



**ORGANOCHLORINE PESTICIDE
RESIDUES IN HERBAL MEDICINE
PROFICIENCY TESTING
PROGRAMME**

APLAC T049

FINAL REPORT

Hong Kong Government Laboratory

in collaboration with

Hong Kong Accreditation Service

June 2006

**Organochlorine Pesticide Residues
in Herbal Medicine
Proficiency Testing Programme
APLAC T049**

FINAL REPORT

Issued by:



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Secretary of Proficiency Testing Advisory Board

Hong Kong Government Laboratory

27th June 2006

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1. Introduction

- 1.1. This report summarised the results of the proficiency testing programme on the quantitative analysis of five organochlorine pesticide residues (namely HCB, α -BHC, β -BHC, γ -BHC and δ -BHC) in herbal medicine.
- 1.2. The programme was organised by Hong Kong Government Laboratory (HKGL), in collaboration with Hong Kong Accreditation Service (HKAS), under the auspices of the Asia-Pacific Laboratory Accreditation Co-operation (APLAC).
- 1.3. A total of 70 organizations from 26 different economies participated (APPENDIX I) and each participant was confidentially assigned with a unique laboratory code in the programme.

2. Objectives

- 2.1. The programme aimed at providing a means for participants to assess their performance in the analysis for the contents of HCB, α -BHC, β -BHC, γ -BHC and δ -BHC in herbal medicine through interlaboratory comparison.
- 2.2. Test results from participants were evaluated according to the robust statistical techniques that outlined in ISO13528:2005^{8.1} "Statistical methods for use in proficiency testing by interlaboratory comparisons" and Analytical Methods Committee of Royal Society of Chemistry^{8.2}. z-score was provided as a numerical indicator to reveal individual participant's proficiency relative to others in the programme.

3. Test Material

- 3.1. About 20 kg of raw radix ginseng were purchased from the local market and confirmed to have incurred with several organochlorine pesticide residues at various concentrations. The samples were initially washed with distilled water to remove dirt and other foreign matters, and freeze-dried (at about -50 °C and < 70mT) for 7 days. The dried sample was powdered, sieved through a 100 μ m sieve and mixed thoroughly in a commercial blender. About 25 g of the sample portions were packed into clean and nitrogen-flush amber glass bottles. All sample-containing bottles were γ -irradiated at a dose of about 0.8 kGy to prevent



microbial growth, then sealed under vacuum and stored at 25 °C prior to dispatch. Selected photographs of test material preparation processes are illustrated in APPENDIX II for reference.

- 3.2. Homogeneity and stability tests of the samples were determined in accordance with the procedures stipulated in APPENDICES III and IV respectively and the results were found to be satisfactory.
- 3.3. 96 sets of powdered sample together with “Instructions to accreditation bodies”, “Receipt form”, “Instructions to participating laboratories” and “Results proforma” were delivered from HKGL to HKAS on 27 Jan 2006 for redirection to participating accreditation bodies. A specimen copy of these instructions was given in APPENDICES V to VIII. Each participant was provided with one bottle of test sample through his accreditation body (AB).

4. Test Procedures

- 4.1. Participants were required to determine the five organochlorine pesticide residues (HCB, α -BHC, β -BHC, γ -BHC and δ -BHC) in the test sample according to their appropriate validated methods or the suggested method given in the “Instructions to participating laboratories”. Results of triplicate analyses in mg/kg with three significant figures or three decimal place, whichever was appropriate, should be reported. Estimation of relative expanded measurement uncertainty of the analysis was also requested.

5. Results and Statistical Evaluation

5.1. General Information

- 5.1.1. Results from all participants were received on or before 18 April 2006.
- 5.1.2. Participation for individual analyte:

	HCB	α -BHC	β -BHC	γ -BHC	δ -BHC
No. of Results Submitted	55	70	68	69	57



5.1.3. Interim report was issued to participants through their AB on 9 May 2006. Participants were requested to check their analytical data before 23 May 2006 to ensure the reported data were correct.

5.2. Participants' test results

5.2.1. Test results submitted by participants were presented in APPENDICES IX to XIII. Their methodologies and techniques employed were tabulated in APPENDIX XIV.

5.2.2. Consensus mean and standard deviation (SD) computed by robust statistics were summarised in APPENDIX XV.

5.3. Performance evaluation

5.3.1. The performance of each participant was evaluated by means of a performance index, z-score which was calculated as:

$$z = \frac{x_i - m}{S}$$

where x_i = mean of individual participant
 m = computed mean from robust test
 S = computed SD from robust test

5.3.2. z-scores of individual participant were presented in APPENDIX XVI and illustrated graphically in APPENDICES XVII to XXI.

5.3.3. z-score is commonly interpreted as follows:

- (a) $|z| \leq 2$ Satisfactory
- (b) $2 < |z| < 3$ Questionable
- (c) $|z| \geq 3$ Unsatisfactory

5.3.4. z-scores marked with asterisk (*) are considered unsatisfactory (i.e. $|z| \geq 3$) and the participants concerned should investigate their results.

5.3.5. Participants having a z-score in the range $2 < |z| < 3$ are encouraged to review their results.



6. Observations and Recommendations

- 6.1. As shown in APPENDIX XVI, performance of participants was in general satisfactory and tabulated as follows:

Performance achieved	Number of Participants (percentage)				
	HCB	α -BHC	β -BHC	γ -BHC	δ -BHC
$ z \leq 2$	53 (96.4 %)	65 (92.9 %)	60 (88.2 %)	63 (91.3 %)	55 (96.5 %)
$2 < z < 3$	1 (1.8 %)	3 (4.3 %)	3 (4.4 %)	3 (4.3 %)	1 (1.8 %)
$ z \geq 3$ (Outliers)	1 (1.8 %)	2 (2.8 %)	5 (7.4 %)	3 (4.3 %)	1 (1.8 %)

In summary, 7 (Laboratory No. 23, 25, 27, 29, 30, 32, 58) out of the 70 participants (10 %) were identified as having reported one or more outlier results; and 12 out of the 319 results submitted were identified as outlier results (3.8 %).

- 6.2. Distributions of participants' test results were relatively wide. The relative standard deviations were found ranging from 43.9% to 56.0%.
- 6.3. Laboratory No. 65 presented the relative standard deviations of 108% and 123% for α -BHC and δ -BHC respectively in his results. It is highly recommended that the participant should cautiously review the instrumental operation and analytical procedures in order to identify the cause of such significant within-laboratory inconsistency.
- 6.4. Large variation of reported measurement uncertainty (MU) was shown amongst participants. For instance, a minimum MU of 0.0000437% and a maximum MU of 67.18%, both at the coverage factor of 2, were reported for the analysis of β -BHC. In addition, about 20% of participants reported their MU smaller than 5%; and about 15% of participants did not report the MU at all. These information probably indicate that some participants might have hesitations to calculate the uncertainty budgets and it was recommended that more trainings should be provided, particularly for those laboratories who claimed that their analytical methods were accredited.



- 6.5. Since there is a substantial number of participants and feedback from them are encouraging, HKGL will consider organization of the second round of this programme.
- 6.6. There are four participants who performed analyses using the suggested method as attached in the instructions to participants, in addition to theirs. The concerned results were not included in the calculation of the consensus values. The participants concerned could evaluate quality of the results generated from application of the concerned method against the consensus means and SD as listed in APPENDIX XV.

7. Remarks

- 7.1. Efforts from all ABs and participants in this programme are gratefully noted. Participants are welcome to forward any comments on this PT and their concerned results to the following address:

Secretary of PTAB
Hong Kong Government Laboratory
7/F. Homantin Government Offices,
88 Chung Hau Street, Homanatin,
Hong Kong SAR.

or email to GL2005PT1@govtlab.gov.hk.

- 7.2. Interlaboratory proficiency testing programmes allow laboratories to demonstrate the comparability of their data. HKGL will continue to conduct similar proficiency testing programmes to assist laboratories in achieving the respective objectives and targets. Suggestions relating to any other PT programmes are all welcome.

8. References

- 8.1. ISO 13528 “Statistical methods for use in proficiency testing by interlaboratory comparisons”, **2005**, Geneva, Switzerland.
- 8.2. Analytical Methods Committee of RSC, *Analyst*, **1989**, 114:1693-1702. International harmonized protocol for proficiency testing.



- 8.3. W. Horwitz. Evaluation of analytical methods used for regulations of food and drugs, *Anal. Chem.* **1982**, 54:67A-76A.
- 8.4. J.N. Miller, J.C. Miller, Statistics and Chemometrics for Analytical Chemistry, 4th edition, **2000**, Prentice Hill.

