

Preparation and testing of the pure water samples

- Paper prepared for the 'Meeting of the Euramet Project 924' at Physikalisch-Technische Bundesanstalt (PTB) on April , 9 -10, 2008
- Including preliminary studies and work for the CCQM P100.1 for mercury (target content = 50 ng Hg/L) and the Euramet 924 project for cadmium, lead and nickel (target contents = 0,2 µg Cd/L; 7,2 µg Pb/L; 20 µg Ni/L) without any matrix elements on the basis of the **WFD 2000/60/EC**
- The way to get stable test samples for the above elements in the shown concentrations after dividing task in the two separate projects is shown by a project of the Bundesanstalt für Materialforschung und –prüfung (BAM) I.1 under the supervision of Dr. Ralf Matschat – the staff: Dr. Holger Scharf, Dr. Jürgen Birkhahn, Dr. Michael Czerwensky, Mrs. Heike Witthuhn, Mrs. Susanne Just, supported by other BAM coworkers in some special tasks

Overview

- Selection of possible vessel materials for storage of the special test samples
- Purification of the vessels
- Required chemicals
- Schemes for gravimetric control for all steps of preparation
- Test preparation for the stability check
- Organisation of the measurements
- Schemes of calibration and first experience with the new AFS method and spectrometer
- Results of the stability tests
- Resumes and conclusions

Material for the sample preparation

- **500 mL Duran-bottles** were selected for CCQM P100.1 (Hg).
- Basics:
 - it was aimed for sample volumes as big as possible in view of possible enrichment procedures,
 - on the other hand 1 L-bottles were regarded as too big in view of their shipping.
 - PFA-bottles of these volumes seemed to too high priced;
 - and on the other hand Duran (borosilicate glass) is the best convenient material for a low-loss storage of low concentrated mercury solutions (based on literature references).
- Because of the high tare weights of the empty bottles, and also of the final masses after filling them with 500 mL, a coarser balance (here a precision balance up to 1,2 kg with a readability of 0,001 g) was selected. Difficulties with the wind deflector (the customary part is too small, all bottles and volumetric vessels of about 500 mL and more are taller than the deflector) could be overcome using a self-made glass construction of a wind deflector with an appropriate height.

Material for the sample preparation

- **100 mL PFA-bottles** were selected for Euramet 924 (Cd, Pb, Ni).
- A priori one has to expect difficulties during the weighings on normal equipped analytical balances, because the bottles have to be strictly dry before the gravimetical procedures for the preparation can be done with an acceptable tara value. Because of this, antistatic weighing conditions are to realise (antistatic seal with wristband, ion shower for elimination of charged surfaces, effective earthing of the body of the balance).

Purification of the bottles

- Acid mixture = 200 mL HNO₃ suprapur + 166 mL HCl suprapur in 2,5 L Milli-Q – water
- the bottles were completely filled with this mixture and leached over a time period of minimum 14 days (4 weeks are better); for finishing the procedure, the bottles were hand-shaken several times with an adequate amount of MilliQ-water and after this filled with water of the same quality for 3 days of repeated leaching – this last procedure was repeated 4 x
- after purification: drying took place under „laminar flow“ – conditions (laminar flow hood).
After drying each bottle was screwed tightly closed and stored in a foil bag made of PE (with ZIP-lock) till the next preparation step.

BrCl --- preparation

- from USEPA Method 1631, Revision E - August 2002
“Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry”
- transformation to the laboratory conditions in BAM working group I.14
 - Approach for 100 mL of the BrCl solution
- KBr and KBrO₃ as pure as possible (min. p.a.), main focus is to guarantee a low Hg-content, - heat in a drying oven at 250°C (for about 1 to 2 h)
- HCl (30 to 37%, suprapur, if not, another possibility is: p.a.)– if any doubt concerning sufficient low Hg blank (in spite of possibly declared low Hg-content or possible faulty lots [what also might happen with products of renowned producers!])
⇒ decrease the Hg content, bubble the acid for at least 15 minutes with argon, nitrogen or air
- 1,08 g KBr are solved in conc. HCl (suprapur) (ca. 40 mL) – with a stirrer for about 1 h
- 1,52 g KBrO₃ are added very slowly while stirring – the colour of the solution changes from yellow to red (reddish) to orange – further stirring for about 1 h is needed – meanwhile filling the vessel with the conc. HCl - before the vessel is closed (otherwise it is possible to get high pressure caused by outgassing of halogene)

BrCl --- Usage

- Addition of this BrCl-solution for stabilisation of the Hg-content in aqueous solutions:
 - ➤ for clear water and filtered samples = addition of 0,5 mL BrCl to 100 mL sample volume
 - ➤ for brownish water and unclear samples = addition of 1,0 mL BrCl to 100 mL sample volume
- Let react for at least 30 min at room temperature, or, to reduce the reaction time, warm to about 50°C, in the case of early fading out of the yellow colour, add the same amount of reagent (sample solution should stay yellow until the next step);
- This kind of stabilisation shall be enough for 7 days in adequate container materials (e.g. borosilicate glass, quartz, PTFE, FEP, PFA --- PE is absolutely improper!!!)

