CORESTA RECOMMENDED METHOD N° 2

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES ON TOBACCO

(May 1997)

PREFACE

The ISO method for the determination of organochlorine pesticide residues (Tobacco and tobacco products - Determination of organochlorine pesticide residues ISO 4389:1981) needs to be replaced as a result of restrictions now in place in many countries on the use of some of the reagents used. In addition, the benefits of new chromatographic columns are available.

This Recommended Method has been compiled by the CORESTA Sub-Group on Pesticide Residues following a Joint Collaborative Study carried out in January 1994. The Sub-Group recommend that this method be submitted to ISO as a replacement for method 4389:1981.

1. SCOPE AND FIELD OF APPLICATION

The method is applicable to the determination of the organochlorine pesticides listed in Section 4.3 in leaf tobacco, manufactured tobacco and tobacco products.

The method has been demonstrated to be free from errors, that may arise from interfering peaks on the chromatogram that originate from nonorganochlorine pesticide substances, when applied to various styles of leaf tobacco. However, the preceding statement cannot be assumed to apply to all forms and variants of tobacco and tobacco product and care must be taken in the interpretation of any unexpected, apparently positive results. Some analysts advocate the use of mass spectrometric confirmation of the chemical structure of compounds detected by chromatography in such circumstances.

2. REFERENCES

3. PRINCIPLE

Dried and milled tobacco is mixed with Florisil and extracted with n-hexane in a special Soxhlet extractor. Pesticides are quantified by gas chromatography equipped with electron capture detector without any further clean-up [1].

4. REAGENTS

4.1 General

All the reagents shall be of analytical pesticide reagent quality (or equivalent). All solvents shall be checked for purity before use by carrying out a blank determination using exactly the same procedure (extraction and gas chromatography) as used for the determination of a sample. The chromatogram obtained from the solvents shall have a baseline without noticeable peaks that could interfere with those from the pesticide residues being determined.

The water used shall be double distilled water or water of a least equivalent purity.

4.2 n-Hexane

4.3 Pesticide standards

certified standards of ≥ 95% purity,

α-HCH, β-HCH, δ-HCH, γ-HCH (Lindane), heptachlor, heptachlor epoxide,
o,p-DDT, o,p-DDE, o,p-DDD, p,p-DDT, p,p-DDE, p,p-DDD,
trans chlordane, aldrin, dieldrin, HCB, α-endosulfan.

Note: trans chlordane is used as an indicator for chlordane (technical mixture).

Chemical names of pesticides according to IUPAC:

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>IUPAC Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCB</td>
<td>Hexachlorobenzene</td>
</tr>
<tr>
<td>Chlordane</td>
<td>1,2,4,5,6,7,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methanoindene</td>
</tr>
<tr>
<td>Heptachlor</td>
<td>1,4,5,6,7,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene</td>
</tr>
<tr>
<td>Heptachlor epoxide</td>
<td>1,4,5,6,7,8-heptachloro-2,3-epoxy-3a,4,7,7a-tetrahydro-4,7-methanoindene</td>
</tr>
<tr>
<td>Lindane</td>
<td>1,2,3,4,5,6-hexachlorocyclohexane</td>
</tr>
<tr>
<td>α-HCH</td>
<td>1,3,5/2,4,6-hexachlorocyclohexane</td>
</tr>
<tr>
<td>β-HCH</td>
<td>1,2,4,5/3,6-hexachlorocyclohexane</td>
</tr>
<tr>
<td>δ-HCH</td>
<td>1,2,3/4,5,6-hexachlorocyclohexane</td>
</tr>
<tr>
<td>α-Endosulfan</td>
<td>(1,4,5,6,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene) sulfite-1</td>
</tr>
<tr>
<td>Aldrin</td>
<td>(1R,4S,4aS,5R,6R,7S,8S,8aR)-1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4,5,8-dimethanonaphthalene</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>(1R,4S,4aS,5R,6R,7S,8S,8aR)-1,2,3,4,10,10-hexachloro-1,4,4a,5,6,7,8,8a-octahydro-6,7-epoxy-1,4,5,8- dimethanonaphthalene</td>
</tr>
<tr>
<td>o,p-DDT</td>
<td>1,1,1-trichloro-2- (2-chlorophenyl)-2-(4-chlorophenyl) ethane</td>
</tr>
<tr>
<td>p,p-DDT</td>
<td>1,1,1-trichloro-2,2-bis (4-chlorophenyl) ethane</td>
</tr>
<tr>
<td>o,p-DDD</td>
<td>1,1-dichloro-2- (2-chlorophenyl)-2-(4-chlorophenyl) ethane</td>
</tr>
<tr>
<td>p,p-DDD</td>
<td>1,1-dichloro-2,2-bis (4-chlorophenyl) ethane</td>
</tr>
<tr>
<td>o,p-DDE</td>
<td>1,1-dichloro-2- (2-chlorophenyl)-2-(4-chlorophenyl) ethylene</td>
</tr>
<tr>
<td>p,p-DDE</td>
<td>1,1-dichloro-2,2-bis (4-chlorophenyl) ethylene</td>
</tr>
</tbody>
</table>

