Measurement of the total nitrogen in water – problem with high blanks

Daniel Žlindra
- implementation of the method in 2003,
- method: ISO 11905-1: peroxodisulfate digestion in autoclave followed by spectrometer determination of nitrate at 220 nm
- SCHOTT DURAN autoclavable bottles 50 mL
- UV-Vis spectrometer Varian Cary 50
- use of control samples:
  ✓ blanks, the same preparation as for the samples
  ✓ IKV (0,995 mg/L), the same preparation as for the samples (digestion included);
  ✓ K 963/05 (~80 mg/L), the same preparation as for the samples (dilution and digestion included)
  ✓ STD 1,25 (1,25 mg/L), the same preparation as for calibration standards (no digestion)
usual values for blanks from 0.02 to maximum 0.10 mg N/L,
in February 2008 blanks started to raise.
IKV 0.995 mg N/L

Date


mg N/L

Higher value
Lower value

1st Meeting of the heads of the laboratories within ICP Forests
Hamburg, Germany, June 9th and 10th 2008, Daniel Žlindra
test: measured 48 blanks, digested in two different autoclaves used two different aged reagents;

<table>
<thead>
<tr>
<th>Autoclave (the same aged reagent)</th>
<th>SFI</th>
<th>BF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>0,18</td>
<td>0,26</td>
</tr>
<tr>
<td>Std deviation</td>
<td>0,11</td>
<td>0,20</td>
</tr>
<tr>
<td>Variance</td>
<td>0,0126</td>
<td>0,0380</td>
</tr>
</tbody>
</table>

1. There’s no significant difference between the autoclaves,
2. There’s no significant difference between the different aged reagents,
3. Conclusion: contamination of the bottles
➢ Cleaning of the bottles according to ISO 11905-1 (blank digestion, washing with dill. HCl, leaving the acid in the bottles just before the use)

➢ Result: 0.303
0.200
0.497
0.288
0.196 mg N/L

➢ Next stage: buying 10 new bottles (leaching of N out of the glass during ages??)
Many thanks to Irena Truden, Magda Špenko and Nataša Filipič

And

Thank you for your attention!