L-Ascorbic acid solution as reducing agent

- before Hg-determination, an addition of 500 μL ^{1*} of a 10% L-ascorbic acid solution (or 200 mg of the solid substance) per 100 mL sample solution is needed, for removing an excess of Br, the yellow colour of BrCl will disappear (otherwise the reduction reaction with tin(II)-chloride will be interfered)

References for a usage of ascorbic acid: DIN EN 13506 from April 2002 and furthermore in the draft of DIN EN ISO 17852 from July 2007 „Water quality – determination of mercury – method using atomic fluorescence spectrometry; German version prEN ISO 17852:2007“)

- ^{1*} better is an addition of 1 mL (according to DIN recommendations)

$\text{NH}_2\text{OH}\cdot\text{HCl}$ as reducing agent

- Hydroxylamine hydrochloride — Dissolve 300 g of $\text{NH}_2\text{OH}\cdot\text{HCl}$ in reagent water and bring to 1.0 L. This solution may be purified by the addition of 1.0 mL of SnCl_2 solution and purging overnight at 500 mL/min with Hg-free N_2 . Flow injection systems may require the use of less SnCl_2 for the purification of this solution.
- Add 0.2-0.25 mL of NH_2OH solution to the BrCl -oxidized sample in the 125-mL sample bottle. Cap the bottle and swirl the sample. The yellow colour will disappear, indicating the destruction of the BrCl . Allow the sample to react for 5 min with periodic swirling to be sure that no traces of halogens remain.
- (Source: EPA Method 1631, Revision E - August 2002)
- **But in this reaction a nitrogen generation is possible and this has a quenching effect to the determination of Hg with the AFS method**

Mercury stock, working and spike solutions

- Selected material: mercury (99,999 %), liquid; Heraeus, 009259
- Stock was prepared at 09/ 27/1995 (directly in a 250 mL volumetric flask)
 - metal drops are solved in 50 mL 1:1 HNO₃ subboiling quality (s.b.)
 - the flask is then filled with (14 mL HNO₃ s.b. in 200 mL water) to the mark

$$m_E (\text{Hg}) = 5,5505 \text{ g (solution named Hg/001)}$$

- 2-step (each 1:100) dilution of this stock solution, this 1:10,000 diluted working solution had a analyte mass of

$$m_W (\text{Hg}) = 2,17809 \cdot 10^{-6} \text{ g}$$

- Preparation of the spike solution by adding 5,5 mL of the working solution to a 500 mL volumetric flask and filling up at 11/ 27/2006 resulting in a concentration of

$$m_S (\text{Hg}) = 24,14 \mu\text{g/kg resp. } 24,52 \mu\text{g/L}$$

this spike solution has the density of 0,99916 g/cm³ at 20°C

Bottle preparation before spiking

- Prefilling of the bottles (gravimetric control) with 499 mL of the mixture - consisting of 2,5 mL BrCl-solution (2,5 mL with Eppendorf) and 496,5 mL MilliQ-water - (all weighing results were put in an Excel-sheet)
- The procedure of supplying water was made under normal laboratory conditions [Note: without any contamination protection] by 5 times pipetting of a 100 mL volume of water into a beaker (graduated, to have control of counting the repeats); then 3,5 mL (2,5 mL + 1 mL) were repipetted to give 496,5 mL volume in the beaker [Note: 2,5 mL for the following addition of the BrCl-solution and 1 mL for later addition of spike solutions (Hg resp. Blank water)].

After this, the beaker content of 496,5 mL water volume was given to the 500 mL-borosilicate-test-bottle. With this procedure a real risk of contamination from the laboratory air is existent. This fact was tolerated as a small contamination risk of a nearly constant amount of Hg which was regarded as being without any relevance for the stability tests.

- Note: This circumstantial procedure was selected, because at that time the assumed contamination risk of an available Metrohm-Diluter wasn't clear. At later preparation of the CCQM test samples this diluter was used as a tool free from any contamination.

- After the addition of water to the bottles, the 2,5 mL BrCl-solution (2,5 mL with Eppendorf) was added to the sum of 499 mL.

