5th Meeting of the Heads of the Laboratories

(17-18 September 2015, Wien)

Again on the Excel sheet for testing water chemical analyses (atmospheric deposition, runoff, soil water)

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Northwest German Forest Research Station (NW-FVA), Göttingen, Germany

1st Meeting of the Heads of the Laboratories

(9.-10. June 2008, Hamburg, Germany)

An excel sheet for testing water chemical analyses (atmospheric deposition, runoff, soil water)

Life+ FutMon - Working Group on QA/QC in Laboratories
Meeting of the Heads of the Laboratories
16-17 June 2011 in Arcachon

Updated Excel sheet for quality check of results of atmospheric deposition and soil water analyses

Complete analysis (all relevant ions analyzed)

↓

Perform the tests described in the ICP Forests manual (either with our Excel sheet or with your own procedures)

↓

If the test fails, repeat (some of) the analyses

Perform the analyses

↓

Send the data to a central data base

↓

Next year (usually in February) someone will submit the data to the ICP Forests central database

The on line submission routine will perform the test

He will see if the test fails and (may be) report back to you

Perform the analyses

↓

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Complete analysis (all relevant ions analyzed)

↓

Perform the tests described in the ICP Forests manual
(either with our Excel sheet or with your own procedures)

↓

If the test fails, repeat (some of) the analyses

The same test are performed by the ICP-Forests on-line submission procedure

If you do the test by yourself, you can repeat the analyses and have good results

If you wait for the on-line submission, you can no more perform the analyses,
and your data will not be validated for the elaborations

Ionic balance

$$PD = 100 * \frac{(\Sigma \text{ cat} - \Sigma \text{ an})}{0.5\,(\Sigma \text{ cat} + \Sigma \text{ an})}$$

$$\Sigma_{\text{anions}} = [HCO_3^-] + [SO_4^{=}] + [NO_3^-] + [Cl^-] + [Org^-]$$

$$\Sigma_{\text{cations}} = [Ca^{++}] + [Mg^{++}] + [Na^+] + [K^+] + [H^+] + [NH_4^+]$$

Comparison between measured (CM) and calculated conductivity (CE)

$$CD = 100 * \frac{(CM-CE)}{CM}$$

for conductivity $\leq$ **100 µS cm^{-1}** $CE_{\infty} = \sum \lambda_i c_i$

for conductivity **>** **100 µS cm^{-1}** $CE = \sum \lambda_i f_i c_i$

λ_i ***equivalent ionic conductance***

C_i ***Concentration of the ion i***

f_i *activity coefficient*

	Units	Factors to µeq L^{-1}	Equivalent conductance at 25°C kS cm^2 eq^{-1}
pH		$10^6 * 10^{-pH}$	0.3500
Ammonium	mg N L^{-1}	71.39	0.0735
Calcium	mg L^{-1}	49.90	0.0595
Magnesium	mg L^{-1}	82.29	0.0531
Sodium	mg L^{-1}	43.50	0.0501
Potassium	mg L^{-1}	25.58	0.0735
Alkalinity	meq L^{-1}	1000	0.0445
Sulphate	mg S L^{-1}	62.37	0.0800
Nitrate	mg N L^{-1}	71.39	0.0714
Chloride	mg L^{-1}	28.21	0.0764

Acceptance threshold values in data validation based on the ionic balance and conductivity.

Conductivity of the sample 25 °C	Ionic balance	Conductivity
≤ 10 µS cm^{-1}	± 20%	±30%
< 20 µS cm^{-1}	± 20%	±20%
> 20 µS cm^{-1}	± 10%	±10%

Ionic balance

$$PD = 100 * \frac{(\Sigma \text{ cat} - \Sigma \text{ an})}{0.5\,(\Sigma \text{ cat} + \Sigma \text{ an})}$$

$$\Sigma_{\text{anions}} = [\,HCO_3^-] + [SO_4^{=}\,] + [\,NO_3^-\,] + [\,Cl^-\,] + [Org^-\,]$$

$$\Sigma_{\text{cations}} = [Ca^{++}\,] + [\,Mg^{++}\,] + [\,Na^+\,] + [\,K^+] + [\,H^+\,] + [\,NH_4^+\,]$$

Evaluation of the contribution of DOC to ion balance

[Org-] = 5.04*DOC - 6.67, String STF (only for BL) equation 1
[Org-] = 6.80*DOC - 12.32, THR, BL equation 2
[Org-] = 4.17*DOC - 5.01 THR, CON equation 3

FALSE() [Org-]= 0 µeq/L Other strings different from STR or THR

IF C23 = "BOF" then no correction to ion balance for DOC

IF C23 = "STF" then ion balance corrected using equation 1
(STF is measured only in the case of broad leaves)

IF C23 = "THR" and D23= "BL" then correction using equation 2
IF C23 = "THR" and D23= "CON" then correction using equation 3

IF C23 ≠ from "THR", "STF" or D23 ≠ from "BL", "CON"

then U23 = "False", i.e. something is wrong!

