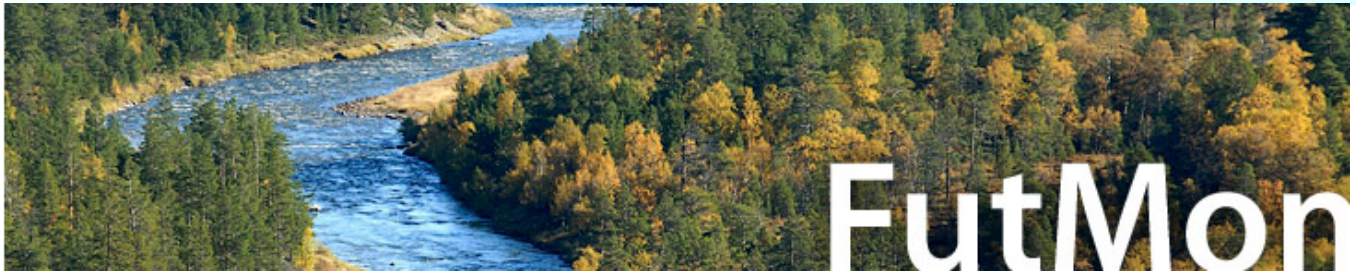
Life+ FutMon - Working Group on QA/QC in Laboratories
Meeting of the Heads of the Laboratories
12-13 October 2009 in Warsaw

Analytical aspects comparison DTN and IC determinations

Gabriele Tartari [1] & Carmen Jacoban [2]

[1]C.N.R. Institute of Ecosystem Study, Verbania (Italy)
[2]Forest Research Station, Campulung Moldovenesc (Romania)



DTN determination

Consiglio Nazionale delle Ricerche
INSTITUTE FOR ECOSYSTEM STUDY
Verbania Pallanza - Italy

Hydrochemistry Laboratory, Analytical Methods - Internal Use.
Gabriele TARTARI

Total Nitrogen in Water

A peroxodisulphate oxidation procedure followed by spectrophotometric determination

PRINCIPLE

Ammonium, nitrite and organic nitrogen, are oxidised to nitrate using potassium peroxodisulphate in a buffer boric acid-sodium hydroxide buffer. The oxidation of the nitrogen compounds is performed in an autoclave at 120 °C, resulting in a pH change of the buffer from 9.7 to 5.0. The resulting nitrate is determined by spectrophotometry at 220 nm. (Note: Organic substances which interfere at this wavelength are assumed to be completely oxidised in the peroxodisulphate digestion procedure).

