JRC Scientific and Technical Reports

Project HORIZONTAL Validation Report
on polychlorinated dibenzodioxins, polychlorinated dibenzofurans and dioxin-like polychlorinated biphenyls

Validation of a horizontal standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludges and treated biowaste using high resolution mass selective detection (HR GC-MS) in a European Intercomparison Exercise

E. Sobiecka, H. van der Sloot, W. Moche, B. M. Gawlik

JRC
EUROPEAN COMMISSION

ies
Institute for
Environment and
Sustainability

The mission of the Institute for Environment and Sustainability is to provide scientific-technical support to the European Union's Policies for the protection and sustainable development of the European and global environment.

European Commission
Joint Research Centre
Institute for Environment and Sustainability

A great deal of additional information on the European Union is available on the Internet.
It can be accessed through the Europa server
http://europa.eu/

Printed in Luxemburg

Project HORIZONTAL Validation Report

Validation of a horizontal standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludges and treated biowaste using high resolution mass selective detection (HR GC-MS) in a European Intercomparison Exercise

E. Sobiecka, H. Van der Sloot, W. Moche, B. M. Gawlik*⌨

Elzbieta Sobiecka, Bernd Manfred Gawlik
European Commission, Joint Research Centre
Institute for Environment and Sustainability
Via Enrico Fermi
21020 Ispra - Italy

Hans van der Sloot
ECN
Emission Characterisation and Reduction
Westerduinweg 3, P.O. Box 1
1755 ZG Petten
The Netherlands

Wolfgang Moche
Umweltbundesamt
Gruppe Umweltanalytik & Chemikalien, Spittelauer Lände 5
1090 Vienna
Austria

⌨ to whom correspondence should be sent
by email to bernd.gawlik@jrc.it

Summary

Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. In the context of this standardization project, a series of draft technical specification were designed upon an extensive desk study, fine-tuned after expert consultations and finally validated in international intercomparisons exercise.

This report summarises the work performed within the validation study of the draft standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludge and treated bio-waste using high resolution mass selective detection (HR GC-MS). It further explains the underlying statistical concept for the calculation of reproducibility and repeatability from intercomparisons data. In addition all single values, results of the statistical evaluation as well as background information on the validation materials used are described and explained.

Abbreviations

Throughout this report the following abbreviations are used:

ANOVA	Analysis of variances		
CAS	Chemical Abstracts System	ISO	International Organization for Standardisation
CEN	Comitteé Européen de Normalisation	JRC	Joint Research Centre
DG	Directorate General	MILC	Measure Interlaboratory Comparison
DL PCB	Dioxins-like Polychlorinated Biphenyls	p	Number of labs
ECD	Electron Capture Detector	PCDD	Polychlorinated Dibenzodioxins
ECN	Energy Research Centre for the Netherlands	PCDF	Polychlorinated Dibnzofurans
EU	European Union	r	Repeatability limit
HR GC-MS	High Resolution Mass Selective Detection	R	Reproducibility limit
IES	Institute for Environment and Sustainability	s_r	Repeatability standard deviation
IT	Information Technology	s_R	Reproducibility standard deviation
		TC	Technical Committee

Table of Contents

Introduction to the validation project

Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. It was created as in response to the European Commission Mandate M 330 given to CEN, asking for the development and validation of those standards in support of forthcoming EU Directives, such as:

- o The revision of the Sewage Sludge Directive 86/278/EEC.
- o The Directive on the biological treatment of biodegradable waste.
- o The initiative on a legal framework for soil monitoring in Europe.

This mandate explicitly considers standards for the entire analytical procedure (i.e., sampling, pre-treatment and analytical measurement methods for inorganic, organic, hygiene and biological parameters). These are grouped into classes according to their physical/chemical properties, which in turn determine the methods needed to quantify the potential impact on human and animal health, plant uptake, soil function and groundwater quality. As the materials generally feature a mixture of different types of contaminants, it is important to provide an integrated answer covering evaluation of all relevant pollutants.

In order to fulfil the requirements of the aforementioned mandate, the European Commissions Joint Research Centre (JRC) and its Directorate-General for Environment (DG ENV) together with the Technical Committees of the European Standardisation Committee (CEN TCs) concerned designed a pre-normative research initiative called Project HORIZONTAL and presented it to the Commission and the Environmental Authorities in the Member States.

After an extensive literature research and careful evaluation of the feasibility of a given horizontal standard, the standards were drafted and finally validated in a European laboratory intercomparison.

The underlying statistical concept, information about the materials used, details about the participants, measurement results obtained as well as the derived performance characteristics obtained for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) are described hereafter.

1.1 Statistical concept underlying the validation

According to the requirements of the work package concerning data handling & interpretation of the project HORIZONTAL-ORG the respective validation intercomparisons have to be evaluated according to the principles laid down in ISO standard 5725-2:1994. In particular repeatability and reproducibility of the draft standard methods have to be determined. The determination of trueness would require the availability of independent reference values for the materials investigated. This, however, is not possible and was not requested in the frame of this work. In the following, the approach chosen is explained.

1.1.1 Introduction to the statistical model

The statistical model used in ISO 5725 for estimation of accuracy of a measurement method assumes that every test result is the sum of three components:

$$y = m + B + e$$

y: test result

m: general mean

B: laboratory component of bias under repeatability conditions

e: random error occurring in every measurement under repeatability conditions

In the workprogram the quantification of term *e* is explicitly asked for (i.e. repeatability and reproducibility). The repeatability variance is measured directly as the variance of the error term *e*, but the reproducibility depends on the sum of the repeatability variance and the between-laboratory variance:

$$\sigma_r = \sqrt{\mathrm{var}(e)}$$

$$\sigma_R = \sqrt{\sigma_L^2 + \sigma_r^2} \qquad \text{with} \quad \sigma_L = \sqrt{\mathrm{var}(B)}$$

However, soil, biowaste and sludge are multi-phase materials, i.e. they contain two or more distinct types of particles which are fundamentally different in their properties and composition. As a consequence, this introduces an important source of variation for the intercomparison exercise which needs to be considered, i.e. the inherent heterogeneity of the materials.

Thus, a contribution of variation between samples *H* is introduced to the general statistical model:

$$y = m + B + e + H$$

Using ANOVA techniques the different variances are calculated and separated for the evaluation.

1.1.2 Requirements for precision experiment

Layout of the experiment

A suite of 10 to 12 different materials (soil, sludge and biowaste) has been made available for the intercomparison exercise. For each parameter investigated, at least 10 to laboratories should be nominated to participate. The same laboratories should be used for different parameters as far as possible. Due to the complexity of analysis and the respective workload to the laboratories, it was decided to propose three materials for the validation of the PCDD, PCDF and DL PCB draft standard.

Each laboratory received two bottles of each material and was be requested to perform 4 independent analysis per material[1] (2 per bottle) using the respective draft standard methods. The 4 analysis per material should be carried out under repeatability conditions (i.e. same operator[2], same equipment, within a short period of time). As far as possible, also the different materials should be measured under repeatability conditions; however, changes of e.g. operator or equipment are permitted, but must be reported. Likewise, different materials can be analysed on different days if necessary.

Equipment used in the experiment needed to be checked prior to the experiment according to the requirements of the draft standard. The results of these checks have to be documented. Similarly, date <u>and</u> time of each measurement had to be recorded for verification of repeatability conditions.

An appropriate timeframe for the entire exercise has been set and was to be respected.

Recruitment of the laboratories

Each sub-workpackage leader of HORIZONTAL was asked to select the laboratories using the information from section 5.2 of ISO 5725-2:1994 and provide the signed questionnaires (see also Annex 1). The workpackage leaders were responsible for providing the laboratories with the draft standard method and explaining the context of this exercise.

Preparation and use of the materials

Materials used for the exercise were prepared according to the general requirements for reference materials as laid down in ISO Guide 34. Materials were accompanied by instructions for use.

Reporting of results

Online submissions of results using an internet-based IT platform as well as XLS-Spreadsheets were used. In case of online data submission, the participating laboratories received a unique and confidential login and password in due time, enabling them to

---

[1] Independent analysis means analysis of independent test portions, applying the entire analytical scheme to this test portion, from e.g. extraction to quantification. For instance it does not mean replicate injections of aliquots into a GC-MS instrument.
[2] Operator in this context may also consist of a fixed team of persons, e.g. one person performing extraction, one clean-up, one quantification.

enter their data in a structured form. For authentication purposes a signed printout had to be submitted by mail.

The online data submission included a detailed questionnaire for additional information on the measurements.

1.1.3 Statistical analysis

Statistical analysis of data followed the requirements of ISO 5725-2:1994 and ISO 5725-5:1998. Appropriate tests for the homogeneity of variance, detection of outliers and normal distribution were applied. Statistical evaluation was done using an Excel Macro, developed, tested and successfully applied in other occasion by ECN. Evaluation was executed jointly by JRC and ECN.

1.2 Validation exercise for PCDD, PCDF and DL PCB

1.2.1 Isomers to be measured

The following PCDD isomers were selected for the validation exercise: 2,3,7,8,-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD.

The following PCDF isomers were selected for the validation exercise: 2,3,7,8,-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; OCDF;

The following DL PCB congeners were selected for the validation exercise:

PCB-77, PCB-81, PCB-105, PCB-114, PCB-118, PCB-123, PCB-126, PCB-156, PCB-157, PCB-167, PCB-169, PCB-189

1.2.2 Samples dispatched for the validation of PCDD, PCDF and DL PCB

After a preliminary rough screening, the following materials were used for the validation round of PCDD, PCDF and DL PCB.

- Compost 1   A pollutant loaded compost material from Vienna
- Sewage Sludge 1   A mixed sewage sludge from Essen
- Soil 3   A sludge amended soil from Barcelona

A more detailed description of background concentrations can be found in Annex 2 to this report. The samples were dispatched simultaneously to all participants using a private courier service.

1.2.3 Draft standards to be followed

The draft standards to be followed could be downloaded following this link, which is situated on the website of the Project HORIZONTAL:

http://www.ecn.nl/docs/society/horizontal/Dioxin_Furan_DL-PCB_Standard_for_validation.pdf

1.2.4 Analytical program

Of each of the three materials 2 bottles had to be analyzed and each bottle had to analyze independently twice. As mentioned above analyses were to be done under repeatability conditions. Results were to be reported referring to DRY MATTER content. The choice, how to apply d.m. correction was free for each participant.

1.2.5 Timing and Submission of data

Dispatch of samples was done on the 18th of October 2006. For users of the Online data submission system (MILC), User Registration was possible from 14th of November 2006 with opening of the MILC Data Submission on 1st of December 2006. The deadline for submission of results has been set for PCDD, PCDF and DL PCB to the 12th of January 2007, but was extended to the end of the same months. After that no further submission was possible.

Alternatively the participants were allowed to submit data electronically as Excel sheet using simply Email.

All data were treated in a confidential way. Any presentation hereafter will refer only to numerical data and it will not be possible to identify the originating laboratory. Lab Codes displayed are NOT related to the order of laboratories hereafter.

In addition to the information provide a Helpdesk was implemented in order to give quick and individual response to the participants during and immediately after the validation study. In case of doubt and suspected transcription errors, further enquires were conducted by JRC.

1.2.6 Participants

The following table lists the participating organizations and entities in the validation exercise for the horizontal PCDD, PCDF and DL PCB standard;

- Austria
    - Umweltbundesamt
- Belgium
    - VITO
- Finland
    - Consulting Engineers Paavo Ristola Ltd.
- France
    - INRA
- Italy
    - European Commission DG JRC IES
- Sweden
    - ALCONTROL AB

1.3 Summary results and derived performance characteristics

The result of the various statistical evaluation including outlier tests, calculation of repeatability and reproducibility standard deviation for the congeners of interest can be found in Annex 3 of this report. In addition, all data submitted by the participants as well as those considered for the calculation of the performance characteristics are listed in Annex 3 to this report.

Based on these calculations the following results were obtained in the validation round upon statistical evaluation according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table1).

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures. The repeatability limit was calculated using the relationship: $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$ with the critical range factor $f = 2$.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures. The reproducibility limit was calculated using the relationship: $R = f \cdot \sqrt{2} \cdot s_R$ with the critical range factor $f = 2$.

Table 1 - Results of the interlaboratory comparison studies of the determination of PCDD, PCDF and DL PCB by HR GC-MS in treated biowaste, sludge and soil. All concentrations are expressed in ug/kg dm.

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	DL PCB 77	6.666	5.01%	11.7%	0.94	2.18	5	0	19	0	19
Compost 1	DL PCB 77	0.132	7.01%	11.7%	0.03	0.04	4	0	16	0	16
Soil 3	DL PCB 77	*0.0258*		*17.4*			3		11	8	19
Sludge 1	DL PCB 81	0.524	10.52%	48.1%	0.15	0.71	5	0	19	0	19
Compost 1	DL PCB 81	*0.0190*		*79.7*			2		8	8	16
Soil 3	DL PCB 81	*0.0022*		*66.5*			3		8	8	16
Sludge 1	DL PCB 105	19.184	7.07%	14.7%	3.80	7.92	4	1	16	0	18
Compost 1	DL PCB 105	0.683	9.19%	19.4%	0.18	0.37	4	0	16	0	16
Soil 3	DL PCB 105	0.096	5.47%	12.3%	0.01	0.03	4	0	15	4	19
Sludge 1	DL PCB 114	1.369	4.91%	28.8%	0.19	1.10	4	1	16	0	20
Compost 1	DL PCB 114	*0.0504*		*60.2*			2		8	8	16
Soil 3	DL PCB 114	*0.0255*		*154.1*			3		10	8	18
Sludge 1	DL PCB 118	32.585	2.59%	12.0%	2.37	10.92	4	1	16	0	20
Compost 1	DL PCB 118	1.945	9.74%	15.6%	0.53	0.85	4	0	16	0	16
Soil 3	DL PCB 118	0.218	7.46%	19.5%	0.05	0.12	5	0	19	0	19
Sludge 1	DL PCB 123	*1.9351*		*89.0*			5		20	0	20
Compost 1	DL PCB 123	*0.1192*		*84.8*			2		12	4	16
Soil 3	DL PCB 123	*0.0081*		*37.8*			3		8	11	19

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	DL PCB 126	0.250	11.41%	33.0%	0.08	0.23	5	0	19	0	19
Compost 1	DL PCB 126	*0.0359*		*17.8*			2		8	8	16
Soil 3	DL PCB 126	*0.0024*		*35.5*			3		10	9	19
Sludge1	DL PCB 156	7.310	3.54%	10.2%	0.72	2.09	4	1	15	1	20
Compost 1	DL PCB 156	0.882	27.30%	28.7%	0.67	0.71	4	0	16	0	16
Soil 3	DL PCB 156	*0.0453*		*12.6*			3		12	7	19
Sludge 1	DL PCB 157	0.864	6.54%	19.3%	0.16	0.47	4	1	16	0	20
Compost 1	DL PCB 157	0.102	17.20%	21.7%	0.05	0.06	4	0	16	0	16
Soil 3	DL PCB 157	*0.0079*		*18.2*			3		8	11	19
Sludge 1	DL PCB 167	*4.0002*		*51.8*			5		20	0	20
Compost 1	DL PCB 167	*0.7498*		*96.5*			2		16	0	16
Soil 3	DL PCB 167	*0.0195*		*16.3*			3		8	11	19
Sludge 1	DL PCB 169	*0.0415*		*81.9*			3		12	6	18
Compost 1	DL PCB 169	*0.0034*		*17.4*			2		8	8	16
Soil 3	DL PCB 169	*0.0122*		*254.8*			3		7	9	16
Sludge 1	DL PCB 189	*1.1565*		*67.2*			5		20	0	20
Compost 1	DL PCB 189	0.177	37.36%	37.4%	0.18	0.18	4	0	16	0	16
Soil 3	DL PCB 189	*0.0076*		*14.7*			3		8	11	19
Sludge 1	Total DL PCB	75.639	1.92%	13.5%	4.07	28.52	4	1	15		
Compost 1	Total DL PCB	4.664	17.71%	24.3%	2.31	3.18	4	0	16		
Soil 3	Total DL PCB	0.370	11.94%	18.9%	0.12	0.20	5	0	19		

