Experiences of the PT Provider

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Introduction

- Changes and Progress of participation
- Planning for the PT 2007 in Windhoek for the first time
- Sample preparation
- Sample distribution
- Evaluation

Changes and Progress in the number of participants

Country	2004	2005	2006	2007
Angola	**1**	**1**	**1**	**0**
Botswana	**2**	**2**	**2**	**4**
Ethiopia	**1**	**1**	**1**	**0**
Kenya	**2**	**2**	**4**	**3**
Lesotho	**1**	**1**	**0**	**1**
Madagascar	**0**	**0**	**2**	**2**
Malawi	**2**	**2**	**2**	**3**
Mauritius	**1**	**3**	**4**	**3**
Mozambique	**2**	**3**	**2**	**0**

Changes and Progress in the number of participants

Country	2004	2005	2006	2007
Namibia	2	2	3	3
Republic of Seychelles	1	2	2	1
Swaziland	1	1	0	1
Tanzania	2	8	5	12
Uganda	1	3	6	5
Zambia	1	4	2	3
Zimbabwe	2	3	3	5
Number of labs participating	**22**	**44**	**39**	**46**

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Changes and Progress Parameters

2004		2005		2006		2007	
Anions	**Cations**	**Anions**	**Cations**	**Anions**	**Cations**	**Anions**	**Cations**
SO4	Ca	SO4	Ca	SO4	Ca	**SO4**	**Ca**
Cl	Mg	Cl	Mg	Cl	Mg	**Cl**	**Mg**
F	Na	F	Na	F	Na	**F**	**Na**
NO3	K	NO3	K	NO3	K	**NO3**	**K**
	Fe	**PO4**	Fe	PO4	Fe	**PO4**	**Fe**
	Mn		Mn		Mn		**Mn**
	Al		Al		Al		**Al**
			Pb		Pb		**Pb**
			Cu		Cu		**Cu**
			Zn		Zn		**Zn**
			Cr		Cr		**Cr**
			Ni		Ni		**Ni**
					As		**As**
					Cd		**Cd**
Total	**11**		**17**		**19**		**19**

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Planning

- Calculation of the target values (masses and volumes)
- Ensure the timorously delivering of requires chemicals (8 weeks) AR / GR grade chemicals, supplied by Merck, Sigma-Aldrich and Strem chemicals were used. Copies of the certificate of analysis are available.
- Ensure enough samples one liter bottles, crates, enough 50 ml beakers, 200 ml beakers and 500ml volumetric flasks,
- 100 liter containers with tap
- Ensure availability of packaging material (boxes, shredded paper, packaging tape, labels, envelopes, paper)

Planning

- Quotations and choice of courier
- Availability and suitability of balances for the different weighings
  - Analytical balance : wires and the salts
  - Top loader : Stock solutions and the 200g weighing
  - 50 kg top loader : Weighing of the final batches - Problem

Sample bottle preparation I

- Bottles were first to arrive
- Wash all 300 bottles which was ordered
- Planned for 50 participants
- Bottles were rinse twice with deionised water
- Bottles & caps were put in the oven @ 60 °C overnight

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Sample bottle preparation II

- Next day – check completely dry
- Closed bottles immediately to prevent them from dust
- Store them in the crates until needed

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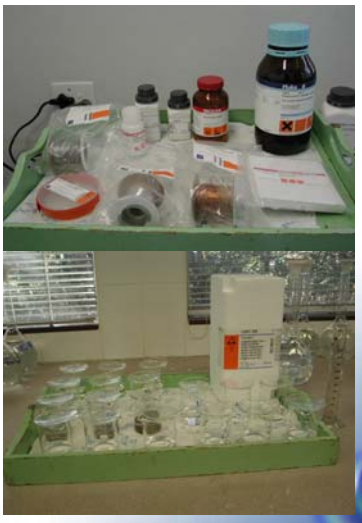
Weighings of wires

- Start of by weighing the different target masses for the 3 levels of each parameter
- Continue with the weighings of the metals where different wires were used



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Calculated Sample mass - cations

Parameter	Chemical	Purity %	Level 1	Level 2	Level 3
Calcium	CaCl2.2H2O	99.5	7.2911	13.7358	23.0648
Magnesium	Mg(NO3)2.6 H2O	99.5	27.006	41.4963	72.8506
Sodium	NaCl	99.6	8.0412	12.6016	18.5693
Potassium	KCl	99.6	2.2736	2.9922	4.4514
Iron	Fe-Wire	99.95	0.1100	0.2034	0.3156
Manganese	Mn-Powder	99.4	0.1061	0.1328	0.2637
Aluminium	Al-wire	99.9995	0.1134	0.1560	0.3222
Lead	Pb(NO3)2	99.7	0.1409	0.1905	0.3811
Copper	Cu-wire	99.999	0.1188	0.2380	0.3947
Zinc	Zn-wire	99.99995	0.138	0.2694	0.5663
Chromium	CrCl3.6 H2O	99	0.2688	0.5554	0.9795
Nickel	Ni-wire	99.9975	0.3649	0.2428	0.3244
Arsenic	As2O3	99.50	0.1853	0.3834	0.1256
Cadmium	CdCl2	99.995	1.1845	0.4688	0.1728

Sample 4, 5 and 6 were constituted as follows with HNO3 acid preservation to a pH 2.1. The samples matrix was pure water. The final weight for the cation samples was 57.08g with the Density (Deionised water) = 0.998g/ml and the temperature 24 ºC.