APPENDIX I: List of Participants

ECONOMIES	ACCREDITATION BODY	NUMBER
ARGENTINA	OAA	1
AUSTRALIA	NATA	3
BRAZIL	INMETRO	1
CANADA	SCC	4
CHILE	INN	5
CHINA	CNAL	6
CZECH REPUBLIC	CAI	3
ECUADOR	OAE	2
FINLAND	FINAS	1
GERMANY	DACH	2
HONG KONG, CHINA	HKAS	6
IRELAND	INAB	1
ISREAL	ISRAC	5
ITALY	SINAL	3
JAPAN	JAB	1
KOREA	KOLAS	4
LATVIA	LNAB	1
NEW ZEALAND	IANZ	3
PHILIPPINES	BPSLAS	4
POLAND	PCA	3
PORTUGAL	IPAC	1
SINGAPORE	SAC	1
SWEDEN	SWEDAC	1
TAIWAN, CHINA	TAF	3
UNITED KINGDOM	UKAS	1
VIETNAM	BoA	4
TOTAL: 26		TOTAL: 70

APPENDIX II: Photographs of Preparation of Test Material

1. Raw radix ginseng purchased from local market
2. Freeze-dried samples
3. Powdered and sieved through 100 μm sieves
4. Packed into amber bottles and disinfected with γ -irradiation
5. Vacuum-sealed and stored at 25 °C prior to dispatch



APPENDIX III: Homogeneity Test

1. Sixteen bottles of powder were randomly selected from a total of 250 bottles.
2. Two test portions (sub-samples) were drawn from each bottle for duplicate analysis on 11 November 2005.
3. Results of the 32 sub-samples on the determination of HCB, α -BHC, β -BHC, γ -BHC and δ -BHC in a random order under the same conditions were as follows:

Sample Code	Concentration (mg/kg) of duplicate analysis									
	HCB		α -BHC		β -BHC		γ -BHC		δ -BHC	
	#1	#2	#1	#2	#1	#2	#1	#2	#1	#2
13	0.120	0.114	0.430	0.442	0.136	0.140	0.248	0.263	1.210	1.288
36	0.128	0.126	0.446	0.448	0.134	0.140	0.255	0.255	1.234	1.269
42	0.126	0.132	0.453	0.428	0.145	0.133	0.277	0.246	1.321	1.227
60	0.123	0.133	0.436	0.465	0.137	0.161	0.254	0.289	1.229	1.473
66	0.120	0.125	0.435	0.450	0.134	0.139	0.249	0.256	1.253	1.288
84	0.126	0.126	0.446	0.456	0.145	0.149	0.263	0.260	1.322	1.350
99	0.117	0.126	0.446	0.448	0.148	0.146	0.267	0.263	1.345	1.326
108	0.123	0.128	0.444	0.460	0.143	0.147	0.255	0.266	1.320	1.357
114	0.139	0.132	0.460	0.446	0.150	0.147	0.270	0.262	1.369	1.362
132	0.125	0.120	0.446	0.425	0.144	0.137	0.261	0.248	1.337	1.279
133	0.126	0.119	0.448	0.434	0.149	0.140	0.262	0.249	1.354	1.321
150	0.120	0.123	0.430	0.444	0.142	0.147	0.253	0.260	1.318	1.348
166	0.118	0.128	0.433	0.453	0.137	0.146	0.248	0.267	1.307	1.353
174	0.116	0.122	0.427	0.444	0.142	0.152	0.250	0.265	1.301	1.386
186	0.117	0.118	0.426	0.432	0.145	0.145	0.253	0.254	1.315	1.325
200	0.127	0.121	0.454	0.441	0.152	0.147	0.269	0.259	1.391	1.348
Mean	0.124		0.443		0.144		0.259		1.320	
SD	0.00554		0.0107		0.00616		0.00938		0.545	
RSD (%)	4.5		2.4		4.3		3.6		4.1	

4. The sampling variance (S_s^2) and analytical variance (S_a^2) were estimated based on one-way analysis of variance (ANOVA), without exclusion of outliers, using the following equations:

$$\bar{x}_i = \frac{1}{n} \sum_{j=1}^n x_{ij}$$

$$\bar{x} = \frac{1}{k} \sum_{i=1}^k \bar{x}_i$$

$$SS_1 = n \cdot \sum_{i=1}^k (\bar{x}_i - \bar{x})^2$$

APPENDIX III: Homogeneity Test (Cont'd)

$$SS_2 = \sum_{i=1}^k \sum_{j=1}^n (x_{ij} - \bar{x}_i)^2$$

sampling variance (S_s^2) = $SS_1 / (k-1)$

analytical variance (S_a^2) = $SS_2 / k(n-1)$

where:

k = number of samples

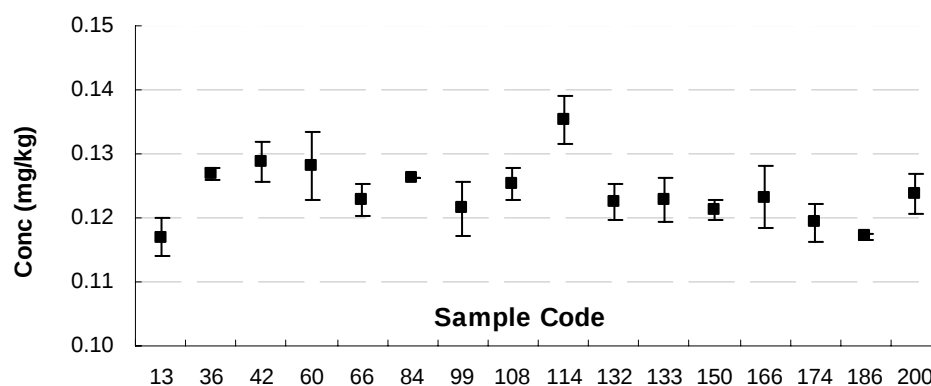
n = number of replicates per sample

5. The values of $\bar{x}$, S_s , S_a , k and the results of F-test were given below:

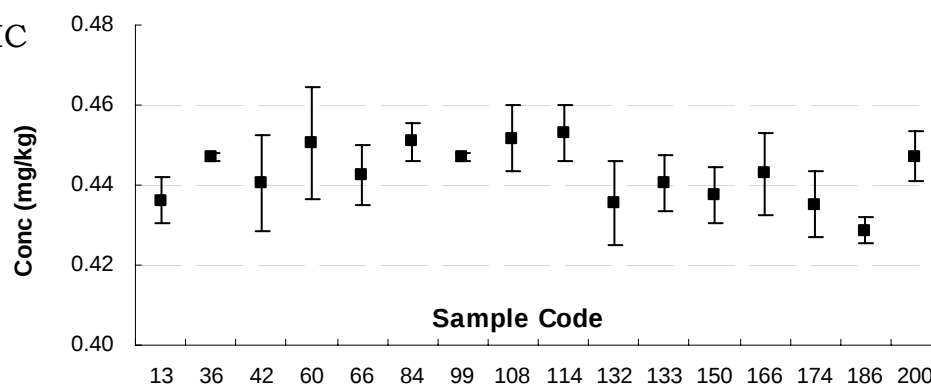
Analyte	$\bar{x}$, mg/kg	S_s^2	S_a^2	F_{test}	$F_{critical}$
HCB	0.124	4.30×10^{-5}	1.91×10^{-5}	2.252	2.352
α -BHC	0.443	1.01×10^{-4}	1.26×10^{-4}	0.800	2.352
β -BHC	0.144	3.88×10^{-5}	3.72×10^{-5}	1.042	2.352
γ -BHC	0.259	5.75×10^{-5}	1.17×10^{-4}	0.492	2.352
δ -BHC	1.320	2.92×10^{-3}	3.01×10^{-3}	0.972	2.352

6. Graphical illustrations of selected samples (n =16) in homogeneity tests

6.1. HCB

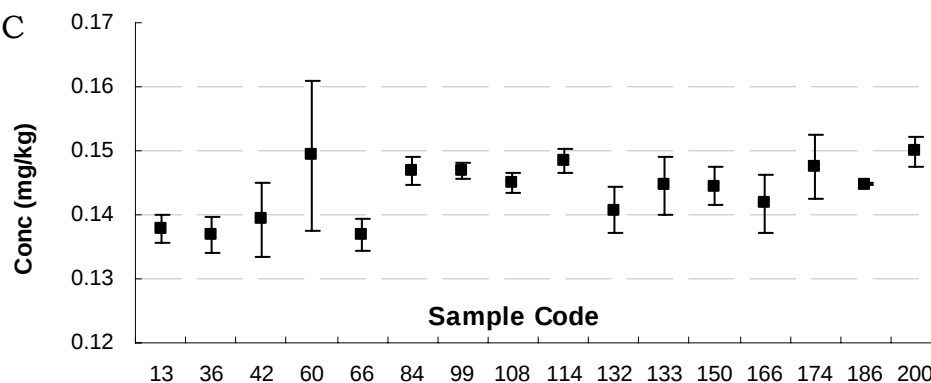


6.2. α -BHC

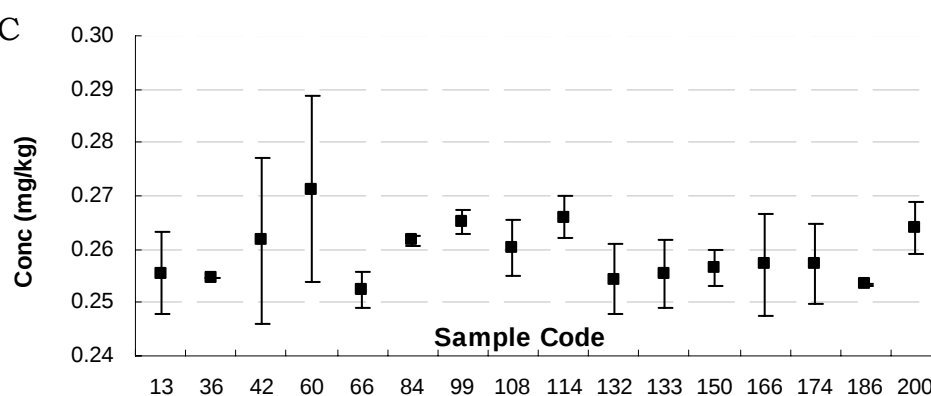


APPENDIX III: Homogeneity Test (Cont'd)

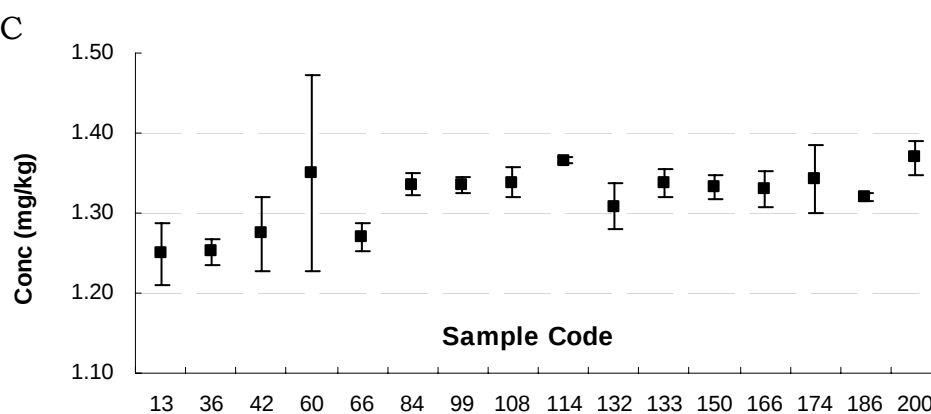
6.3. β -BHC



6.4. γ -BHC



6.5. δ -BHC



7. Since the test statistic (F_{test}) was less than the critical value (F_{critical}), it was concluded that no significant difference between the sampling and the analytical variances; and the sample was considered homogeneous.