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,4,6,7,8-HpCDD	185.260	11.65%	20.0%	60.44	103.75	6	0	24	4	28
Compost 1	1,2,3,4,6,7,8-HpCDD	175.856	7.85%	40.7%	38.65	200.49	5	0	20	0	20
Soil 3	1,2,3,4,6,7,8-HpCDD	6.538	8.15%	18.8%	1.49	3.45	4	2	15	0	23
Sludge 1	1,2,3,4,7,8-HxCDD	3.498	6.90%	14.1%	0.68	1.38	4	1	15	5	24
Compost 1	1,2,3,4,7,8-HxCDD	0.74		17.6			2		8	12	20
Soil 3	1,2,3,4,7,8-HxCDD	0.07		41.9			2		5	18	23
Sludge 1	1,2,3,6,7,8-HxCDD									24	24
Compost 1	1,2,3,6,7,8-HxCDD	3.61		23.3			3		12	8	20
Soil 3	1,2,3,6,7,8-HxCDD	0.33		26.7			3		5	18	23
Sludge 1	1,2,3,7,8,9-HxCDD	6.427	14.43%	48.7%	2.60	8.77	4	1	16	4	24
Compost 1	1,2,3,7,8,9-HxCDD	2.086	9.86%	30.1%	0.58	1.76	4	0	14	6	20
Soil 3	1,2,3,7,8,9-HxCDD	0.43		174.0			3		5	18	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,7,8-PeCDD	2.285	18.09%	54.0%	1.16	3.46	4	0	16	8	24
Compost 1	1,2,3,7,8-PeCDD	0.54		12.9			2		8	12	20
Soil 3	1,2,3,7,8-PeCDD	0.10		15.2			1		4	19	23
Sludge 1	OCDD	1478	13.83%	22.8%	572.63	942.60	6	0	24	0	24
Compost 1	OCDD	1031	11.19%	42.9%	323.00	1238.75	5	0	20	0	20
Soil 3	OCDD	56.9	5.10%	24.7%	8.13	39.34	5	1	19	0	23
Sludge 1	2,3,7,8-TCDD	0.77		32.9			3		9	15	24
Compost 1	2,3,7,8-TCDD	0.17		12.4			1		4	16	20
Soil 3	2,3,7,8-TCDD	0.55		74.8			1		2	22	24
Sludge 1	OCDF	196.966	13.90%	22.0%	76.68	121.33	6	0	24	0	24
Compost 1	OCDF	26.395	24.25%	27.6%	17.92	20.41	4	1	16	0	20
Soil 3	OCDF	42.273	9.34%	21.4%	11.06	25.37	5	1	19	0	23
Sludge 1	1,2,3,4,6,7,8-HpCDF	95.411	8.97%	19.2%	23.96	51.19	6	0	24	0	24
Compost 1	1,2,3,4,6,7,8-HpCDF						5		20	0	20
Soil 3	1,2,3,4,6,7,8-HpCDF	12.424	20.04%	21.6%	6.97	7.52	6	0	23	0	23
Sludge 1	1,2,3,4,7,8,9-HpCDF	7.407	23.63%	34.7%	4.90	7.20	5	0	20	4	24
Compost 1	1,2,3,4,7,8,9-HpCDF	1.98		73.9			4		12	8	20
Soil 3	1,2,3,4,7,8,9-HpCDF	0.21		21.8			2		6	17	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,4,7,8-HxCDF	*14.09*		*66.3*			5	0	20	4	24
Compost 1	1,2,3,4,7,8-HxCDF	*2.42*		*41.1*			4		16	4	20
Soil 3	1,2,3,4,7,8-HxCDF	*0.46*		*165.1*			2		10	13	23
Sludge 1	1,2,3,7,8,9-HxCDF	*2.32*		*82.0*			4		15	9	24
Compost 1	1,2,3,7,8,9-HxCDF	*0.22*		*36.1*			4		6	14	20
Soil 3	1,2,3,7,8,9-HxCDF	*0.05*		*47.3*			2		4	19	23
Sludge 1	1,2,3,6,7,8-HxCDF	7.397	10.87%	21.3%	2.25	4.40	5	0	20	4	24
Compost 1	1,2,3,6,7,8-HxCDF	*2.50*		*93.1*			4		13	7	20
Soil 3	1,2,3,6,7,8-HxCDF	*0.19*		*18.0*			2		7	16	23
Sludge 1	2,3,4,6,7,8-HxCDF	*9.64*		*25.6*			5		19	5	24
Compost 1	2,3,4,6,7,8-HxCDF	*2.35*		*40.6*			5		15	5	20
Soil 3	2,3,4,6,7,8-HxCDF	*1.99*		*222.5*			2		11	12	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,7,8-PeCDF	5.203	8.32%	23.8%	1.21	3.47	4	0	15	9	24
Compost 1	1,2,3,7,8-PeCDF	1.49		35.5			4		12	8	20
Soil 3	1,2,3,7,8-PeCDF	0.17		9.9			2		5	18	23
Sludge 1	2,3,4,7,8-PeCDF	9.784	31.88%	33.3%	8.73	9.12	5	0	20	4	24
Compost 1	2,3,4,7,8-PeCDF	*2.10*		*36.1*			4		14	6	20
Soil 3	2,3,4,7,8-PeCDF	*1.24*		*47.8*			2		5	18	23
Sludge 1	2,3,7,8-TCDF	10.033	6.57%	14.4%	1.85	4.05	4	1	16	8	28
Compost 1	2,3,7,8-TCDF	*2.46*		*26.0*			4		15	5	20
Soil 3	2,3,7,8-TCDF	*0.58*		*78.8*			2		7	16	23

Abbreviations: sr Repeatability standard deviation; SR Reproducibility standard deviation; r Repeatability limit (comparing two measurements); R Reproducibility limit (comparing two measurements); p Number of labs; */* determination not possible.

1.4 Annexes

Annex 1: Model questionnaire to be filled by the participating laboratories

Annex 3: Report on the validation materials used

Annex 2: Statistical calculations

Annex 3: Data submitted

Annex 1:

Model questionnaire to be filled by the participating laboratories

Model questionnaire to be filled by the participating laboratories

Name of laboratory:
Contact person:
Contact details: email:
        Phone:
        Fax:
Mail address of lab:

Dispatch address of lab for shipment of samples (no PO boxes!):

Title of measurement method (copy attached):

Our laboratory is willing to participate in the precision experiment for this draft standard method.

<div align="center">

Yes ☐   No ☐

</div>

As participant we understand that:
- All essential apparatus, chemicals and other requirements specified in the method must be available in our laboratory when the programme begins
- Specified timing requirements such as starting and finishing date of the programme must be rigidly met
- The method must be strictly adhered to
- Samples must be handled in accordance with instructions
- A qualified operator must perform the measurements

Having studied the method and having made a fair appraisal of our capabilities and facilities, we feel that we will be adequately prepared for cooperative testing of this method.

Comments:

………………………………………………………………………………….
            Signature   Date

Annex 2:

Report on the validation materials used

Abstract

This report gives an overview on the available analytical information on the following raw materials to be used for the production of validation materials of the so-called Project HORIZONTAL:

- Four sludge materials from Düsseldorf, Germany,

- An agricultural soil material from Reading, United Kingdom;

- A compost material from Vienna, Austria;

- A compost material from Korschenbroich, Germany;

- A sludge-amended, agricultural soil from Pavia Province, Italy;

- A sludge-amended soil from Barcelona, Spain

- A sludge-amended soil from Essen, Germany

- A long-term sludge exposed soil from Hohenheim, Germany

List of Abbreviations

Throughout this report the following abbreviations are used.

AOX	absorbable organic halogens		LoD	limit of detection
C_{org}	organic carbon content		LUA	Landesumweltamt
C_{total}	total carbon content		N_{total}	total nitrogen content
CAT	cation exchangeable		NH_4-N	Ammonium nitrogen
CDD	chlorinated dibenzodioxin		NO_3-N	Nitrate nitrogen
CDF	chlorinated dibenzofuran		NP	nonylphenol
DEHP	di(2-ethylhexyl)phthalate		NRW	North Rhine Westphalia
DM	dry matter		O	octa
EPA	Environment Protection Agency		P	poly
EU	European Union		PAH	polycyclic aromatic hydrocarbon
FM	fresh matter		PCB	polychlorinated biphenyl
Hp	hepta		Pe	penta
Hx	hexa		T	tetra
IES	Institute for Environment and Sustainability		TEQ	toxicity equivalent
IRMM	Institute for Reference Materials and Measurements		UBA	Umweltbundesamt
JRC	Joint Research Centre		WHO	World Health Organization
LAS	linear alkylsulfonates		WWTP	waste water treatment plant

1 Introduction

This report gives an overview on the available analytical information on the following raw materials to be used for the production of validation materials of the so-called Project HORIZONTAL:

- Four sludge materials from Düsseldorf, Germany,

- An agricultural soil material from Reading, United Kingdom;

- A compost material from Vienna, Austria;

- A compost material from Korschenbroich, Germany;

- A sludge-amended, agricultural soil from Pavia Province, Italy;

- A sludge-amended soil from Barcelona, Spain

- A sludge-amended soil from Essen, Germany

- A long-term sludge exposed soil from Hohenheim, Germany

The following analytical information was gathered partly before and during the sampling of the raw materials, to be used for the production of the HORIZONTAL validation materials. The material were sampled by IES and shipped to IRMM in the course of the year 2005. The information gathered was then completed by various analytical screenings for PAHs and PCBs done by the Institute for Reference Materials and Measurements, Geel, Belgium, for phthalates done by UBA, Berlin, Germany, for PBDE done by IIQAB-CSIC, Barcelona, Spain, for trace elements and some selected major and minor elements by the Institute for Environment and Sustainability, Ispra, Italy.

The work compiled hereafter is based on the numerous additional efforts of the scientists working at various members of the consortium Project HORIZONTAL-Org and contributing organisations.

This work is gratefully acknowledged.

2 Overview on property values

2.1 Sludge materials from Düsseldorf, Germany

The various sewage sludge materials originate from various installations in the North Rhine Westphalia and were produced and sampled by staff from the Landesumweltamt (LUA) NRW under the responsibility from Dr. K. Furtmann.

In total, four sludge materials (Sludge A and D from a major municipal WWTP, Sludge B from a municipal WWTP with industrial input, and Sludge C from a municipal WWTP with high PCB-Content,) were obtained and will be blended to two final materials. Before sampling the following analytical data for a typical sample were received.

Table 1 – Analytical data obtained on an average sludge sample in LUA NRW (with courtesy of K. Furtmann, LUA, Düsseldorf)

Parameter	Concentration
PCB	120 ug/kg
DEHP	110 mg/kg
PAH	5 mg/kg (EPA)
PCDD/F	15 ng TE/kg
PBDE	400 ug/kg
NP	40 mg/kg
LAS	3 g/kg
AOX	300 mg/kg

Subsequent screening led to the information displayed hereafter. It should be stressed that the data were obtained as SCREENING information on the UNTREATED or partially treated raw materials. Therefore, the final target values, which are relevant for the validation intercomparison can be different.

Table 2 – Analytical data obtained on a first screening on the sludge samples from LUA NRW

	Sewage sludge A Dusseldorf	sewage sludge D Dusseldorf
PCB (ng/g)		
28	62	35
52	101	65
101	31	38
118	49	40
153	30	33
105	24	11
138	46	38
156	<1	<1
180	34	23
170	23	19
PAH (ng/g)		
Naphtalene	34	381
Acenaphtylene	15	43
Acenaphthene	81	108
Fluorene	94	1167
Phenantrene		3440
Anthracene	22	344
Flouranthene	316	4817
Pyrene	235	3011
Benz(a)anthracene	473	791
Chrysene	691	1078
Benz(b)fluoranthene	538	1688
Benz(k)fluoranthene	228	635
Benz(a)pyrene	383	1114
Indeno(1,2,3-c,d)pyrene	92	229
Dibenzo(a,h)anthracene	71	70
Benzo(g,h,i)perylene	80	185

Table 3 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	DBP	DCHP	DEHP	Water
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Sludge D (1)		0.135		41.474	3.85
Sludge B (2)	0.538	0.034		30.634	5.47
Sludge A (3)	0.184	0.037		31.399	1.46
Sludge C (4)		0.354	1.528	6.678	2.29

Table 4 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Sludge 2 (B)
Tetra-BDE-47	55.4
Penta-BDE-100	9.59
Penta-BDE-99	69.4
Hexa-BDE-154	5.91
Hexa-BDE-153	7.72
Hepta-BDE-183	5.09
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	9.70
Deca-BDE-209	2216
TOTAL	2379

Table 5 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena, IES, Ispra, Spain). Note that these data are based on single measurements!

	Cd	Co	Cr	Cu	Mn	Ni	Pb	Sb	Tl	V	Zn
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Sludge 1 (D)	2.65	29.0	53.3	359	1231	33.8	78.4	4.38	< 0.05	23.2	786
Sludge 2 (B)	1.19	31.1	62.6	202	278	29.9	72.2	2.51	< 0.05	11.8	625
Sludge 3 (A)	1.68	36.0	62.1	332	847	41.6	119	4.51	< 0.05	11.6	1237
Sludge 4 (C)	5.63	19.8	116	273	726	51.1	473	6.18	< 0.05	44.4	2015

Table 6 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	SiO2 (%)	Al2O3 (%)	CaO (%)	K2O (%)	Fe2O3 (%)	MgO (%)	TiO2 (PPM)	S (PPM)	P2O5 (PPM)
Sludge 1 (D)	21.54	5.8	8.44	0.99	10.3	1.01	4367	<15	50448
Sludge 2 (B)	10.67	3.66	6.92	0.46	14.91	0.77	5217	<15	57633
Sludge 3 (A)	7.31	6.63	6.84	0.35	12.87	0.68	3733	<15	60369
Sludge 4 (C)	43.79	9.65	5.27	1.63	5.22	1.07	5628	<15	23945

Sample	Na2O (%)	Cl (PPM)	Pb (PPM)	Zn (PPM)	Cu (PPM)	Ni (PPM)	Mn (PPM)	Cr (PPM)
Sludge 1 (D)	0.3	2403	101	1002	350	15	1944	132
Sludge 2 (B)	0.31	315	97	879	172	12	514	180
Sludge 3 (A)	0.31	1281	153	1567	265	16	1440	168
Sludge 4 (C)	0.55	231	628	2625	371	81	1101	244

2.2 Agricultural soil material from Reading, United Kingdom

The material was proposed by the University of Reading (S. Nortcliff) and was sampled from a site called "*Frogmore Farm*" which was featured in the "*Metals*" Report for HORIZONTAL. This site is close to Reading with soils developed on flintyloamy periglacial materials over Chalk, has a long and well documented history of sludge application. The focus of the work of Nortcliff *et al.* undertook at this site and the monitoring and control at the site (by Thames Water and the subsequent subsidiary bodies dealing with sludge application to soil) was on metals (and metal loads), with no analysis or indeed any form of investigation in to organics in the broadest sense.