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O90 µeq (mg C)-1

Estimation of the organic ion charge from DOC

Correction parameters from	user			
Sampling typology	STF	THR	CON	SW
Slope	5.04	6.80	4.17	8.64
Intercept	-6.67	-12.32	-5.01	0.00

Correction parameters from Mosello et al. 2008				from manual
Sampling typology	STF	THR	CON	SW
Slope	5.04	6.80	4.17	8.64
Intercept	-6.67	-12.32	-5.01	0.00

REFERENCE

Mosello R., T. Amoriello, S. Benham, N. Clarke, J. Derome, K. Derome, G. Genouw, N. Koenig, A. Orrù, G. Tartari, A. Thimonier, E. Ulrich and A. J. Lindroos. 2008. Validation of chemical analyses of atmospheric deposition on forested sites in Europe: 2. DOC concentration as an estimator of the organic ion charge. J. Limnol., 67(1): 1-14.

e-mail corresponding author: r.mosello@ise.cnr.it

Tab 7a – Broad-leaves - Statistical values (nu

and significance of the regressions f

Type of solution	BROAD LEAVES	Units
BOF	N	-
BOF	pH range	u
BOF	pH mean± σ	u
BOF	DOC range	mg C l^{-1}
BOF	DOC mean± σ	mg C l^{-1}
BOF	∑ cat range	µeq l^{-1}
BOF	∑ cat mean± σ	µeq l^{-1}
BOF	∑ an range	µeq l^{-1}

Results | S cat - S an | Xm-Xc corrected | Na-Cl | Notes

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J. Limnol., 67(1): ??-??, 2008

Validation of chemical analyses of atmospheric deposition on forested sites in Europe: 2. DOC concentration as an estimator of the organic ion charge

Rosario MOSELLO*, Tiziana AMORIELLO[1)], Sue BENHAM[2)], Nicholas Clarke[3)], John DEROME[4)], Kirsti DEROME[4)], Gerrit GENOUW[5)], Nils KOENIG[6)], Arianna ORRÙ, Gabriele TARTARI, Anne THIMONIER[7)], Erwin ULRICH[8)] and Antti-Jussi LINDROOS[4)]

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[4)]Finnish Forest Research Institute, P.O. Box 16, 96301 Rovaniemi, Finland
[5)]Research Institute for Nature and Forest, Geraardsbergen, Belgium
[6)]Nordwestdeutsche Forstliche Versuchsanstalt, Graetzelstr. 2, 37079 Goettingen, Germany
[7)]WSL, Birmensdorf, Switzerland
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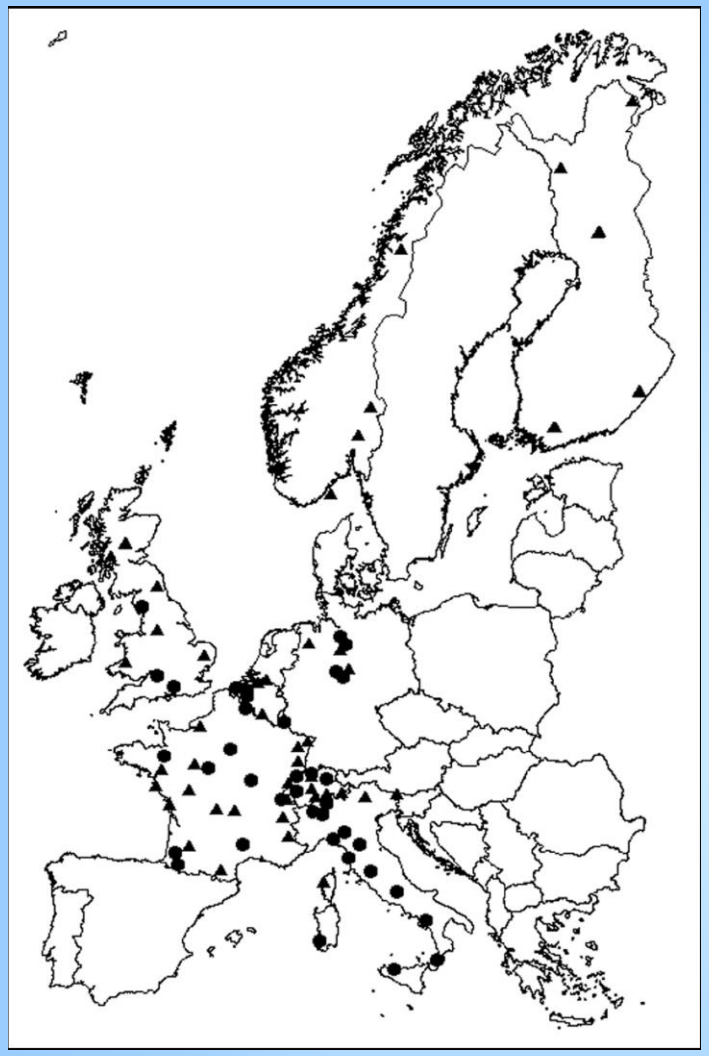
* e-mail corresponding author: r.mosello@ise.cnr.it



About 6000 analyses of deposition samples done from 8 different laboratories

Samples were representative of different geographic and climatic situations

Evaluation of the formal charge of organic carbon

$$\Sigma \text{ cations} - \Sigma \text{ anions} = ß_1 * DOC + ß_0$$

$$[Org^-] = \beta_1 * DOC + \beta_0$$

where $[Org^-]$ and β_0 units are μeq l-1, DOC and β_1 units are mg C l^{-1} and μeq (mg C)$^{-1}$.