APPENDIX IV: Stability Test

1. Stability test was monitored regularly on a monthly basis from January 2006 to May 2006 which covered the time when the samples were analyzed by participants.
2. Samples selected for the stability test were stored at (i) a room temperature of 25 °C and (ii) at an elevated temperature of 37 °C.
3. One sample from each temperature set was randomly selected and analyzed in triplicate under the same operational conditions as in homogeneity test.
4. Stability of samples was evaluated from the absolute difference between the mean value obtained in homogeneity test ($\bar{x}$) and the mean value of each stability test ($\bar{y}_i$). According to ISO13528:2005^{8.1}, the absolute difference should not be more than 30% of the standard deviation of the proficiency test (σ_R).

$$\text{ie. } |\bar{x} - \bar{y}_i| \leq 0.3\sigma_R$$

σ_R was estimated using Horwitz^{8.3} equation of $2 c^{-0.15}$ where c was the mean concentration (in percent) of analyte from homogeneity test.

5. Results of the stability tests at 25 °C were summarized below:

5.1. Date: 24 January 2006; Sample code: 115

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.116	0.118	0.120	0.118	0.124	0.217	0.0652	0.006
α -BHC	0.448	0.456	0.455	0.453	0.443	0.180	0.0539	0.010
β -BHC	0.157	0.146	0.154	0.152	0.144	0.212	0.0637	0.008
γ -BHC	0.275	0.241	0.255	0.257	0.259	0.195	0.0584	0.002
δ -BHC	1.328	1.362	1.404	1.365	1.320	0.152	0.0457	0.045

5.2. Date: 24 February 2006; Sample code: 113

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.109	0.111	0.125	0.115	0.124	0.217	0.0652	0.009
α -BHC	0.418	0.428	0.444	0.430	0.443	0.180	0.0539	0.013
β -BHC	0.157	0.152	0.154	0.154	0.144	0.212	0.0637	0.010
γ -BHC	0.258	0.253	0.262	0.258	0.259	0.195	0.0584	0.001
δ -BHC	1.325	1.366	1.380	1.357	1.320	0.152	0.0457	0.037

APPENDIX IV: Stability Test (Cont'd)

5.3. Date: 24 March 2006; Sample code: 117

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.118	0.117	0.115	0.117	0.124	0.217	0.0652	0.007
α -BHC	0.444	0.439	0.432	0.439	0.443	0.180	0.0539	0.004
β -BHC	0.150	0.152	0.156	0.153	0.144	0.212	0.0637	0.009
γ -BHC	0.261	0.262	0.268	0.264	0.259	0.195	0.0584	0.005
δ -BHC	1.349	1.373	1.321	1.348	1.320	0.152	0.0457	0.028

5.4. Date: 24 April 2006; Sample code: 45

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.128	0.125	0.118	0.124	0.124	0.217	0.0652	0
α -BHC	0.462	0.460	0.478	0.467	0.443	0.180	0.0539	0.024
β -BHC	0.144	0.141	0.139	0.141	0.144	0.212	0.0637	0.003
γ -BHC	0.268	0.266	0.276	0.270	0.259	0.195	0.0584	0.011
δ -BHC	1.336	1.316	1.325	1.326	1.320	0.152	0.0457	0.006

5.5. Date: 18 May 2006; Sample code: 39

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.119	0.119	0.109	0.116	0.124	0.217	0.0652	0.008
α -BHC	0.447	0.451	0.442	0.447	0.443	0.180	0.0539	0.004
β -BHC	0.151	0.145	0.142	0.146	0.144	0.212	0.0637	0.002
γ -BHC	0.256	0.256	0.251	0.254	0.259	0.195	0.0584	0.005
δ -BHC	1.372	1.337	1.366	1.358	1.320	0.152	0.0457	0.038

6. Results of the stability tests at 37 °C were summarized below:

6.1. Date: 24 February 2006; Sample code: 106

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.115	0.119	0.125	0.119	0.124	0.217	0.0652	0.005
α -BHC	0.424	0.438	0.423	0.428	0.443	0.180	0.0539	0.015
β -BHC	0.152	0.156	0.149	0.152	0.144	0.212	0.0637	0.008
γ -BHC	0.254	0.263	0.256	0.258	0.259	0.195	0.0584	0.001
δ -BHC	1.288	1.331	1.278	1.299	1.320	0.152	0.0457	0.021

APPENDIX IV: Stability Test (Cont'd)

6.2. Date: 24 March 2006; Sample code: 33

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.116	0.114	0.123	0.118	0.124	0.217	0.0652	0.006
α -BHC	0.424	0.416	0.447	0.429	0.443	0.180	0.0539	0.014
β -BHC	0.153	0.148	0.158	0.153	0.144	0.212	0.0637	0.009
γ -BHC	0.253	0.247	0.267	0.256	0.259	0.195	0.0584	0.003
δ -BHC	1.323	1.266	1.378	1.322	1.320	0.152	0.0457	0.002

6.3. Date: 24 April 2006; Sample code: 52

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.122	0.122	0.127	0.124	0.124	0.217	0.0652	0
α -BHC	0.464	0.451	0.478	0.464	0.443	0.180	0.0539	0.021
β -BHC	0.145	0.144	0.148	0.146	0.144	0.212	0.0637	0.002
γ -BHC	0.272	0.262	0.279	0.271	0.259	0.195	0.0584	0.012
δ -BHC	1.299	1.266	1.323	1.296	1.320	0.152	0.0457	0.024

6.4. Date: 18 May 2006; Sample code: 159

Analyte	#1	#2	#3	$\bar{y}_i$	$\bar{x}$	σ_R	$0.3\sigma_R$	$ \bar{x} - \bar{y}_i $
HCB	0.120	0.112	0.126	0.119	0.124	0.217	0.0652	0.005
α -BHC	0.439	0.473	0.425	0.445	0.443	0.180	0.0539	0.002
β -BHC	0.162	0.157	0.171	0.163	0.144	0.212	0.0637	0.019
γ -BHC	0.255	0.269	0.249	0.258	0.259	0.195	0.0584	0.001
δ -BHC	1.325	1.392	1.252	1.323	1.320	0.152	0.0457	0.003

- Since the differences between the mean value from homogeneity test ($\bar{x}$) and the mean value from individual stability test ($\bar{y}_i$) were found to be less than $0.3\sigma_R$, the samples were considered stable upon a six-month storage (from November 2005 to May 2006) at both 25 °C and 37 °C.

APPENDIX V: Instructions to Accreditation Bodies

GOVERNMENT LABORATORY OF HONG KONG SPECIAL ADMINISTRATIVE REGION IN COLLABORATION WITH HONG KONG ACCREDITATION SERVICE

PROFICIENCY TESTING PROGRAMME

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES IN HERBAL MEDICINE (APLAC T049)

INSTRUCTIONS TO ACCREDITATION BODIES

1. **Sample**

Each participating laboratory is given a sample bottle containing about 25 g powdered sample.

Upon receipt of the sample bottle from the Hong Kong Government Laboratory (HKGL), participating accreditation bodies should check the sample for damage, if any, and acknowledge receipt of the sample by returning the Receipt Form to HKGL via email (GL2005PT1@govtlab.gov.hk).

2. **Tests to be performed**

Each participating laboratory is required to determine the levels of α -BHC, β -BHC, γ -BHC, δ -BHC and HCB (mg/kg) in the sample. The determination should be conducted in triplicate. (BHC represents hexachlorocyclohexane and HCB represents hexachlorobenzene).

3. **Sample distribution**

The sample, Instructions to Laboratories and Results Proforma (hard copy and soft copy) should be distributed to participating laboratories as soon as possible.

Laboratories will have about four weeks to analyse the sample.

4. **Documents to be submitted**

Accreditation bodies should collect the Result Proforma from the participating laboratories on or before dd mm 2006 for record purposes while participating laboratories shall forward a soft copy of the Results Proforma via e-mail to HKGL before dd mm 2006 for data treatment.

5. **Enquiries**

Please contact:-

Dr. Della Wai-mei SIN,
Secretary, Proficiency Testing Advisory Board
Analytical and Advisory Services Division
Government Laboratory, HKSAR
Homantin Government Offices, 88 Chung Hau Street,
Homantin, Kowloon, HKSAR
Fax: +852 2714 4083, Tel.: +852 2762 3859
Email: GL2005PT1@govtlab.gov.hk

APPENDIX VI: Receipt Form

GOVERNMENT LABORATORY OF HONG KONG SPECIAL ADMINISTRATIVE REGION
IN COLLABORATION WITH
HONG KONG ACCREDITATION SERVICE

PROFICIENCY TESTING PROGRAMME

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES IN HERBAL
MEDICINE (APLAC T049)

RECEIPT FORM

<To be completed by Participating Accreditation Body/ Laboratories>

From:
Participating Accreditation Body/ Laboratories

Contact Person

Fax:

To: Dr. Della Wai-mei SIN,
Secretary, Proficiency Testing Advisory
Board
Analytical and Advisory Services Division
Government Laboratory, HKSAR
Homantin Government Offices,
88 Chung Hau Street,
Kowloon, HKSAR.

Tel.:

Email:

Date:

Please complete the shaded area.

Fax: +852 2714 4083

Tel.: +852 2762 3859

Email: GL2005PT1@govtlab.gov.hk

This is to acknowledge the receipt of the sample.

After inspection, the sample was found (☐ damaged / ☐ intact)* and were deemed (☐ suitable / ☐ not suitable)* for testing.