The analytical information produced in the context of the screening of the raw material is displayed below.

Table 7 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	DBP	DCHP	DEHP	Water
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Soil 3 (Reading)		0.032		0.119	6.69

Table 8 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	Co	Cr	Cu	Mn	Ni	Pb	Sb	Tl	V	Zn
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 3 (Reading)	0.15	7.06	27.9	13.8	152	9.01	26.7	3.00	< 0.05	25.8	93.1

Table 9 – Analytical data obtained on a first screening on the sludge-amended soil from Reading (courtesy of IRMM)

Parameter	Concentration
PCB	**ng/g**
28	<1
52	<1
101	<1
118	<1
153	<1
105	<1
138	<1
156	<1
180	<1
170	<1
PAH	**ng/g**
Naphtalene	<10
Acenaphtylene	21
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	<10
Flouranthene	818
Pyrene	776
Benz(a)anthracene	565

9

Parameter	Concentration
Chrysene	608
Benz(b)fluoranthene	824
Benz(k)fluoranthene	329
Benz(a)pyrene	799
Indeno(1,2,3-c,d)pyrene	779
Dibenzo(a,h)anthracene	118
Benzo(g,h,i)perylene	394

Table 10 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 3 (Reading)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	1.03
Hexa-BDE-154	0.03
Hexa-BDE-153	nq
Hepta-BDE-183	nq
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	272
TOTAL	273

Table 11 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	Co	Cr	Cu	Mn	Ni	Pb	Sb	Tl	V	Zn
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 3 (Reading)	0.15	7.06	27.9	13.8	152	9.01	26.7	3.00	< 0.05	25.8	93.1

Table 12 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	SiO2 (%)	Al2O3 (%)	CaO (%)	K2O (%)	Fe2O3 (%)	MgO (%)	TiO2 (PPM)	S (PPM)	P2O5 (PPM)
Soil 3 (Reading)	79.36	4.77	1.12	0.96	1.94	0.17	4107	443	2102

Sample	Na2O (%)	Cl (PPM)	Pb (PPM)	Zn (PPM)	Cu (PPM)	Ni (PPM)	Mn (PPM)	Cr (PPM)
Soil 3 (Reading)	0.42	13	45	69	69	69	216	92

Table 13 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	Hg µg/g
Soil 3 (Reading)	0.12

2.3 Compost from Vienna, Austria

The fresh compost material was obtained from the Austrian Federal Environment Agency (UBA, Vienna), which had used a sub-batch of the raw material for national intercomparson. The remainder of the material was stored at 4°C until shipment to IRMM for further processing. The following analytical information was provided by UBA Austria and completed with various screenings.

Table 14 – Analytical data on compost material received from UBA Austria
Inorganic and sum parameters

Parameter	Unit	Sample fraction used	Observed mean
B CAT	mg/l F.M.	Fresh sample, <10mm	6.1
K CAT	mg/l F.M.	Fresh sample, <10mm	2624
Mg CAT	mg/l F.M.	Fresh sample, <10mm	242
P CAT	mg/l F.M.	Fresh sample, <10mm	49
B CAT	% D.M.	Fresh sample, <10mm	0.0017
K CAT	% D.M.	Fresh sample, <10mm	0.72
Mg CAT	% D.M.	Fresh sample, <10mm	0.07
P CAT	% D.M.	Fresh sample, <10mm	0.01
NO3-N	mg/kg F.M.	Fresh sample, <10mm	3.5
NH4-N	mg/kg F.M.	Fresh sample, <10mm	230
Ctotal	% D.M.	<45°dry, milled	29
Corg	% D.M.	<45°dry, milled	27
Ntotal	% D.M.	<45°dry, milled	1.7
P	mg/kg D.M.	<45°dry, milled	2596
K	mg/kg D.M.	<45°dry, milled	11019
K	% D.M.	<45°dry, milled	1.10
B	mg/kg D.M.	<45°dry, milled	60
Cd	mg/kg D.M.	<45°dry, milled	0.46
Cr	mg/kg D.M.	<45°dry, milled	25
Cu	mg/kg D.M.	<45°dry, milled	46
Hg	mg/kg D.M.	<45°dry, milled	0.20
Ni	mg/kg D.M.	<45°dry, milled	18
Pb	mg/kg D.M.	<45°dry, milled	45
Zn	mg/kg D.M.	<45°dry, milled	198
Ca	mg/kg D.M.	<45°dry, milled	68776
Ca	% D.M.	<45°dry, milled	6.9
Mo	mg/kg D.M.	<45°dry, milled	0.8
S	mg/kg D.M.	<45°dry, milled	2137
Fe	mg/kg D.M.	<45°dry, milled	9959
Mn	mg/kg D.M.	<45°dry, milled	418
Na	mg/kg D.M.	<45°dry, milled	742
Co	mg/kg D.M.	<45°dry, milled	4.1
AOX	mg/kg D.M.	<30° dry, milled	62

Table 15 – Analytical data on compost material received from UBA Austria
Polycyclic aromatic hydrocarbons

PAH	Unit	Result
Naphthaline	µg/kg DM	9.3
Acenaphthylene	µg/kg DM	8.6
Acenaphthene	µg/kg DM	5
Fluorene	µg/kg DM	8.0
Phenanthrene	µg/kg DM	89
Anthracene	µg/kg DM	27
Fluoranthene	µg/kg DM	487
Pyrene	µg/kg DM	380

PAH	Unit	Result
Benzo(a)anthracene	µg/kg DM	278
Chrysene	µg/kg DM	317
Benzo(b)fluoranthene	µg/kg DM	365
Benzo(k)fluoranthene	µg/kg DM	193
Benz(a)pyrene	µg/kg DM	320
Indeno(1,2,3-c,d)pyrene	µg/kg DM	233
Dibenz(a,h)anthracene	µg/kg DM	67
Benzo(g,h,i)perylene	µg/kg DM	225
Sum EPA	µg/kg DM	3013
Sum EPA	mg/kg DM	3.0

Table 16 – Analytical data on compost material received from UBA Austria
Sum PCDDs and PCBs

Parameter			
Dioxine	TEQ (ITEF)	ng/kg DM	7.3
PCB	TEQ (WHO)	ng/kg DM	3.5
	Σ Ballschmiter	mg/kg DM	0.05

Table 17 – Analytical data on compost material obtained by screening in IRMM

Parameter	Result in ng/g
PCB	
28	2
52	2
101	4
118	3
153	10
105	1
138	8
156	1
180	5
170	<1
PAH	
Naphtalene	<10
Acenaphtylene	<10
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	26
Fluoranthene	611
Pyrene	510
Benz(a)anthracene	888
Chrysene	957
Benz(b)fluoranthene	1531
Benz(k)fluoranthene	547
Benz(a)pyrene	1101
Indeno(1,2,3-c,d)pyrene	416
Dibenzo(a,h)anthracene	81
Benzo(g,h,i)perylene	295

Table 18 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Compost 1 (Vienna)
Tetra-BDE-47	4.02
Penta-BDE-100	0.19
Penta-BDE-99	2.59
Hexa-BDE-154	nq
Hexa-BDE-153	0.23
Hepta-BDE-183	0.04
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	1.44
Deca-BDE-209	17.4
TOTAL	25.9

Table 19 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	DBP	DCHP	DEHP	Water
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Compost 1 (Vienna)		0.058		1.426	5.57

Table 20 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	Co	Cr	Cu	Mn	Ni	Pb	Sb	Tl	V	Zn
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Compost 1 (Vienna)	0.39	7.36	31.9	41.0	365	12.7	49.5	0.04	0.79	0.13	208

Table 21 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	SiO2 (%)	Al2O3 (%)	CaO (%)	K2O (%)	Fe2O3 (%)	MgO (%)	TiO2 (PPM)	S (PPM)	P2O5 (PPM)
Compost 1 (Vienna)	20.63	4.31	6.17	4.26	1.99	2.49	1602	<15	10521

Sample	Na2O (%)	Cl (PPM)	Pb (PPM)	Zn (PPM)	Cu (PPM)	Ni (PPM)	Mn (PPM)	Cr (PPM)
Compost 1 (Vienna)	0.35	3496	81	375	79	55	653	60

Table 22 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	Hg µg/g
Compost 1 (Vienna)	0.17

13

2.4 Agricultural soil, sludge amended soil from Pavia, Italy

This sludge-amended soil material was obtained during a monitoring campaign, which aimed at a generic description of the over-all soil quality in Pavia Province, Italy. The material, which was collected from the upper horizon, originates from a small farm called "*Cascina Novello*". During the characterisation of the site, the following analytical information was obtained on a pooled sample of a sub-area of the farm of 20 X 20 m^2.

Table 23 – Analytical data on Pavia soil

Parameter	Result
Al	7.13 Wgt%
As	22.4 mg/kg
Cd	0.79 mg/kg
Cr	59 mg/kg
Cu	30.8 mg/kg
Hg	0.08 mg/kg
Ni	34.4 mg/kg
Pb	24.6 mg/kg
Zn	95 mg/kg
C	0.91 Wgt %
2,3,7,8-TCDD	0.047 pg/g
1,2,3,7,8-PeCDD	0.15 pg/g
1,2,3,4,7,8-HxCDD	0.19 pg/g
1,2,3,6,7,8-HxCDD	1.5 pg/g
1,2,3,7,8,9-HxCDD	0.74 pg/g
1,2,3,4,6,7,8-HpCDD	26 pg/g
OCDD	382 pg/g
2,3,7,8-TCDF	0.68 pg/g
1,2,3,7,8-PeCDF	0.53 pg/g
2,3,4,7,8-PeCDF	0.71 pg/g
1,2,3,4,7,8-HxDF	1.00 pg/g
1,2,3,6,7,8-HxDF	0.66 pg/g
2,3,4,6,7,8-HxDF	1.6 pg/g
1,2,3,7,8,9-HxDF	0.27 pg/g
1,2,3,4,6,7,8-HpDF	12 pg/g
1,2,3,4,7,8,9-HpDF	0.68 pg/g
OCDF	33 pg/g
I-TEQ	2.0 pg/g
WHO-TEQ	1.7 pg/g

In addition, the screening performed at IRMM did not reveal significant quantities of PCBs and PAHs, which were all below the LoDs (1 ng/g for PCBs and 10 ng/g for PAHs, respectively).

Table 24 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	DBP	DCHP	DEHP	Water
	µg/g TM	µg/g TM	µg/g TM	µg/g TM	Wgt. %
Soil 5 (Pavia)		0.005		0.011	1.54

Table 25 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 5 (Pavia)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	0.39
Hexa-BDE-154	nq
Hexa-BDE-153	nq
Hepta-BDE-183	0.08
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	670
TOTAL	671

Table 26 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	Co	Cr	Cu	Mn	Ni	Pb	Sb	Tl	V	Zn
	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g
Soil 5 (Pavia)	0.33	18.4	57.3	22.5	426	30.5	20.6	2.00	< 0.05	38.1	87.8

Table 27 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	SiO2 (%)	Al2O3 (%)	CaO (%)	K2O (%)	Fe2O3 (%)	MgO (%)	TiO2 (PPM)	S (PPM)	P2O5 (PPM)
Soil 5 (Pavia)	69.39	12.9	1.45	2.24	4.25	1.16	6118	255	1789

Sample	Na2O (%)	Cl (PPM)	Pb (PPM)	Zn (PPM)	Cu (PPM)	Ni (PPM)	Mn (PPM)	Cr (PPM)
Soil 5 (Pavia)	1.84	62	38	108	55	66	597	110

Table 28 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	Hg μg/g
Soil 5 (Pavia)	0.06

2.5 *Sludge-amended-soil from Barcelona, Spain*

The sludge-amended soil material from Barcelona sampled upon indication from the Barcelo'- Group in Barcelona.

Table 29 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	μg/g dm	μg/g dm	μg/g dm	μg/g dm	Wgt. %
Soil 2 (Lleida T.)		0.015		0.183	11.38

Table 30 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 2 (Lleida T.)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	1.59
Hexa-BDE-154	0.45
Hexa-BDE-153	nq
Hepta-BDE-183	0.48
Octa-BDE-196	1.60
Octa-BDE-197	nq
Octa-BDE-203	nq
Deca-BDE-209	1000
TOTAL	1004

Table 31 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	*Co*	*Cr*	*Cu*	*Mn*	*Ni*	*Pb*	*Sb*	*Tl*	*V*	*Zn*
	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g	μg/g
Soil 2 (Lleida T.)	0.59	14.1	32.7	53.6	405	18.6	18.4	2.24	< 0.05	31.8	111

Table 32 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	*SiO2 (%)*	*Al2O3 (%)*	*CaO (%)*	*K2O (%)*	*Fe2O3 (%)*	*MgO (%)*	*TiO2 (PPM)*	*S (PPM)*	*P2O5 (PPM)*
Soil 2 (Lleida T.)	44.43	10.67	14.29	2.53	3.44	2.04	4116	780	3396

Sample	*Na2O (%)*	*Cl (PPM)*	*Pb (PPM)*	*Zn (PPM)*	*Cu (PPM)*	*Ni (PPM)*	*Mn (PPM)*	*Cr (PPM)*
Soil 2 (Lleida T.)	0.64	65	26	125	59	17	547	65

Table 33 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	*Hg μg/g*
Soil 2 (Lleida T.)	0.10

2.6 Sludge amended soil from Essen, Germany

The German sludge-amended soil from Essen, which was provided as the three sludge materials by LUA NRW, did not feature significant concentrations of the PCB congeners 28, 52, 101, 118, 153, 105, 138, 156, 180, 170, but had detectable amounts of some PAHs.

Table 34 – Analytical screening data on the German sludge-amended soil.

Parameter	Concentration (ng/g)
Naphtalene	<10
Acenaphtylene	<10
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	<10
Fluoranthene	28
Pyrene	20
Benz(a)anthracene	24
Chrysene	47
Benz(b)fluoranthene	76
Benz(k)fluoranthene	20
Benz(a)pyrene	35
Indeno(1,2,3-c,d)pyrene	35
Dibenzo(a,h)anthracene	10
Benzo(g,h,i)perylene	26

Table 35 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	DBP	DCHP	DEHP	Water
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Soil 4 (Essen)		0.011		0.302	0.55

Table 36 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 4 (Essen)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	nq
Hexa-BDE-154	nq
Hexa-BDE-153	nq
Hepta-BDE-183	nq
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	1.28
Deca-BDE-209	19.1
TOTAL	20.3

Table 37 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	*Co*	*Cr*	*Cu*	*Mn*	*Ni*	*Pb*	*Sb*	*Tl*	*V*	*Zn*
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 4 (Essen)	0.52	5.45	26.1	8.05	320	4.03	27.3	2.73	< 0.05	29.5	78.1

Table 38 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	*SiO2 (%)*	*Al2O3 (%)*	*CaO (%)*	*K2O (%)*	*Fe2O3 (%)*	*MgO (%)*	*TiO2 (PPM)*	*S (PPM)*	*P2O5 (PPM)*
Soil 4 (Essen)	79.47	4.42	0.85	0.6	0.86	0.07	2163	189	2019

Sample	*Na2O (%)*	*Cl (PPM)*	*Pb (PPM)*	*Zn (PPM)*	*Cu (PPM)*	*Ni (PPM)*	*Mn (PPM)*	*Cr (PPM)*
Soil 4 (Essen)	0.45	19	42	87	683	60	462	61

Table 39 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	*Hg µg/g*
Soil 4 (Essen)	0.04

2.7 Long-term sludge exposed soil from Hohenheim-Stuttgart, Germany

Similarly, an additional sludge exposed soil was sampled at the University of Hohenheim, Stuttgart, were a test soil was long-term exposed to elevated concentrations of sewage sludge.