		Broad Leaves		Conifers
	units	THR	STF	THR
N	-	1454	597	1657
pH range	u	4.0 - 7.9	3.8 - 8.1	4.1 – 7.0
pH mean± σ	u	5.8±0.6	5.6±0.6	5.3±0.5
DOC range	mg C L^{-1}	0-37	14246	0-40
DOC mean± σ	mg C L^{-1}	8±6	11±7	10±7
∑ cat range	µeq L^{-1}	37-2736	30-5287	13-2601
∑ cat mean± σ	µeq L^{-1}	418±321	593±539	316±278
∑ an range	µeq L^{-1}	29-2606	22-5303	250102
∑ an mean± σ	µeq L^{-1}	377±304	545±523	279±265
∑ ca t- ∑ an range	µeq L^{-1}	258	263	225
∑ cat - ∑ an mean± σ	µeq L^{-1}	41±59	48±58	37±41
Slope β1	µeq $(mg\ C)^{-1}$	**6,80±0,16**	**5.04±0.25**	**4.17±0.11**
Intercept β0	µeq L^{-1}	**-12,32±1,63**	**-6.67±3.29**	**-5.01±1.32**
P-value		<0,0001	<0.0001	<0,0001
R^2		0.56	0.4	0.47

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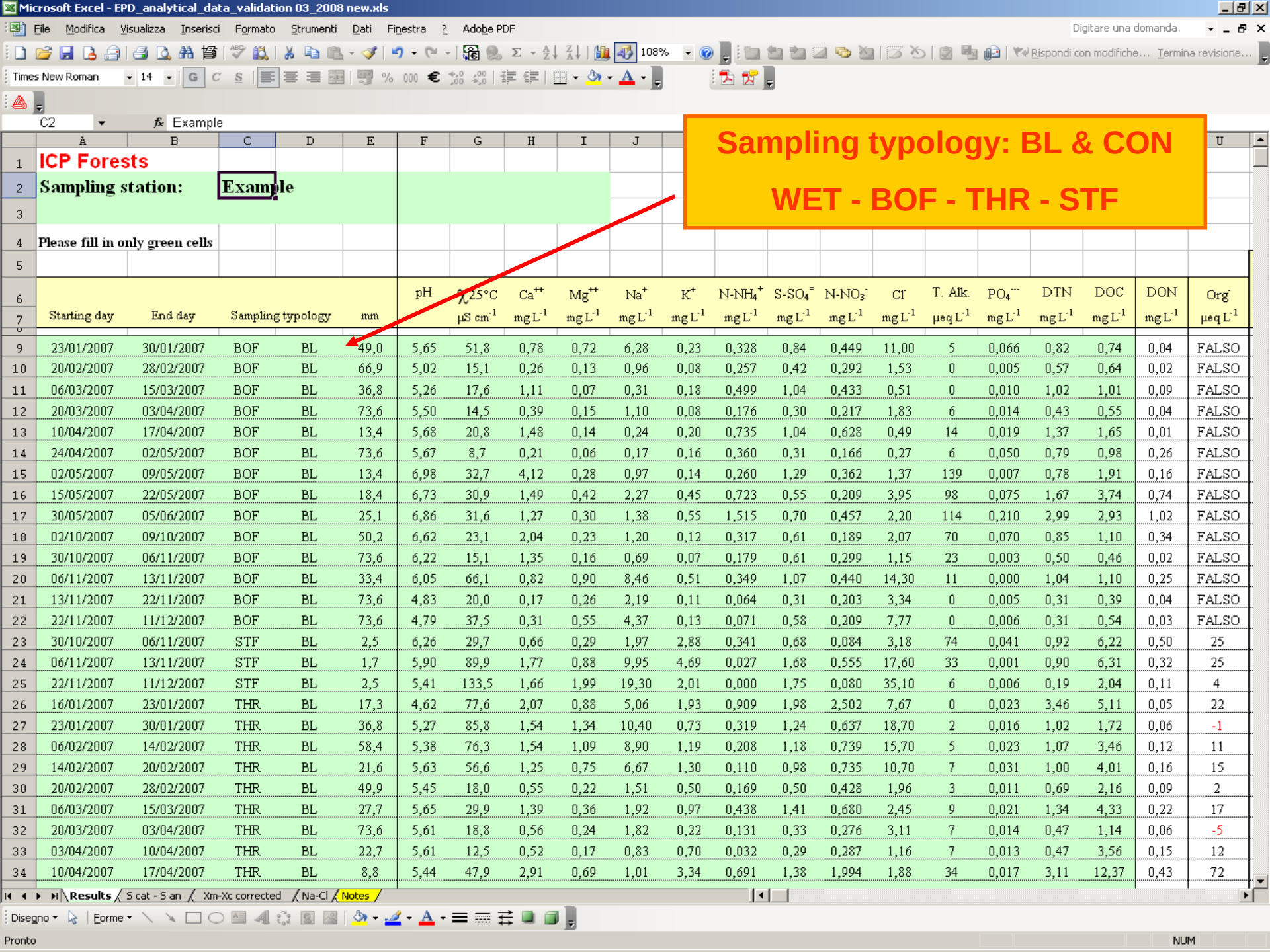