Remark(s) :

Tick as appropriate

APPENDIX VII: Instruction to Participating Laboratories

GOVERNMENT LABORATORY OF HONG KONG SPECIAL ADMINISTRATIVE REGION IN COLLABORATION WITH HONG KONG ACCREDITATION SERVICE

PROFICIENCY TESTING PROGRAMME

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES IN HERBAL MEDICINE (APLAC T049)

INSTRUCTIONS TO PARTICIPATING LABORATORIES

Laboratory Code Number:

1. **Sample**

Please check whether a sealed amber bottle containing about 25 g powdered sample is provided.

2. **Tests to be performed**

Each participating laboratory is required to determine the levels (mg/kg) of α -BHC, β -BHC, γ -BHC, δ -BHC and HCB in the sample. The determination should be conducted in triplicate. (BHC represents hexachlorocyclohexane and HCB represents hexachlorobenzene).

Storage details: Participating laboratories are recommended to store the sample bottle unopened in a secure environment at or below 25°C. Opened bottle should be kept in dry box or desiccator when not in use. Avoid direct exposure to sunlight.

Methods used: Participating laboratories should use routine test methods which would normally be used to test customer supplied samples and record the method used on the results sheet.

(Note: A suggested method in APPENDIX I is also provided for interested participants who would like to use, in addition to their accredited method(s), in this proficiency test. A second sample bottle would be given to these participants; and the results obtained from the suggested method and their accredited method(s) should be reported in separate sheets)

3. **Reporting**

Report all the replicate data in the Results Proforma and complete both Part I & II. (Report the Measurement Uncertainty ($\pm$ % base) for each result.

4. **Documents to be submitted**

Submit the completed Results Proforma to your accreditation body, if applicable, **AND** to Hong Kong Government Laboratory (HKGL) on or before dd mm 2006 via e-mail (GL2005PT1@govtlab.gov.hk) using the soft copy of Results Proforma provided.

5. **Enquires**

Please contact HKGL or your accreditation body, if applicable, for general enquiry concerning the program. Detailed information of the program may be obtained from:-

Dr. Della Wai-mei SIN, Secretary,

Proficiency Testing Advisory Board

Analytical and Advisory Services Division,

Government Laboratory, HKSAR

Homantin Government Offices, 88 Chung Hau Street, Homantin, Kowloon, HKSAR

Fax: +852 2714 4083; Tel.: +852 2762 3859;

Email: GL2005PT1@govtlab.gov.hk

APPENDIX VIII: Results Proforma

GOVERNMENT LABORATORY OF HONG KONG SPECIAL ADMINISTRATIVE REGION
IN COLLABORATION WITH
HONG KONG ACCREDITATION SERVICE

PROFICIENCY TESTING PROGRAMME

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES IN HERBAL
MEDICINE (APLAC T049)

RESULTS PROFORMA

<Both Part I & II shall be completed>

Laboratory Code Number: _____ Date of Analysis: _____

Name of Laboratory: _____

Part I: Analytical Results

Analyte	Concentration (in mg/kg) [§]				Measurement uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Relative expanded uncertainty (%)	Coverage factor (k)
α-BHC						
β-BHC						
γ-BHC						
δ-BHC						
HCB						

§ Report values to 3 significant figure or 3 decimal places, whichever is appropriate

Note: *It is the responsibility of the participants to avoid collusion and falsification of result so as to ensure a reliable and effective assessment to be made by this proficiency testing programme*

APPENDIX VIII: Results Proforma (Cont'd)

GOVERNMENT LABORATORY OF HONG KONG SPECIAL ADMINISTRATIVE REGION IN COLLABORATION WITH HONG KONG ACCREDITATION SERVICE

PROFICIENCY TESTING PROGRAMME

DETERMINATION OF ORGANOCHLORINE PESTICIDE RESIDUES IN HERBAL MEDICINE (APLAC T049)

Part II: Test Parameters

☐ Tick as appropriate

1. Analytical technique(s): ☐ GC-ECD
☐ GC-MSD
☐ Others _____
(Please specify)
2. Instrument Model: ☐ GC-ECD _____
☐ GC-MSD _____
☐ Others _____
(Please specify)
3. GC Column, if appropriate: _____

(Please specify)
4. Solvent(s) used for sample preparation: _____

(Please specify)
5. Extraction technique used: _____
(Please specify)
6. Clean-up procedure, if any: _____

7. Other comments: _____

[Note: The accreditation body under which your laboratory is accredited may be informed of your results and performance in this programme.]

- End of Results Proforma -

APPENDIX IX: Participants' Test Results for HCB

Lab Code	Concentration (mg/kg)				Measurement Uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Rel. Expanded Uncertainty (%)	Coverage Factor
1	0.128	0.097	0.146	0.124	40.0	2
2	---	---	---	---	---	---
3	0.218	0.234	0.248	0.233	19	2
4	0.322	0.317	0.320	0.320	14	---
5	0.155	0.148	0.159	0.154	11	2
6	0.0725	0.0602	0.0357	0.0561	13.8	2
7	0.15	0.14	0.19	0.16	---	---
8	0.122	0.133	0.137	0.131	11.9	2
9	0.192	0.185	0.195	0.191	30	2
10	0.210	0.203	0.237	0.217	35	2
11	---	---	---	---	---	---
12	0.192	0.192	0.212	0.199	17	2
13	0.02	0.02	0.03	0.023	---	---
14	0.238	0.256	0.249	0.248	6.40	2.571
15	---	---	---	---	---	---
16	0.151	0.139	0.145	0.145	0.02	2
17	0.344	0.351	0.368	0.354	---	---
18	0.098	0.094	0.097	0.096	---	---
19	---	---	---	---	---	---
20	0.158	0.205	0.176	0.180	---	---
21	0.121	0.127	0.123	0.124	30	2
22	0.21	0.222	0.216	0.216	6	2
23	0.232	0.263	0.270	0.255	2.65E-06	2
24	0.105	0.106	0.107	0.106	29	2
25	4.63	4.24	4.38	4.42	25	2
26	0.079	0.072	0.077	0.076	5.36	2
27	0.096	0.093	0.099	0.096	32	2
28	---	---	---	---	---	---
29	---	---	---	---	---	---
30	---	---	---	---	---	---
31	0.227	0.225	0.235	0.229	31	2
32	0.15	0.12	0.13	0.13	---	---
33	0.214	0.232	0.245	0.230	34	2
34	0.067	0.070	0.089	0.075	---	---
35	0.157	0.148	0.187	0.164	19.8	2.23
36	0.302	0.295	0.310	0.302	0.078	2
37	0.015	0.016	0.015	0.015	22.00	2
38	0.134	0.141	0.147	0.141	3	---
39	---	---	---	---	---	---
40	0.251	0.239	0.248	0.246	15.5	2
41	0.143	0.153	0.156	0.151	10	---
42	0.096	0.069	0.061	0.075	24	2
43	0.266	0.284	0.275	0.275	9.8	3
44	0.12	0.11	0.11	0.11	50	2
45	0.093	0.091	0.082	0.089	---	---
46	---	---	---	---	---	---
47	0.29	0.3	0.29	0.29	0.02	2
48	0.205	0.209	0.205	0.206	11	2
49	---	---	---	---	---	---
50	---	---	---	---	---	---
51	0.148	0.150	0.150	0.149	15	2
52	0.233	0.222	0.205	0.220	---	---
53	0.437	0.479	0.433	0.450	58.0	2
54	0.0675	0.0705	0.0832	0.0737	35	2
55	0.162	0.160	0.169	0.164	32.8	2
56	0.2375	0.2375	0.2457	0.240	4	2
57	0.035	0.039	0.038	0.037	20	2
58	0.30	0.32	0.32	0.31	7.79	2.365
59	0.126	0.155	0.136	0.139	1.76	4.53
60	---	---	---	---	---	---
61	0.182	0.202	0.198	0.194	20	2
62	0.236	0.249	0.256	0.247	10.2	4.3
63	0.091	0.086	0.088	0.088	20	2
64	0.13	0.13	0.13	0.13	28.86	2
65	---	---	---	---	---	---
66	0.231	0.235	0.236	0.234	26.1	2
67	---	---	---	---	---	---
68	0.037	0.044	0.039	0.040	11.17	---
69	---	---	---	---	---	---
70	0.048	0.054	0.052	0.052	5.8	2