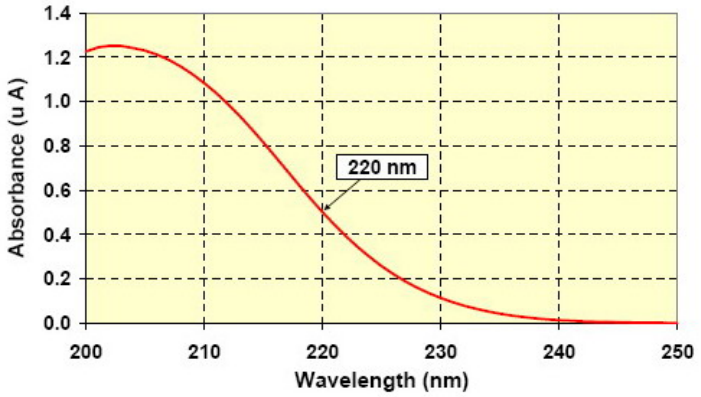


Fig. 1 - Absorption spectrum of a nitrate solution between 200 nm to 250 nm

DTN determination

Total nitrogen UV determination 220 nm OP 1 cm from 2000 to 2009 CNR-ISE Italy

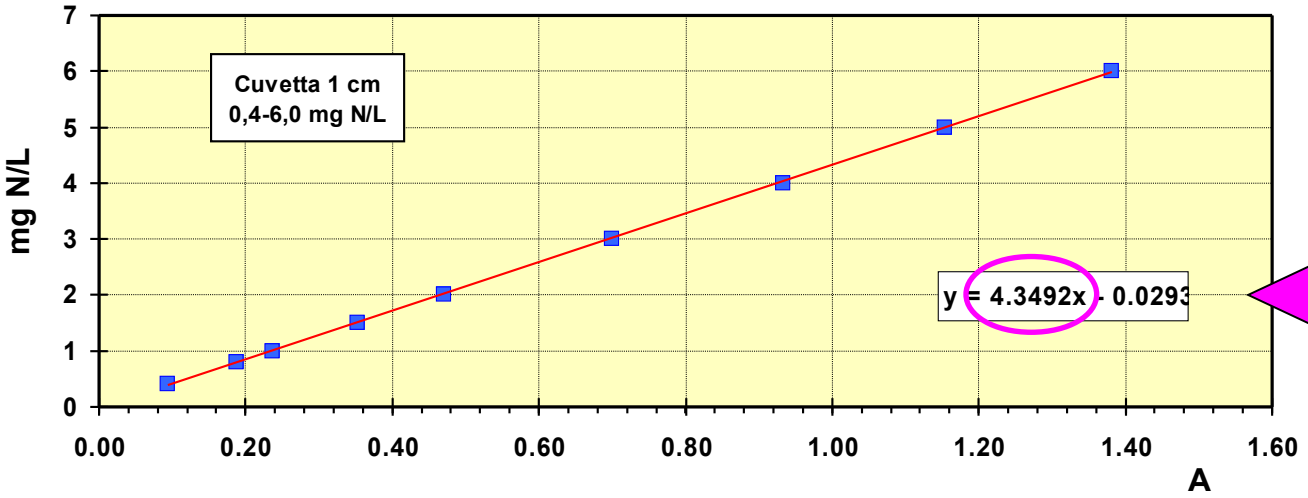
		Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	Standard 8	Standard 9
NT 0.4-6	Media	0.094	0.188	0.237	0.353	0.470	0.700	0.934	1.154	1.381
	Med.+2SD	0.101	0.196	0.251	0.372	0.491	0.725	0.978	1.211	1.430
	Med.-2SD	0.088	0.179	0.222	0.334	0.448	0.675	0.889	1.098	1.332
	R.S,D	3.6	2.3	3.1	2.7	2.3	1.8	2.4	2.4	1.8
	n° dati	25	23	22	20	24	23	24	20	24
	mg N/L	0.4	0.8	1.0	1.5	2.0	3.0	4.0	5.0	6.0
	RF medio	0.2361	0.2349	0.2367	0.2350	0.2348	0.2333	0.2334	0.2308	0.2302

Total nitrogen UV determination 220 nm OP 1 cm - Carmen Jacoban 4-5 May 2009

		Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	Standard 8	Standard 9
NT 0.4-6	Media	0.111	0.201	0.240	0.358	0.476	0.697	0.926	1.154	1.406
	mg N/L	0.4	0.8	1.0	1.5	2.0	3.0	4.0	5.0	6.0
	RF medio	0.2786	0.2509	0.2405	0.2384	0.2381	0.2323	0.2314	0.2308	0.2343