Table 1: Protocol of the test bottle preparation (brutto values);
weighings carried out at analytical balance Sartorius LC1200S

	Sample- Name	Bottle type	Mass [dry, empty]	Mass after addition 2,5 mL BrCl- solution	Mass after addition 496,5 mL MilliQ- water	Mass after addition 1000 µL of spike-solution
Bottle 01	HgBW1	500 mL- glass bottle	350,705	353,617	847,641	848,636
Bottle 02	HgStab01	500 mL-glassb.	350,367	353,245	847,235	848,228
Bottle 03	HgStab02	500 mL-glassb.	351,692	354,604	848,541	849,536
Bottle 04	HgStab03	500 mL-glassb.	350,161	353,077	847,008	848,001
Bottle 05	HgStab04	500 mL-glassb.	351,63	354,528	848,473	849,465
Bottle 06	HgBW2	500 mL-glassb.	350,888	353,801	847,784	848,779
Bottle 07	HgStab05	500 mL-glassb.	351,589	354,495	848,435	849,428
Bottle 08	HgStab06	500 mL-glassb.	351,869	354,786	848,728	849,723
Bottle 09	HgStab07	500 mL-glassb.	350,615	353,531	847,49	848,488
Bottle 10	HgStab08	500 mL-glassb.	350,886	353,806	847,662	848,656
Bottle 11	HgBW3	500 mL-glassb.	350,509	353,426	847,673	848,671
Bottle 12	HgStab09	500 mL-glassb.	351,88	354,799	848,731	849,725
Bottle 13	HgStab10	500 mL-glassb.	352,381	355,293	849,24	850,236
Bottle 14	HgStab11	500 mL-glassb.	351,399	354,315	848,273	849,267
Bottle 15	HgStab12	500 mL-glassb.	352,485	355,4	849,432	850,424

Dispenser calibration

Tables 2 + 3 show the measuring results of the gravimetric control of the piston stroke dispenser Hamilton MicroLab1000 in the scale 1000 μL

- (Tabelle 2): with MilliQ-water on Sartorius AC210S respectively
 - (Tabelle 3): with the prepared Hg-spike solution from the 500 mL-volumetric flask for the prefilled glass bottles on Sartorius LC1200S
- (subsequent the used symbols)

M	= mean value of n repeated measurements
s	= standard deviation of n repeated measurements
RSD%	= relative standard deviation of n repeated measurements = VK = Unprecision $cv [\%] = s : M * 100\%$
VB/2	= half bandwidth of the confidence interval for a probability of error of 5%
n	= number of the repeated measurements
d	= incorrectness $[\%] d = (M - TARGET) : TARGET * 100\%$
F [%]	= margin of error DIN 12650 $[\%] F[\%] = d + 2 * cv$
F [μL]	= margin of error DIN 12650 $[\mu\text{L}] F[\mu\text{L}] = F[\%] * M : 100$
Z	= correction factor Z $[\mu\text{L}/\text{mg}]$ is a proportion for the dependence of the buoyant force of the weighing from temperature-, air pressure- and air moisture, divided by the density of dest. waters at the measuring temperature

Table 2

Test parameter		Weighing the delivered volume (of MilliQ water) of the piston stroke dispenser MicroLab1000 adjusted to 1 mL ; the volumetric dosing tip is above the solution						
		Nr.	mH ₂ O [g]	mH ₂ O,corr [g]	V _{corr,T°C} [mL]	V _{corr,20°C} [mL]	ΔV _{Ref-Real} [mL]	ΔV _{Ref-Real} [%]
User	Jürgen Birkhahn	1	0,9893	0,9903	0,9928	0,9928	0,0072	0,7187
Organisation unit	BAM I.14	2	0,9931	0,9941	0,9966	0,9966	0,0034	0,3374
Date of testing	11/27/2006	3	0,9938	0,9948	0,9973	0,9973	0,0027	0,2671
Notations	syringe A = 1 mL	4	0,9939	0,9949	0,9974	0,9974	0,0026	0,2571
Identification	method 2	5	0,9937	0,9947	0,9972	0,9972	0,0028	0,2772
Target-Volume [mL]	1	6	0,9939	0,9949	0,9974	0,9974	0,0026	0,2571
Max. error (produc.) [μL]	6	7	0,9938	0,9948	0,9973	0,9973	0,0027	0,2671
Reference temperature [°C]	20	8	0,9937	0,9947	0,9972	0,9972	0,0028	0,2772
Real - temperature t [°C]	23,2	9	0,9936	0,9946	0,9971	0,9971	0,0029	0,2872
Density (H ₂ O), 20°C [g/mL]	0,9982	10	0,9938	0,9948	0,9973	0,9973	0,0027	0,2671
Density (H ₂ O), t°C [g/mL]	0,9975	M	0,9933	0,9943	0,9968	0,9968	0,0032	0,3213
Air pressure [mbar]	1022,582808	s	0,0014	0,0014	0,0014	0,0014		
Relative air moisture [%]	30	RSD%	0,1420	0,1420	0,1420	0,1420		
Tube material	FEP	VB/2	0,0010	0,0010	0,0010	0,0010		
Expansion coeff. [1/K]	0,0000032	n	10	10	10	10		
Producer	Hamilton/CH	d				-0,3213		
		F [%]				-0,0373		
		F [μL]				-0,0004		
		Z				1,0035611		