“---” denotes data/information was not provided by participants

APPENDIX X: Participants' Test Results for α -BHC

Lab Code	Concentration (mg/kg)				Measurement Uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Rel. Expanded Uncertainty (%)	Coverage Factor
1	0.521	0.449	0.510	0.493	15.7	2
2	0.345	0.282	0.264	0.297	29	2
3	0.508	0.540	0.558	0.535	18	2
4	0.435	0.441	0.454	0.443	14	---
5	0.275	0.270	0.279	0.275	11	2
6	0.145	0.156	0.148	0.150	12	2
7	0.31	0.31	0.39	0.34	---	---
8	0.235	0.259	0.270	0.254	14.1	2
9	0.483	0.480	0.492	0.485	32	2
10	0.580	0.524	0.544	0.549	29	2
11	0.22	0.23	0.20	0.22	40	2
12	0.450	0.496	0.477	0.474	19	2
13	0.22	0.23	0.21	0.22	---	---
14	0.537	0.572	0.553	0.554	5.67	2.447
15	0.287	0.264	---	0.276	0.146	2
16	0.468	0.479	0.504	0.484	0.03	2
17	0.268	0.263	0.191	0.241	---	---
18	0.272	0.258	0.273	0.268	---	---
19	0.398	0.396	0.379	0.391	0.079	2
20	0.358	0.495	0.348	0.400	---	---
21	0.323	0.297	0.298	0.306	30	2
22	0.594	0.588	0.594	0.592	6	2
23	0.306	0.333	0.279	0.306	5.79E-06	2
24	0.264	0.262	0.267	0.264	28	2
25	0.655	0.606	0.644	0.635	25	2
26	0.541	0.543	0.542	0.542	0.92	2
27	0.226	0.221	0.235	0.227	28	2
28	0.006	0.007	0.007	0.007	0.13	2
29	0.851	0.843	0.88	0.858	8.3	2
30	1.28	1.31	1.29	1.29	---	---
31	0.526	0.521	0.540	0.529	10	2
32	1.37	1.48	1.40	1.41	---	---
33	0.447	0.458	0.476	0.460	34	2
34	0.413	0.430	0.445	0.430	---	---
35	0.416	0.407	0.477	0.433	17.9	2.23
36	0.452	0.462	0.47	0.461	0.111	2
37	0.028	0.032	0.029	0.030	22.22	2
38	0.32	0.34	0.347	0.336	2.5	---
39	0.241	0.258	0.250	0.250	0.05	2
40	0.554	0.530	0.544	0.543	14.2	2
41	0.406	0.423	0.461	0.430	10	---
42	0.324	0.216	0.198	0.246	28	2
43	0.719	0.712	0.716	0.716	1.4	3
44	0.28	0.26	0.27	0.270	50	2
45	0.269	0.264	0.257	0.263	---	---
46	0.511	0.515	0.503	0.510	17.112	2
47	0.63	0.64	0.62	0.63	0.02	2
48	0.497	0.564	0.498	0.520	19	2
49	0.2667	0.2654	0.2683	0.267	9.4	2
50	0.109	0.100	0.102	0.104	5.3	4.3
51	0.444	0.457	0.437	0.446	15	2
52	0.531	0.534	0.520	0.528	---	---
53	0.802	0.931	0.791	0.841	25.2	2
54	0.307	0.278	0.310	0.298	35	2
55	0.381	0.354	0.407	0.381	27.1	2
56	0.5547	0.5525	0.5725	0.560	3	2
57	0.107	0.117	0.107	0.110	14	2
58	0.66	0.65	0.64	0.65	2.43	2.306
59	0.150	0.166	0.159	0.158	1.33	2.29
60	0.16	0.15	---	0.16	10	1.66
61	0.48	0.515	0.513	0.503	20	2
62	0.466	0.499	0.508	0.491	11.2	4.3
63	0.214	0.208	0.211	0.211	20	2
64	0.37	0.38	0.38	0.38	28.86	2
65	0.012	0.12	0.029	0.054	47.75	2
66	0.567	0.560	0.558	0.562	25.6	2
67	0.557	0.555	0.557	0.556	5.08	2
68	0.324	0.318	0.290	0.311	5.84	---
69	0.343	0.347	0.333	0.341	3.8	2
70	0.226	0.242	0.250	0.240	5.8	2

“---” denotes data/information was not provided by participants

APPENDIX XI: Participants' Test Results for β -BHC

Lab Code	Concentration (mg/kg)				Measurement Uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Rel. Expanded Uncertainty (%)	Coverage Factor
1	0.265	0.208	0.250	0.241	24.5	2
2	0.113	0.102	0.093	0.103	20	2
3	0.184	0.243	0.215	0.214	20	2
4	0.189	0.197	0.192	0.193	14	---
5	0.122	0.114	0.124	0.120	12	2
6	0.0921	0.101	0.0957	0.0963	12.3	2
7	0.22	0.26	0.23	0.24	---	---
8	0.118	0.128	0.131	0.126	11.3	2
9	0.207	0.212	0.218	0.212	33	2
10	0.251	0.229	0.238	0.239	34	2
11	0.11	0.10	0.10	0.10	45	2
12	0.218	0.247	0.254	0.240	29	2
13	0.28	0.33	0.32	0.31	---	---
14	0.261	0.272	0.271	0.268	4.04	2.179
15	0.310	0.269	---	0.290	0.258	2
16	0.091	0.096	0.097	0.095	0.01	2
17	0.291	0.311	0.321	0.308	---	---
18	0.115	0.137	0.136	0.129	---	---
19	0.210	0.208	0.193	0.204	0.089	2
20	0.126	0.162	0.130	0.139	---	---
21	0.391	0.375	0.381	0.382	30	2
22	0.288	0.252	0.258	0.266	6	2
23	1.361	1.491	1.474	1.442	4.37E-05	2
24	0.146	0.144	0.146	0.145	26	2
25	0.315	0.314	0.310	0.313	25	2
26	0.082	0.081	0.087	0.083	6.02	2
27	0.090	0.086	0.092	0.089	32	2
28	0.002	0.003	0.002	0.002	0.04	2
29	0.651	0.692	0.686	0.676	8.3	2
30	0.82	0.76	0.80	0.80	---	---
31	0.253	0.256	0.261	0.257	21	2
32	0.76	0.58	0.65	0.66	---	---
33	0.221	0.226	0.238	0.228	34	2
34	0.255	0.278	0.300	0.278	---	---
35	0.192	0.182	0.236	0.203	21.8	2.23
36	0.310	0.305	0.316	0.310	0.079	2
37	0.008	0.009	0.008	0.008	12.95	2
38	0.193	0.204	0.212	0.203	2.5	---
39	0.143	0.151	0.147	0.147	0.05	2
40	0.255	0.245	0.245	0.248	15.1	2
41	0.158	0.161	0.150	0.156	10	---
42	0.141	0.098	0.091	0.110	25	2
43	0.315	0.291	0.305	0.304	12.1	3
44	0.16	0.16	0.16	0.16	50	2
45	0.138	0.135	0.138	0.137	---	---
46	0.277	0.273	0.276	0.276	26.930	2
47	0.33	0.31	0.32	0.32	0.02	2
48	0.231	0.233	0.232	0.232	23	2
49	0.2161	0.2182	0.2224	0.219	9.35	2
50	0.352	0.333	0.342	0.342	2.8	4.3
51	0.186	0.190	0.198	0.191	15	2
52	0.227	0.232	0.215	0.225	---	---
53	0.362	0.406	0.416	0.395	33.3	2
54	0.146	0.142	0.160	0.149	35	2
55	0.203	0.203	0.215	0.207	35.6	2
56	0.2620	0.2405	0.2580	0.253	5	2
57	ND	ND	ND	ND	18	2
58	0.75	0.74	0.75	0.75	5.91	2.228
59	0.096	0.096	0.097	0.096	2.15	2.01
60	---	---	---	---	---	---
61	0.188	0.195	0.205	0.196	20	2
62	0.181	0.201	0.222	0.201	25.3	4.3
63	0.150	0.141	0.138	0.143	20	2
64	0.26	0.26	0.24	0.25	28.86	2
65	0.040	0.032	0.037	0.036	67.18	2
66	0.252	0.252	0.256	0.253	25.7	2
67	0.265	0.264	0.270	0.266	6.68	2
68	0.510	0.470	0.320	0.433	23.11	---
69	0.162	0.167	0.166	0.165	11.5	2
70	---	---	---	---	---	---

“---” denotes data/information was not provided by participants

“ND” denotes not detected

APPENDIX XII: Participants' Test Results for γ -BHC

Lab Code	Concentration (mg/kg)				Measurement Uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Rel. Expanded Uncertainty (%)	Coverage Factor
1	0.362	0.305	0.347	0.338	17.5	2
2	0.242	0.206	0.223	0.224	16	2
3	0.309	0.346	0.353	0.336	20	2
4	0.212	0.235	0.220	0.222	14	---
5	0.235	0.237	0.242	0.238	11	2
6	0.208	0.226	0.199	0.211	14.8	2
7	0.25	0.27	0.27	0.26	---	---
8	0.166	0.184	0.189	0.179	13.6	2
9	0.356	0.359	0.373	0.363	33	2
10	0.471	0.430	0.443	0.448	28	2
11	0.17	0.18	0.17	0.17	42	2
12	0.322	0.393	0.383	0.366	26	2
13	0.33	0.38	0.35	0.35	---	---
14	0.473	0.493	0.487	0.484	3.87	2.131
15	0.319	0.276	---	0.298	0.272	2
16	0.284	0.285	0.305	0.291	0.02	2
17	0.478	0.52	0.529	0.509	---	---
18	0.181	0.182	0.193	0.185	---	---
19	0.260	0.260	0.250	0.257	0.075	2
20	0.228	0.304	0.235	0.256	---	---
21	0.287	0.309	0.309	0.302	30	2
22	0.456	0.450	0.450	0.452	6	2
23	0.155	0.160	0.158	0.158	1.87E-06	2
24	0.201	0.199	0.200	0.200	31	2
25	0.450	0.444	0.437	0.444	25	2
26	0.346	0.349	0.346	0.347	1.44	2
27	1.140	1.11	1.18	1.143	22	2
28	0.0006	0.001	0.001	0.001	0.02	2
29	0.469	0.455	0.476	0.466	6.9	2
30	1.59	1.63	1.67	1.63	---	---
31	0.397	0.396	0.405	0.399	16	2
32	1.55	1.42	1.50	1.49	---	---
33	0.401	0.414	0.419	0.411	34	2
34	0.317	0.341	0.364	0.341	---	---
35	0.329	0.307	0.369	0.335	17.3	2.23
36	0.248	0.252	0.258	0.253	0.067	2
37	0.022	0.025	0.022	0.023	20.53	2
38	0.277	0.293	0.302	0.291	2.5	---
39	0.165	0.170	0.160	0.165	0.05	2
40	0.416	0.401	0.404	0.407	13.8	2
41	0.303	0.307	0.318	0.309	10	---
42	0.218	0.145	0.136	0.166	27	2
43	0.531	0.506	0.516	0.518	7.1	3
44	0.22	0.22	0.21	0.22	50	2
45	0.197	0.192	0.180	0.190	---	---
46	0.665	0.668	0.644	0.659	28.400	2
47	0.46	0.45	0.45	0.45	0.02	2
48	0.386	0.423	0.391	0.400	21	2
49	0.3278	0.3325	0.3248	0.328	7.9	2
50	0.520	0.512	0.560	0.531	5.6	4.3
51	0.362	0.363	0.348	0.358	15	2
52	0.395	0.407	0.407	0.403	---	---
53	0.590	0.669	0.613	0.624	44.3	2
54	0.221	0.202	0.220	0.214	35	2
55	0.278	0.258	0.299	0.278	29.1	2
56	0.4442	0.4491	0.4331	0.442	3	2
57	0.084	0.090	0.084	0.086	26	2
58	0.55	0.54	0.55	0.55	6.80	2.306
59	0.539	0.532	0.538	0.536	2.16	2.13
60	0.59	0.48	---	0.54	---	---
61	0.362	0.385	0.375	0.374	20	2
62	0.365	0.394	0.399	0.386	11.8	4.3
63	0.164	0.159	0.162	0.162	20	2
64	0.29	0.28	0.29	0.29	28.86	2
65	---	---	---	---	---	---
66	0.446	0.442	0.443	0.444	25.6	2
67	0.444	0.443	0.448	0.445	5.94	2
68	0.050	0.074	0.080	0.068	23.34	---
69	0.304	0.322	0.319	0.315	6.3	2
70	0.225	0.312	0.332	0.290	22	2