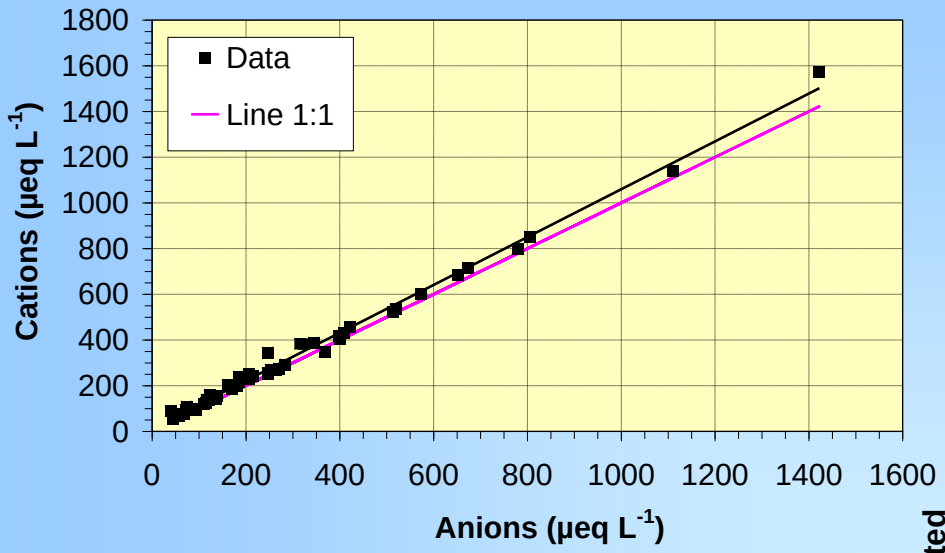
ICP Forests

Sampling station: Example

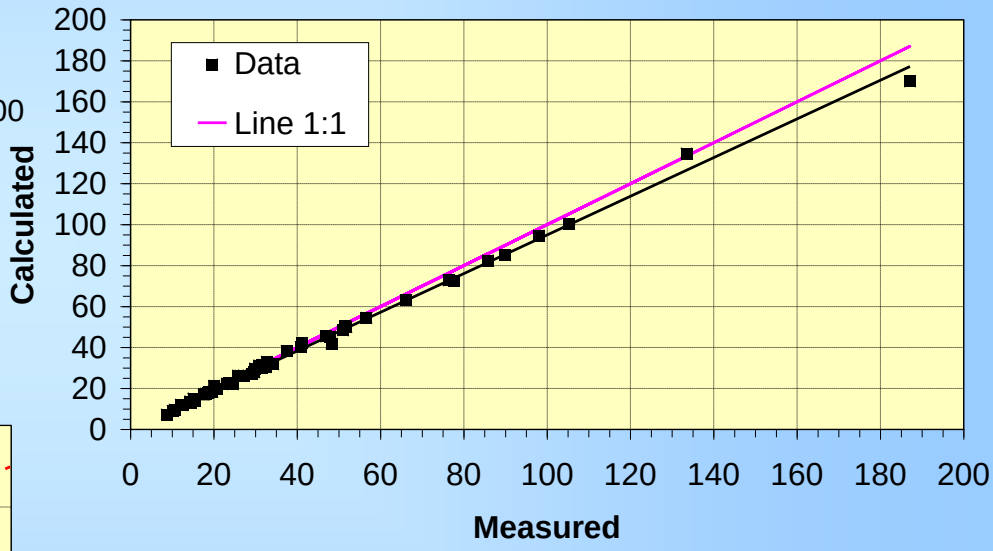
Please fill in only green cells

Starting day	End day	Sampling typology		mm	pH	χ 25°C	Ca^{++}	Mg^{++}	Na^{+}	K^{+}	$N\text{-}NH_4^{+}$	$S\text{-}SO_4^{=}$	$N\text{-}NO_3^{-}$	Cl^{-}	T. Alk.	PO_4^{---}	DTN	DOC	DON	Org
						µS cm^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	µeq L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	mg L^{-1}	µeq L^{-1}
23/01/2007	30/01/2007	BOF	BL	49,0	5,65	51,8	0,78	0,72	6,28	0,23	0,328	0,84	0,449	11,00	5	0,066	0,82	0,74	0,04	FALSO
20/02/2007	28/02/2007	BOF	BL	66,9	5,02	15,1	0,26	0,13	0,96	0,08	0,257	0,42	0,292	1,53	0	0,005	0,57	0,64	0,02	FALSO
06/03/2007	15/03/2007	BOF	BL	36,8	5,26	17,6	1,11	0,07	0,31	0,18	0,499	1,04	0,433	0,51	0	0,010	1,02	1,01	0,09	FALSO
20/03/2007	03/04/2007	BOF	BL	73,6	5,50	14,5	0,39	0,15	1,10	0,08	0,176	0,30	0,217	1,83	6	0,014	0,43	0,55	0,04	FALSO
10/04/2007	17/04/2007	BOF	BL	13,4	5,68	20,8	1,48	0,14	0,24	0,20	0,735	1,04	0,628	0,49	14	0,019	1,37	1,65	0,01	FALSO
24/04/2007	02/05/2007	BOF	BL	73,6	5,67	8,7	0,21	0,06	0,17	0,16	0,360	0,31	0,166	0,27	6	0,050	0,79	0,98	0,26	FALSO
02/05/2007	09/05/2007	BOF	BL	13,4	6,98	32,7	4,12	0,28	0,97	0,14	0,260	1,29	0,362	1,37	139	0,007	0,78	1,91	0,16	FALSO
15/05/2007	22/05/2007	BOF	BL	18,4	6,73	30,9	1,49	0,42	2,27	0,45	0,723	0,55	0,209	3,95	98	0,075	1,67	3,74	0,74	FALSO
30/05/2007	05/06/2007	BOF	BL	25,1	6,86	31,6	1,27	0,30	1,38	0,55	1,515	0,70	0,457	2,20	114	0,210	2,99	2,93	1,02	FALSO
02/10/2007	09/10/2007	BOF	BL	50,2	6,62	23,1	2,04	0,23	1,20	0,12	0,317	0,61	0,189	2,07	70	0,070	0,85	1,10	0,34	FALSO
30/10/2007	06/11/2007	BOF	BL	73,6	6,22	15,1	1,35	0,16	0,69	0,07	0,179	0,61	0,299	1,15	23	0,003	0,50	0,46	0,02	FALSO