Table 3

Test parameter		Weighing the delivered volume (of Hg-spike-solution) of the piston stroke dispenser MicroLab1000 adjusted to 1 mL ; the volumetric dosing tip is above the solution						
		Nr.	m _{H2O} [g]	m _{H2O,corr} [g]	V _{corr,T°C} [mL]	V _{corr,20°C} [mL]	ΔV _{Ref-Real} [mL]	ΔV _{Ref-Real} [%]
User	Jürgen Birkhahn	1	0,9909	0,9919	0,9944	0,9944	0,0056	0,5581
Organisation unit	BAM I.14	2	0,9954	0,9964	0,9989	0,9989	0,0011	0,1065
Date of testing	11/27/2006	3	0,9955	0,9965	0,9990	0,9990	0,0010	0,0965
Notations	Syringe A = 1 mL	4	0,9960	0,9970	0,9995	0,9995	0,0005	0,0463
Identification	method 2	5	0,9954	0,9964	0,9989	0,9989	0,0011	0,1065
Target-Volume [mL]	1	6	0,9922	0,9932	0,9957	0,9957	0,0043	0,4277
Max. error (produc.) [μL]	6	7	0,9946	0,9956	0,9981	0,9981	0,0019	0,1868
Reference temperature [°C]	20	8	0,9954	0,9964	0,9989	0,9989	0,0011	0,1065
Real - temperature t [°C]	23,2	9	0,9944	0,9954	0,9979	0,9979	0,0021	0,2069
Density (H ₂ O), 20°C [g/mL]	0,9982	10	0,9916	0,9926	0,9951	0,9951	0,0049	0,4879
Density (H ₂ O), t°C [g/mL]	0,9975	M	0,9941	0,9952	0,9977	0,9977	0,0023	0,2330
Air pressure [mbar]	1022,582808	s	0,0019	0,0019	0,0019	0,0019		
Relative air moisture [%]	30	RSD%	0,1869	0,1869	0,1869	0,1869		
Tube material	FEP	VB/2	0,0013	0,0013	0,0013	0,0013		
Expansion coeff. [1/K]	0,0000032	n	10	10	10	10		
Producer	Hamilton/CH	d				-0,2330		
		F [%]				0,1408		
		F [μL]				0,0014		
		Z				1,0035611		

Table 4: Protocol of the test bottle preparation for the stability test (net values)

	Sample-Name	Mass [dry,empty]	Mass of addition 2,5 mL BrCl-solution	Mass of addition 496,5 mL MilliQ-water	Mass of addition 1000 µL of spike-solution	Mass of addition 1000 µL of Blank-water	Mass [total]
Bottle 01	HgBW1	350,705	2,912	494,024		0,995	848,636
Bottle 02	HgStab01	350,367	2,878	493,99	0,993		848,228
Bottle 03	HgStab02	351,692	2,912	493,937	0,995		849,536
Bottle 04	HgStab03	350,161	2,916	493,931	0,993		848,001
Bottle 05	HgStab04	351,63	2,898	493,945	0,992		849,465
Bottle 06	HgBW2	350,888	2,913	493,983		0,995	848,779
Bottle 07	HgStab05	351,589	2,906	493,94	0,993		849,428
Bottle 08	HgStab06	351,869	2,917	493,942	0,995		849,723
Bottle 09	HgStab07	350,615	2,916	493,959	0,998		848,488
Bottle 10	HgStab08	350,886	2,92	493,856	0,994		848,656
Bottle 11	HgBW3	350,509	2,917	494,247		0,998	848,671
Bottle 12	HgStab09	351,88	2,919	493,932	0,994		849,725
Bottle 13	HgStab10	352,381	2,912	493,947	0,996		850,236
Bottle 14	HgStab11	351,399	2,916	493,958	0,994		849,267
Bottle 15	HgStab12	352,485	2,915	494,032	0,992		850,424
Mean value:		351,2704	2,91113333	493,974867	0,99408333		849,1508 67

Hg-Content of spike and test solutions

- From this calculation the mean Hg content of the spike solution for the test bottles results in
= 24,43 ng Hg per added mL
- And this lead to a mean Hg content of the prepared test bottles of
= 49,04 ng Hg/L
- [this is demonstrated in the next table 5]

The measurements with the AFS-apparatus ‚Mercur‘ were carried out on the same basis, since all calibration standards were also prepared volumetrically.

From this point of view the value above was the basic value for the beginning of the stability studies.

