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

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1st **Meeting of the heads of the laboratories within ICP Forests**

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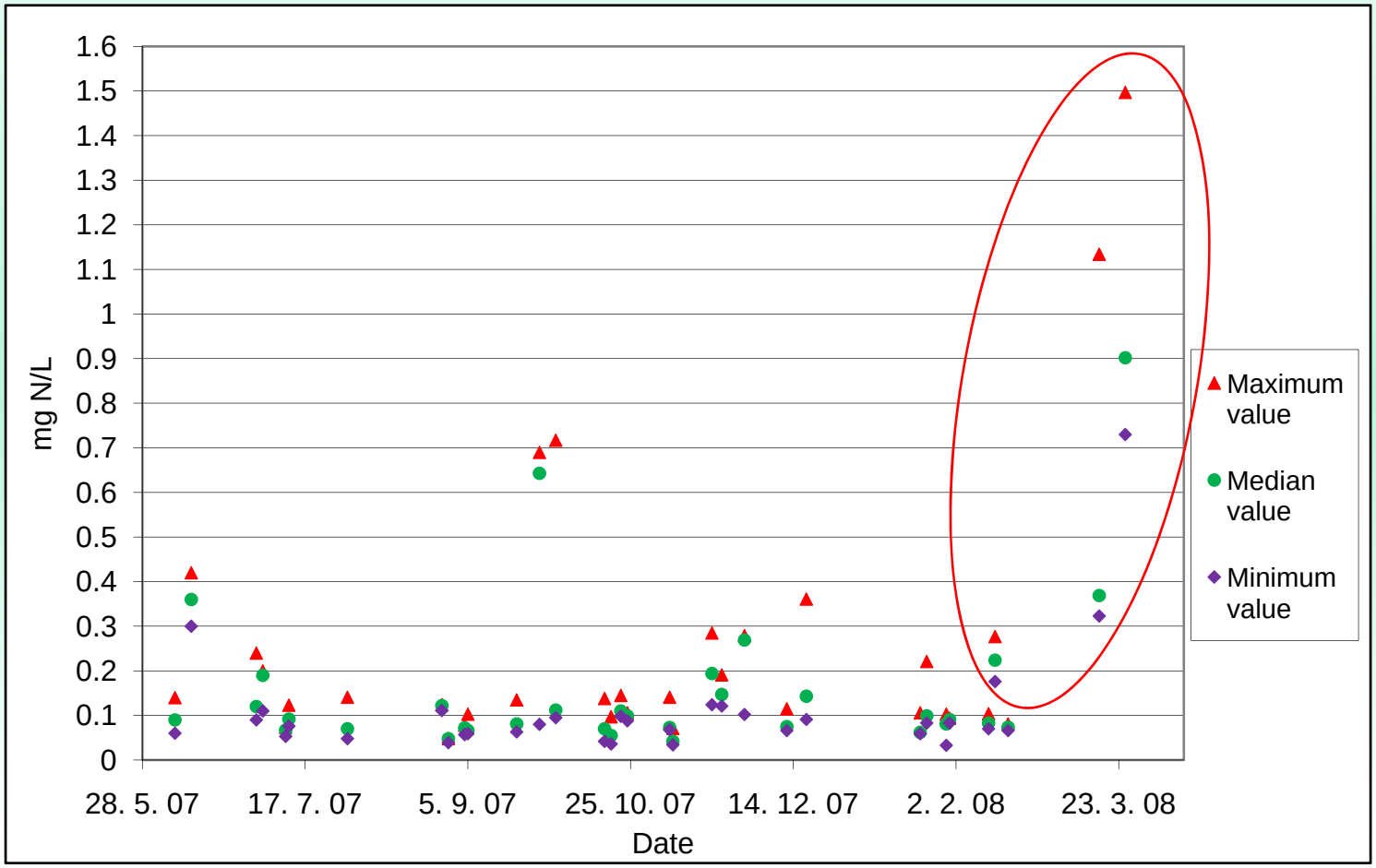
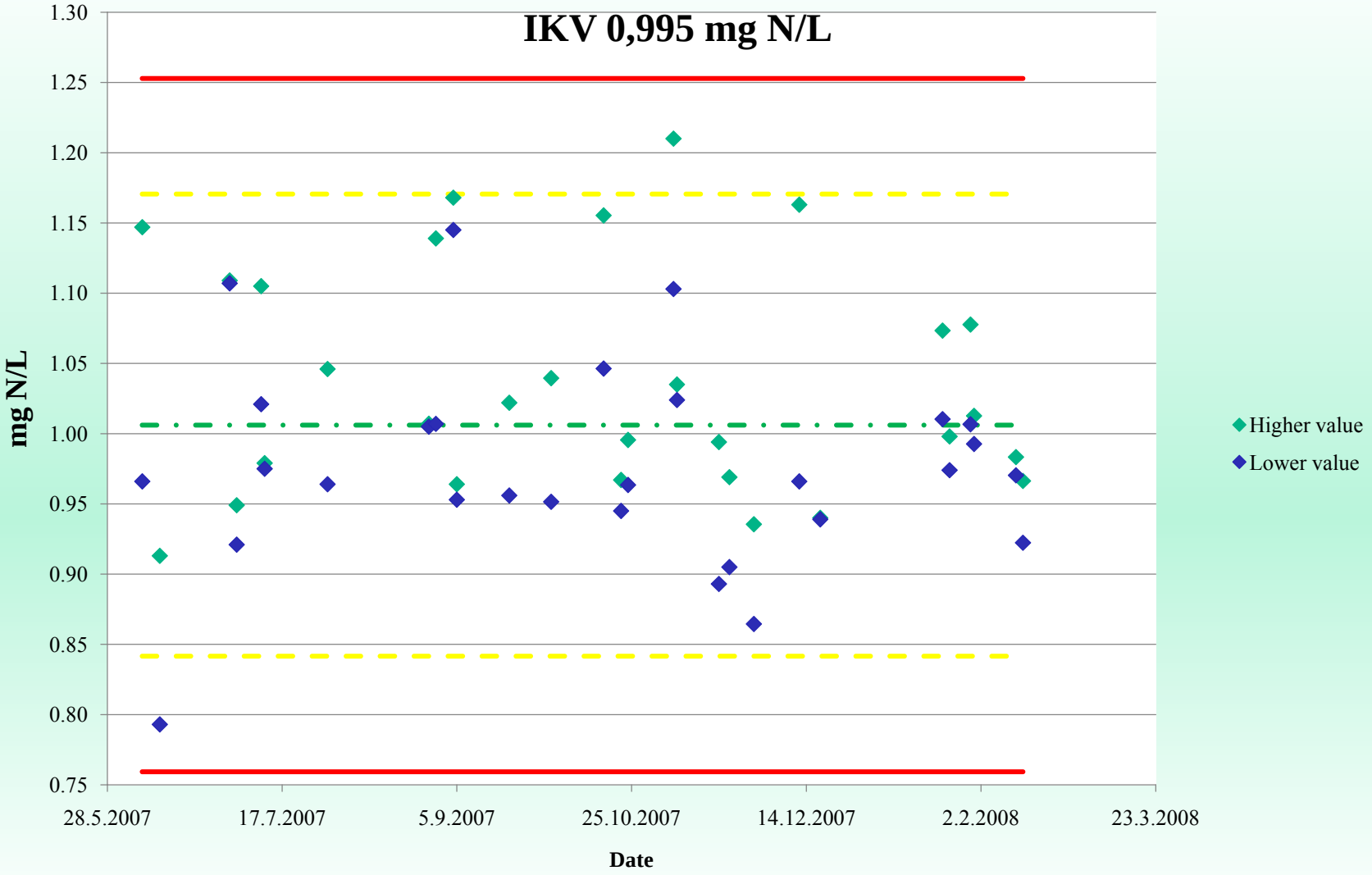
- implementation of the method in 2003,
- method: ISO 11905-1: peroxodisulfate digestion in autoclave followed by spectrometer determination of nitrate at 220 nm
- SCHOTT DURAN autoclavable bottles 50 mL
- UV-Vis spectrometer Varian Cary 50
- use of control samples:
  - ✓ blanks, the same preparation 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 (dillution 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
mg N/L
1.30
1.25
1.20
1.15
1.10
1.05
1.00
0.95
0.90
0.85
0.80
0.75
28.5.2007
17.7.2007
5.9.2007
25.10.2007
14.12.2007
2.2.2008
23.3.2008
Date
Higher value
Lower value

➢ test: measured 48 blanks, digested in two different autoclaves used two different aged reagents;

| Autoclave (the same aged reagent) | SFI | BF |
|---|---|---|
| Average | 0,18 | 0,26 |
| Std deviation | 0,11 | 0,20 |
| Variance | 0,0126 | 0,0380 |

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!