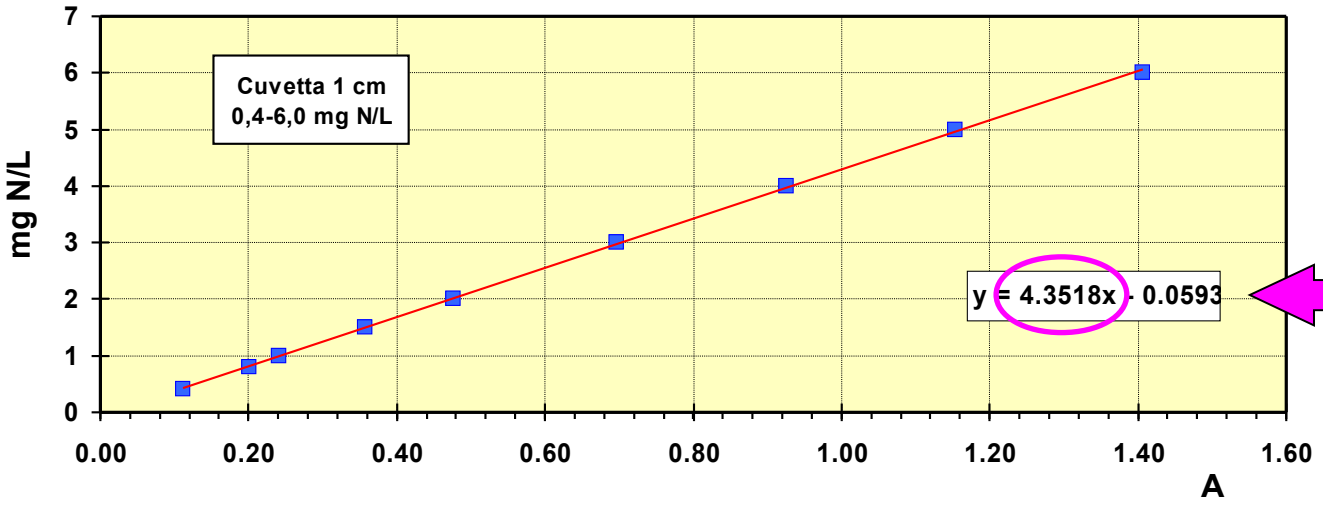
Diff.	-0.017	-0.013	-0.004	-0.005	-0.006	0.003	0.008	0.000	-0.025