Table 5: Documentation of the relatively small fluctuations of the finished preparation

Sample-Name	Content of test-sample ng Hg / L (single-sample)	Fill-Volume of the test-bottles (calculated from the mean density = 0,999132917 g/mL) [mL]	Fill-Volume for blank-samples (calculated from the mean density of the water-spiking with MicroLab1000 = 0,996786931 g/mL) [mL]
HgBW1	0		499,536044
HgStab01	49,0371356	498,293062	
HgStab02	49,0388101	498,276047	
HgStab03	49,0392041	498,272043	
HgStab04	49,0396966	498,267039	
HgBW2	0		499,495915
HgStab05	49,0393026	498,271043	
HgStab06	49,0378251	498,286056	
HgStab07	49,0359537	498,305072	
HgStab08	49,0461004	498,201983	
HgBW3	0		499,767788
HgStab09	49,0387116	498,277048	
HgStab10	49,0377266	498,287056	
HgStab11	49,0364461	498,300068	
HgStab12	49,0294542	498,371129	
Mean value:	49,038	498,28397	499,599916

Organisation of measurements:

For stability testing of the freshly before prepared test bottles

- **01. Test** = day of preparation = Mo. 11/27/2006
= first measurement
- 8 spiked bottles
 - After daily analyses were 4 bottles for meantime storage in a refrigerator, and
 - 4 bottles for storage at room temperature
- 2 blank bottles
 - After daily analyses was 1 bottle for meantime storage in a refrigerator, and
 - 1 bottle for storage at room temperature

Scheme of calibration

[values from the ,Mercur' protocol on 11/27/2006]

	Target [ng Hg/L]	Ints	SD (3time- measurm.)	RSD/% (3time- measurm.)	Real [ng Hg/L]	Real as % of target
Kal-Null	0	0,000535	0,000017	3,206	0,057377049	
Kal-Std1	1,986	0,000787	0,000038	4,907	2,12295082	106,895812
Kal-Std2	9,93	0,00172	0,000032	1,87	9,770491803	98,3936737
Kal-Std3	19,986	0,002954	0,000022	0,768	19,8852459	99,4958766
Kal-Std4	29,819	0,004146	0,000028	0,679	29,6557377	99,452489
Kal-Std5	39,72	0,00539	0,000008	0,149	39,85245902	100,333482
Kal-Std6	49,65	0,006581	0,000045	0,693	49,6147541	99,9290113

Results of the just now prepared stability test samples

(driftcorrected); all values in [ng Hg / L]

Arranged as the of measurement cycle	Target content of the single test sample	Measured content At Date: 11/27/2006	Note:
KalStd-1	Intensity value =	0,000787	Intensity value
KalStd-3 as control sample; Drift-Basis	19,986	19,85	Real value
HgBW1	0	2,1390177	Driftcorrected value
HgStab01	49,03713561	65,528784	Driftcorrected value
HgStab02	49,03881009	57,144365	Driftcorrected value
HgStab04	49,03969663	56,848855	Driftcorrected value
HgStab05	49,03930261	50,343456	Driftcorrected value
KalStd-3 as control sample; Drift-Basis	19,986	20,05	Real value
HgBW2	0	2,4045705	Driftcorrected value
HgStab07	49,03595369	54,093394	Driftcorrected value
HgStab09	49,03871159	65,228173	Driftcorrected value
HgStab10	49,03772659	53,947149	Driftcorrected value
HgStab12	49,02945415	56,059537	Driftcorrected value
KalStd-3 as control sample; Drift-Basis	19,986	19,79	Real value

