

# Application Data Sheet

## No. 68

### GC-MS

Gas Chromatograph Mass Spectrometer

## GC-MS/MS Analysis of Pesticides in Drinking Water

According to Japan's list of drinking water quality control substances, pesticides are included as supplemental items subject to analysis. Designed to complement the standards, the Ministry of Health, Labour and Welfare encourages water utilities to monitor pesticide levels and achieve specified targets. Among the 102 listed pesticides, 84 are simultaneously analyzed using solid-phase extraction and GC-MS. In this datasheet, those pesticides were analyzed using GC-MS/MS and Multiple Reaction Monitoring (MRM) mode.

### Experimental

Analytical conditions are shown in Table 1.

Table 1: Analytical Conditions

|                   |  |                       |         |
|-------------------|--|-----------------------|---------|
| GC-MS             | :GCMS-TQ8030   |                       |         |
| Column            | :Rtx-5MS (Length 30 m, 0.25 mm I.D., df=0.25 μm)                                     |                       |         |
| Glass liner       | :Custom Sky Liner, Splitless Single Taper Gooseneck w/Wool (RESTEK, catalog# 567366) |                       |         |
| [GC]              |  | [MS]                  |         |
| Injection Temp.   | :250°C   | Interface Temp.       | :250 °C |
| Column Oven Temp. | :80°C(2 min)→(20°C /min)→180°C →(5°C /min)→280°C(3 min)                              | Ion Source Temp.      | :230 °C |
| Injection Mode    | :Splitless (High Pressure Injection 250 kPa, 2.3 min)                                | Data Acquisition Mode | :MRM    |
| Flow Control Mode | :Linear Velocity (44.5 cm/sec)   |                       |         |
| Injection Volume  | :2 μL  |                       |         |