DTN calibrations comparison



CNR-ISE Italy
Average 10 years
calibrations 2000-2009

Slope



Carmen Jacoban
May 2009
One calibration

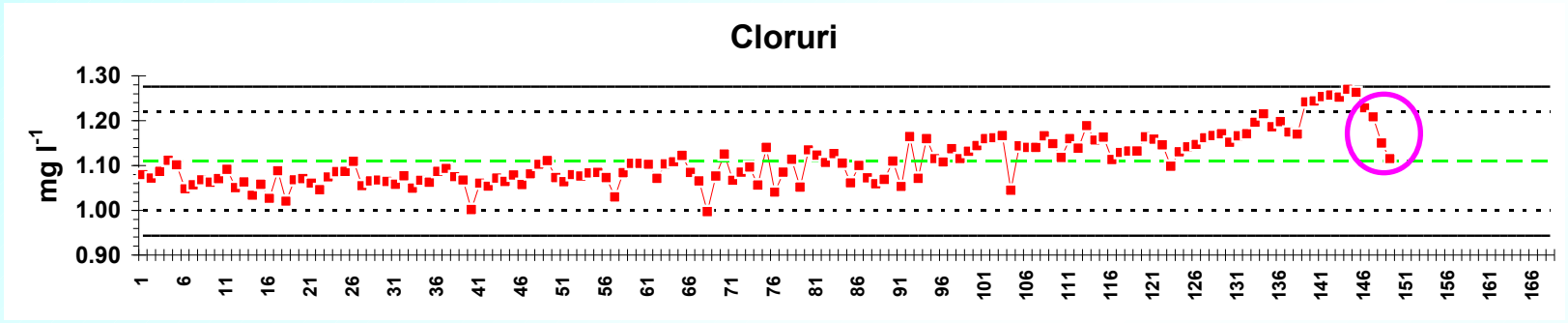
Slope

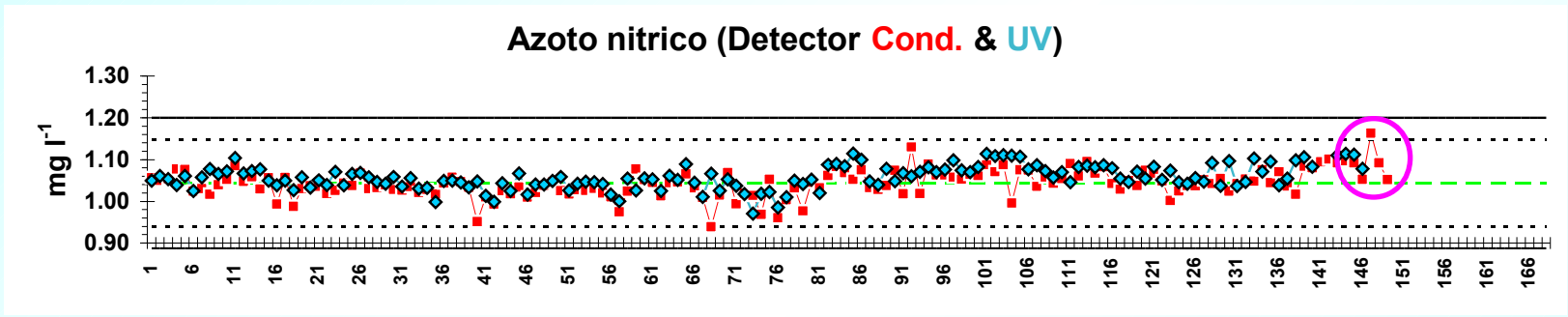
IC determination

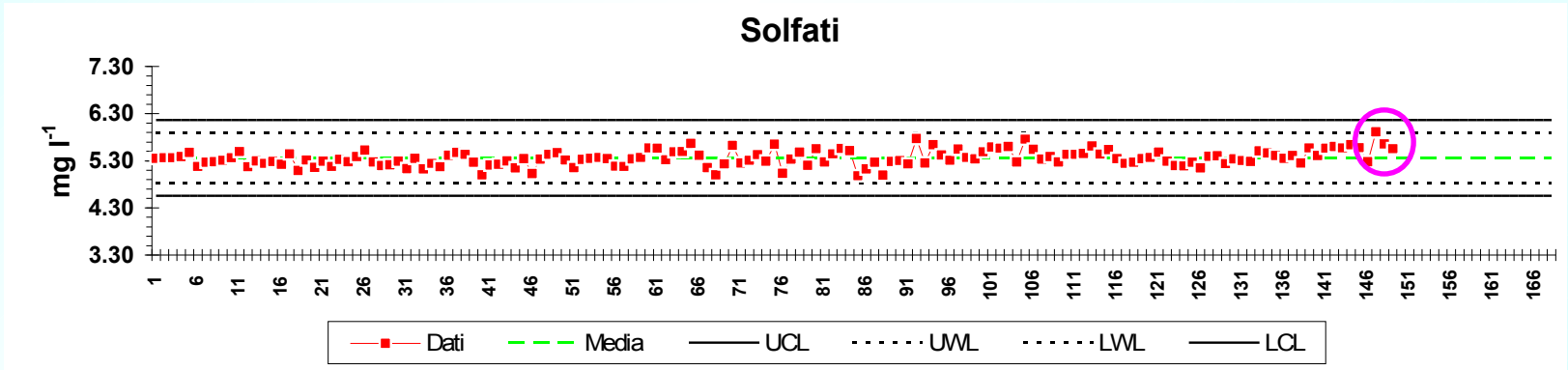
Instruments:

Dionex ICS 3000 cations
Dionex DX 500 cations *Gabriele Tartari*
Dionex DX 320 anions