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Traceability of the weighings

- Since traceability is very important in a PT scheme
- Biggest problem for 2007 - Balances had no possibility for a printer connection
- Tried various option to borrow a balance – without success

Documentation of wires

- Solution for the problem - Pictures were taken of all the weighings with a digital camera
- Pictures were downloaded, printed and cut out
- Paste it next to the written weighing for proof of the traceability

Digestion of metals

- Digestion of the pure metals e.g.
- Water and HNO3 acid was added for digestion / As 32 % NaOH
- Left on a hot plate at very low temperature setting until the metals were completely dissolved

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Weighing of the salts

- Continue with weighing of the salts
- Weigh the substances for three levels
- Continue to prepare the stock solution

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Calculated Sample mass - Anions

Parameter	Chemical	Purity %	Level 1	Level 2	Level 3
Sulphate	K2SO4	99.5	7.0676	10.3072	13.6371
Chloride	KCl	99.6	11.0492	13.5912	17.365
Fluoride	KF	100	0.2000	0.3404	0.5938
Nitrate	KNO3	99.3	3.1201	7.2868	12.3361
Phosphate	KH2PO4	99.9	1.5061	2.9053	3.6030

Sample 1, 2 and 3 were constituted as follows without acid preservation. The sample matrix was pure water. The final weight for the cation samples was 57.08g with the Density (Deionised water) = 0.998 g/ml and the temperature 24 ºC.

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Preparation of stock solutions

- Fill the 500 ml volumetric flask by weight
- Wash accurately into a 500ml volumetric flask
- Repeat for all the parameters

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Documentation of Stock solutions

- Pictures were again taken of all the weighings with a digital camera
- Downloaded, printed and cut out
- Paste next to the written weighing for proof of the traceability

Labeling of the bottles

- Prepare labels for each sample bottle with a short description of the information
- Print labels
- Stick on the samples bottles for identification of the samples
- Put sellotape over the labels – to protect the labels
- Bottles were ready for the filling process

Preparation of final batches

- Obtain a suitable balance
- Find a suitable container
- Made special rack for the stirrer in order to mix the samples properly

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Preparations for the 200g weighings

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Preparation of the 200g weighings

- Weigh the empty container
- Weigh the calculated amount of the different stock solutions with the density taken into consideration
- Add some water into the big container
- Add the calculated amount of the stock solution (by weight)
- Rinse over in the 100 l container
- Fill by weight

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Preparation of final batch

- 50 liters of each sample were prepared
- Pure water spiked with parameter of interest
- Nitric acid was added to the cations for preservation (pH 2)

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pH adjustment

- Stirring took place for continuously during the process
- Filled by weight
- Final stirring for 15 minutes
- Document the pH

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Homogeneity

- All analytes were physically dissolved
- Proper stirring ensure the homogeneity of the samples
- Conductivity check on the first samples and the last samples – basically NO difference
- Documentation

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Documentation of information of batch

- All the readings of the balance were once again downloaded, cut out and pasted next to the weighings
- The weigings of the final batch was also documented
- pH and temperature were documented

Samples dispensing

- Samples bottles were filled after each batch
- Put in the crate
- Tank was washed properly in between the batches
- Start to prepare for the next batch

Storing

- Space was limited in the fridge
- Crates were very handy – stacked all the samples
- All samples were stored at 4 ° C until all six batches were prepared

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Packaging

- Strong packaging was once again a requirement
- Flat cartons needed to be fold into boxes
- Staple it together

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Preparation of the documentation

- Hard copies of the forms for the results and the method information were included in each box
- Labels of all the participants were prepared

Packaging of the samples

Packaging of the samples

- Packed six polyethylene bottles into each box
- Shredded paper was used for the packaging material
- sealed with packaging tape

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Packaging of the samples

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Packaging of the samples

Packaging

Ready for pick up

- Samples ready to be picked up by the courier for distribution to the local coordinators

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Loading

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Information to courier

- Supplied the correct address list of the local distributors to the courier with the total weight of one parcel
  - Determine the weight of bottle filled with deionised water
  - Determine the weight of empty box
  - Determine the weight of envelope filled with documentation

Loading completed

Shipment

- The courier was Kuehne & Nagel in Namibia
- Participants were notified by e-mail to inform them that the samples are on their way

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Shipment

- All samples were shipped to the address of the local distributor.
- Samples were delivered with a lot of frustration and problems and the PT deadline needed to be change for some of the laboratories
- No leakage problems were reported

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Evaluation

- Results were received by fax or e-mail
- Deadline was extended on request because of courier problems
- The last results were received on the 04th October 2007
- Angola informed that they experience problems with the samples and the customs clearance.
- Lesotho also informed me that they experince customs problems.