#### MRM Monitoring *m/z*

| Compound Name         | Quantitative Transition |        | Qualitative Transition |        | Compound Name            | Quantitative Transition |        | Qualitative Transition |        |
|-----------------------|-------------------------|--------|------------------------|--------|--------------------------|-------------------------|--------|------------------------|--------|
|                       | Precursor>Product       | CE (V) | Precursor>Product      | CE (V) |                          | Precursor>Product       | CE (V) | Precursor>Product      | CE (V) |
| Dichlorvos            | 184.9>109.0             | 18     | 184.9>93.0             | 13     | Isofenphos               | 213.1>185.1             | 6      | 213.1>121.1            | 18     |
| Dichlobenil           | 170.9>136.0             | 13     | 170.9>100.0            | 23     | Captan                   | 149.0>105.1             | 5      | 149.0>79.0             | 19     |
| Etridiazole           | 210.9>182.9             | 10     | 210.9>139.9            | 20     | Dimepiperate             | 145.1>112.1             | 9      | 145.1>69.1             | 18     |
| Chloroneb             | 205.9>190.9             | 12     | 205.9>140.9            | 19     | Phenthoate               | 274.0>121.0             | 11     | 274.0>125.0            | 18     |
| Isoprocarb            | 136.1>121.1             | 9      | 136.1>103.1            | 23     | Procymidone              | 283.1>96.0              | 10     | 283.1>68.1             | 24     |
| Molinate              | 126.1>55.0              | 18     | 126.1>83.1             | 6      | Butamifos oxon           | 244.0>216.0             | 7      | 244.0>136.1            | 15     |
| Fenobucarb            | 150.1>121.1             | 9      | 150.1>103.1            | 23     | Methodathion             | 145.0>85.0              | 8      | 145.0>58.0             | 18     |
| Trifluralin           | 306.1>264.0             | 7      | 306.1>206.1            | 17     | 9-Bromoanthracene (ISTD) | 256.0>177.1             | 18     | 256.0>151.1            | 30     |
| Benfluralin           | 292.1>264.0             | 9      | 292.1>206.1            | 14     | alpha-Endosulfan         | 240.9>205.9             | 13     | 240.9>170.0            | 26     |
| Pencycuron            | 180.1>125.0             | 10     | 180.1>89.0             | 29     | Butamifos                | 286.1>202.1             | 17     | 286.1>185.0            | 27     |
| Dimethoate            | 125.0>79.0              | 10     | 125.0>62.0             | 8      | Napropamide              | 128.1>72.1              | 7      | 128.1>100.1            | 9      |
| Simazine              | 201.1>173.1             | 6      | 201.1>186.1            | 7      | Flutolanil               | 173.0>145.0             | 18     | 173.0>95.0             | 27     |
| Atrazine              | 215.2>200.1             | 8      | 215.2>173.1            | 6      | Isoxathion oxon          | 161.1>105.0             | 11     | 161.1>77.0             | 25     |
| Diazinon oxon         | 273.1>137.1             | 18     | 273.1>217.0            | 10     | Isoprothiolane           | 290.1>204.1             | 5      | 290.1>118.0            | 14     |
| Propyzamide           | 172.9>144.9             | 15     | 172.9>109.0            | 27     | Pretilachlor             | 238.1>162.2             | 11     | 238.1>146.2            | 10     |
| Pyroquilon            | 173.1>130.1             | 20     | 173.1>144.1            | 23     | Fenthion oxon sulfoxide  | 262.1>247.1             | 11     | 262.1>109.0            | 22     |
| Diazinon              | 304.1>179.2             | 10     | 304.1>162.1            | 9      | CNP-amino                | 287.0>108.1             | 19     | 287.0>217.0            | 13     |
| Anthracene-d10 (ISTD) | 188.2>160.1             | 20     | 188.2>158.1            | 30     | Fenthion oxon sulfone    | 294.1>104.1             | 19     | 294.1>230.2            | 8      |
| Disulfoton            | 274.1>88.0              | 6      | 274.1>60.0             | 22     | Buprofezin               | 172.1>57.1              | 18     | 172.1>131.1            | 6      |
| Chlorothalonil        | 265.9>230.9             | 19     | 265.9>169.9            | 23     | Isoxathion               | 312.9>177.0             | 7      | 312.9>130.0            | 17     |