Dionex ICS 3000 *Carmen Jacoban*

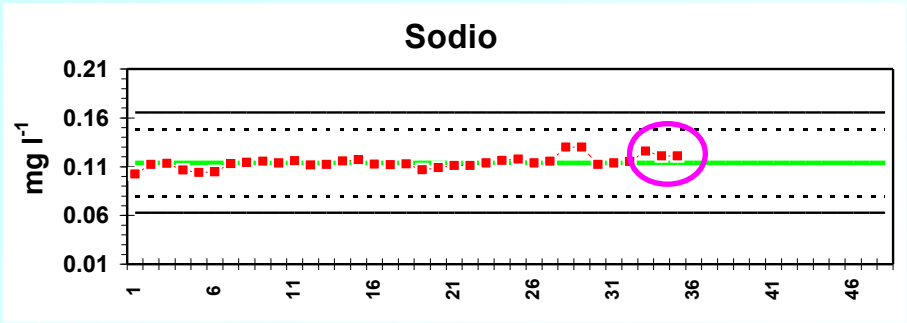


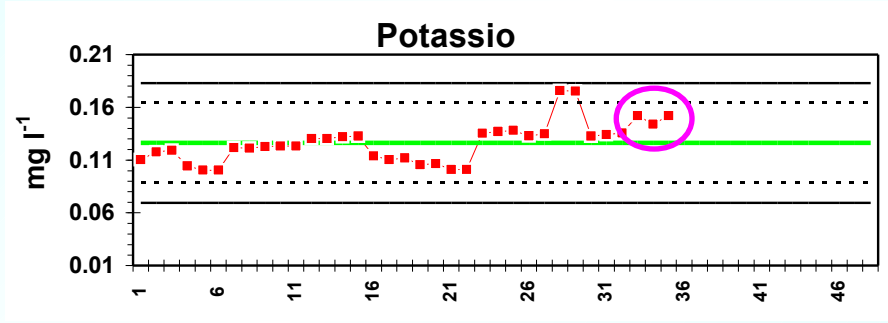


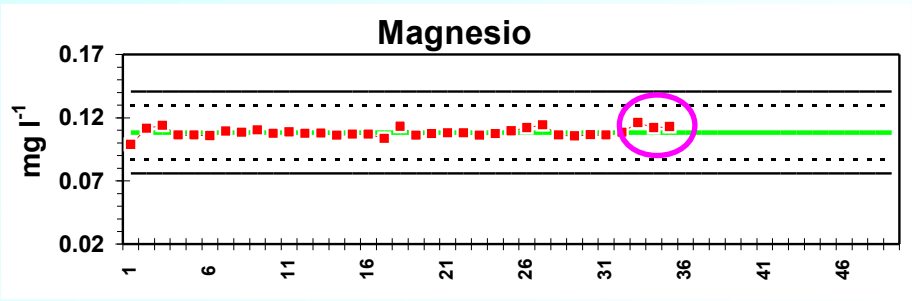


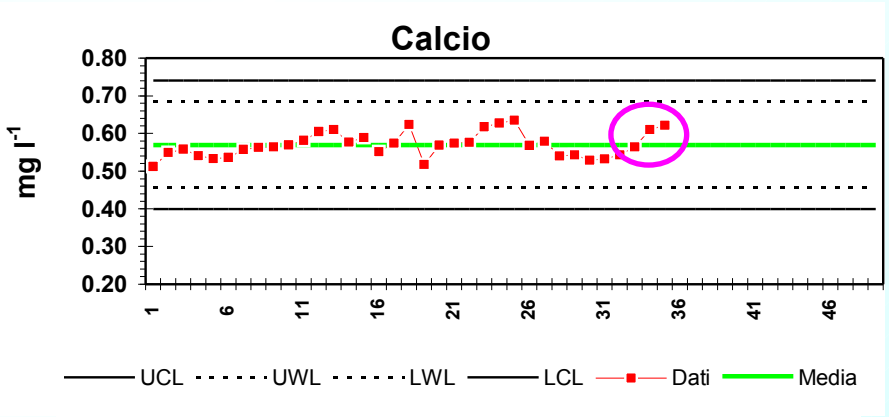
CNR-ISE control chart from Jul 2008-Apr 2009