4.4 Internal Standard
OBSOLETE - This method was set up and validated at a time when few multi-residue methods were available. Nowadays, more efficient and powerful multi-residue methods are being applied.

Mirex - this is an obsolete pesticide that has been superseded (The Pesticide Manual, 9th Edition, British Crop Protection Council).

Note: Mirex is a generic name for dodecachloropentacyclo [5.2.1.0^2.6.0^1.9.0^5.8] decane.

4.5 Toluene

4.6 Internal Standard Stock Solution

Weigh accurately approximately 0.02 g to a precision of ± 0.0001 g of mirex into a volumetric flask (100 cm^3 capacity) and dilute to volume with n-hexane.

4.6.1 Internal Standard Solution

Pipette an aliquot of the internal standard stock solution (5 cm^3) into a volumetric flask (200 cm^3 capacity) and dilute to volume with n-hexane. This will give a solution with a concentration of approximately 5 µg/cm^3. This solution should be stored in a dark place at <5°C and is stable for at least 6 months.

4.7 Standard Pesticide Solutions

All pesticide solutions should be stored in a dark place at <5°C. These solutions are stable for at least 6 months.

4.7.1 Individual Standard Stock Solutions

Prepare Individual Standard Stock Solutions by weighing accurately approximately 0.02 g to a precision of ± 0.0001 g of each pesticide into individual volumetric flasks (100 cm^3 capacity) and dilute to volume with n-hexane. This will give solutions with concentrations of approximately 200 µg/cm^3.

Note: Due to the reduced solubility of ß-HCH this stock solution should be prepared in toluene.

4.7.2 Mixed Stock Solution A

Pipette an aliquot of each Individual Standard Stock Solution (5 cm^3) into a single volumetric flask (200 cm^3 capacity) and dilute to volume with n-hexane. This will give a solution with concentrations of approximately 5 µg/cm^3.

4.7.3 Mixed Stock Solution B

Pipette an aliquot of Mixed Stock Solution A (1 cm^3) into a single volumetric flask (10 cm^3 capacity) and dilute to volume with n-hexane. This will give a solution with concentrations of approximately 0.5 µg/cm^3.
4.7.4 Standard Calibrating Solution

Pipette an aliquot of the Mixed Stock Solution A (1 cm$^3$) and the Internal Standard Solution (1 cm$^3$) into a volumetric flask (100 cm$^3$ capacity) and dilute to volume with n-hexane. This will give a solution with concentrations of approximately 0.05 µg/cm$^3$ of each pesticide and Internal Standard.

4.8 Florisil 60-100 mesh

Note: Florisil is the trade name of a special selected variety of magnesium silicate. The mesh size range designated as 60 to 100 mesh corresponds to a mesh aperture size range of 250 to 150 µm.

4.8.1 Requirement

The quality of the Florisil is one of the most critical features of the method of test. The activity of the Florisil needs to be sufficient to retain impurities present in the extract from the sample while allowing the pesticide residues to be eluted. The Florisil shall first be pre-treated as described in 4.8.2. Only Florisil that passes the subsequent verification test described in 4.8.3 shall be used.