| Iprobenfos            | 204.0>91.0              | 8      | 204.0>122.0            | 15     | beta-Endosulfan          | 240.9>205.9             | 18     | 240.9>170.0            | 23     |
| Tolclofos-methyl oxon | 249.0>199.0             | 26     | 249.0>233.9            | 15     | Fenthion sulfoxide       | 278.0>109.0             | 20     | 278.0>169.1            | 14     |
| Fenitrothion oxon     | 244.0>109.0             | 16     | 244.0>90.0             | 18     | Fenthion sulfone         | 310.0>109.0             | 24     | 310.0>105.1            | 16     |
| Bromobutide           | 232.2>176.1             | 10     | 232.2>114.1            | 9      | Mepronil                 | 269.1>119.1             | 18     | 269.1>227.1            | 5      |
| Terbucarb             | 205.2>177.1             | 8      | 205.2>145.1            | 18     | Chlornitrofen            | 318.9>288.9             | 12     | 318.9>238.0            | 10     |
| Malaoxon              | 127.1>99.0              | 7      | 127.1>109.0            | 10     | Edifenphos               | 310.0>173.0             | 13     | 310.0>109.1            | 25     |
| Simetryn              | 213.2>170.1             | 10     | 213.2>185.1            | 7      | Propiconazole-1          | 259.1>69.0              | 13     | 259.1>173.0            | 18     |
| Tolclofos-methyl      | 265.0>249.9             | 15     | 265.0>219.9            | 23     | Endosulfate              | 271.8>236.8             | 18     | 271.8>234.8            | 19     |
| Alachlor              | 188.1>160.1             | 10     | 188.1>131.1            | 22     | Propiconazole-2          | 259.0>69.0              | 11     | 259.0>172.9            | 19     |
| Metalaxyl             | 249.2>190.2             | 6      | 249.2>146.1            | 18     | EPN oxon                 | 141.0>77.0              | 18     | 141.0>51.0             | 30     |
| Fenthion oxon         | 262.0>247.0             | 8      | 262.0>109.0            | 26     | Thenylchlor              | 288.1>141.0             | 13     | 288.1>174.1            | 7      |
| Dithiopyr             | 354.1>306.0             | 7      | 354.1>286.0            | 17     | Pyributicarb             | 165.1>108.1             | 10     | 165.1>93.0             | 25     |
| Fenitrothion          | 277.0>260.1             | 7      | 277.0>109.0            | 20     | Iprodione                | 314.0>244.9             | 11     | 314.0>56.0             | 25     |
| Esprocarb             | 222.1>91.0              | 19     | 222.1>162.2            | 7      | Pyridaphenthion          | 340.0>199.1             | 8      | 340.0>109.0            | 22     |
| Malathion             | 173.1>127.1             | 7      | 173.1>99.0             | 18     | Chrysenes-d12 (ISTD)     | 240.2>236.1             | 30     | 240.2>238.2            | 20     |
| Thiobencarb           | 257.1>100.1             | 7      | 257.1>72.1             | 23     | EPN                      | 157.0>77.0              | 24     | 157.0>110.0            | 14     |
| Chlorpyrifos oxon     | 298.0>241.8             | 14     | 298.0>269.9            | 6      | Piperophos               | 320.2>122.1             | 10     | 320.2>81.0             | 26     |
| Fenthion              | 278.1>109.0             | 18     | 278.1>169.0            | 18     | Bifenox                  | 341.1>309.9             | 6      | 341.1>188.8            | 19     |
| Chlorpyrifos          | 314.0>257.9             | 19     | 314.0>285.9            | 7      | Anilofos                 | 226.1>184.0             | 5      | 226.1>157.0            | 13     |
| Isofenphos oxon       | 229.1>201.0             | 10     | 229.1>121.1            | 24     | Pyriproxyfen             | 136.1>78.0              | 20     | 136.1>96.0             | 14     |
| Phthalide             | 242.8>214.8             | 18     | 242.8>178.9            | 26     | Mefenacet                | 192.0>136.0             | 17     | 192.0>109.0            | 28     |
| Dimethametryn         | 212.1>122.1             | 13     | 212.1>94.0             | 22     | Cafenstrole              | 188.2>119.1             | 22     | 188.2>82.0             | 20     |
| Pendimethalin         | 252.1>162.1             | 11     | 252.1>191.1            | 8      | Etofenprox               | 163.1>135.1             | 10     | 163.1>107.1            | 19     |
| Methyldymron          | 107.1>106.1             | 13     | 107.1>77.0             | 25     |                          |                         |        |                        |        |