Evaluation

- Results were typed into a spreadsheet
- Copied and paste into different parameter files
- All the files were created for the different laboratories in Excel
- Excel files were converted to a pdf format to reduce the size of the file and to ensure all the participants will be able to read the file.
- Precision tests were run on the balances
- Measurement uncertainty was taken in consideration according to the method told by Angelique in the 2005 workshop

Payment

- Payments were made by bank drafts, transfers and cheques
- Some payments were made, but the money is still outstanding
- NamWater still experiences problems to identify the payments within NamWater due to insufficient information from bank/participant
- Some payments were not yet made at all

Successes of 4rd PT

- Increased and continued enthusiasm - Tanzania was the country with most participants !
- Local distributors are very important and I very helpful and reliable specially with the courier problems
- Five form 51 laboratories did not submit results (3 due to courier problems)

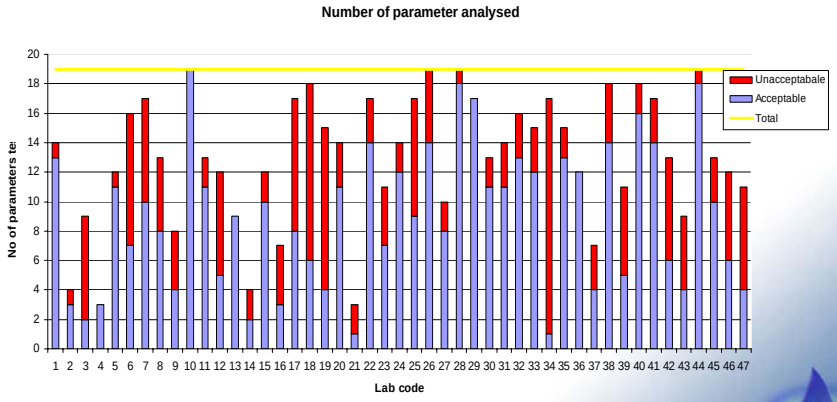
Confidentiality

- Confidentiality was once again very important
- PT round require a high degree of confidentiality from the provider
- Lab codes were changed
- It is also the responsibility of everybody involved to keep all the data and items of information relating to inter-laboratory confidential

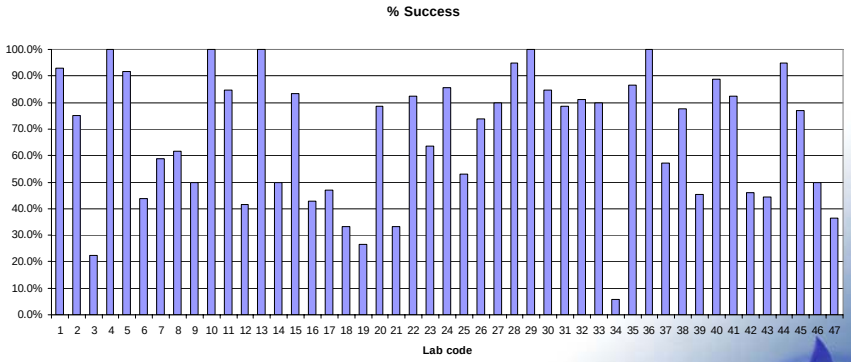
Conclusions

- Participation is an important and a valuable tool for a laboratory to uncover errors and improve on their performance
- Valuable method of quality control where suitable reference materials are not available
- The performance certificate can be used to proof competence in in the testing field
- It is a regular, external and independent check on the data quality of the laboratory

Number of parameters analyzed



% Success



Challenges for 2008

- The results should be used as a motivation to improve performance and apply corrective actions if necessary
- Strive to improve the success
- Increase the number of analyzed parameters
- Reporting of results again caused problems with incorrect units (e.g as N and not NO3 and as P and not PO4
- Try and rectify the analyses not determined due to a lack of chemicals or problems with equipment
- Instrumentation or method should be stipulated clearly
- Once again very high standard deviations in the 2007 PT scheme

Problems experienced

- Dedicated time for the preparation and evaluation period without interruptions
- The PT provider had a limited number of staff
- Contract with local electricity supplier - results to be reported by 12h00 every day
- Accreditation requirement for NamWater laboratory was delayed
- Late confirmations and requests of participation caused problems and unnecessary rearrangements with the courier
- The initial return date for the results was set as the 31st of August 2007 with an extension of three weeks for some of the laboratories due to transportation problems. Five laboratories did not submit results at all.
- Follow-up of participation where people did not respond on e-mails

Problems

- Late submitting of results due to courier problems delay the submitting of the evaluation report
- Receipt of results by fax – unclear and difficult to get hold of the participant
- Five labs did submit results at all for unknown reasons
- Three labs did not take part due to courier problems

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PTB Donation

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Thank you

- **PTB**
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- Margaret Ngobeni
- **University of Stuttgart**
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- **Participants**