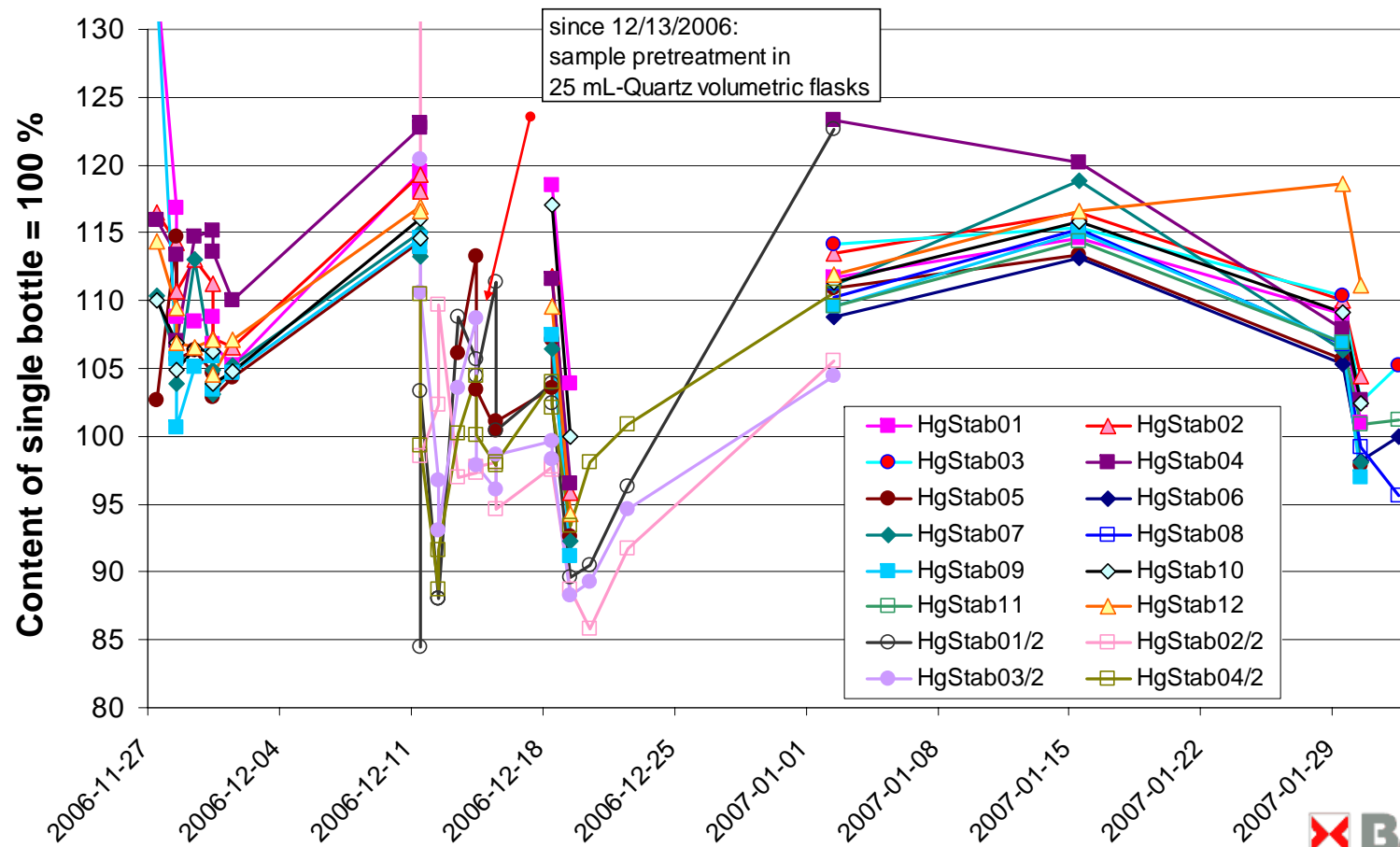
Further organisation of the stability measurements:

- With this scheme the following stability tests were executed, with changing drift-bases and daily new prepared calibration and calibration standards; also, if necessary (due to their shorttime stability), with new prepared calibration stock solutions.
- 02. Test = 1 day after preparation Di. 11/28/2006
- 03. Test = 2 days after preparation Mi. 11/29/2006
- 04. Test = 4 days after preparation Fr. 12/01/2006
- 05. Test = 7 days after preparation Mo. 12/04/2006 (canceld, concerned staff member not available)
- 06. Test = 14 days after preparation Mo. 12/11/2006
- 07. Test = 21 days after preparation Mo. 12/18/2006
- 08. Test = 35 days after preparation Di. 01/02/2007
- additional measurements (starting at 01/02/2007) with the longtime stored samples
 - 4 spiked bottles (2x meantime storage in a refrigerator, 2x storage at room temperature)
 - 1 blank bottle (1x storage at room temperature)
- 09. Test = 49 days after preparation Mo. 01/15/2007
- 10. Test = 63 days after preparation Mo. 01/29/2007

AFS-Measurements of the stability of an amount of about 49,04 ng Hg
 Figure 1 in 1 L prepared test-solution stabilized with BrCl

[corrected for the target value of the single bottle (= recovery rate in %); including drift control, but without a blank-correction (blank = about 3,7 ng Hg/L)]

Note: HgStab01, 02, 03 and 04/2 were tests prepared later in 500 mL volumetric flasks



First resume:

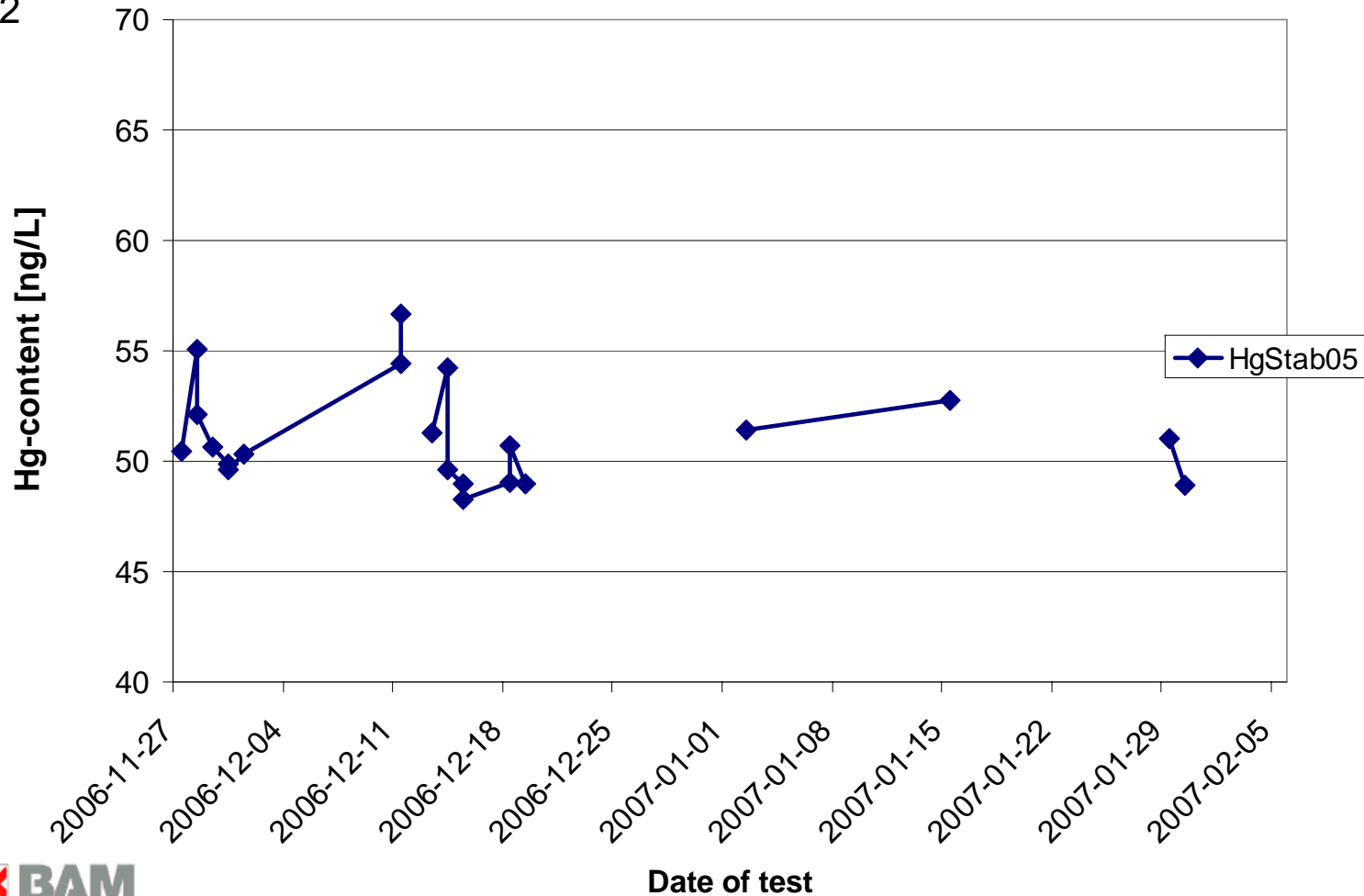
- The values shown in with Figure 1 were corrected for the target values of the single-test bottles (only in this manner it is really comparable). These values reflect the potential sample specific fluctuations of the recovery rates; but the instabilities and the failures of the apparatus used were also inherent at the time of executing the measurement scheme.
- Because of this, one can only conclude that these fluctuations caused by sample instability were in a maximum range of
about ± 20 % of the target value.

Evaluation of the stability of one special test bottle

- Using the test bottle „HgStab05“ as an example, Figure 2 is documented that a slightly increased Hg-content can be indeed stated compared to the target value of 49,04 ng Hg/L, but the value can be considered as stable in a range of $\pm 7 - 8$ ng Hg/L from the target value over the whole time period of the investigations, of about 60 days.
- With this, we have a tolerable percental margin of fluctuation of about ± 15 % for the stability over the declared time period of 60 days.