06/11/2007	13/11/2007	BOF	BL	33,4	6,05	66,1	0,82	0,90	8,46	0,51	0,349	1,07	0,440	14,30	11	0,000	1,04	1,10	0,25	FALSO
13/11/2007	22/11/2007	BOF	BL	73,6	4,83	20,0	0,17	0,26	2,19	0,11	0,064	0,31	0,203	3,34	0	0,005	0,31	0,39	0,04	FALSO
22/11/2007	11/12/2007	BOF	BL	73,6	4,79	37,5	0,31	0,55	4,37	0,13	0,071	0,58	0,209	7,77	0	0,006	0,31	0,54	0,03	FALSO
30/10/2007	06/11/2007	STF	BL	2,5	6,26	29,7	0,66	0,29	1,97	2,88	0,341	0,68	0,084	3,18	74	0,041	0,92	6,22	0,50	25
06/11/2007	13/11/2007	STF	BL	1,7	5,90	89,9	1,77	0,88	9,95	4,69	0,027	1,68	0,555	17,60	33	0,001	0,90	6,31	0,32	25
22/11/2007	11/12/2007	STF	BL	2,5	5,41	133,5	1,66	1,99	19,30	2,01	0,000	1,75	0,080	35,10	6	0,006	0,19	2,04	0,11	4
16/01/2007	23/01/2007	THR	BL	17,3	4,62	77,6	2,07	0,88	5,06	1,93	0,909	1,98	2,502	7,67	0	0,023	3,46	5,11	0,05	22
23/01/2007	30/01/2007	THR	BL	36,8	5,27	85,8	1,54	1,34	10,40	0,73	0,319	1,24	0,637	18,70	2	0,016	1,02	1,72	0,06	-1
06/02/2007	14/02/2007	THR	BL	58,4	5,38	76,3	1,54	1,09	8,90	1,19	0,208	1,18	0,739	15,70	5	0,023	1,07	3,46	0,12	11
14/02/2007	20/02/2007	THR	BL	21,6	5,63	56,6	1,25	0,75	6,67	1,30	0,110	0,98	0,735	10,70	7	0,031	1,00	4,01	0,16	15
20/02/2007	28/02/2007	THR	BL	49,9	5,45	18,0	0,55	0,22	1,51	0,50	0,169	0,50	0,428	1,96	3	0,011	0,69	2,16	0,09	2
06/03/2007	15/03/2007	THR	BL	27,7	5,65	29,9	1,39	0,36	1,92	0,97	0,438	1,41	0,680	2,45	9	0,021	1,34	4,33	0,22	17
20/03/2007	03/04/2007	THR	BL	73,6	5,61	18,8	0,56	0,24	1,82	0,22	0,131	0,33	0,276	3,11	7	0,014	0,47	1,14	0,06	-5
03/04/2007	10/04/2007	THR	BL	22,7	5,61	12,5	0,52	0,17	0,83	0,70	0,032	0,29	0,287	1,16	7	0,013	0,47	3,56	0,15	12
10/04/2007	17/04/2007	THR	BL	8,8	5,44	47,9	2,91	0,69	1,01	3,34	0,691	1,38	1,994	1,88	34	0,017	3,11	12,37	0,43	72

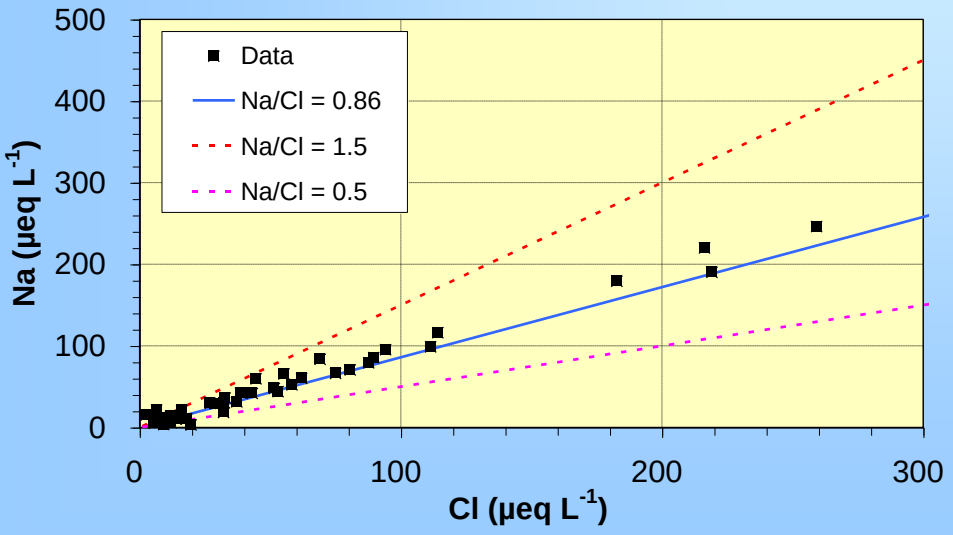
Ion balance



Conductivity (µS cm^{-1} 25°C)