Table 40 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g TM	µg/g TM	µg/g TM	µg/g TM	Wgt. %
Soil 1 (Stuttgart)		0.045		0.263	17.65

Table 41 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 1 (Stuttgart)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	2.30
Hexa-BDE-154	0.06
Hexa-BDE-153	0.04
Hepta-BDE-183	0.04
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	498
TOTAL	500

Table 42 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	*Co*	*Cr*	*Cu*	*Mn*	*Ni*	*Pb*	*Sb*	*Tl*	*V*	*Zn*
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 1 (Stuttgart)	0.69	12.7	36.1	26.2	504	18.3	25.2	2.62	< 0.05	26.6	142

Table 43 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	*SiO2 (%)*	*Al2O3 (%)*	*CaO (%)*	*K2O (%)*	*Fe2O3 (%)*	*MgO (%)*	*TiO2 (PPM)*	*S (PPM)*	*P2O5 (PPM)*
Soil 1 (Stuttgart)	71.94	10.06	1.33	1.86	3.66	0.88	7874	275	3571

Sample	*Na2O (%)*	*Cl (PPM)*	*Pb (PPM)*	*Zn (PPM)*	*Cu (PPM)*	*Ni (PPM)*	*Mn (PPM)*	*Cr (PPM)*
Soil 1 (Stuttgart)	1.23	50	47	212	85	69	991	129

Table 44 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	*Hg µg/g*
Soil 1 (Stuttgart)	1.77

Annex 3:

Statistical calculations

Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDD**

Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)

Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

	General calc.parm.
T1=	3.51713E+03
T2=	6.98259E+05
T3=	20
T4=	80
T5=	2.8576E+03
n=	variabel
p=	5
N-table=	4

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	65.2000	9.394	4		-1.57	0.68	Fail		65.2000	9.3940		4	3	-110.66
46L	165.0000	17.321	4		-0.15	1.25			165.0000	17.3205		4	3	-10.86
12L	174.3071	5.665	4		-0.02	0.41			174.3071	5.6649		4	3	-1.55
4L	237.2750	6.365	4		0.87	0.46	Fail		237.2750	6.3652		4	3	61.42
23L	237.5000	22.174	4		0.87	1.61	! Fail		237.5000	22.1736		4	3	61.64
Tot.gem	175.856	12.184 ng/kg		1%-level:	1.72	(1.73)		5	175.8564	()		5	4	
Tot.std=	70.600	7.251		5%-level:	1.57	(1.53)								

RESULTS: Mean =   **175.85643**   ng/kg

Repeatability variance	S2r =	190.50416				
Repeatability std.	Sr =	**13.80232**	-->	7.85%	r =	38.6465
Between lab variance	S2L =	4936.69354				
Reproducibility var.	S2R =	5127.19770				
Reproducibility std.	SR =	**71.60445**	-->	40.72%	R =	200.4925
Remarks:	none					

Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

Compost 1-1,2,3,4,6,7,8-HpCDD

Sample: **Sludge 1**

Element: **1,2,3,4,6,7,8-HpCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDD)

Mandel's k statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDD)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDD)
Sludge 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 185.3 [ng/kg]

	General calc.parm.
T1=	4.44623E+03
T2=	8.44178E+05
T3=	24
T4=	96
T5=	8.3859E+03
n=	variabel
p=	6
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	143.3000	35.740	4		-1.31	1.66	!	Fail	143.3000	35.7401		4	3	-41.96
12L	166.5336	4.754	4		-0.59	0.22			166.5336	4.7542		4	3	-18.73
23L	177.5000	23.629	4		-0.24	1.09			177.5000	23.6291		4	3	-7.76
19L	185.0000	28.308	4		-0.01	1.31			185.0000	28.3078		4	3	-0.26
46L	202.5000	9.574	4		0.54	0.44			202.5000	9.5743		4	3	17.24
4L	236.7250	6.633	4		1.61	0.31	Fail		236.7250	6.6334		4	3	51.47
Tot.gem	185.260	18.106 ng/kg		1%-level:	1.87	(1.77)		6	185.2598	()		6	5	
Tot.std=	31.992	12.870		5%-level:	1.66	(1.54)								

RESULTS: **Mean =** **185.25977** *ng/kg*

Repeatability variance	**S2r =**	465.88199				
Repeatability std.	**Sr =**	**21.58430**	-->	11.65%	**r =**	60.4360
Between lab variance	**S2L =**	907.02290				
Reproducibility var.	**S2R =**	1372.90489				
Reproducibility std.	**SR =**	**37.05273**	-->	20.00%	**R =**	103.7476
Remarks:	none					
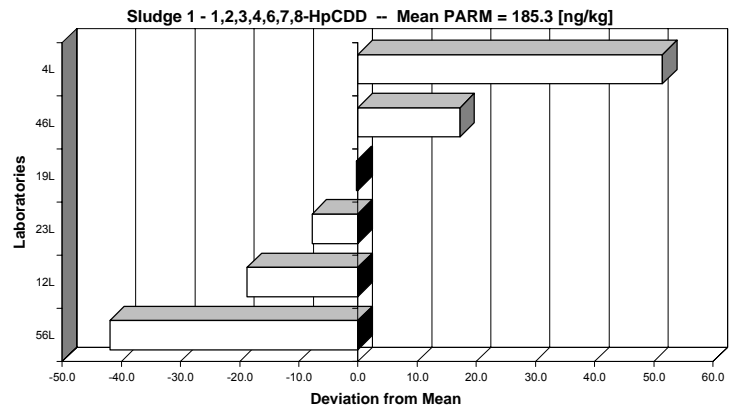
Sludge 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 185.3 [ng/kg]
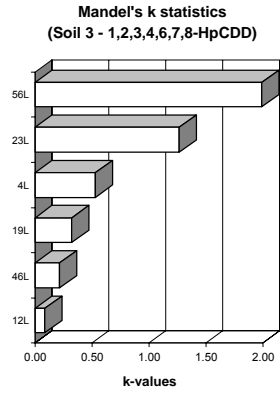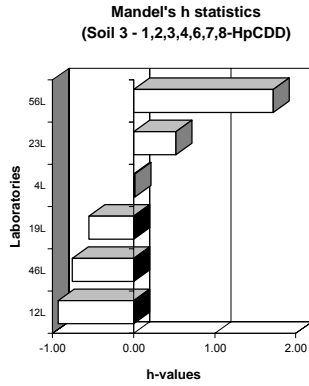
Sludge 1-1,2,3,4,6,7,8-HpCDD

Mandel's h statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDD)

Mandel's k statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDD)

Unit: ng/kg

Mandel's k statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDD)
Soil 3 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 6.54 [ng/kg]

	General calc.parm.
T1=	9.80624E+01
T2=	6.55749E+02
T3=	15
T4=	57
T5=	3.1232E+00
n=	variabel
p=	4
N-table=	4

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
12L	5.5306	0.137	4		-0.94	0.09		Fail	5.5306	0.1371		4	3	-1.01
46L	6.0000	0.346	4		-0.76	0.22			6.0000	0.3464		4	3	-0.54
19L	6.5467	0.519	3		-0.56	0.32			6.5467	0.5193		3	2	0.01
4L	8.0750	0.850	4		0.01	0.53			8.0750	0.8500		4	3	1.54
23L	9.4500	2.027	4		0.52	1.27			-	-	,23L	-	-	2.91
56L	12.6750	3.188	4	!	1.72	1.99	!! Fail		-	-	,56L	-	-	6.14
Tot.gem	8.046	1.178 ng/kg		1%-level:	1.87	(1.77)			4	6.5381	(23L ,56L)		4	3
Tot.std=	2.689	1.190		5%-level:	1.66	(1.54)			2					

RESULTS:	**Mean =**	**6.53806**	*ng/kg*			
Repeatability variance	**S2r =**	0.28392				
Repeatability std.	**Sr =**	**0.53284**	-->	8.15%	**r =**	1.4920
Between lab variance	**S2L =**	1.23350				
Reproducibility var.	**S2R =**	1.51742				
Reproducibility std.	**SR =**	**1.23184**	-->	18.84%	**R =**	3.4491
Remarks:	2 Labs rejected! (23L ,56L)					
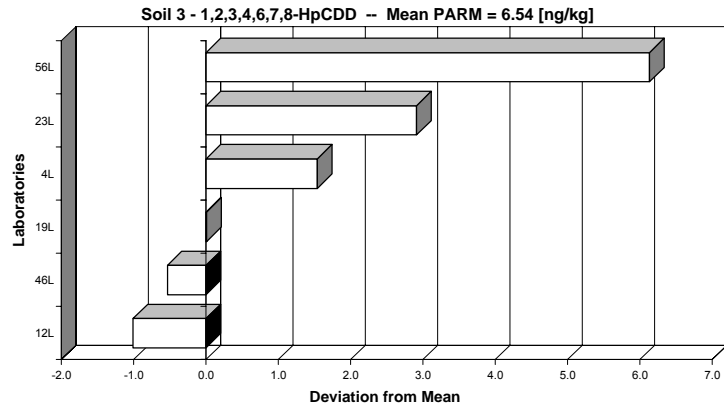
Soil 3 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 6.54 [ng/kg]
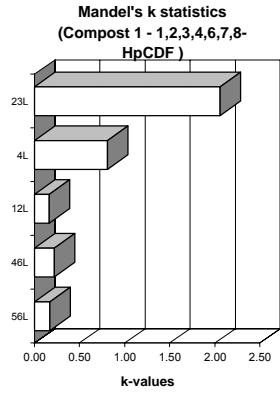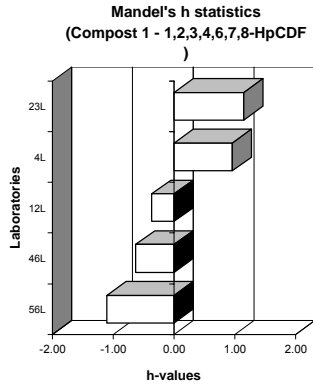
Soil 3-1,2,3,4,6,7,8-HpCDD

Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDF**

Mandel's h statistics
(Compost 1 - 1,2,3,4,6,7,8-HpCDF)

Mandel's k statistics
(Compost 1 - 1,2,3,4,6,7,8-HpCDF)

Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDF)
#NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	4

End Result:

LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	8.3871	1.009	4		-1.11	0.18			#NUM!	-	,56L	-	-	#NUM!
46L	11.5000	1.291	4		-0.63	0.23			-	-	,46L	-	-	#NUM!
12L	13.2804	0.944	4		-0.36	0.16			-	-	,12L	-	-	#NUM!
4L	22.0000	4.651	4		0.96	0.81			-	-	,4L	-	-	#NUM!
23L	23.2500	11.786	4		1.15	2.06	!!		-	-	,23L	-	-	#NUM!
Tot.gem	15.684	3.936 ng/kg		*1%-level:*	1.72	(1.73)			#NUM!	(4L,23L,12L,56L,46L)			-1	
Tot.std=	6.589	4.654		*5%-level:*	1.57	(1.53)								

RESULTS: **Mean =**   **#NUM!**   ng/kg

Repeatability variance	**S2r =**	**#DIV/0!**					
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!		**r =**	#DIV/0!
Between lab variance	**S2L =**	**#NUM!**					
Reproducibility var.	**S2R =**	**#NUM!**					
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!		**R =**	#NUM!
Remarks:		5 Labs rejected! (4L,23L,12L,56L,46L)					
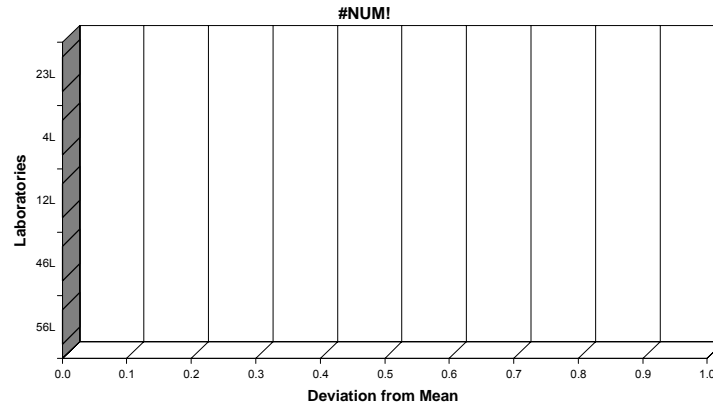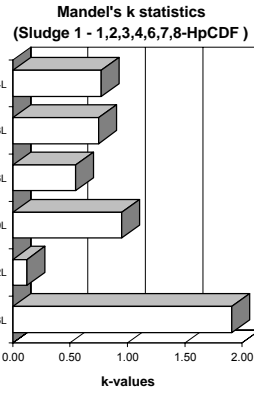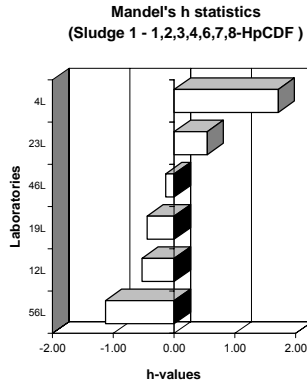
#NUM!

Compost 1-1,2,3,4,6,7,8-HpCDF

Sample: **Sludge 1**

Element: **1,2,3,4,6,7,8-HpCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDF)

Mandel's k statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDF)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDF)
Sludge 1 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 95.41 [ng/kg]

General calc.parm.