4.8.2 Pre-treatment

(a) Heat sufficient Florisil for the experiment (portion) in a quartz cup in a muffle furnace at 550°C for a minimum of 5 hours.

(b) Cool in a desiccator without desiccant and transfer to a round-bottom flask. Add water (equivalent to 5% w/w) to the Florisil and homogenise by rotation for approximately 1 hour.

(c) Store the portion of deactivated Florisil in a tightly closed glass container to allow equilibration for 48 hours before use.

4.8.3 Verification of activity level

The activity of the Florisil is checked by an extraction step of dieldrin solution, which must have a concentration equivalent to that of an extract from tobacco containing 1.0 µg/g of this pesticide. The activity is correct when the recovery of dieldrin is greater than 95%.

The activity of the Florisil should be checked each time a new portion is prepared.

5. APPARATUS

Note: It is essential to clean all glassware very thoroughly before use and to avoid the use of plastic containers and stopcock grease, otherwise impurities may be introduced into the solvents. All volumetric flasks and pipettes shall comply with class A of ISO 1042 and class A of ISO 648 respectively.

In addition to normal laboratory glassware the following apparatus is required:
OBSOLETE - This method was set up and validated at a time when few multi-residue methods were available. Nowadays, more efficient and powerful multi-residue methods are being applied.

5.1 Rotary evaporator
5.2 Tobacco mill with 2 mm mesh
5.3 Oven with ventilation
5.4 Muffle furnace
5.5 Heating mantles
5.6 Soxhlet extractor for continuous extraction (see Fig. 1)
5.7 Reflux condenser
5.8 Desiccator
5.9 Quartz cup

5.10 Gas chromatograph

5.10.1 Basic requirements

The gas chromatograph shall be operated in accordance with the manufacturers instructions. The injection port, oven and detector shall each be equipped with a separate heating unit.

The conditions given in 5.10.2 to 5.10.7 have been found to work on a particular make of instrument and are given for guidance. If other conditions are used they should be validated prior to use.

5.10.2 Temperatures

Injection port temperature shall be between 180 and 210°C. Detector temperature shall be between 290 and 340°C. The following temperature program and gas flow rates are given as an example. If any other conditions are used they should be sufficient to achieve satisfactory separation of all components and similar to that given in the specimen chromatogram (Fig. 2).

**Temperature program**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Temperature/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial temperature</td>
<td>40°C</td>
</tr>
<tr>
<td>Initial time</td>
<td>2 min</td>
</tr>
<tr>
<td>Temperature profile 1</td>
<td>20°C/min from 40°C to 150°C</td>
</tr>
<tr>
<td>Temperature profile 2</td>
<td>3°C/min from 150°C to 270°C</td>
</tr>
<tr>
<td>Final time</td>
<td>15 min at 270°C</td>
</tr>
<tr>
<td>Total GC run time</td>
<td>62.5 min</td>
</tr>
</tbody>
</table>
**OBSOLETE** - This method was set up and validated at a time when few multi-residue methods were available. Nowadays, more efficient and powerful multi-residue methods are being applied.

5.10.3 Gas flow rates
Gas flow rates should be set according to instrument manufacturers guidance and analyst experience. The following are examples.

- Carrier gas: helium, 4 cm³/min
- Make-up gas: nitrogen, 30 cm³/min
- Septum purge: 5 cm³/min
- Split vent: 30 cm³/min

5.10.4 Injection mode
2µl splitless with split valve closed for 1 min after injection.

5.10.5 Injection device
Use an automated injector or any suitable alternative means of injection. For manual injection the use of a micro syringe capable of injecting 1 to 5 µl portions is recommended. Before solutions are injected with the syringe, rinse it at least 10 times with n-hexane then 5 times with the solution. After injection rinse the syringe 5 times with n-hexane.

5.10.6 Column
A recommended column is 30 m x 0.32 mm i.d x 0.25 µm film thickness DB5 (5% methyl phenyl silicone) fused silica capillary. The performance of the column should be sufficient to achieve satisfactory separation of all components and similar to that given in the specimen chromatogram (Fig. 2).