## Results

The standard sample mixture of 84 pesticides at the concentration of 5 µg/L was analyzed 5 times. The overlay mass chromatograms from 5 injections and the repeatability are shown in Fig. 1 and Table 2, respectively.

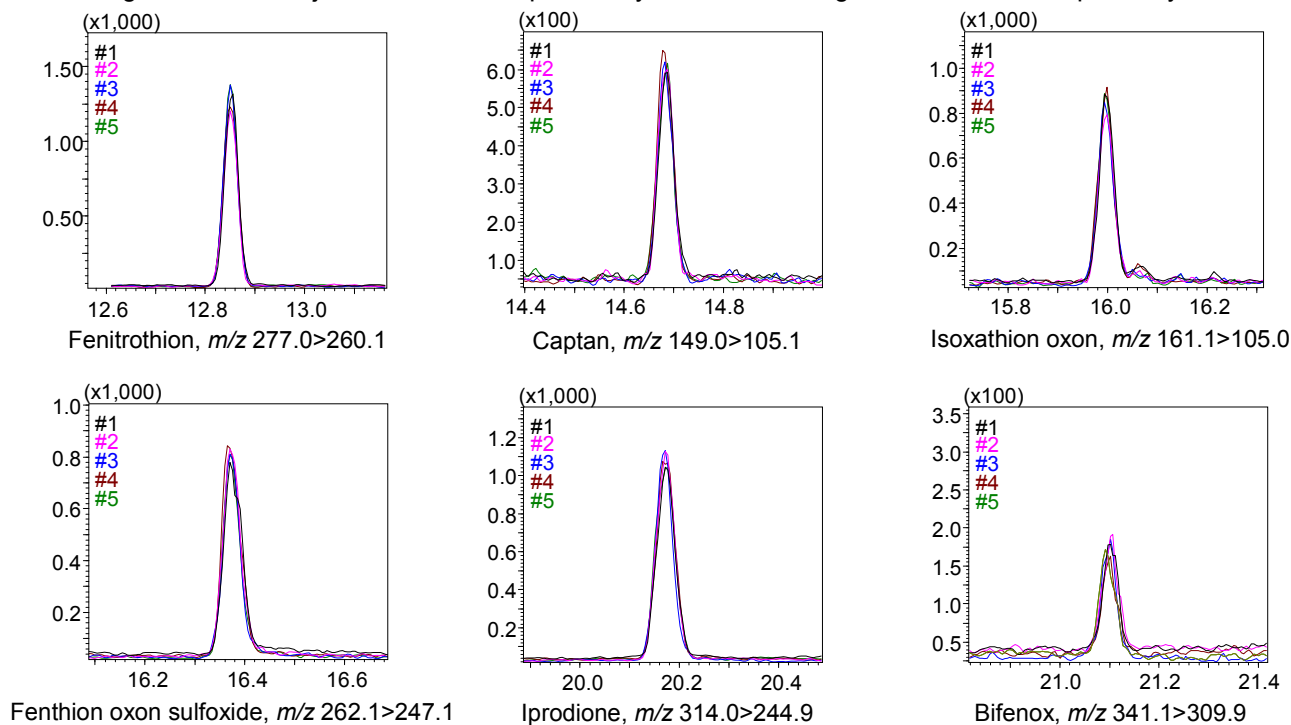


Fig. 1: Overlay mass chromatograms from 5 injections

Table 2: Repeatability (n=5, area ratio)

| Compound Name         | %RSD | Compound Name     | %RSD | Compound Name           | %RSD |
|-----------------------|------|-------------------|------|-------------------------|------|
| Dichlorvos            | 1.62 | Metalaxyl         | 3.01 | Pretilachlor            | 7.26 |
| Dichlobenil           | 0.73 | Fenthion oxon     | 1.88 | Fenthion oxon sulfoxide | 5.72 |
| Etridiazole           | 3.38 | Dithiopyr         | 0.66 | CNP-amino               | 1.04 |
| Chloroneb             | 0.89 | Fenitrothion      | 5.72 | Fenthion oxon sulfone   | 1.19 |
| Isoprocarb            | 0.47 | Esprocarb         | 1.30 | Buprofezin              | 2.14 |
| Molinate              | 1.25 | Malathion         | 0.82 | Isoxathion              | 8.25 |
| Fenobucarb            | 0.65 | Thiobencarb       | 2.83 | beta-Endosulfan         | 5.28 |
| Trifluralin           | 1.71 | Chlorpyrifos oxon | 4.14 | Fenthion sulfoxide      | 3.17 |
| Benfluralin           | 2.09 | Fenthion          | 1.17 | Fenthion sulfone        | 9.61 |
| Pencycuron            | 0.23 | Chlorpyrifos      | 2.40 | Mepronil                | 3.62 |
| Dimethoate            | 2.98 | Isofenphos oxon   | 2.03 | Chlornitrofen           | 1.82 |
| Simazine              | 1.17 | Phthalide         | 1.03 | Edifenphos              | 1.06 |
| Atrazine              | 3.51 | Dimethametryn     | 1.37 | Propiconazole-1         | 7.70 |
| Diazinon oxon         | 1.37 | Pendimethalin     | 3.38 | Endosulfate             | 2.98 |
| Propyzamide           | 1.39 | Methyldymron      | 2.29 | Propiconazole-2         | 5.75 |
| Pyroquilon            | 1.36 | Isofenphos        | 2.93 | EPN oxon                | 2.31 |
| Diazinon              | 3.15 | Captan            | 7.46 | Thenylchlor             | 5.43 |
| Disulfoton            | 3.37 | Dimepiperate      | 3.64 | Pyributicarb            | 0.88 |
| Chlorothalonil        | 1.57 | Phenthoate        | 2.65 | Iprodione               | 3.03 |
| Iprobenfos            | 1.29 | Procymidone       | 0.87 | Pyridaphenthion         | 3.78 |
| Tolclofos-methyl oxon | 1.56 | Butamifos oxon    | 4.28 | EPN                     | 2.85 |
| Fenitrothion oxon     | 3.75 | Methidathion      | 2.27 | Piperophos              | 5.48 |
| Bromobutide           | 4.98 | alpha-Endosulfan  | 1.78 | Bifenox                 | 7.02 |
| Terbucarb             | 1.08 | Butamifos         | 5.57 | Anilofos                | 2.48 |
| Malaoxon              | 2.64 | Napropamide       | 2.38 | Pyriproxyfen            | 2.39 |
| Simetryn              | 3.14 | Flutolanil        | 1.40 | Mefenacet               | 1.70 |
| Tolclofos-methyl      | 2.33 | Isoxathion oxon   | 2.71 | Cafenstrole             | 3.14 |
| Alachlor              | 1.12 | Isoprothiolane    | 4.96 | Etofenprox              | 1.10 |

First Edition: September, 2012



Shimadzu Corporation

www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

© Shimadzu Corporation, 2012