T1=	2.28987E+03
T2=	2.24067E+05
T3=	24
T4=	96
T5=	1.3184E+03
n=	variabel
p=	6
N-table=	4

					Mandel's statistics			End Result:						
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	76.5573	16.319	4		-1.13	1.91	!!	Fail	76.5573	16.3190		4	3	-18.85
12L	86.5354	1.072	4		-0.53	0.13			86.5354	1.0724		4	3	-8.88
19L	87.8750	8.107	4		-0.45	0.95			87.8750	8.1074		4	3	-7.54
46L	93.0000	4.690	4		-0.14	0.55			93.0000	4.6904		4	3	-2.41
23L	104.5000	6.403	4		0.54	0.75			104.5000	6.4031		4	3	9.09
4L	124.0000	6.579	4	!	1.71	0.77	Fail		124.0000	6.5793		4	3	28.59
Tot.gem	95.411	7.195 ng/kg		1%-level:	1.87	(1.77)		6	95.4113	()		6	5	
Tot.std=	16.714	5.076		5%-level:	1.66	(1.54)								

RESULTS: **Mean =**   **95.41127**   *ng/kg*

Repeatability variance	**S2r =**	73.24592				
Repeatability std.	**Sr =**	**8.55838**	-->	8.97%	**r =**	23.9635
Between lab variance	**S2L =**	261.04540				
Reproducibility var.	**S2R =**	334.29132				
Reproducibility std.	**SR =**	**18.28364**	-->	19.16%	**R =**	51.1942
Remarks:	none					

Sludge 1 - 1,2,3,4,6,7,8-HpCDF   -- Mean PARM = 95.41 [ng/kg]

Sludge 1-1,2,3,4,6,7,8-HpCDF

Mandel's h statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDF)

Mandel's k statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDF)
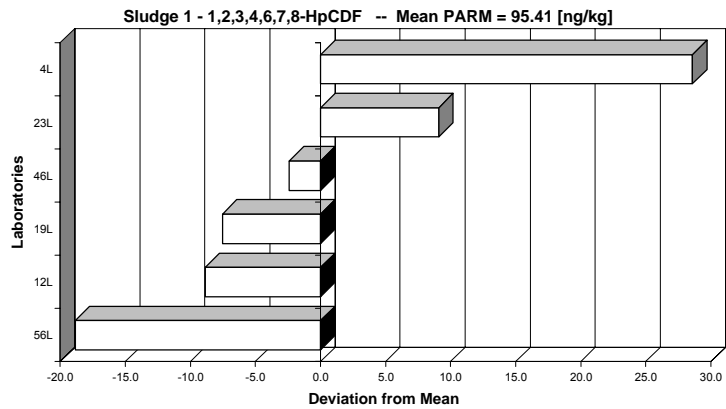
Sample: **Soil 3**

Element: **1,2,3,4,6,7,8-HpCDF**
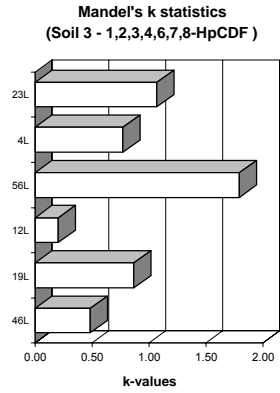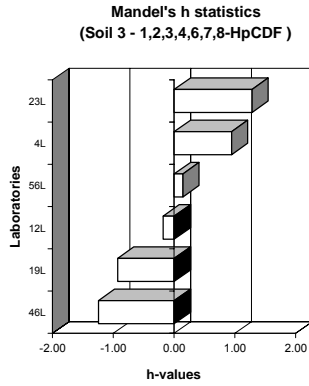
Mandel's k statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDF)
Soil 3 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 12.42 [ng/kg]

	General calc.parm.
T1=	2.87261E+02
T2=	3.63801E+03
T3=	23
T4=	89
T5=	1.0541E+02
n=	variabel
p=	6
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	10.4000	1.200	4		-1.25	0.49			10.4000	1.2000		4	3	-2.02
19L	10.9133	2.147	3		-0.93	0.87			10.9133	2.1472		3	2	-1.51
12L	12.1329	0.510	4		-0.18	0.21			12.1329	0.5098		4	3	-0.29
56L	12.6475	4.441	4		0.14	1.80	!!		12.6475	4.4413		4	3	0.22
4L	13.9500	1.907	4		0.94	0.77			13.9500	1.9070		4	3	1.53
23L	14.5000	2.646	4		1.28	1.07			14.5000	2.6458		4	3	2.08
Tot.gem	12.424	2.142 ng/kg		1%-level:	1.87	(1.77)		6	12.4239	()		6	5	
Tot.std=	1.622	1.353		5%-level:	1.66	(1.54)								

RESULTS: **Mean =**   **12.42394**   *ng/kg*

Repeatability variance	**S2r =**	6.20036			
Repeatability std.	**Sr =**	**2.49005**	--> 20.04%	**r =**	6.9721
Between lab variance	**S2L =**	1.00491			
Reproducibility var.	**S2R =**	7.20527			
Reproducibility std.	**SR =**	**2.68426**	--> 21.61%	**R =**	7.5159
Remarks:	none				

Soil 3 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 12.42 [ng/kg]

Soil 3-1,2,3,4,6,7,8-HpCDF

Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDD**

Mandel's h statistics
(Compost 1 - 1,2,3,4,6,7,8-HpCDD)

h-values

Mandel's k statistics
(Compost 1 - 1,2,3,4,6,7,8-HpCDD)

k-values

Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

	General calc.parm.
T1=	3.51713E+03
T2=	6.98259E+05
T3=	20
T4=	80
T5=	2.8576E+03
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	65.2000	9.394	4		-1.57	0.68	Fail		65.2000	9.3940		4	3	-110.66
46L	165.0000	17.321	4		-0.15	1.25			165.0000	17.3205		4	3	-10.86
12L	174.3071	5.665	4		-0.02	0.41			174.3071	5.6649		4	3	-1.55
4L	237.2750	6.365	4		0.87	0.46	Fail		237.2750	6.3652		4	3	61.42
23L	237.5000	22.174	4		0.87	1.61	! Fail		237.5000	22.1736		4	3	61.64
Tot.gem	175.856	12.184 ng/kg		1%-level:	1.72	(1.73)		5	175.8564	()		5	4	
Tot.std=	70.600	7.251		5%-level:	1.57	(1.53)								

RESULTS: Mean = **175.85643** ng/kg

Repeatability variance	S2r =	190.50416			
Repeatability std.	Sr =	**13.80232**	--> 7.85%	r =	38.6465
Between lab variance	S2L =	4936.69354			
Reproducibility var.	S2R =	5127.19770			
Reproducibility std.	SR =	**71.60445**	--> 40.72%	R =	200.4925
Remarks:	none				

Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

Deviation from Mean

Compost 1-1,2,3,4,6,7,8-HpCDD

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,7,8,9-HpCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,4,7,8,9-HpCDF)
Sludge 1 - 1,2,3,4,7,8,9-HpCDF -- Mean PARM = 7.41 [ng/kg]

Sample: **Sludge 1**

Element: **1,2,3,4,7,8,9-HpCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,7,8,9-HpCDF)

Mandel's k statistics
(Sludge 1 - 1,2,3,4,7,8,9-HpCDF)

	General calc.parm.
T1=	1.48141E+02
T2=	1.16636E+03
T3=	20
T4=	80
T5=	4.5943E+01
n=	variabel
p=	5
N-table=	4

					Mandel's statistics					End Result:					
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
12L	5.3948	0.033	4		-0.97	0.02			5.3948	0.0328		4	3	-2.01
46L	6.5250	2.327	4		-0.42	1.33			6.5250	2.3272		4	3	-0.88
19L	6.5900	0.250	4		-0.39	0.14			6.5900	0.2505		4	3	-0.82
4L	7.7000	0.440	4		0.14	0.25			7.7000	0.4397		4	3	0.29
56L	10.8255	3.105	4	!	1.65	1.77	!! Fail		10.8255	3.1051		4	3	3.42
Tot.gem	7.407	1.231 ng/kg		*1%-level:*	1.72	(1.73)			5	*7.4071*	()		5	4	
Tot.std=	2.078	1.391		*5%-level:*	1.57	(1.53)								

RESULTS: Mean = **7.40706** *ng/kg*

Repeatability variance	**S2r =**	3.06290			
Repeatability std.	**Sr =**	**1.75011**	--> 23.63%	**r =**	4.9003
Between lab variance	**S2L =**	3.55091			
Reproducibility var.	**S2R =**	6.61380			
Reproducibility std.	**SR =**	**2.57173**	--> 34.72%	**R =**	7.2008
Remarks:	none				

Sludge 1 - 1,2,3,4,7,8,9-HpCDF -- Mean PARM = 7.41 [ng/kg]
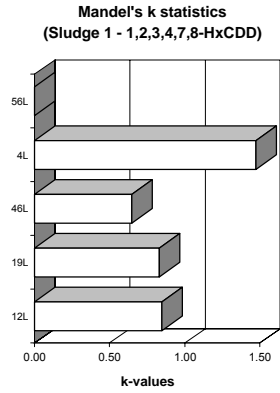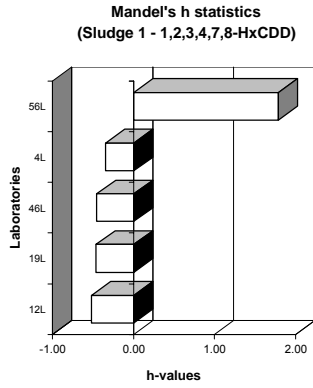
Sludge 1-1,2,3,4,7,8,9-HpCDF

Sample: **Sludge 1**

Element: **1,2,3,4,7,8-HxCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,7,8-HxCDD)

Mandel's k statistics
(Sludge 1 - 1,2,3,4,7,8-HxCDD)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,7,8-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,4,7,8-HxCDD)
Sludge 1 - 1,2,3,4,7,8-HxCDD -- Mean PARM = 3.5 [ng/kg]

	General calc.parm.
T1=	5.25284E+01
T2=	1.86187E+02
T3=	15
T4=	57
T5=	6.4029E-01
n=	variabel
p=	4
N-table=	3

					Mandel's statistics				End Result:					
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
12L	3.0721	0.199	4		-0.52	0.85		Fail	3.0721	0.1993		4	3	-0.43
19L	3.3850	0.195	4		-0.47	0.83		Fail	3.3850	0.1954		4	3	-0.11
46L	3.4333	0.153	3		-0.46	0.65		Fail	3.4333	0.1528		3	2	-0.06
4L	4.1000	0.346	4		-0.34	1.47			4.1000	0.3464		4	3	0.60
56L	16.5000	-	1	!!	1.79		Fail		-	-	,56L	-	-	13.00
Tot.gem	6.098	0.223 ng/kg		*1%-level:*	1.72	(1.77)			4	3.4976	(56L)	4	3	
Tot.std=	5.827	0.085		*5%-level:*	1.57	(1.59)			1					

RESULTS: **Mean =**   **3.49761**   *ng/kg*

Repeatability variance	**S2r =**	0.05821				
Repeatability std.	**Sr =**	**0.24126**	-->	6.90%	**r =**	0.6755
Between lab variance	**S2L =**	0.18428				
Reproducibility var.	**S2R =**	0.24249				
Reproducibility std.	**SR =**	**0.49243**	-->	14.08%	**R =**	1.3788
Remarks:	1 Lab rejected! (56L)					

Sludge 1 - 1,2,3,4,7,8-HxCDD -- Mean PARM = 3.5 [ng/kg]

Sludge 1-1,2,3,4,7,8-HxCDD

Mandel's h statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDD)

Mandel's k statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDD)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,6,7,8-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,6,7,8-HxCDD)
Sludge 1 - 1,2,3,6,7,8-HxCDD -- Mean PARM = 8.28 [ng/kg]

	General calc.parm.
T1=	1.65567E+02
T2=	1.41503E+03
T3=	20
T4=	80
T5=	1.9356E+01
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	6.9450	1.087	4		-0.80	0.96			6.9450	1.0868		4	3	-1.33
12L	7.4968	0.260	4		-0.47	0.23			7.4968	0.2596		4	3	-0.78
56L	7.6000	2.028	4		-0.41	1.79	!!		7.6000	2.0281		4	3	-0.68
46L	8.2000	0.548	4		-0.05	0.48			8.2000	0.5477		4	3	-0.08
4L	11.1500	0.889	4	!!	1.72	0.78		Fail	11.1500	0.8888		4	3	2.87
Tot.gem	8.278	0.962 ng/kg		1%-level:	1.72	(1.73)			5	8.2784	()		5	4
Tot.std=	1.666	0.675		5%-level:	1.57	(1.53)								

RESULTS: **Mean =**   **8.27836**   *ng/kg*

Repeatability variance	**S2r =**	1.29037			
Repeatability std.	**Sr =**	**1.13594**	-->	13.72%	**r =** 3.1806
Between lab variance	**S2L =**	2.45273			
Reproducibility var.	**S2R =**	3.74310			
Reproducibility std.	**SR =**	**1.93471**	-->	23.37%	**R =** 5.4172
Remarks:		none			

Sludge 1 - 1,2,3,6,7,8-HxCDD -- Mean PARM = 8.28 [ng/kg]

Sludge 1-1,2,3,6,7,8-HxCDD

Mandel's h statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDF)

Mandel's k statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDF)

Sample: **Sludge 1**

Element: **1,2,3,6,7,8-HxCDF**
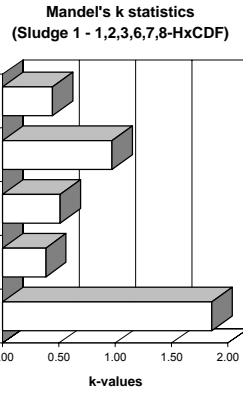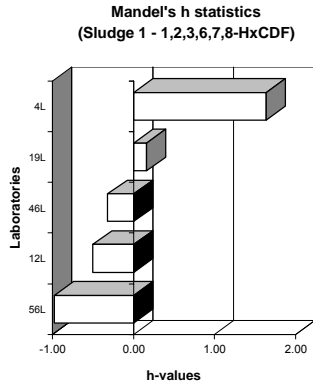
Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,6,7,8-HxCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,6,7,8-HxCDF)
Sludge 1 - 1,2,3,6,7,8-HxCDF -- Mean PARM = 7.4 [ng/kg]

	General calc.parm.
T1=	1.47943E+02
T2=	1.12613E+03
T3=	20
T4=	80
T5=	9.6995E+00
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	6.0250	1.493	4		-0.97	1.86	!!	Fail	6.0250	1.4930		4	3	-1.37
12L	6.6857	0.309	4		-0.50	0.38			6.6857	0.3094		4	3	-0.71
46L	6.9500	0.412	4		-0.32	0.51			6.9500	0.4123		4	3	-0.45
19L	7.6250	0.782	4		0.16	0.97			7.6250	0.7820		4	3	0.23
4L	9.7000	0.356	4	!	1.63	0.44	Fail		9.7000	0.3559		4	3	2.30
Tot.gem	7.397	0.671 ng/kg		*1%-level:*	1.72	(1.73)			5	7.3971	()		5	4
Tot.std=	1.409	0.496		*5%-level:*	1.57	(1.53)								

RESULTS: **Mean =** **7.39714** *ng/kg*

Repeatability variance	**S2r =**	0.64663			
Repeatability std.	**Sr =**	**0.80413**	-->	10.87%	**r =** 2.2516
Between lab variance	**S2L =**	1.82432			
Reproducibility var.	**S2R =**	2.47095			
Reproducibility std.	**SR =**	**1.57193**	-->	21.25%	**R =** 4.4014
Remarks:	**none**				

Sludge 1 - 1,2,3,6,7,8-HxCDF -- Mean PARM = 7.4 [ng/kg]

Sludge 1-1,2,3,6,7,8-HxCDF

Sample: **Compost 1**

Element: **1,2,3,7,8,9-HxCDD**

Mandel's h statistics
(Compost 1 - 1,2,3,7,8,9-HxCDD)

Laboratories: 56L, 4L, 46L, 12L

h-values: -1.00 0.00 1.00 2.00

Mandel's k statistics
(Compost 1 - 1,2,3,7,8,9-HxCDD)