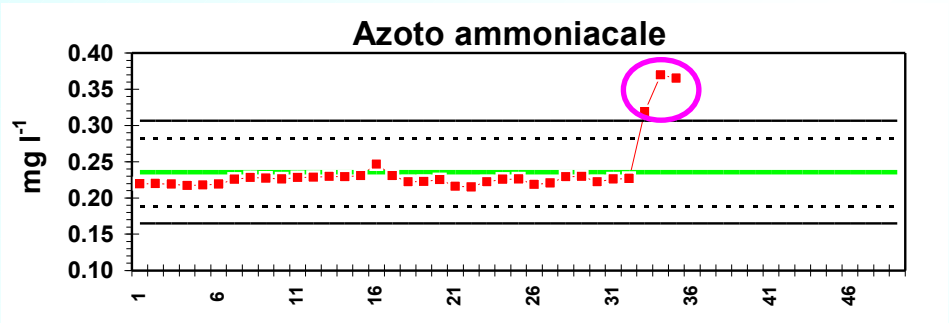
Carmen's data 2 May 2009









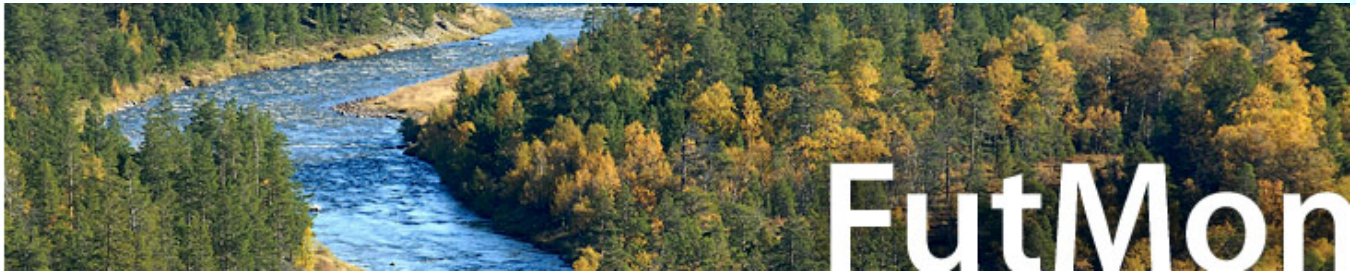


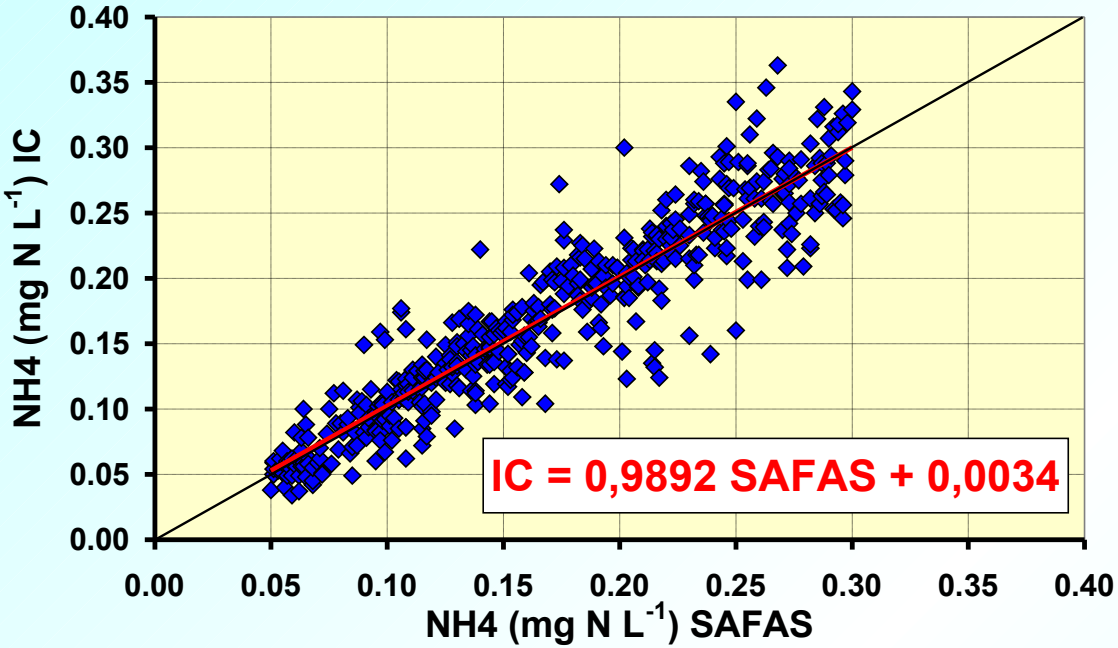
UCL	**Upper Control Limit (massimo accettato)**	**= Media + 3 D.S. consigliata**
UWL	**Upper Warning Limit (massimo consigliato)**	**= Media + 2 D.S. consigliata**
LWL	**Lower Control Limit (minimo consigliato)**	**= Media - 2 D.S. consigliata**
LCL	**Lower Warning Limit (minimo accettato)**	**= Media - 3 D.S. consigliata**

