quantification of organophosphorus insecticides spiked into bivalve samples

| Analyte | | Hard clam | | Freshwater clam | | |
|---|---|---|---|---|---|---|
| | Spikedlevel (ng/g) | Recovery (%) | RSD (%) | Recovery (%) | RSD (%) | LOQ (ng/g) |
| Fenamiphos | 5 | 98.52 | 5.27 | 97.36 | 3.71 | 5 |
| | 25 | 99.89 | 6.72 | 98.59 | 5.32 | |
| Fenthion | 5 | 98.15 | 6.17 | 99.36 | 5.42 | 5 |
| | 25 | 99.47 | 7.96 | 99.58 | 8.35 | |
| Methamidophos | 5 | 96.38 | 8.32 | 97.21 | 7.41 | 5 |
| | 25 | 98.71 | 8.95 | 98.56 | 8.23 | |
| Phoxim | 5 | 100.52 | 2.83 | 99.17 | 10.15 | 5 |
| | 25 | 102.57 | 3.58 | 100.46 | 9.81 | |
| Profenophos | 5 | 99.52 | 7.83 | 98.62 | 6.72 | 5 |
| | 25 | 100.65 | 6.56 | 99.86 | 8.29 | |
| Trichlorfon | 5 | 98.56 | 3.82 | 99.23 | 9.21 | 5 |
| | 25 | 99.16 | 5.61 | 100.72 | 8.53 | |
| Chlorpyrifos | 5 | 96.35 | 8.57 | 95.47 | 9.36 | 5 |
| | 25 | 97.68 | 9.13 | 98.72 | 11.35 | |
| Diazinon | 5 | 99.62 | 5.73 | 98.48 | 7.73 | 5 |
| | 25 | 100.76 | 9.25 | 99.39 | 8.39 | |
| Fenitrothion | 5 | 103.31 | 7.68 | 105.06 | 6.53 | 5 |
| | 25 | 107.26 | 5.37 | 108.37 | 8.16 | |
| Formothion | 5 | 105.72 | 6.51 | 102.61 | 5.32 | 5 |
| | 25 | 108.61 | 9.82 | 104.37 | 7.85 | |
| Malathion | 5 | 107.42 | 10.31 | 105.82 | 8.67 | 5 |
| | 25 | 113.56 | 12.67 | 108.35 | 10.23 | |
| Triazophos | 5 | 112.32 | 8.27 | 108.32 | 7.35 | 5 |
| | 25 | 114.53 | 7.36 | 110.48 | 9.18 | |

**Table S3.** MS/MS fragmentation conditions for LC-amenable 8 quinolones

| Analyte | Retention Time | Quantification ion (Collision energy) m/z (eV) | Identification ion (Collision energy) m/z (eV) |
|---|---|---|---|
| Ciprofloxacin | 9.66±0.02 | 332→314 (8) | 332→231 (11) |
| Danofloxacin | 10.37±0.02 | 358→340 (9) | 358→283 (18) |
| Difloxacin | 11.13±0.02 | 400→356 (10) | 400→299 (17) |
| Enrofloxacin | 10.21±0.02 | 360→316 (6) | 360→245 (8) |
| Fleroxacin | 7.98±0.02 | 370→326 (25) | 370→269 (19) |
| Flumequine | 15.93±0.02 | 262→244 (14) | 262→202 (14) |
| Marbofloxacin | 7.79±0.02 | 363→345 (17) | 363→72 (26) |
| Sarafloxacin | 11.78±0.02 | 386→368 (12) | 386→342 (16) |