Laboratories: 56L, 4L, 46L, 12L

k-values: 0.00 0.50 1.00 1.50
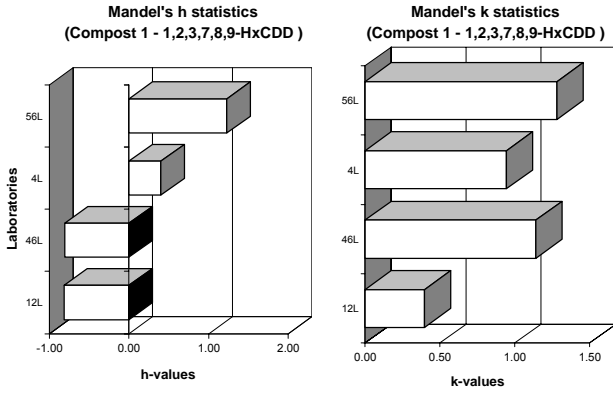
Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,7,8,9-HxCDD)
Mandel's h statistics (Compost 1 - 1,2,3,7,8,9-HxCDD)
Compost 1 - 1,2,3,7,8,9-HxCDD   -- Mean PARM = 2.09 [ng/kg]

	General calc.parm.
T1=	2.75803E+01
T2=	5.80691E+01
T3=	14
T4=	52
T5=	4.2296E-01
n=	variabel
p=	4
N-table=	4

				Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
12L	1.5451	0.087	4		-0.82	0.40		Fail	1.5451	0.0875		4	3	-0.54
46L	1.5500	0.252	4		-0.81	1.14		Fail	1.5500	0.2517		4	3	-0.54
4L	2.3500	0.208	4		0.40	0.94	Fail		2.3500	0.2082		4	3	0.26
56L	2.9000	0.283	2		1.23	1.28	Fail		2.9000	0.2828		2	1	0.81
Tot.gem	2.086	0.208 ng/kg		1%-level:	1.49	(1.67)			4	2.0863	()		4	3
Tot.std=	0.661	0.086		5%-level:	1.42	(1.5)								

RESULTS: **Mean =**   **2.08627**   *ng/kg*

Repeatability variance	**S2r =**	0.04230			
Repeatability std.	**Sr =**	**0.20566**	-->	9.86%	**r =** 0.5758
Between lab variance	**S2L =**	0.35081			
Reproducibility var.	**S2R =**	0.39311			
Reproducibility std.	**SR =**	**0.62698**	-->	30.05%	**R =** 1.7555
Remarks:	none				
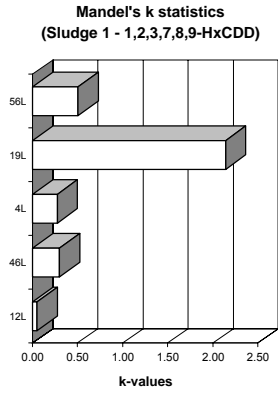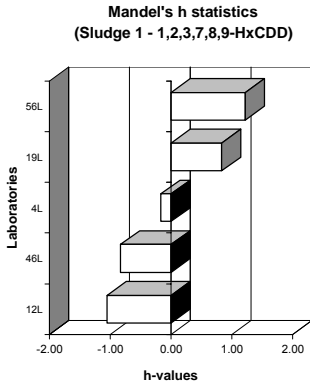
Compost 1 - 1,2,3,7,8,9-HxCDD   -- Mean PARM = 2.09 [ng/kg]

Laboratories: 56L, 4L, 46L, 12L

Deviation from Mean: -0.6 -0.4 -0.2 0.0 0.2 0.4 0.6 0.8 1.0

Compost 1-1,2,3,7,8,9-HxCDD

Unit: ng/kg

Mandel's h statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)

Mandel's k statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)

Sample: **Sludge 1**

Element: **1,2,3,7,8,9-HxCDD**

Mandel's k statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)
Sludge 1 - 1,2,3,7,8,9-HxCDD -- Mean PARM = 6.43 [ng/kg]

	General calc.parm.
T1=	1.02832E+02
T2=	7.70893E+02
T3=	16
T4=	64
T5=	1.0314E+01
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
12L	3.9329	0.149	4		-1.05	0.05			3.9329	0.1493		4	3	-2.49
46L	4.5750	0.862	4		-0.83	0.30			4.5750	0.8617		4	3	-1.85
4L	6.5500	0.785	4		-0.17	0.28			6.5500	0.7853		4	3	0.12
19L	9.5125	6.083	4		0.83	2.14	!!	-	-	-	,19L	-	-	3.09
56L	10.6500	1.434	4		1.22	0.50			10.6500	1.4341		4	3	4.22
Tot.gem	7.044	1.863 ng/kg		*1%-level:*	1.72	(1.73)		4	*6.4270*	(19L)		4	3	
Tot.std=	2.963	2.403		*5%-level:*	1.57	(1.53)		1						

RESULTS: **Mean =**   **6.42698**   *ng/kg*

Repeatability variance	**S2r =**	0.85953				
Repeatability std.	**Sr =**	**0.92711**	-->	14.43%	**r =**	2.5959
Between lab variance	**S2L =**	8.95154				
Reproducibility var.	**S2R =**	9.81107				
Reproducibility std.	**SR =**	**3.13226**	-->	48.74%	**R =**	8.7703
Remarks:	1 Lab rejected! (19L)					
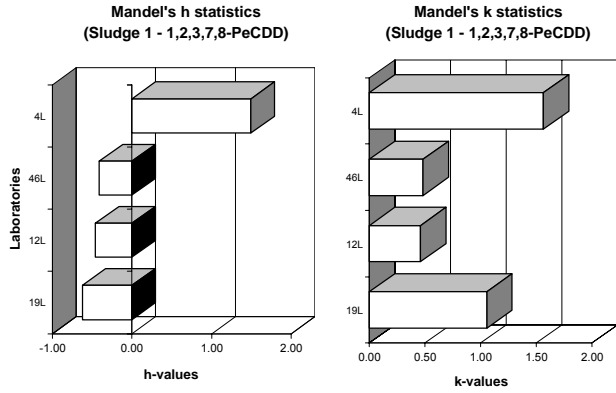
Sludge 1 - 1,2,3,7,8,9-HxCDD -- Mean PARM = 6.43 [ng/kg]

Sludge 1-1,2,3,7,8,9-HxCDD

Sample: **Sludge 1**

Element: **1,2,3,7,8-PeCDD**

**Mandel's h statistics
(Sludge 1 - 1,2,3,7,8-PeCDD)**

**Mandel's k statistics
(Sludge 1 - 1,2,3,7,8-PeCDD)**

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,7,8-PeCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8-PeCDD)
Sludge 1 - 1,2,3,7,8-PeCDD -- Mean PARM = 2.29 [ng/kg]

	General calc.parm.
T1=	3.65631E+01
T2=	1.00307E+02
T3=	16
T4=	64
T5=	2.0500E+00
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	1.5463	0.436	4		-0.63	1.06		Fail	1.5463	0.4365		4	3	-0.74
12L	1.7445	0.190	4		-0.46	0.46		Fail	1.7445	0.1901		4	3	-0.54
46L	1.8000	0.200	4		-0.41	0.48		Fail	1.8000	0.2000		4	3	-0.49
4L	4.0500	0.645	4	!!	1.49	1.56	!	Fail	4.0500	0.6455		4	3	1.76
Tot.gem	2.285	0.368 ng/kg		1%-level:	1.49	(1.67)			4	2.2852	()		4	3
Tot.std=	1.182	0.217		5%-level:	1.42	(1.5)								

RESULTS: **Mean =** **2.28519** *ng/kg*

Repeatability variance	**S2r =**	0.17083			
Repeatability std.	**Sr =**	**0.41332**	-->	18.09%	r = 1.1573
Between lab variance	**S2L =**	1.35340			
Reproducibility var.	**S2R =**	1.52423			
Reproducibility std.	**SR =**	**1.23460**	-->	54.03%	R = 3.4569
Remarks:	none				

Sludge 1 - 1,2,3,7,8-PeCDD -- Mean PARM = 2.29 [ng/kg]
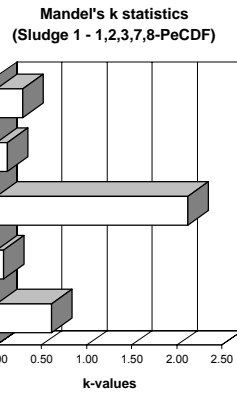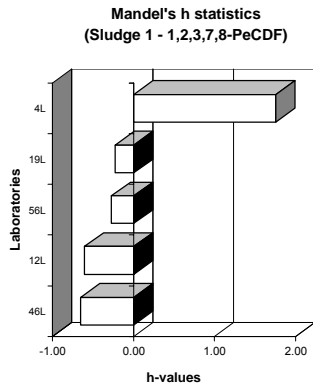
Sludge 1-1,2,3,7,8-PeCDD

Sample: **Sludge 1**

Element: **1,2,3,7,8-PeCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,7,8-PeCDF)

Mandel's k statistics
(Sludge 1 - 1,2,3,7,8-PeCDF)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,7,8-PeCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8-PeCDF)
Sludge 1 - 1,2,3,7,8-PeCDF -- Mean PARM = 5.2 [ng/kg]

	General calc.parm.
T1=	7.87769E+01
T2=	4.29418E+02
T3=	15
T4=	57
T5=	2.0598E+00
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	4.4667	0.850	3		-0.65	0.62			4.4667	0.8505		3	2	-0.74
12L	4.5167	0.114	4		-0.60	0.08			4.5167	0.1142		4	3	-0.69
56L	4.8500	2.937	4		-0.28	2.12	!!		-	-	,56L	-		-0.35
19L	4.9025	0.170	4		-0.22	0.12			4.9025	0.1698		4	3	-0.30
4L	6.9250	0.403	4	!!	1.76	0.29			6.9250	0.4031		4	3	1.72
Tot.gem	5.132	0.895 ng/kg		1%-level:	1.72	(1.73)		4	5.2027	(56L)		4	3	
Tot.std=	1.021	1.178		5%-level:	1.57	(1.53)								

RESULTS: **Mean =**   **5.20273**   *ng/kg*

Repeatability variance	**S2r =**	0.18725			
Repeatability std.	**Sr =**	**0.43272**	-->	8.32%	r =	1.2116
Between lab variance	**S2L =**	1.35136			
Reproducibility var.	**S2R =**	1.53861			
Reproducibility std.	**SR =**	**1.24041**	-->	23.84%	R =	3.4731
Remarks:	1 Lab rejected! (56L)				
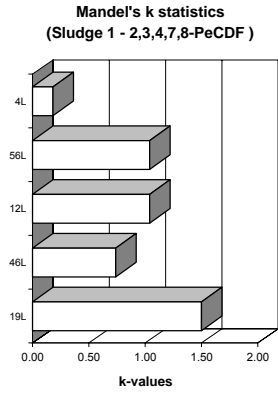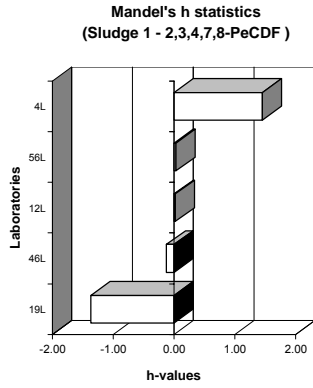
Sludge 1 - 1,2,3,7,8-PeCDF -- Mean PARM = 5.2 [ng/kg]

Sample: **Sludge 1**

Element: **2,3,4,7,8-PeCDF**

Mandel's h statistics (Sludge 1 - 2,3,4,7,8-PeCDF)

Mandel's k statistics (Sludge 1 - 2,3,4,7,8-PeCDF)

Unit: ng/kg

Mandel's k statistics (Sludge 1 - 2,3,4,7,8-PeCDF)
Mandel's h statistics (Sludge 1 - 2,3,4,7,8-PeCDF)
Sludge 1 - 2,3,4,7,8-PeCDF -- Mean PARM = 9.78 [ng/kg]

	General calc.parm.
T1=	1.95673E+02
T2=	1.96752E+03
T3=	20
T4=	80
T5=	1.4596E+02
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	7.2850	4.683	4		-1.37	1.50			7.2850	4.6834		4	3	-2.50
46L	9.5500	2.313	4		-0.13	0.74			9.5500	2.3130		4	3	-0.23
12L	9.8250	3.238	4		0.02	1.04			9.8250	3.2377		4	3	0.04
56L	9.8333	3.249	4		0.03	1.04			9.8333	3.2494		4	3	0.05
4L	12.4250	0.574	4		1.45	0.18			12.4250	0.5737		4	3	2.64
Tot.gem	9.784	2.811 ng/kg		1%-level:	1.72	(1.73)		5	9.7837	()		5	4	
Tot.std=	1.822	1.511		5%-level:	1.57	(1.53)								

RESULTS: | **Mean =** | **9.78367** | ng/kg

Repeatability variance	S2r =	9.73094			
Repeatability std.	Sr =	**3.11945**	-->	31.88%	r =	8.7344
Between lab variance	S2L =	0.88695			
Reproducibility var.	S2R =	10.61789			
Reproducibility std.	SR =	**3.25851**	-->	33.31%	R =	9.1238
Remarks:	none				

Sludge 1 - 2,3,4,7,8-PeCDF -- Mean PARM = 9.78 [ng/kg]

Sludge 1-2,3,4,7,8-PeCDF

Mandel's h statistics
(Sludge 1 - 2,3,7,8-TCDF)

Mandel's k statistics
(Sludge 1 - 2,3,7,8-TCDF)
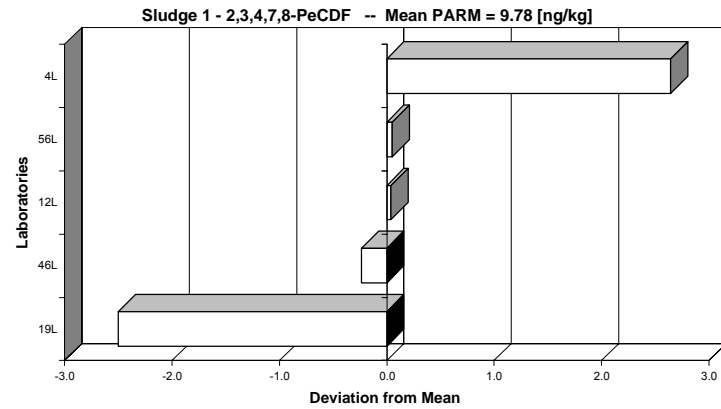
Sample: **Sludge 1**

Element: **2,3,7,8-TCDF**
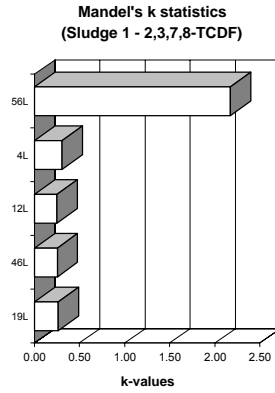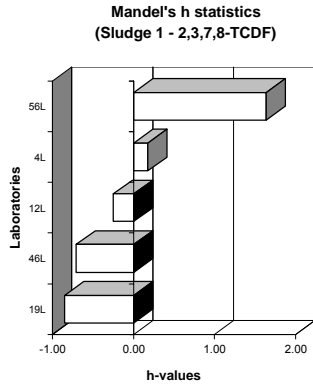
Unit: ng/kg

Mandel's k statistics (Sludge 1 - 2,3,7,8-TCDF)
Mandel's h statistics (Sludge 1 - 2,3,7,8-TCDF)
Sludge 1 - 2,3,7,8-TCDF -- Mean PARM = 10.03 [ng/kg]

General calc.parm.

