Analytical methods, and analytical techniques in food and environmental monitoring

WP 2
Saša Popov (ESV)
Analysis of food and environmental samples - **classification by analytical techniques**:

- instrumental analysis;
- non-instrumental analysis-standard analytical methods (gravimetry, volumetry, sensory....);

**Instrumental analysis classification:**

- Spectrometric (UV-VIS, IR, FTIR, AAS, ICP-OES,...);
- Chromatografic (gas chromatography, liquid chromatography, TLC,...);
- Electroanalytical methods (potenciometry, voltametry, conductometry,...)
- Others (MS, MS/MS, NMR, Coupled tehniqes (GC/MS, GC/MSMS, LC/MS, LC/MSMS,...));

All kinds of listed techniques are used to better define the product performance!
Some techniques in Enological station Vršac
Nature of sample characteristics (food safety and environment):

- Quality of sample – composition of sample (proteins, fats, sugars, acids, macroelements, microelements....);
- **Contaminants-health safety** (heavy metals, pesticides, mycotoxins, PAHs, PCBs, antibiotics, dioxins, phenols....);
- microbiological profile;

Modern science and analytics today, attaches increasing importance of contaminants in food and environmental samples!
Determination of pesticide contaminants in fruits and vegetables

- QueChers method of sample preparation

(10 g sample + 10 ml ACN + ISTD + QuEChERS tube), extraction, cleaning, centrifugation, optional evaporation;
Finished sample preparation
- **Analysis by GC / MSD**

  (injection (liquid), separation on chromatographic column (HP5-MS), ionization (EI), detection (MS));

  Analysis over 100 active components of pesticides (organochlorines, organophosphates, pyrethrins, triazines, others);
Possible problems - errors:

Losses in sample preparation
- strictly use the ISTD during the preparation (PCB 52, Triphenyl phosphate,...);
- quantitatively transferr extracts and carefully evaporate;

Influence of matrix
- additional sample cleaning;
- LLE (with hexane/20% NaCl (w/w) mixture) leads to significant reduction of matrix co-extracts;
- select different Qualyfiers which you record with MSD;

Confirmation some active component
- spike sample;
- additional sample concentration;
Scan chromatogram of mentha piperita
Comparison of MS spectra chlorpyrifos from sample and library of MS spectra
SIM chromatogram of *mentha piperita*
SIM chromatogram of spiked mentha piperita
SIM chromatogram of mentha piperita
### Enoloska Stanica

**Quantitation Report**

- **Data Path:** D:\Data\TRI_PEST_1\USORC1\2016\NOVEMBER\14_11_2016_4\n- **Data File:** 7726_CaI_Nana_SIM.D
- **Acq On:** 14 Nov 2016 13:23
- **Operator:**
- **Sample:** 7726_CaI_Nana_SIM
- **Misc:**
- **ALS Vial:** 4  **Sample Multiplier:** 1

**Quant Time:** Jun 05 14:40:08 2017  
**Quant Method:** D:\Methods\TRI_PEST_1_OF_SIM_10_2016.M
- **Quant Title:** TRI_PEST_1_OF_SCAN
- **Last Update:** Thu Oct 27 21:25:12 2016
- **Response via:** Initial Calibration

**Internal Standards**

<table>
<thead>
<tr>
<th>Compound</th>
<th>R.T. Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev (Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) PCB 52</td>
<td>10.146</td>
<td>292</td>
<td>395060</td>
<td>0.50 ug/ml</td>
</tr>
</tbody>
</table>