**Table S4.** MS/MS fragmentation conditions for LC- or GC-amenable 12 organophosphorus insecticides

| Analyte | LC/GC | Retention Time | Quantification ion (Collision energy) m/z (eV) | Identification ion (Collision energy) m/z (eV) |
|---|---|---|---|---|
| Fenamiphos | LC | 10.31±0.02 | 304→202 (37) | 304→217 (24) |
| Fenthion | LC | 10.84±0.02 | 279→169 (20) | 279→247 (14) |
| Methamidophos | LC | 2.74±0.02 | 142→94 (14) | 142→125 (13) |
| Phoxim | LC | 10.99±0.02 | 299→129 (10) | 299→153 (10) |
| Profenophos | LC | 11.45±0.02 | 373→128 (55) | 373→303 (19) |
| Trichlorfon | LC | 4.34±0.02 | 257→79(28) | 257→109 (18) |
| Chlorpyrifos | GC | 10.45±0.02 | 314→258 (15) | 314→286 (5) |
| Diazinon | GC | 8.75±0.02 | 304→179 (15) | 304→162 (5) |
| Fenitrothion | GC | 10.16±0.02 | 277→109 (20) | 277→260 (5) |
| Formothion | GC | 9.33±0.02 | 224→125 (20) | 224→155 (10) |
| Malathion | GC | 10.26±0.02 | 173→127 (5) | 173→99 (15) |
| Triazophos | GC | 13.26±0.02 | 257→162 (5) | 257→119 (30) |

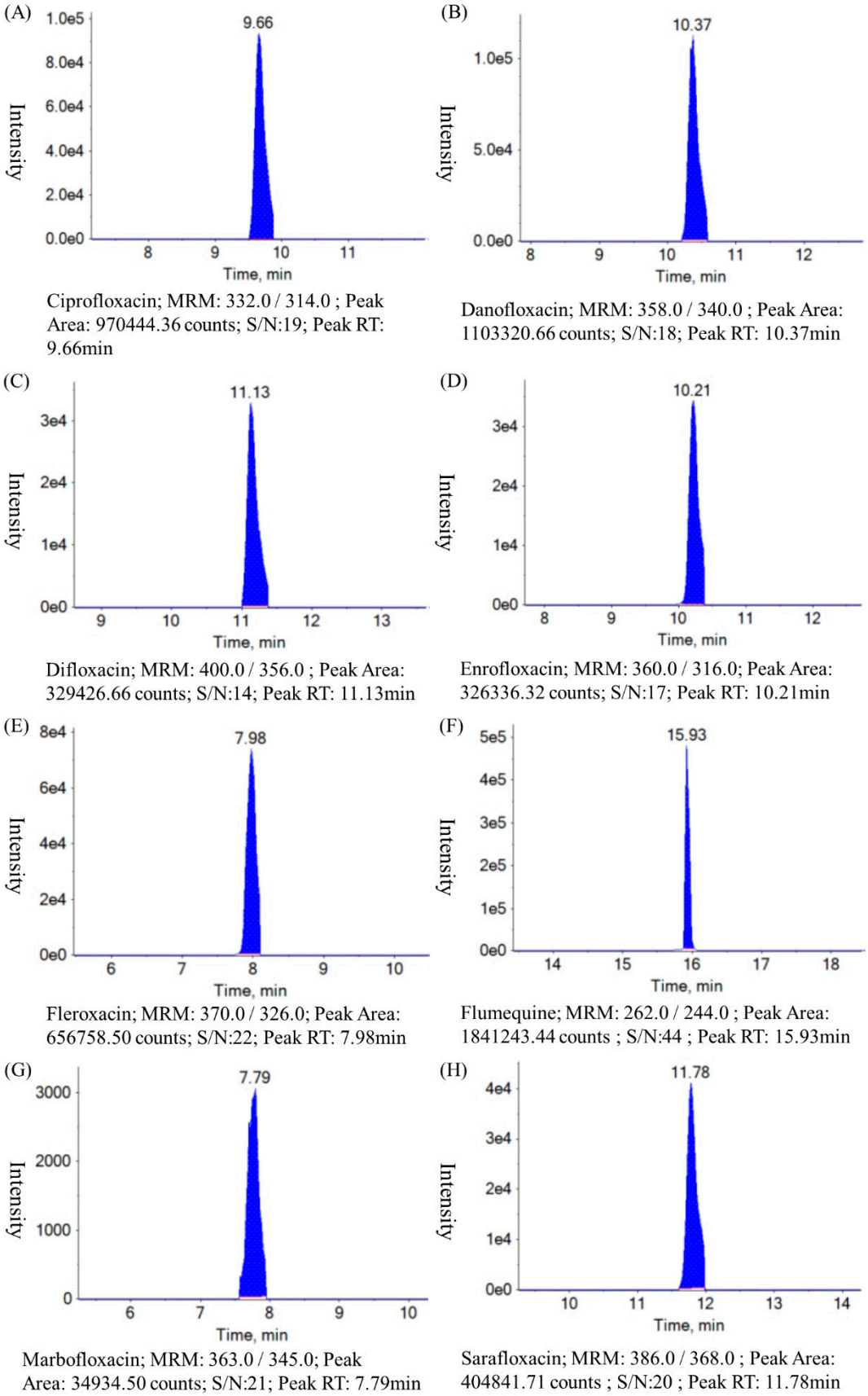


**Figure S1.** LC-MS/MS chromatogram of the detected quinolones at the quantification ion at LOQ (5 ng/g) in the bivalve samples.