5.10.7 Detector
An Electron Capture Detector shall be used with a sensitivity sufficient to detect (twice baseline noise) a 2 µl injection of a 0.0015 µg/cm³ p,p-DDT solution.

6. SAMPLING AND PREPARATION OF SAMPLE

6.1 Sampling
Sample the tobacco or tobacco product in accordance with ISO 4874 [3]. Give particular attention to ensuring that the test sample is representative of the product as received.

6.2 Preparation of test sample
(a) Tobacco is dried in a hot air cabinet with ventilation at 50°C for 2 hours. The water content shall be approximately 5% after drying.

(b) Grind the tobacco through a 2 mm mesh taking care to avoid heating above 50°C. Alternatively the tobacco may be received in a milled form in which case ensure that the moisture content is less than 10%.

(c) Store the tobacco is sealed containers in the dark. If samples are kept for longer than one month prior to analysis they should be stored in a freezer at a temperature of less than -8°C.
7. PROCEDURE

7.1 Test portions
Weigh the tobacco test portions (about 5 g) to a precision of ± 0.01 g into a beaker (50 cm³ capacity), add deactivated Florisil (5 g) and mix thoroughly. Carry out the procedure described in 7.2 and 7.3.

7.2 Extraction

7.2.1 Add deactivated Florisil (5 g) to a Soxhlet extractor.

7.2.2 Add the test portion as prepared in 7.1 without mixing so that two separate layers are formed. (For recovery determinations appropriate fortifying solutions should be added at this stage, by pipette to the top of the test portion layer).

7.2.3 Add n-hexane (60 cm³) and Mirex Internal Standard Solution (1 cm³) into a suitable round-bottom flask (150-250 cm³ capacity).

7.2.4 Connect the extraction apparatus together ensuring good seals between all joints and turn on the heaters.

7.2.5 Regulate the heating element and the tap on the Soxhlet extractor to give a distillation rate at least 200 cm³/hour.

The level of n-hexane above the tobacco must be kept constant by adjusting the tap on the Soxhlet extractor. Do not allow the round bottom flask to become dry, if this happens the apparatus dimensions may be too large. Total extraction time is 4.5 hours.

7.2.6 After extraction and cooling for at least 30 minutes, an aliquot of the extract is taken for analysis by gas chromatography. No volumetric adjustment is made.

7.3 Linearity
Pipette an aliquot of Mixed Stock Solution A (10 cm³, 5 cm³ and 1 cm³ respectively) into three individual volumetric flasks (100 cm³ capacity). To each volumetric flask add Internal Standard Solution (1 cm³) and dilute to volume with n-hexane. Pipette an aliquot of Mixed Stock Solution B (1 cm³) into two individual volumetric flasks (100 cm³ and 200 cm³ capacity respectively). To each volumetric flask add Internal Standard Solution (1 cm³ and 2 cm³ respectively) and dilute to volume with n-hexane. This will give solutions with concentrations of approximately 0.5, 0.25, 0.05, 0.005 and 0.0025 µg/cm³ respectively (equivalent to approximately 6, 3, 0.6, 0.06 and 0.03 µg/g on tobacco).

These solutions should be used to determine the linearity of detector response. This need only be determined when using a detector for the first time.

7.4 Calibration
If the detector response was found to be linear a single level calibration may be used. The 0.05 µg/cm³ Standard Calibrating Solution should be used for single level calibration.
7.5 **Gas Chromatography**

Set up the gas chromatograph and equilibrate the system. Check that reproducible results are obtained from triplicate injections of the Standard Calibrating Solution (± 5% of the mean). Carry out duplicate injections of each sample bracketed by single injections of standard and calculate the mean values.

Specimen chromatograms of a standard and a tobacco extract are attached (Fig. 2&3).