T1=	1.60521E+02
T2=	1.63166E+03
T3=	16
T4=	64
T5=	5.2140E+00
n=	variabel
p=	4
N-table=	4

						Mandel's statistics						End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std		AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	8.7725	0.642	4		-0.85	0.26				8.7725	0.6422		4	3	-1.26
46L	9.1750	0.624	4		-0.71	0.25				9.1750	0.6238		4	3	-0.86
12L	10.4827	0.608	4		-0.25	0.25				10.4827	0.6081		4	3	0.45
4L	11.7000	0.753	4	!	0.17	0.31				11.7000	0.7528		4	3	1.67
56L	15.8750	5.319	4		1.64	2.17	!!	Fail		-	-	,56L	-		5.84
Tot.gem	11.201	1.589 ng/kg		*1%-level:*	1.72	(1.73)			4	*10.0325*	(56L)		4	3	
Tot.std=	2.855	2.086		*5%-level:*	1.57	(1.53)			1						

RESULTS: **Mean =**   **10.03255**   *ng/kg*

Repeatability variance	**S2r =**	0.43450			
Repeatability std.	**Sr =**	**0.65916**	-->	6.57%	**r =**	1.8457
Between lab variance	**S2L =**	1.66009			
Reproducibility var.	**S2R =**	2.09459			
Reproducibility std.	**SR =**	**1.44727**	-->	14.43%	**R =**	4.0524
Remarks:	1 Lab rejected! (56L)				

Sludge 1 - 2,3,7,8-TCDF -- Mean PARM = 10.03 [ng/kg]

Sludge 1-2,3,7,8-TCDF

Sample: **Compost 1**

Element: **OCDD**

**Mandel's h statistics
(Compost 1 - OCDD)**
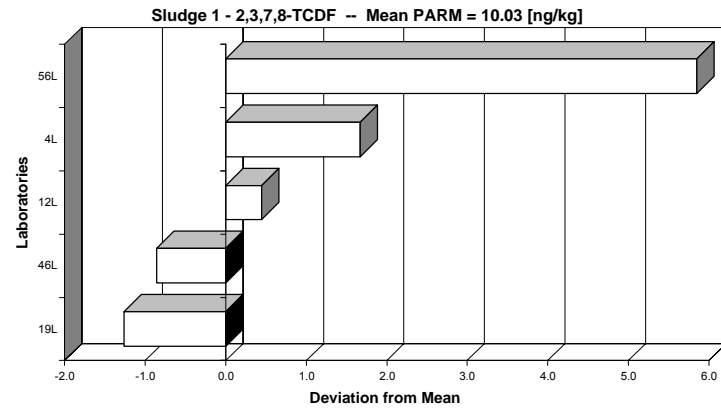
**Mandel's k statistics
(Compost 1 - OCDD)**
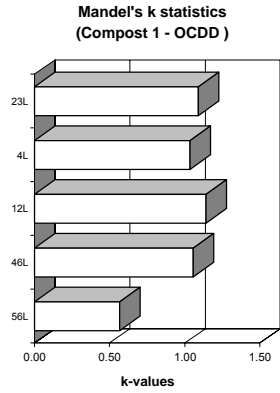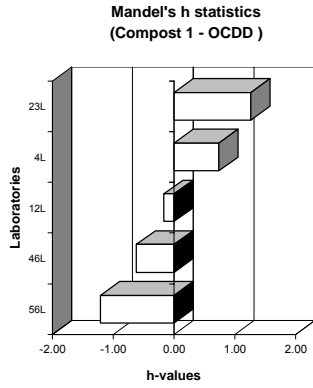
Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDD)
Mandel's h statistics (Compost 1 - OCDD)
Compost 1 - OCDD -- Mean PARM = 1031 [ng/kg]

	General calc.parm.
T1=	2.06128E+04
T2=	2.42164E+07
T3=	20
T4=	80
T5=	1.9961E+05
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	510.2750	65.527	4		-1.21	0.57		Fail	510.2750	65.5274		4	3	-520.37
46L	765.0000	121.792	4		-0.62	1.06		Fail	765.0000	121.7922		4	3	-265.64
12L	956.3812	131.426	4		-0.17	1.14		Fail	956.3812	131.4264		4	3	-74.26
4L	1346.5500	119.590	4		0.73	1.04	Fail		1346.5500	119.5901		4	3	315.91
23L	1575.0000	125.831	4		1.26	1.09	Fail		1575.0000	125.8306		4	3	544.36
Tot.gem	1030.641	112.833 ng/kg		1%-level:	1.72	(1.73)		5	1030.6412	()		5	4	
Tot.std=	430.983	26.825		5%-level:	1.57	(1.53)								

RESULTS: Mean = **1030.64124** *ng/kg*

Repeatability variance	S2r =	13307.04038				
Repeatability std.	Sr =	**115.35615**	-->	11.19%	r =	322.9972
Between lab variance	S2L =	182419.65199				
Reproducibility var.	S2R =	195726.69237				
Reproducibility std.	SR =	**442.41010**	-->	42.93%	R =	1238.7483
Remarks:	none					
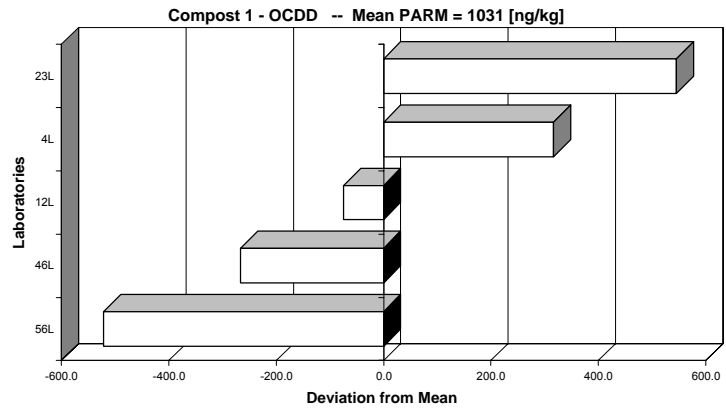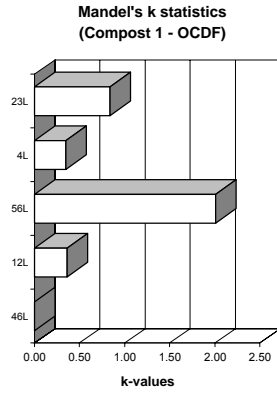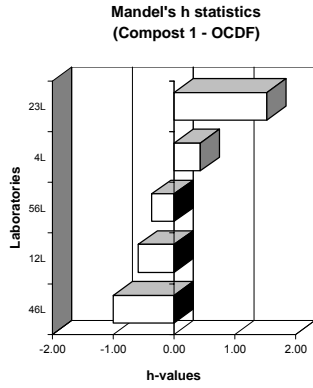
Compost 1 - OCDD -- Mean PARM = 1031 [ng/kg]

Compost 1-OCDD

Sample: **Compost 1**

Element: **OCDF**

Mandel's h statistics
(Compost 1 - OCDF)

Mandel's k statistics
(Compost 1 - OCDF)

Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDF)
Mandel's h statistics (Compost 1 - OCDF)
Compost 1 - OCDF -- Mean PARM = 27.54 [ng/kg]

	General calc.parm.
T1=	2.75390E+02
T2=	7.92465E+03
T3=	10
T4=	20
T5=	8.9112E+01
n=	variabel
p=	5
N-table=	2

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	21.0000		2		-1.00				21.0000			2	1	-6.54
12L	23.7055	1.540	2		-0.59	0.36			23.7055	1.5399		2	1	-3.83
56L	25.1397	8.487	2		-0.37	2.01	!		25.1397	8.4874		2	1	-2.40
4L	30.3500	1.485	2		0.43	0.35			30.3500	1.4849		2	1	2.81
23L	37.5000	3.536	2		1.53	0.84	Fail		37.5000	3.5355		2	1	9.96
Tot.gem	27.539	3.010 ng/kg		1%-level:	1.72	(2.05)			5	27.5390	()		5	4
Tot.std=	6.526	3.310		5%-level:	1.57	(1.81)								

RESULTS: Mean =   **27.53904**   ng/kg

Repeatability variance	S2r =	17.82245				
Repeatability std.	Sr =	**4.22166**	-->	15.33%	r =	11.8207
Between lab variance	S2L =	33.67234				
Reproducibility var.	S2R =	51.49480				
Reproducibility std.	SR =	**7.17599**	-->	26.06%	R =	20.0928
Remarks:	none					

Compost 1 - OCDF -- Mean PARM = 27.54 [ng/kg]

Compost 1-OCDF
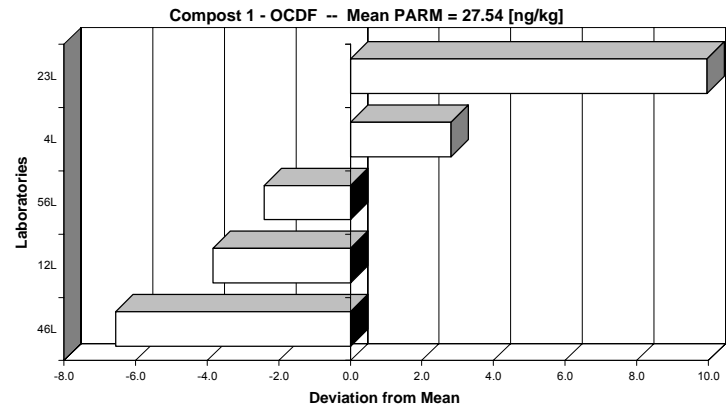
Sample: **Sludge 1**

Element: **OCDD**
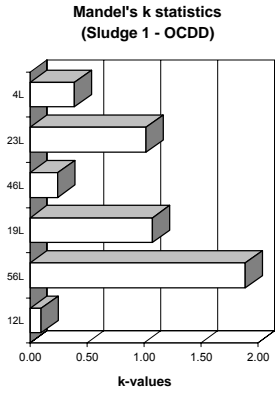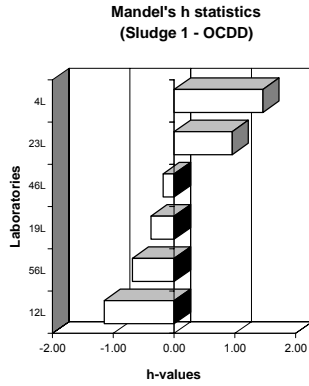
Mandel's k statistics (Sludge 1 - OCDD)
Mandel's h statistics (Sludge 1 - OCDD)

Sludge 1 - OCDD -- Mean PARM = 1479 [ng/kg]

Mandel's h statistics (Sludge 1 - OCDD)

Mandel's k statistics (Sludge 1 - OCDD)

h-values

k-values

Laboratories

	General calc.parm.
T1=	3.54869E+04
T2=	5.41109E+07
T3=	24
T4=	96
T5=	7.5285E+05
n=	variabel
p=	6
N-table=	4

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
12L	1149.0802	20.297	4		-1.15	0.10		Fail	1149.0802	20.2969		4	3	-329.54
56L	1281.7500	387.063	4		-0.69	1.89	!!		1281.7500	387.0631		4	3	-196.87
19L	1370.0000	220.303	4		-0.38	1.08			1370.0000	220.3028		4	3	-108.62
46L	1425.0000	50.000	4		-0.19	0.24			1425.0000	50.0000		4	3	-53.62
23L	1750.0000	208.167	4		0.95	1.02			1750.0000	208.1666		4	3	271.38
4L	1895.9000	79.712	4		1.46	0.39	Fail		1895.9000	79.7116		4	3	417.28
Tot.gem	1478.622	160.924 ng/kg		1%-level:	1.87	(1.77)		6	1478.6217	()		6	5	
Tot.std=	286.286	138.255		5%-level:	1.66	(1.54)								

RESULTS: Mean = **1478.62170** *ng/kg*

Repeatability variance	**S2r =**	41825.07246				
Repeatability std.	**Sr =**	**204.51179**	-->	13.83%	**r =**	572.6330
Between lab variance	**S2L =**	71503.20658				
Reproducibility var.	**S2R =**	113328.27904				
Reproducibility std.	**SR =**	**336.64266**	-->	22.77%	**R =**	942.5994
Remarks:	none					

Sludge 1 - OCDD -- Mean PARM = 1479 [ng/kg]

Laboratories

Deviation from Mean

Sludge 1-OCDD

Sample: **Soil 3**

Element: **OCDD**

Mandel's h statistics
(Soil 3 - OCDD)
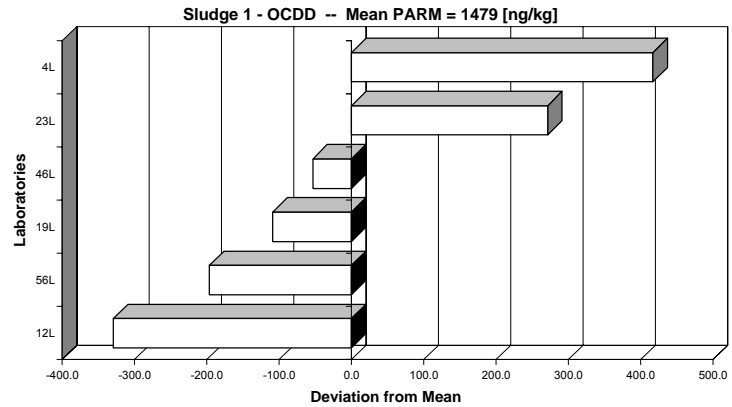
Mandel's k statistics
(Soil 3 - OCDD)
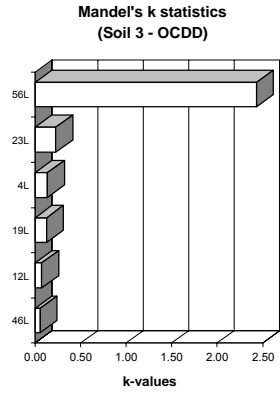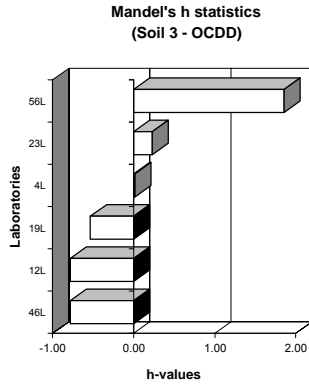
Unit: ng/kg

Mandel's k statistics (Soil 3 - OCDD)
Mandel's h statistics (Soil 3 - OCDD)
Soil 3 - OCDD -- Mean PARM = 56.89 [ng/kg]

	General calc.parm.
T1=	1.08561E+03
T2=	6.49269E+04
T3=	19
T4=	73
T5=	1.1791E+02
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	45.0000	1.155	4		-0.78	0.06			45.0000	1.1547		4	3	-11.89
12L	45.0026	1.524	4		-0.78	0.07			45.0026	1.5236		4	3	-11.89
19L	52.1667	2.650	3		-0.53	0.13			52.1667	2.6502		3	2	-4.72
4L	68.0250	2.837	4		0.01	0.14			68.0250	2.8371		4	3	11.14
23L	74.2500	4.787	4		0.23	0.23			74.2500	4.7871		4	3	17.36
56L	121.1750	50.812	4	!	1.85	2.43	!!	Fail	-	-	,56L	-	-	64.29
Tot.gem	67.603	10.627 ng/kg		1%-level:	1.87	(1.77)		5	56.8888	(56L)		5	4	
Tot.std=	28.894	19.727		5%-level:	1.66	(1.54)		1						