“---” denotes data/information was not provided by participants

APPENDIX XIII: Participants' Test Results for δ -BHC

Lab Code	Concentration (mg/kg)				Measurement Uncertainty	
	Replicate 1	Replicate 2	Replicate 3	Mean	Rel. Expanded Uncertainty (%)	Coverage Factor
1	2.36	2.13	1.96	2.15	18.7	2
2	0.829	0.752	0.700	0.760	17	2
3	0.935	1.21	1.24	1.13	16	2
4	2.405	2.400	2.410	2.405	10	---
5	0.619	0.644	0.647	0.637	11	2
6	0.930	1.050	0.938	0.973	7.8	2
7	1.55	1.64	1.77	1.65	---	---
8	0.840	0.865	---	0.853	4.06	2
9	1.83	1.84	1.82	1.83	31	2
10	2.73	2.17	2.27	2.39	33	2
11	0.98	1.05	0.90	0.98	32	2
12	1.12	0.986	1.01	1.04	22	2
13	1.47	0.94	1.10	1.17	---	---
14	1.924	2.079	2.149	2.05	9.69	2.776
15	1.009	0.936	---	0.973	0.461	2
16	---	---	---	---	---	---
17	2.349	2.922	2.916	2.729	---	---
18	0.479	0.481	0.484	0.481	---	---
19	1.449	1.448	1.386	1.428	0.077	2
20	1.08	1.38	1.15	1.20	---	---
21	1.444	1.331	1.714	1.496	30	2
22	---	---	---	---	---	---
23	0.225	0.232	0.234	0.230	3.07E-06	2
24	0.858	0.810	0.844	0.837	30	2
25	---	---	---	---	---	---
26	3.758	3.751	3.755	3.755	0.13	2
27	---	---	---	---	---	---
28	0.0006	0.002	0.002	0.002	0.02	2
29	5.156	5.109	5.204	5.156	3.9	2
30	---	---	---	---	---	---
31	2.061	2.044	2.094	2.066	33	2
32	---	---	---	---	---	---
33	2.254	2.158	2.216	2.21	34	2
34	---	---	---	---	---	---
35	1.432	1.434	1.696	1.521	21.6	2.23
36	1.660	1.682	1.702	1.681	0.333	2
37	---	---	---	---	---	---
38	1.303	1.286	1.273	1.287	2	---
39	0.625	0.611	0.639	0.625	0.05	2
40	2.052	2.032	2.066	2.050	13.4	2
41	1.40	1.43	1.56	1.46	10	---
42	0.954	0.648	0.592	0.73	27	2
43	2.60	2.40	2.50	2.50	12.2	3
44	0.52	0.52	0.53	0.52	50	2
45	1.153	1.116	1.103	1.124	---	---
46	1.533	1.597	1.554	1.562	8.094	2
47	2.21	2.21	2.28	2.23	0.07	2
48	1.61	1.8	1.61	1.67	20	2
49	1.6547	1.7097	1.6545	1.67	8.14	2
50	0.341	0.335	0.369	0.35	6.0	4.3
51	1.82	1.79	1.45	1.69	15	2
52	1.67	1.79	1.87	1.78	---	---
53	2.696	3.031	2.957	2.895	36.8	2
54	2.38	2.23	2.45	2.35	35	2
55	1.263	1.25	1.405	1.306	25.8	2
56	1.982	1.996	2.064	2.01	3	2
57	0.382	0.407	0.393	0.394	21	2
58	2.49	2.50	2.51	2.50	5.33	2.262
59	0.602	0.549	0.568	0.573	0.41	2.87
60	---	---	---	---	---	---
61	---	---	---	---	---	---
62	---	---	---	---	---	---
63	---	---	---	---	---	---
64	1.32	1.37	1.30	1.33	28.86	2
65	0.394	6.325	1.167	2.629	47.26	2
66	2.028	2.039	2.011	2.026	25.6	2
67	2.155	2.131	2.110	2.132	5.10	2
68	2.250	2.210	1.650	2.037	16.47	---
69	1.552	1.547	1.572	1.557	3.14	2
70	---	---	---	---	---	---

“---” denotes data/information was not provided by participants

APPENDIX XIV: Methodologies & Techniques Used by Participants

Lab Code	Instrumentation	GC Column	Cleanup	Accreditation Status
1	GC-MSD	1701P	SPE	NO
2	GC-ECD	---	---	NO
3	GC-ECD	VF-5MS	GPC	NO
4	GC-ECD	HP5	---	---
5	GC-ECD	DB5, DB17	GPC	YES
6	GC-ECD, GC-MS/MS	RTX-CLP	---	NO
7	GC-MSD	DB1	SPE	YES
8	GC-ECD	DB-XLB, DB-35	Florisil	YES
9	GC-ECD, GC-MSD	DB-17, HP-5MS	GPC, Florisil	YES
10	GC-ECD, GC-MSD	---	Liquid-liquid partition	YES
11	GC-MSD	RTX-5Sil MS	NO	NO
12	GC-MSD	VF-5MS	DSPE	NO
13	GC-MSD	Alltech AT-5MS	NO	YES
14	GC-ECD, GC-MSD	DB-608	Florisil	NO
15	GC-MSD	VF-5MS	NO	NO
16	GC-ECD	SPB-608	---	YES (α - & β -BHC only)
17	GC-ECD	DB17	Acid treatment	YES
18	GC-ECD, GC-MSD	HP5, DB-35MS	GPC, Florisil	YES
19	GC-ECD	HP-5	---	YES
20	GC-ECD	CP-Sil5CB, CP-Sil9CB	GPC	NO
21	GC-ECD, GC-MSD	HP-1701, HP-5MS	Florisil	YES
22	GC-ECD	DB-608	Florisil	YES
23	GC-ECD	HP-5	NO	NO
24	GC-ECD, GC-MSD	DB-35MS	Florisil	YES
25	GC-MSD	HP-5MS	GPC	NO
26	GC-ECD	SPB-1701	---	NO
27	GC-ECD	HP35	GPC	YES
28	GC-ECD	RTX-5	Florisil	---
29	GC-MSD	DB-5MS	Florisil	---
30	GC-ECD, GC-MSD	HP-5MS	NO	NO
31	GC-ECD	SE-54	---	YES (except HCB)
32	GC-MSD	HP-5MS	---	NO
33	GC-MSD	DB1701	---	YES
34	GC-ECD	---	C ₁₈ SPE	NO
35	GC-ECD	phenyl (5%) methylsiloxane	GPC + H ₂ SO ₄	NO
36	GC-ECD	Zebron	SPE Florisil	YES
37	GC-ECD	HP-608	Al activated	---
38	GC-MSD	HP-5	NO	YES
39	GC-ECD	DB-608	NO	NO
40	GC-ECD	DB1701	Conc. H ₂ SO ₄	YES
41	GC-ECD	---	Florisil	NO
42	GC-ECD, GC-MSD	BPX-50 + BPX-5; DB-XLB	GPC	YES
43	GC-MSD	DB-5	GPC	YES
44	GC-MSD	HP-5MS	GPC	YES
45	GC-ECD	HP-5M	GPC	NO
46	GC-ECD, GC-MSD	HP-5	Florisil	YES
47	GC-ECD	DB-5	NO	---
48	GC-ECD	HP-5MS	GPC	NO
49	GC-ECD	HP-5	NO	YES
50	GC-ECD	SPB-5	NO	NO
51	GC-ECD, GC-MSD	DB-5	---	NO
52	GC-ECD	DB-1701, DB-1	Silica gel SPE	YES
53	GC-ECD	SPB-5	SPE	---
54	GC-ECD	DB-608, RTXOC-Pesticides	SPE	NO
55	GC-ECD, GC-MSD	DB-17MS, DB-5MS	GPC; SPE	YES
56	GC-ECD	DB-1701	---	YES
57	GC-MSD	HP-5MS	LLE + Florisil	YES
58	GC-ECD	HP-5	Florisil	YES
59	GC-ECD	CP-Sil 8CB	Silica gel SPE	---
60	GC-MSD	Zebron ZB5MS	---	NO
61	GC-ECD	HP-5MS, DB-1, HP35	Neutral Alumina SPE	YES (except δ -BHC)
62	GC-ECD	DB-1	Basic Alumina SPE	NO
63	GC-ECD, GC-uECD	DB5MS	GPC	YES
64	GC-MSD	HP-5MS	GPC; SPE	NO
65	GC-ECD	DB-624	SPE	NO
66	GC-ECD	HP-5	---	YES
67	GC-ECD	DB-1701	Conc. H ₂ SO ₄	YES
68	GC-MSD	5MS	---	NO
69	GC-ECD	DB-5	Florisil	YES
70	GC-MSD	DB-5	SPE Florisil	NO

“---” denotes data/information was not provided by participants

The above summary was based on the information given in Part II of the *Results Proforma*