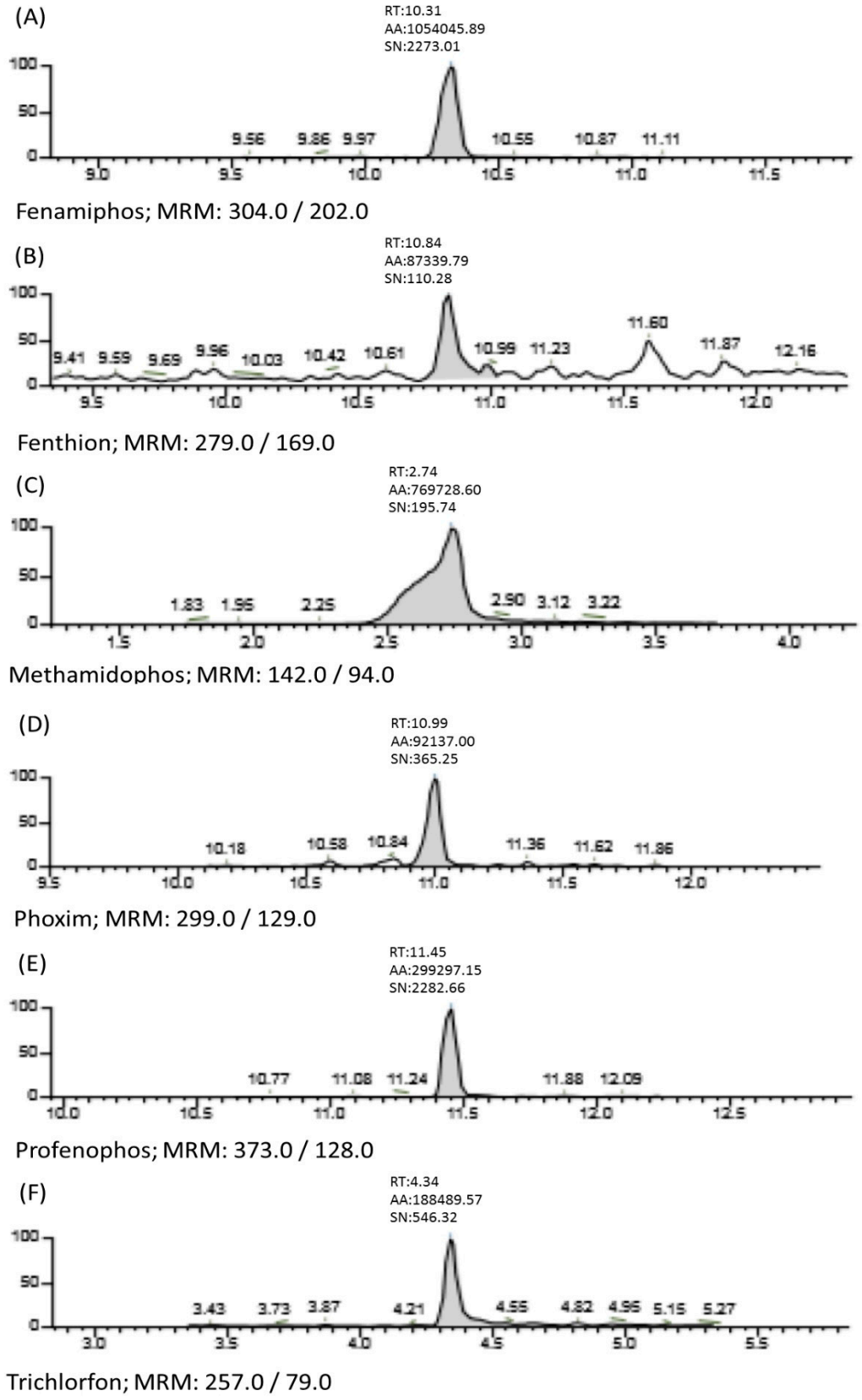


**Figure S2.** LC-MS/MS chromatograms of the detected organophosphorus insecticide at the quantification ion at LOQ (5 ng/g) in the bivalve samples.