### 8. EXPRESSION OF RESULTS

#### 8.1 Method of calculation and formula

The amounts of pesticides are determined by the internal standard method. The residue \( R_{pest} \) expressed in µg/g dried tobacco is calculated as follows:

\[
R_{pest} = \frac{A(pest) \times ECD_{pest} \times Q_{st}}{(A(mirex) \times W \times ((100-M)/100))}
\]

where:

- \( A(pest) \) = peak (area or height) of pesticide in sample extract
- \( A(mirex) \) = peak (area or height) of mirex in sample extract
- \( Q_{st} \) = quantity of internal standard added in the extraction solution in µg (approx. 5 µg)
- \( W \) = weight of tobacco analysed in g (approx. 5 g)
- \( M \) = percentage moisture content of tobacco (approx. 5%)
- \( ECD_{pest} \) = response factor for the pesticide

\[
ECD_{pest} = \frac{[pest]_{st}/A(pest)_{st}}{[mirex]_{st}/A(mirex)_{st}}
\]

where: \( A(pest)_{st} \) and \( A(mirex)_{st} \) represent the peak (area or height) of the pesticide and mirex in the standard solution respectively.

\( [pest]_{st} \) and \( [mirex]_{st} \) represent the concentration of the pesticide and mirex in the standard solution respectively.

The response factor is that appropriate for the sample calculated, based upon the responses obtained from the Standard Calibrating Solutions bracketing the sample.

#### 8.2 Repeatability, Reproducibility, Recovery and Detection Limit

Repeatability, reproducibility and recovery were determined by the Joint Experiment 34 (JE 34) of the CORESTA Sub-Group on Pesticide Residues. Values were obtained for 4 different spiking levels F1-F4 as indicated in table 1 which is a summary of the major results obtained from JE 34. For more information about the statistical evaluation of results refer to CORESTA Sub-Group on Pesticide Residues-Document 94/3a.

Besides for the 17 pesticides mentioned in 1, JE 34 examined also the possible analyses of endrin and ß-endosulfan. Results (in document 94/3a) show that those two pesticides can not be analysed by the method.
OBSOLETE - This method was set up and validated at a time when few multi-residue methods were available. Nowadays, more efficient and powerful multi-residue methods are being applied.

9. TEST REPORT

The test report shall show the method used and the results obtained (in µg/g). It shall indicate the amounts of each of the individual pesticide residues identified. It shall also mention any operating conditions not specified in this International Standard or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for complete identification of the sample.
OBSOLETE - This method was set up and validated at a time when few multi-residue methods were available. Nowadays, more efficient and powerful multi-residue methods are being applied.
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Table 1

Results of Joint Experiment 34 of the CORESTA Sub-Group on pesticide residue (JE34/1/F1, JE34/1/F2, JE34/1/F3 and JE34/1/F4), including: spiking level, mean of recovery (Rec.), standard deviation of repeatability within labs (S\text{RL}), standard deviation of reproducibility (S\text{RR}) and detection limit.

Number of laboratories : 12, Number of samples per laboratory at each level : 2, Number of GC replicates per sample : 2

Number of determinations per pesticide at each level : $= 12 \text{ labs} 	imes 2 \text{ samples} \times 2 \text{ replicates} = 48 \text{ determinations}$

Outlayers : lab 7 and extreme lab means according to tables 14 to 17 in document 94/3a.