Ratio Na/Cl

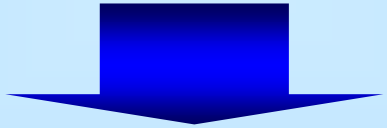


Conductivity:	higher than calculated	lower than calculated
Anions higher than cations	One or more cation(s) underestimated	One or more anion(s) overestimated
Anions lower than cations	One or more anion(s) underestimated	One or more cation(s) overestimated

$$TN = N\text{-}NO_3^- + N\text{-}NH_4^+ + (N\text{-}NO_2^-) + Org_N$$

$$Org_N = TN - N\text{-}NO_3^- - N\text{-}NH_4^+$$

The concentration of organic nitrogen can not be negative



$$TN - N\text{-}NO_3^- - N\text{-}NH_4^+ >= 0$$

Ionic balance (version 2011)

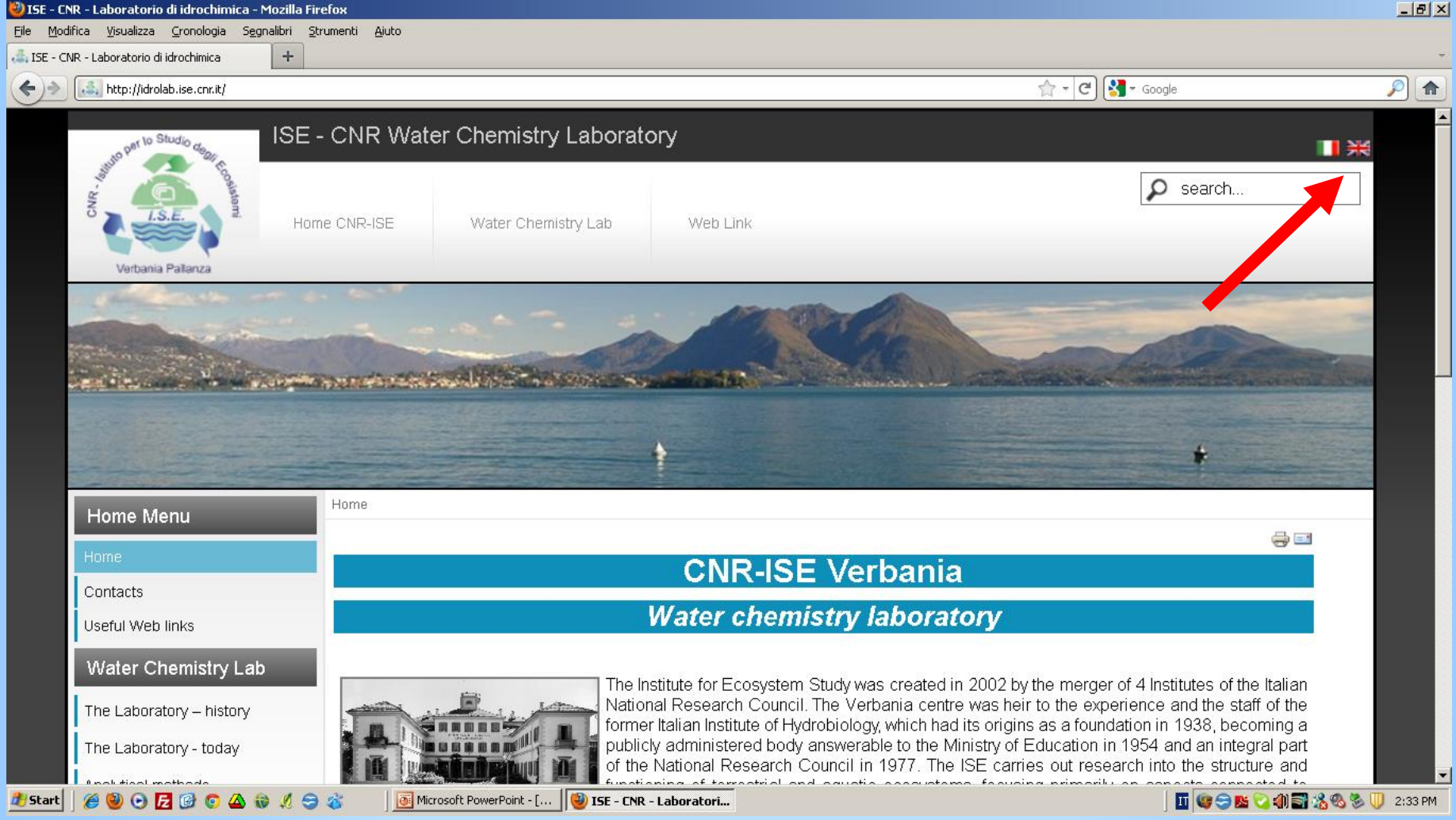
$$PD = 100 * \frac{(\Sigma \text{ cat} - \Sigma \text{ an})}{0.5\,(\Sigma \text{ cat} + \Sigma \text{ an})}$$

$$\Sigma_{\text{anions}} = \text{Alk} + [SO_4^{=}] + [NO_3^{-}] + [Cl^{-}] + [Org^{-}]$$

$$\Sigma_{\text{cations}} = [Ca^{++}] + [Mg^{++}] + [Na^{+}] + [K^{+}] + [H^{+}] + [NH_4^{+}] + [Al^{+++}] + [Fe^{+++}] + [Mg^{++}]$$