APPENDIX XV: Consensus Mean and Standard Deviation

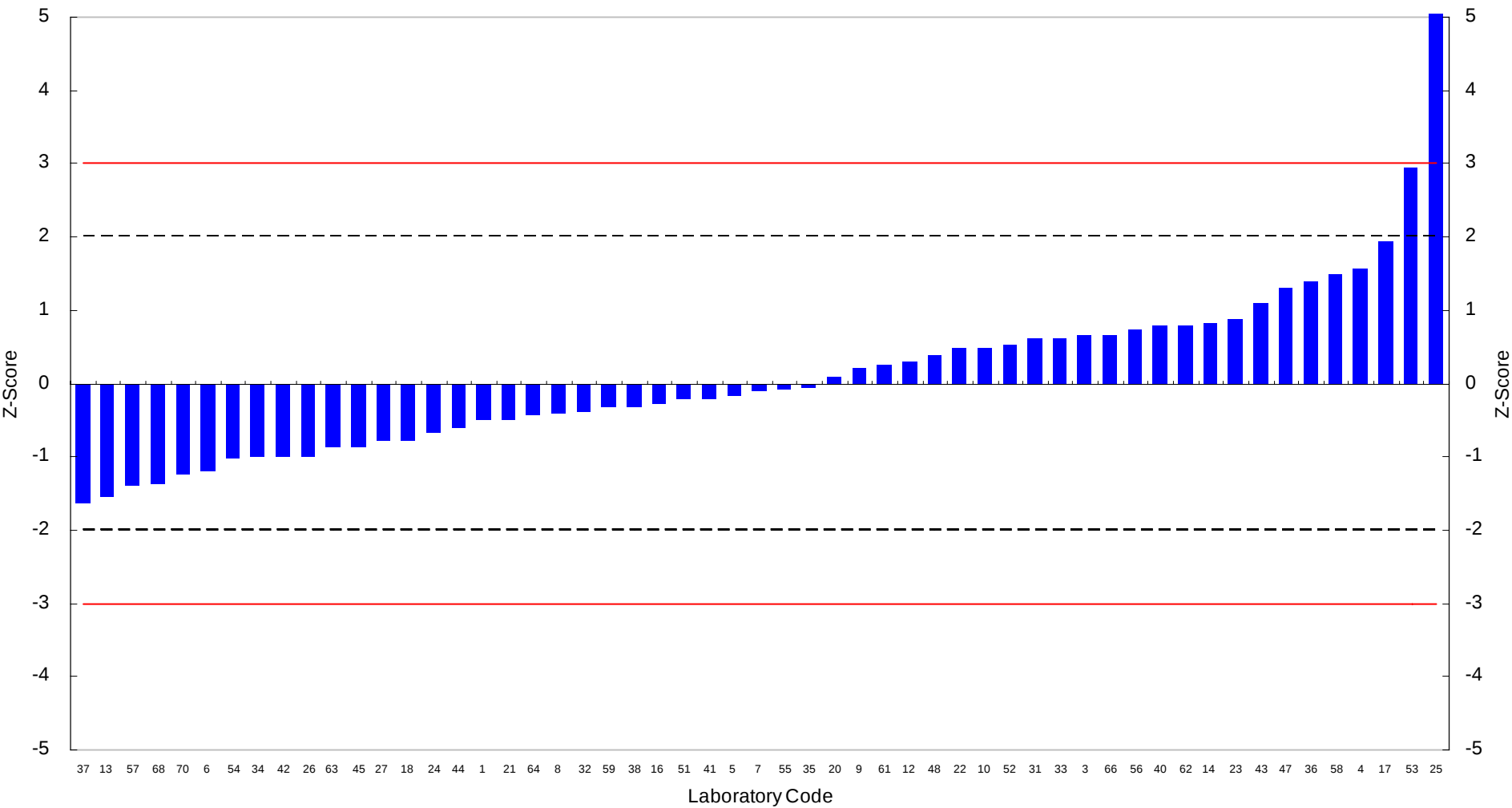
1. All participants' results were included in the computation of the mean and SD, in mg/kg, by robust test.
2. The statistical parameters were reported as follows:

	HCB	α-BHC	β-BHC	γ-BHC	δ-BHC
Mean	0.170	0.393	0.219	0.339	1.54
SD	0.095	0.188	0.103	0.149	0.816

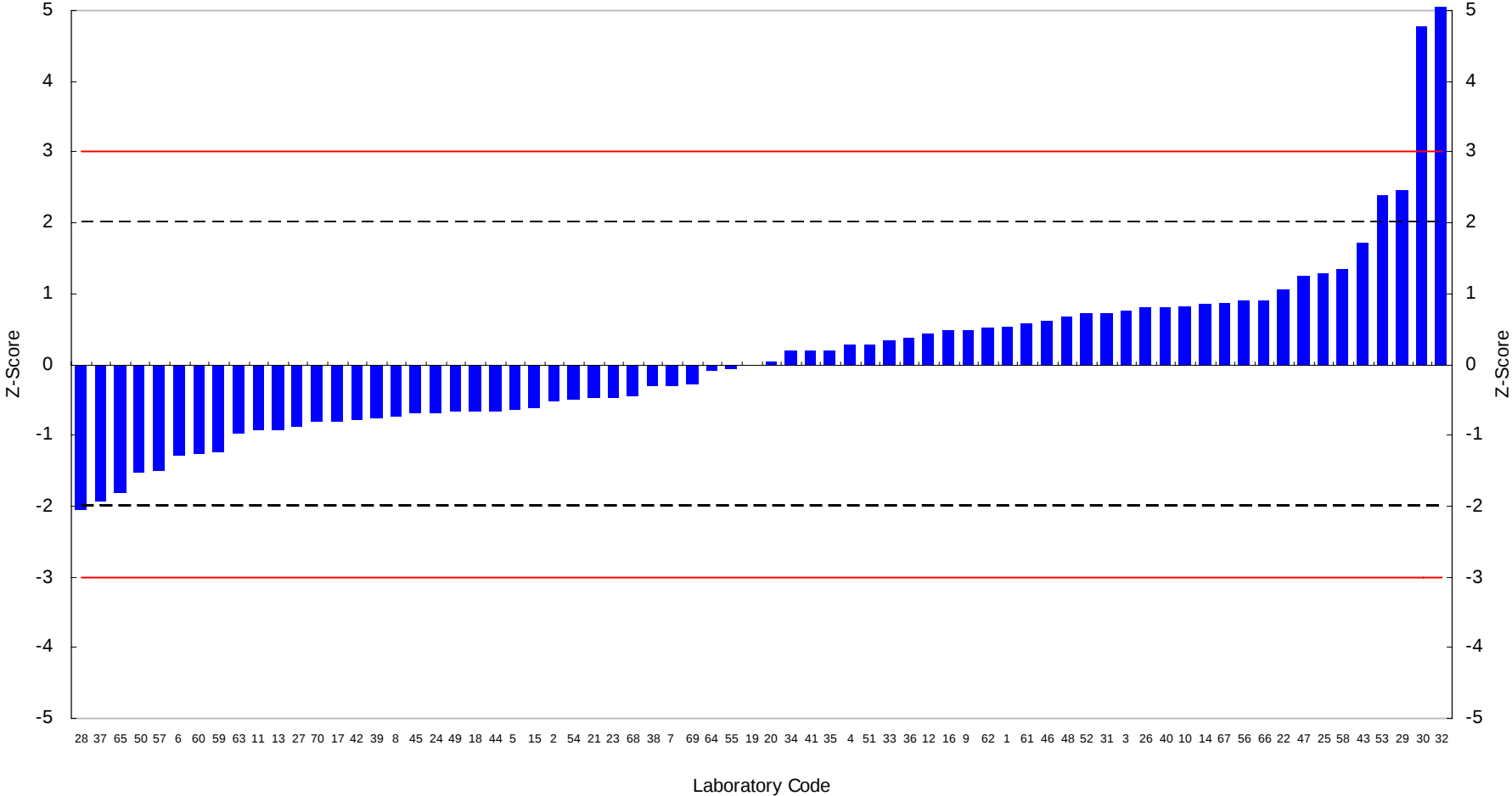
APPENDIX XVI: z-Scores of Participants

Lab Code	HCB	α -BHC	β -BHC	γ -BHC	δ -BHC
1	-0.49	0.53	0.21	0.00	0.75
2	---	-0.51	-1.1	-0.77	-0.95
3	0.67	0.76	-0.05	-0.02	-0.50
4	1.6	0.27	-0.26	-0.78	1.1
5	-0.17	-0.63	-0.96	-0.67	-1.1
6	-1.2	-1.3	-1.2	-0.85	-0.69
7	-0.11	-0.30	0.17	-0.50	0.14
8	-0.41	-0.74	-0.91	-1.1	-0.84
9	0.22	0.49	-0.06	0.16	0.36
10	0.49	0.83	0.20	0.73	1.0
11	---	-0.93	-1.1	-1.1	-0.69
12	0.30	0.43	0.20	0.18	-0.61
13	-1.5	-0.92	0.88	0.10	-0.45
14	0.82	0.86	0.48	0.98	0.63
15	---	-0.62	0.69	-0.27	-0.69
16	-0.26	0.48	-1.2	-0.32	---
17	1.9	-0.81	0.86	1.1	1.5
18	-0.77	-0.66	-0.87	-1.03	-1.3
19	---	-0.01	-0.15	-0.55	-0.13
20	0.10	0.04	-0.77	-0.55	-0.41
21	-0.49	-0.46	1.6	-0.25	-0.05
22	0.48	1.1	0.46	0.76	---
23	0.89	-0.46	12*	-1.2	-1.6
24	-0.67	-0.68	-0.71	-0.93	-0.86
25	45*	1.3	0.91	0.70	---
26	-0.99	0.79	-1.3	0.06	2.7
27	-0.78	-0.88	-1.3	5.4*	---
28	---	-2.0	-2.1	-2.3	-1.9
29	---	2.5	4.4*	0.85	4.4*
30	---	4.8*	5.6*	8.6*	---
31	0.62	0.72	0.37	0.41	0.65
32	-0.39	5.4*	4.3*	7.7*	---
33	0.63	0.36	0.09	0.49	0.82
34	-1.0	0.20	0.57	0.01	---
35	-0.06	0.22	-0.15	-0.02	-0.02
36	1.4	0.36	0.89	-0.58	0.18
37	-1.6	-1.9	-2.0	-2.1	---
38	-0.31	-0.30	-0.15	-0.32	-0.31
39	---	-0.76	-0.70	-1.2	-1.1
40	0.80	0.80	0.29	0.46	0.63
41	-0.20	0.20	-0.61	-0.20	-0.09
42	-1.0	-0.78	-1.1	-1.2	-0.99
43	1.1	1.7	0.82	1.2	1.2
44	-0.60	-0.65	-0.57	-0.82	-1.2
45	-0.86	-0.69	-0.80	-1.0	-0.51
46	---	0.62	0.55	2.1	0.03
47	1.3	1.3	0.98	0.77	0.85
48	0.38	0.67	0.13	0.41	0.17
49	---	-0.67	0.00	-0.07	0.17
50	---	-1.5	1.2	1.3	-1.5
51	-0.22	0.28	-0.27	0.13	0.18
52	0.53	0.72	0.06	0.43	0.29
53	2.9	2.4	1.7	1.9	1.7
54	-1.0	-0.50	-0.68	-0.83	1.0
55	-0.07	-0.06	-0.12	-0.40	-0.28
56	0.74	0.89	0.33	0.69	0.58
57	-1.4	-1.5	---	-1.7	-1.4
58	1.5	1.4	5.1*	1.4	1.2
59	-0.33	-1.2	-1.2	1.3	-1.2
60	---	-1.3	---	1.3	---
61	0.25	0.58	-0.22	0.24	---
62	0.81	0.52	-0.17	0.32	---
63	-0.86	-0.96	-0.74	-1.2	---
64	-0.42	-0.08	0.33	-0.35	-0.25
65	---	-1.8	-1.8	---	1.34
66	0.67	0.90	0.33	0.70	0.60
67	---	0.87	0.46	0.71	0.73
68	-1.4	-0.44	2.1	-1.8	0.61
69	---	-0.27	-0.52	-0.16	0.02
70	-1.2	-0.81	---	-0.33	---