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Spiking level (µg/g) F1</th>
<th>Rec. \ \</th>
<th>S\text{RL} \ \</th>
<th>S\text{RR} \</th>
<th>Spiking level (µg/g) F2</th>
<th>Rec. \ \</th>
<th>S\text{RL} \ \</th>
<th>S\text{RR} \</th>
<th>Spiking level (µg/g) F3</th>
<th>Rec. \ \</th>
<th>S\text{RL} \ \</th>
<th>S\text{RR} \</th>
<th>Spiking level (µg/g) F4</th>
<th>Rec. \ \</th>
<th>S\text{RL} \ \</th>
<th>S\text{RR} \</th>
<th>Detection limit (µg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alpha-HCH</td>
<td>0.0995</td>
<td>79 \ \ 12 \ \ 27</td>
<td>0.4975 \ \ 102 \ \ 14 \ \ 18</td>
<td>0.9950</td>
<td>105 \ \ 8 \ \ 13</td>
<td>4.9750</td>
<td>95 \ \ 11 \ \ 18</td>
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</tr>
<tr>
<td>Beta-HCH</td>
<td>0.1000</td>
<td>89 \ \ 17 \ \ 25</td>
<td>0.5000 \ \ 99 \ \ 10 \ \ 15</td>
<td>1.0000</td>
<td>100 \ \ 6 \ \ 11</td>
<td>5.0000</td>
<td>86 \ \ 5 \ \ 14</td>
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<tr>
<td>Gamma-HCH (lindane)</td>
<td>0.1005</td>
<td>100 \ \ 26 \ \ 34</td>
<td>0.5025 \ \ 101 \ \ 15 \ \ 16</td>
<td>1.0050</td>
<td>109 \ \ 9 \ \ 19</td>
<td>5.0250</td>
<td>100 \ \ 7 \ \ 29</td>
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</tr>
<tr>
<td>Delta-HCH</td>
<td>0.1020</td>
<td>62 \ \ 13 \ \ 27</td>
<td>0.5100 \ \ 88 \ \ 7 \ \ 18</td>
<td>1.0200</td>
<td>96 \ \ 9 \ \ 15</td>
<td>5.1000</td>
<td>90 \ \ 12 \ \ 22</td>
<td></td>
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<tr>
<td>HCB</td>
<td>0.1000</td>
<td>109 \ \ 9 \ \ 19</td>
<td>0.5000 \ \ 105 \ \ 16 \ \ 18</td>
<td>1.0000</td>
<td>105 \ \ 8 \ \ 13</td>
<td>5.0000</td>
<td>88 \ \ 8 \ \ 16</td>
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<tr>
<td>Heptachlor</td>
<td>0.1020</td>
<td>100 \ \ 13 \ \ 19</td>
<td>0.5100 \ \ 105 \ \ 13 \ \ 15</td>
<td>1.0200</td>
<td>107 \ \ 12 \ \ 16</td>
<td>5.1000</td>
<td>101 \ \ 13 \ \ 25</td>
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<tr>
<td>Aldrin</td>
<td>0.1000</td>
<td>83 \ \ 13 \ \ 24</td>
<td>0.5000 \ \ 100 \ \ 14 \ \ 14</td>
<td>1.0000</td>
<td>107 \ \ 8 \ \ 14</td>
<td>5.0000</td>
<td>103 \ \ 7 \ \ 25</td>
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<tr>
<td>Heptachlor epoxide</td>
<td>0.1025</td>
<td>84 \ \ 15 \ \ 29</td>
<td>0.5125 \ \ 89 \ \ 16 \ \ 19</td>
<td>1.0250</td>
<td>97 \ \ 16 \ \ 18</td>
<td>5.1250</td>
<td>91 \ \ 11 \ \ 24</td>
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</tr>
<tr>
<td>Trans-chlordane</td>
<td>0.0995</td>
<td>86 \ \ 8 \ \ 23</td>
<td>0.4975 \ \ 96 \ \ 6 \ \ 14</td>
<td>0.9950</td>
<td>102 \ \ 4 \ \ 10</td>
<td>4.9750</td>
<td>99 \ \ 6 \ \ 18</td>
<td></td>
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</tr>
<tr>
<td>o,p-DDE</td>
<td>0.1010</td>
<td>105 \ \ 12 \ \ 25</td>
<td>0.5050 \ \ 106 \ \ 9 \ \ 11</td>
<td>1.0100</td>
<td>110 \ \ 4 \ \ 7</td>
<td>5.0500</td>
<td>101 \ \ 8 \ \ 19</td>
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</tr>
<tr>
<td>Alpha-Endosulfan</td>
<td>0.1010</td>
<td>73 \ \ 14 \ \ 36</td>
<td>0.5050 \ \ 81 \ \ 9 \ \ 15</td>
<td>1.0100</td>
<td>83 \ \ 10 \ \ 18</td>
<td>5.0500</td>
<td>77 \ \ 10 \ \ 23</td>
<td></td>
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