Applicability of validation tests to different type of solutions

	ion balance	conductivity	Na/Cl	nitrogen
wet-only	yes	yes	yes	yes
bulk open field	yes	yes	yes	yes
throughfall	no	yes	yes	yes
stemflow	no	yes	yes	yes
runoff	yes	yes	no	yes
soil water	yes	yes	no	yes



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CNR-ISE Verbania

Water chemistry laboratory

The Institute for Ecosystem Study was created in 2002 by the merger of 4 Institutes of the Italian National Research Council. The Verbania centre was heir to the experience and the staff of the former Italian Institute of Hydrobiology, which had its origins as a foundation in 1938, becoming a publicly administered body answerable to the Ministry of Education in 1954 and an integral part of the National Research Council in 1977. The ISE carries out research into the structure and functioning of terrestrial and aquatic ecosystems, focusing primarily on aspects connected to anthropogenic pressure and global changes. The knowledge gained provides the scientific basis for deciding on the most appropriate protective and corrective action to be taken, and supports the bodies responsible for formulating policies for the protection of the environment and the recovery of damaged environments.

Research activity is conducted in two main areas:

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Analytical Quality Control
http://idrolab.ise.cnr.it/index.php?option=com_content&view=article&id=60&Itemid=62&lang=en
Google

Analytical Quality Control

Since the seventies the laboratory has worked on perfecting a system of rigorous quality control of all the analytical methods used. The development of increasingly sophisticated equipment, allowing the generation of a large quantity of data, has inevitably led to greater focus on the quality of these data. In addition, the existence of monitoring networks active both nationally and internationally, involving many different laboratories, raise the question of the comparability of the results. Lastly, environmental monitoring performed to comply with national and European legislation makes it essential for participating laboratories to reach common quality standards.

However, analytical activity is only one phase of the measuring process. Sampling procedures also have a marked impact on the significance of the end results, i.e. on their representativity and the descriptive value of the system under examination. The most frequent errors are the non-representativity of the samples and the risk of contamination.The ultimate quality of the data is also affected by the methods of preservation and treatment of the samples (filtration, vial collection, homogenization, dilution, etc), the structure of the laboratory, the analytical staff and a number of other variables.

The demands of precision and accuracy have been a prime concern in the Institute from its inception, with great care being taken with the organisation of analyses in aspects relating to the structure of the laboratory as well as to actual analytical practice.

Since the 80s repeatability control charts have been applied to blanks, calibration standards, natural and synthetic samples, with comparison and optimisation of different analytical techniques and their limits of detection, and with regular participation in proficiency tests at national and international level. All this has made it possible to further optimise the analytical methods used in studying the temporal evolution of surface waters and atmospheric deposition

Currently, internal and external AQC are performed regularly in the water chemistry laboratory of the CNR-ISE of Verbania.