Stability test with AFS; Example: HgStab05 (target = 49,04 ng Hg/L; blank = 3,7 ng Hg/L)

Figure 2

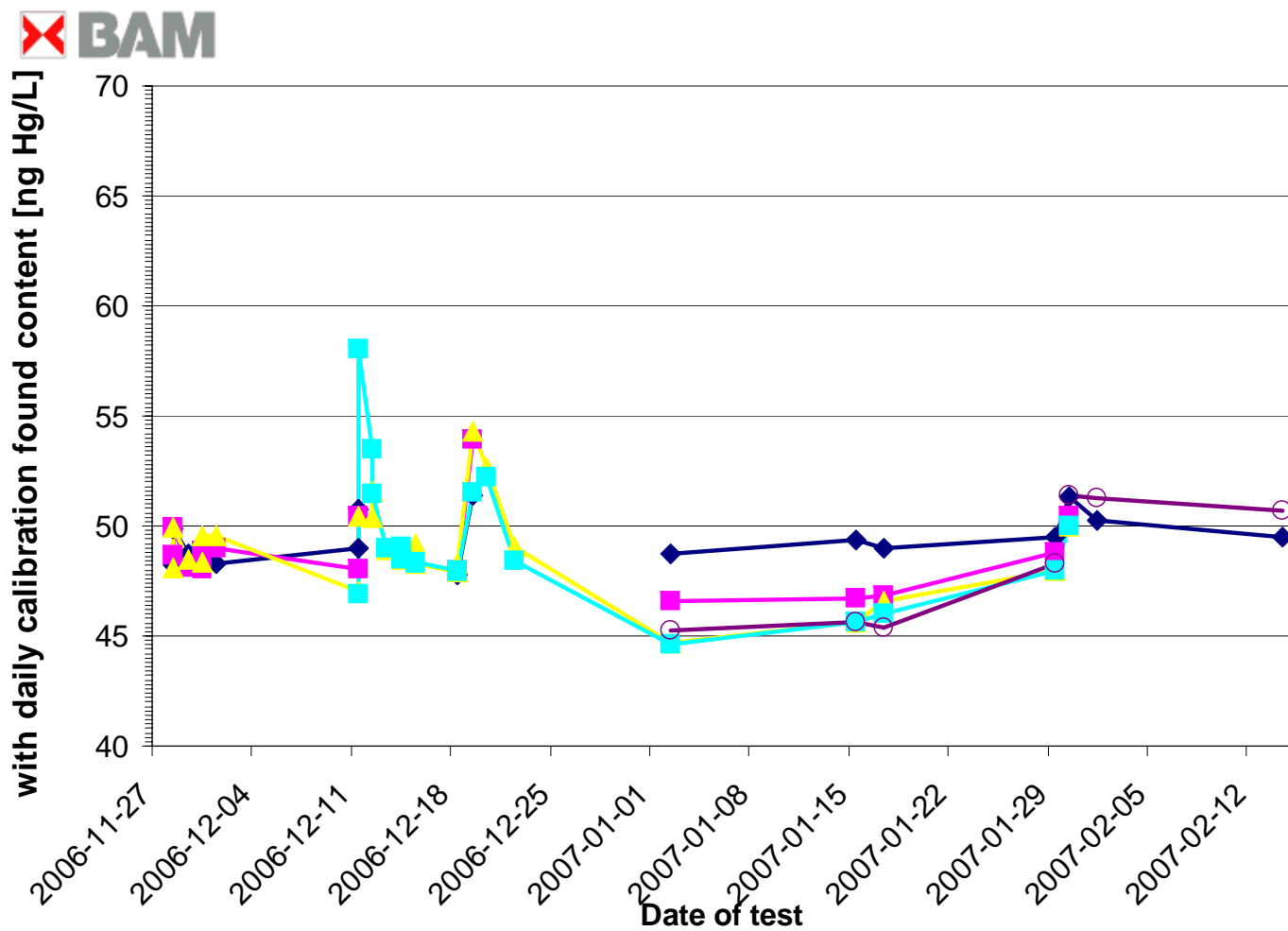


Second resume:

- With the example of the calibration values of the calibration standard 6 - used as QC- respectively Drift-Control-sample - the following Figure 3 is documenting once more that the calibration point, very well adjusted to the concentration of the stability test samples, shows the same fluctuation pattern tendencies as seen in Figure 2 for a single test sample.

Stability test with AFS; Example: KalStd-6 as QualityControl-sample (target = 49,65 ng Hg/L)

Figure 3 [at several positions of the measurement scheme]



Conclusions

- After all measurements, stability of prepared solutions could be stated as being robust enough and thus allowing the preparation of the samples for the CCQM P100.1 (Hg) intercomparison run
- It is possible to prepare solutions with such a small content of mercury with a stability sufficient for a time period of about 60 days
- Basis for this is a careful, gravimetrically controlled, low contamination handling of the desired test bottles during preparation

Further conclusions

- The second stability series for the elements Cd, Pb, Ni in Euramet 924 test samples wasn't such a big problem, because of higher contents of the desired elements.
- Another risk - the used material PFA - was a little bit more complicated because of the electrostatic properties of this material.
- The stability of Cd, Pb, Ni in acidly solutions (HNO_3) was proven to be satisfactory for a period longer than 60 days.