CNR-ISE control chart from Mar-Apr 2009

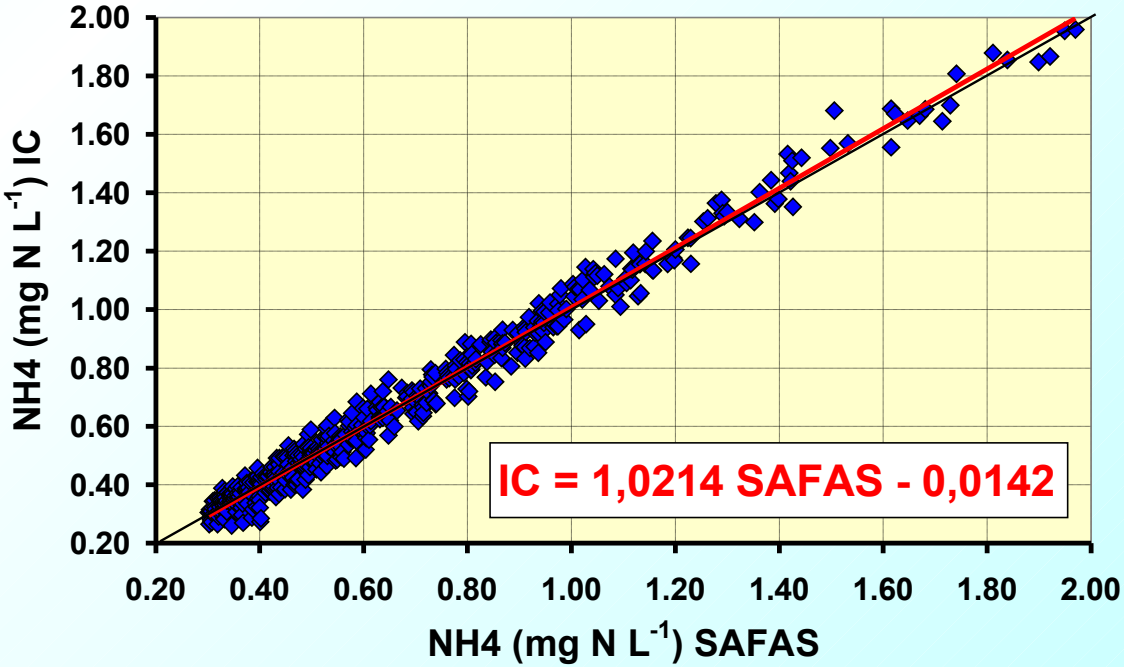
Carmen's data 2 May 2009

Thank you for your attention





CNR-ISE Italy



N-NH4 comparison SPEC Phe - IC

Jen 2005 - Jun 2006

Tolal Range (mg L^{-1})	0.05 - 2.00	
Half Range on mediane (mg L^{-1})	0.050 - 0.30	0.30 - 2.00
N. data	476	508
Test Wilcoxon (a 0.001)	OK	OK
Probability	0.037	0.78
IC = a + b x SPEC		
r	0.9316	0.9924
Test Slope = 1 (0.001)	OK	NON OK
Slope	0.989	1.021
Test Intercept = 0 (0.001)	OK	OK
Intercept	0.003	-0.014
DIFFERENCE from data	OK	OK
DIFFERENCE (µg N L^{-1})	SPEC - IC	
Min	-98	-176
25 %ile	-16	-29
Mediane	**-3**	**-1**
75 %ile	12	29
Max	97	128
Average	-2	0
DIFFERECE absolute (µg N L^{-1})		
25 %ile	7	13
Mediane	**15**	**29**
75 %ile	26	52
Max	98	176
Average	20	34

CNR-ISE Italy