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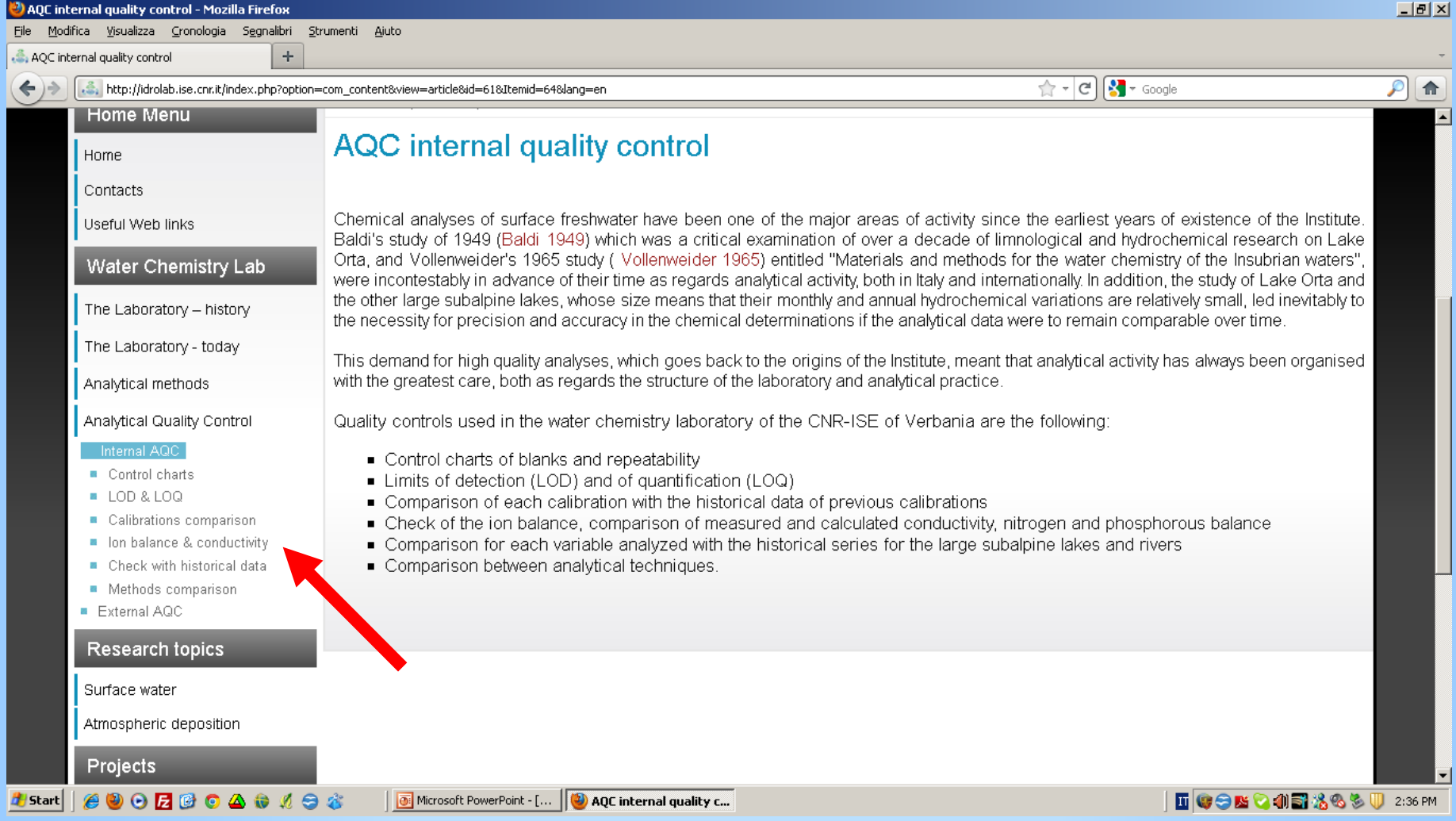
AQC internal quality control - Mozilla Firefox
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AQC internal quality control
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AQC internal quality control
Chemical analyses of surface freshwater have been one of the major areas of activity since the earliest years of existence of the Institute. Baldi's study of 1949 (Baldi 1949) which was a critical examination of over a decade of limnological and hydrochemical research on Lake Orta, and Vollenweider's 1965 study (Vollenweider 1965) entitled "Materials and methods for the water chemistry of the Insubrian waters", were incontestably in advance of their time as regards analytical activity, both in Italy and internationally. In addition, the study of Lake Orta and the other large subalpine lakes, whose size means that their monthly and annual hydrochemical variations are relatively small, led inevitably to the necessity for precision and accuracy in the chemical determinations if the analytical data were to remain comparable over time.
This demand for high quality analyses, which goes back to the origins of the Institute, meant that analytical activity has always been organised with the greatest care, both as regards the structure of the laboratory and analytical practice.
Quality controls used in the water chemistry laboratory of the CNR-ISE of Verbania are the following:
Control charts of blanks and repeatability
Limits of detection (LOD) and of quantification (LOQ)
Comparison of each calibration with the historical data of previous calibrations
Check of the ion balance, comparison of measured and calculated conductivity, nitrogen and phosphorous balance
Comparison for each variable analyzed with the historical series for the large subalpine lakes and rivers
Comparison between analytical techniques.
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Ion balance & conductivity - Mozilla Firefox

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Ion balance & conductivity

http://idrolab.ise.cnr.it/index.php?option=com_content&view=article&id=77&Itemid=81&lang=en

- Analytical methods
- Analytical Quality Control
  - Internal AQC
    - Control charts
    - LOD & LOQ
    - Calibrations comparison
    - Ion balance & conductivity
    - Check with historical data
    - Methods comparison
  - External AQC

Research topics

- Surface water
- Atmospheric deposition

Projects

- CONECOFOR
- FutMon
- ICP Waters
- Other projects

Publications

- Papers
- Reports

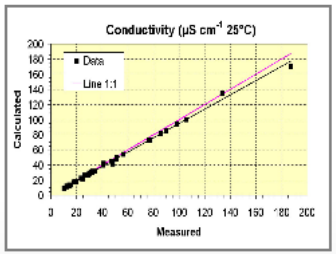
In the case of atmospheric deposition with a high organic content, a correction is used for the contribution of the organic anions calculated from the DOC and suggested in Mosello 2008.

Another check is the nitrogen and phosphorous balance, performed by checking that the total forms of phosphorous and nitrogen are higher than the respective sums of the inorganic forms.

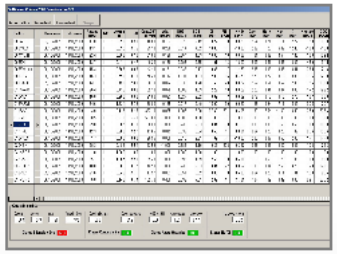
$$\text{Total P} > \text{P-PO}_4 \qquad \text{Total N} > (\text{N-NO}_3 + \text{N-NO}_2 + \text{N-NH}_4)$$

These validations of the results, performed immediately after the analyses have been performed (usually within 3-5 days of sampling), make it possible to re-analyze in a short time the samples that do not pass the quality control described above.

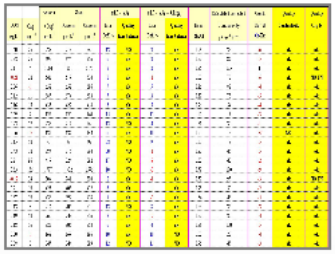
[Click on the images to enlarge]



Conductivity comparison



Data base for rain analysis with quality control



An example applied to rain:
Excel file downloadable from
http://www.icp-forests.org/WGqual_lab.htm
updated version June 2011
rain_analytical_data_validation_2011

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http://idrolab.ise.cnr.it/images/doc/analytical_data_validation_2011.xls