**Target Compounds**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Q Value</th>
<th>R.T. Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev (Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2) Dichlorvos</td>
<td></td>
<td>5.875</td>
<td>109</td>
<td>2026524</td>
<td>5.69 ug/ml</td>
</tr>
<tr>
<td>3) Methacetifos</td>
<td></td>
<td>8.592</td>
<td>125</td>
<td>960</td>
<td>Below Cal</td>
</tr>
<tr>
<td>4) Sulcotep</td>
<td>11.815</td>
<td>322</td>
<td>5</td>
<td>Below Cal</td>
<td>1</td>
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<tr>
<td>5) Ponofos</td>
<td>13.541</td>
<td>109</td>
<td>58441</td>
<td>0.03 ug/ml</td>
<td>30</td>
</tr>
<tr>
<td>6) Propetamphos</td>
<td>13.903</td>
<td>138</td>
<td>14647</td>
<td>0.03 ug/ml</td>
<td>53</td>
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<tr>
<td>7) Diazinon</td>
<td>14.866</td>
<td>179</td>
<td>52778</td>
<td>0.16 ug/ml</td>
<td>5</td>
</tr>
<tr>
<td>8) Etrifos</td>
<td>15.202</td>
<td>292</td>
<td>116</td>
<td>Below Cal</td>
<td>1</td>
</tr>
<tr>
<td>9) Dichloflention</td>
<td>16.430</td>
<td>279</td>
<td>13283</td>
<td>0.06 ug/ml</td>
<td>1</td>
</tr>
<tr>
<td>10) Chlorpyrifos methyl</td>
<td></td>
<td>16.639</td>
<td>286</td>
<td>17</td>
<td>0.00 ug/ml</td>
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<td>11) Penchlorphos</td>
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<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
<td>51</td>
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<tr>
<td>12) Pirimiphos methyl</td>
<td></td>
<td>18.146</td>
<td>290</td>
<td>294960</td>
<td>7.19 ug/ml</td>
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<td>13) Malathion</td>
<td>18.723</td>
<td>173</td>
<td>6815</td>
<td>0.89 ug/ml</td>
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<td>14) Chlorpyrifos</td>
<td>19.472</td>
<td>197</td>
<td>116513</td>
<td>0.45 ug/ml</td>
<td>84</td>
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<td>15) Bromophos methyl</td>
<td></td>
<td>0.000</td>
<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
</tr>
<tr>
<td>16) Pirimiphos ethyl</td>
<td></td>
<td>20.718</td>
<td>333</td>
<td>16</td>
<td>Below Cal</td>
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<tr>
<td>17) Chlorfenvinphos</td>
<td></td>
<td>0.000</td>
<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
</tr>
<tr>
<td>18) Methidathion</td>
<td></td>
<td>0.000</td>
<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
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<tr>
<td>19) Bromophos ethyl</td>
<td></td>
<td>0.000</td>
<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
</tr>
<tr>
<td>20) Tetrachlorvinphos</td>
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<td>22.932</td>
<td>329</td>
<td>2293</td>
<td>0.05 ug/ml</td>
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<tr>
<td>21) Ethion</td>
<td>0.000</td>
<td>0</td>
<td>M.D.</td>
<td>0.00 ug/ml</td>
<td>51</td>
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<td>22) Carbenophenthiol</td>
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<td>26.771</td>
<td>157</td>
<td>20132</td>
<td>0.06 ug/ml</td>
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<td>23) Azinphos methyl</td>
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<td>29.769</td>
<td>160</td>
<td>4664</td>
<td>10.18 ug/ml</td>
</tr>
<tr>
<td>24) Azinphos ethyl</td>
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<td>30.709</td>
<td>132</td>
<td>3201</td>
<td>0.18 ug/ml</td>
</tr>
</tbody>
</table>

( # ) = qualifier out of range  ( m ) = manual integration  (+) = signals summed
Determination of metals in water, soil and sludge samples by ICP-OES (US EPA 200.7)

Sample preparation:
-Microwave digestion, filtration, evaporation, dilution;
ICP-OES determination:
- conditions settings;
- calibration;
- ICP-OES determination;
Possible problems - errors:

- Bad sample preparation (losses, low preparation temperature, contamination, ...);
- Influence of matrix (bad choice of characteristic emission lines,...);
- Bad conditions on ICP-OES (spectral shift of the characteristic lines, not using of ISTD,...)

WHAT CAN WE DO?

- Analysis of CRM-correction of sample preparation and ICP-OES determination;
- Check and set conditions on ICP-OES (the optics with special standards solution, choice of different emission lines, speed of sample uptakes, ....);
- Spike samples;
Example of bad determination on ICP-OES soil samples—determination of CEC
Example of good determination on ICP-OES soil samples—determination of CEC
Example of influence ISTD on ICP-OES determination soil samples
Effect of stability on ICP-OES determination of CRM sample (strawberry leaves)
Effect of stability on ICP-OES determinations of CRM sample (strawberry leaves) – power failure
FOR SUCCESSFUL ANALYSIS

you must know, look, think, feel and do analytics