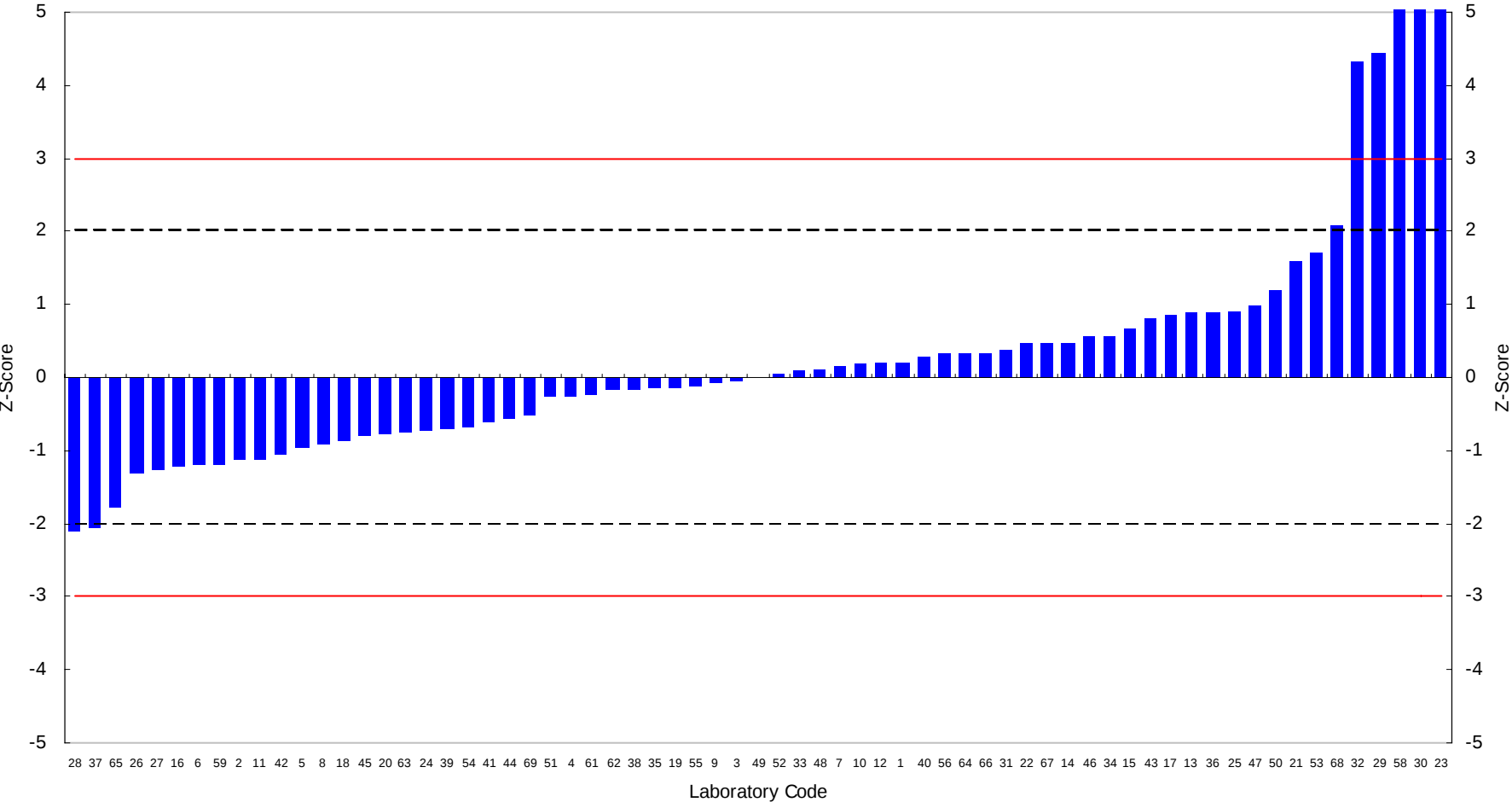
APPENDIX XVII: z-Scores of Participants for HCB



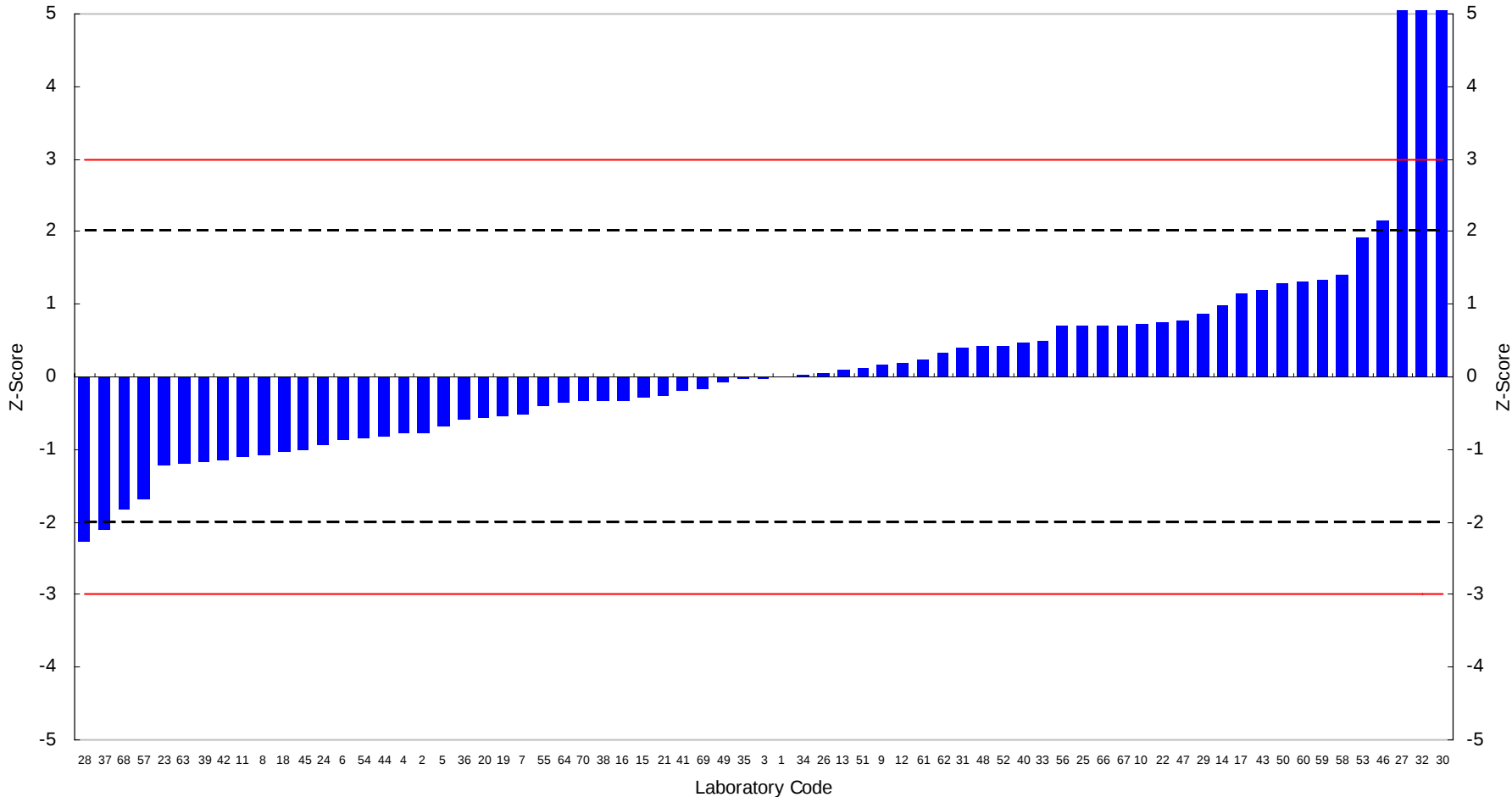
APPENDIX XVIII: z-Scores of Participants for α -BHC



APPENDIX XIX: z-Scores of Participants for β-BHC



APPENDIX XX: z-Scores of Participants for γ -BHC



APPENDIX XXI: z-Scores of Participants for δ -BHC