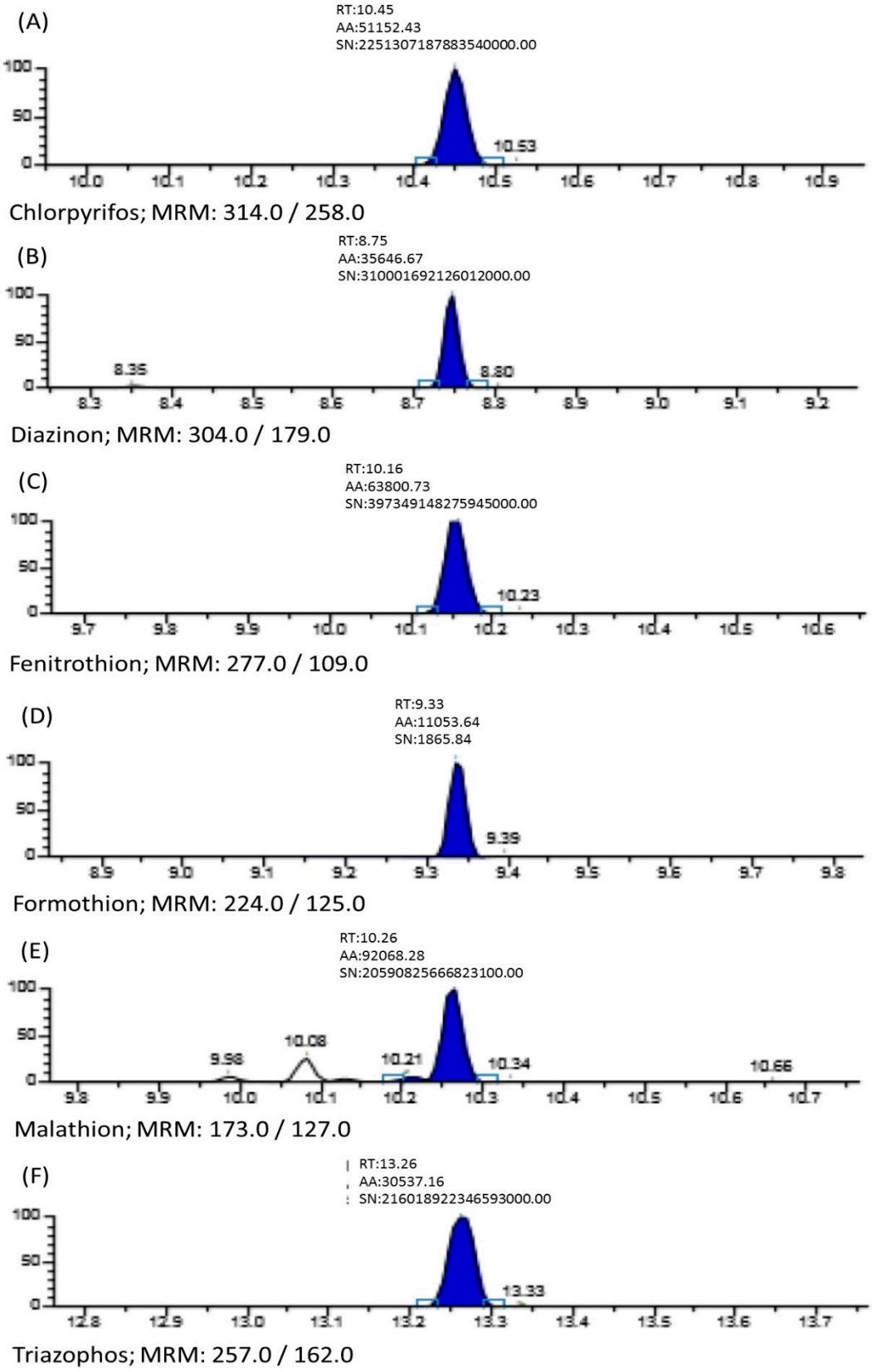


**Figure S3**. GC-MS/MS chromatograms of the detected organophosphorus insecticide at the quantification ion at LOQ (5 ng/g) in the bivalve samples.