RESULTS: Mean = **56.88884** *ng/kg*

Repeatability variance	**S2r =**	8.42204			
Repeatability std.	**Sr =**	**2.90208**	-->	5.10%	r =	8.1258
Between lab variance	**S2L =**	188.96055			
Reproducibility var.	**S2R =**	197.38259			
Reproducibility std.	**SR =**	**14.04929**	-->	24.70%	R =	39.3380
Remarks:	1 Lab rejected! (56L)				
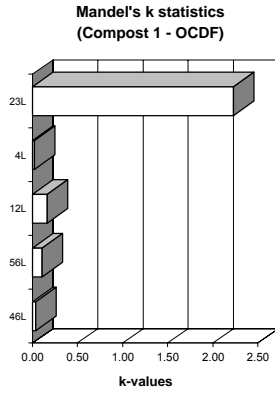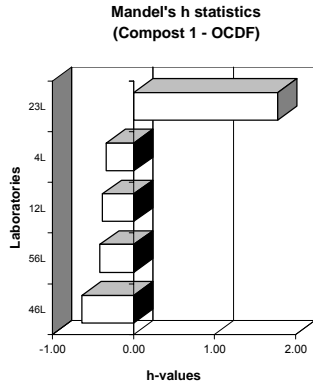
Soil 3 - OCDD -- Mean PARM = 56.89 [ng/kg]

Soil 3-OCDD

Sample: **Compost 1**

Element: **OCDF**

**Mandel's h statistics
(Compost 1 - OCDF)**

**Mandel's k statistics
(Compost 1 - OCDF)**

Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDF)
Mandel's h statistics (Compost 1 - OCDF)
Compost 1 - OCDF -- Mean PARM = 26.4 [ng/kg]

	General calc.parm.
T1=	4.22325E+02
T2=	1.14161E+04
T3=	16
T4=	64
T5=	4.9164E+02
n=	variabel
p=	4
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	19.5000	2.380	4		-0.64	0.04			19.5000	2.3805		4	3	-6.90
56L	27.3802	6.858	4		-0.42	0.11			27.3802	6.8578		4	3	0.98
12L	28.5762	10.444	4		-0.38	0.16			28.5762	10.4439		4	3	2.18
4L	30.1250	1.452	4		-0.34	0.02			30.1250	1.4523		4	3	3.73
23L	106.0000	142.738	4	!!	1.78	2.23	!!		-	,23L	-	-		79.60
Tot.gem	42.316	32.774 ng/kg		*1%-level:*	1.72	(1.73)		4	26.3953		(23L)	4	3	
Tot.std=	35.835	61.577		*5%-level:*	1.57	(1.53)		1						

RESULTS: Mean =   **26.39533**   *ng/kg*

Repeatability variance	S2r =	40.96990				
Repeatability std.	Sr =	**6.40077**	-->	24.25%	r =	17.9222
Between lab variance	S2L =	12.15150				
Reproducibility var.	S2R =	53.12140				
Reproducibility std.	SR =	**7.28844**	-->	27.61%	R =	20.4076
Remarks:	1 Lab rejected! (23L)					

Compost 1 - OCDF -- Mean PARM = 26.4 [ng/kg]

Compost 1-OCDF

Sample: **Sludge 1**

Element: **OCDF**

Mandel's h statistics (Sludge 1 - OCDF)
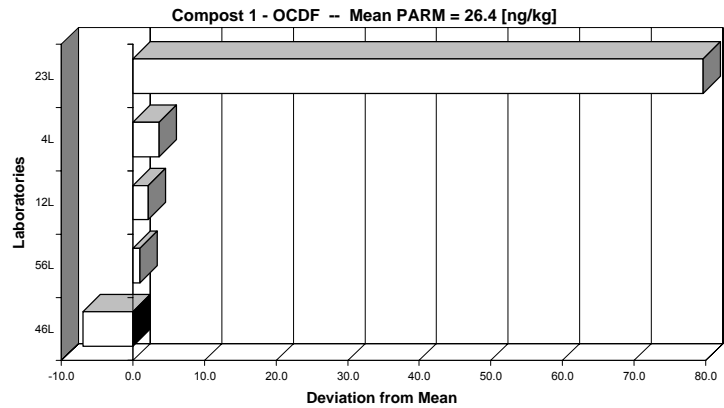
Mandel's k statistics (Sludge 1 - OCDF)
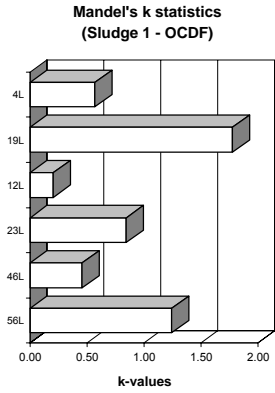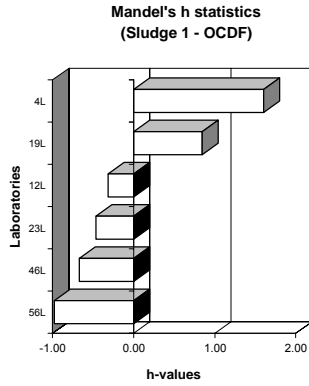
Mandel's k statistics (Sludge 1 - OCDF)
Mandel's h statistics (Sludge 1 - OCDF)
Sludge 1 - OCDF -- Mean PARM = 197 [ng/kg]

	General calc.parm.
T1=	4.72719E+03
T2=	9.57403E+05
T3=	24
T4=	96
T5=	1.3498E+04
n=	variabel
p=	6
N-table=	4

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
56L	161.4505	34.053	4		-0.98	1.24	Fail		161.4505	34.0534		4	3	-35.52
46L	172.5000	12.583	4		-0.67	0.46			172.5000	12.5831		4	3	-24.47
23L	180.0000	23.094	4		-0.47	0.84			180.0000	23.0940		4	3	-16.97
12L	185.3981	5.617	4		-0.32	0.21			185.3981	5.6169		4	3	-11.57
19L	227.5000	48.720	4		0.84	1.78	!!		227.5000	48.7203		4	3	30.53
4L	254.9500	15.587	4		1.60	0.57	Fail		254.9500	15.5875		4	3	57.98
Tot.gem	196.966	23.276 ng/kg		1%-level:	1.87	(1.77)		6	196.9664	()		6	5	
Tot.std=	36.266	15.804		5%-level:	1.66	(1.54)								

RESULTS: Mean = **196.96644** ng/kg

Repeatability variance	S2r =	749.91486				
Repeatability std.	Sr =	**27.38457**	-->	13.90%	r =	76.6768
Between lab variance	S2L =	1127.73420				
Reproducibility var.	S2R =	1877.64907				
Reproducibility std.	SR =	**43.33185**	-->	22.00%	R =	121.3292
Remarks:	none					

Sludge 1 - OCDF -- Mean PARM = 197 [ng/kg]

Laboratories: 4L, 19L, 12L, 23L, 46L, 56L

Deviation from Mean

Sludge 1-OCDF

Sample: **Soil 3**

Element: **OCDF**

Mandel's h statistics (Soil 3 - OCDF)
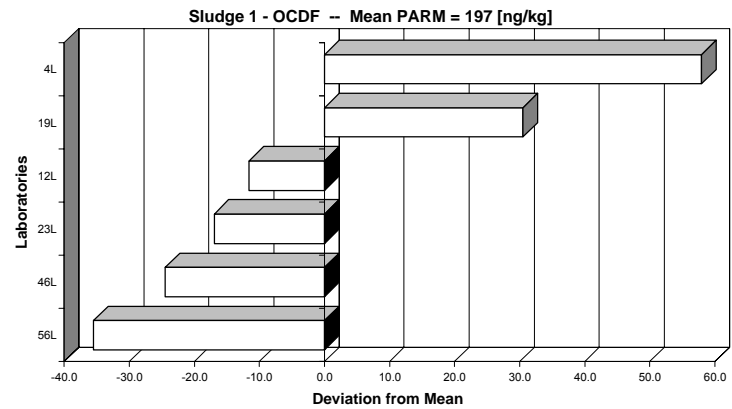
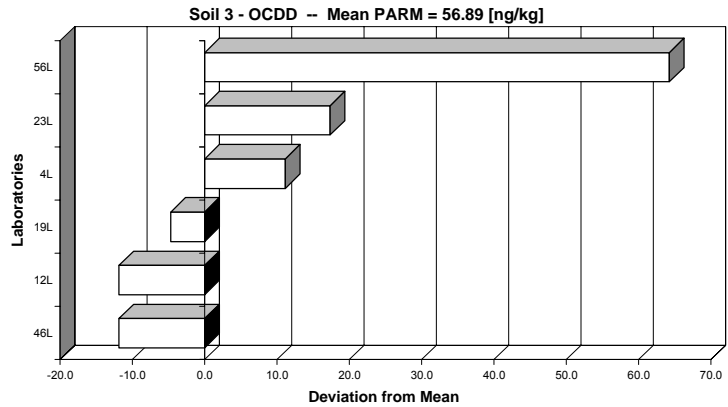**Mandel's k statistics (Soil 3 - OCDF)**
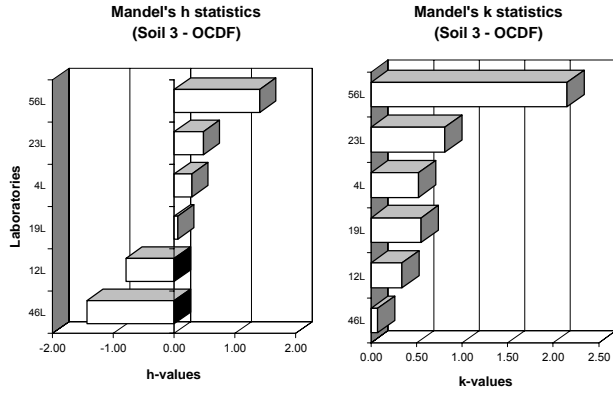
Unit: ng/kg

Mandel's k statistics (Soil 3 - OCDF)
Mandel's h statistics (Soil 3 - OCDF)
Soil 3 - OCDF -- Mean PARM = 42.27 [ng/kg]

	General calc.parm.
T1=	7.99768E+02
T2=	3.47346E+04
T3=	19
T4=	73
T5=	2.1843E+02
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	30.5000	0.577	4		-1.44	0.08		Fail	30.5000	0.5774		4	3	-11.77
12L	37.0921	2.586	4		-0.79	0.34			37.0921	2.5857		4	3	-5.18
19L	45.7000	4.204	3		0.06	0.55			45.7000	4.2036		3	2	3.43
4L	48.0750	4.001	4		0.29	0.53			48.0750	4.0011		4	3	5.80
23L	50.0000	6.164	4		0.48	0.81			50.0000	6.1644		4	3	7.73
56L	59.3776	16.396	4		1.41	2.15	!!	Fail	-		,56L	-	-	17.10
Tot.gem	45.124	5.655 ng/kg		1%-level:	1.87	(1.77)		5	42.2734	(56L)		5	4	
Tot.std=	10.143	5.580		5%-level:	1.66	(1.54)		1						

RESULTS: **Mean =**   **42.27342**   *ng/kg*

Repeatability variance	**S2r =**	15.60183				
Repeatability std.	**Sr =**	**3.94992**	-->	9.34%	**r =**	11.0598
Between lab variance	**S2L =**	66.46505				
Reproducibility var.	**S2R =**	82.06688				
Reproducibility std.	**SR =**	**9.05908**	-->	21.43%	**R =**	25.3654
Remarks:	1 Lab rejected! (56L)					

Soil 3 - OCDF -- Mean PARM = 42.27 [ng/kg]

Soil 3-OCDF

Sample: **Sludge 1**

Element: **DL PCB 77**

Mandel's h statistics (Sludge 1 - DL PCB 77)
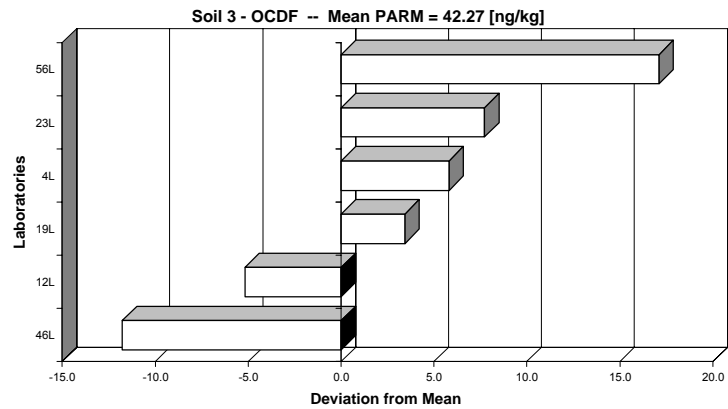
Mandel's k statistics (Sludge 1 - DL PCB 77)
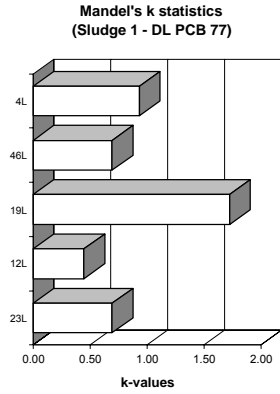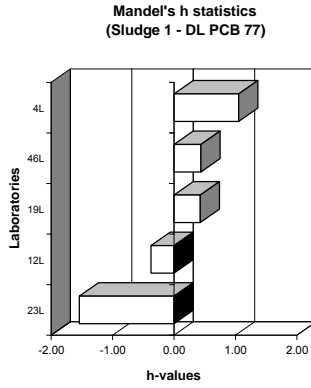
Mandel's k statistics (Sludge 1 - DL PCB 77)
Mandel's h statistics (Sludge 1 - DL PCB 77)
Sludge 1 - DL PCB 77 -- Mean PARM = 6.67 [ng/kg]

	General calc.parm.
T1=	1.26359E+02
T2=	8.48262E+02
T3=	19
T4=	73
T5=	1.5623E+00
n=	variabel
p=	5
N-table=	4

LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	5.5750	0.250	4		-1.54	0.69		Fail	5.5750	0.2500		4	3	-1.09
12L	6.4025	0.161	4		-0.37	0.45			6.4025	0.1611		4	3	-0.26
19L	6.9667	0.621	3		0.42	1.72	!		6.9667	0.6208		3	2	0.30
46L	6.9750	0.250	4		0.44	0.69			6.9750	0.2500		4	3	0.31
4L	7.4123	0.336	4		1.05	0.93	Fail		7.4123	0.3359		4	3	0.75
Tot.gem	6.666	0.324 ng/kg		1%-level:	1.72	(1.73)			5	6.6663	()		5	4
Tot.std=	0.708	0.177		5%-level:	1.57	(1.53)								

RESULTS: Mean =   **6.66628**   ng/kg

Repeatability variance	S2r =	0.11159				
Repeatability std.	Sr =	**0.33405**	-->	5.01%	r =	0.9353
Between lab variance	S2L =	0.49272				
Reproducibility var.	S2R =	0.60431				
Reproducibility std.	SR =	**0.77737**	-->	11.66%	R =	2.1766
Remarks:	none					

Sludge 1 - DL PCB 77 -- Mean PARM = 6.67 [ng/kg]

Sludge 1-DL PCB 77

Sample: **Sludge 1**

Element: **DL PCB 81**

**Mandel's h statistics
(Sludge 1 - DL PCB 81)**
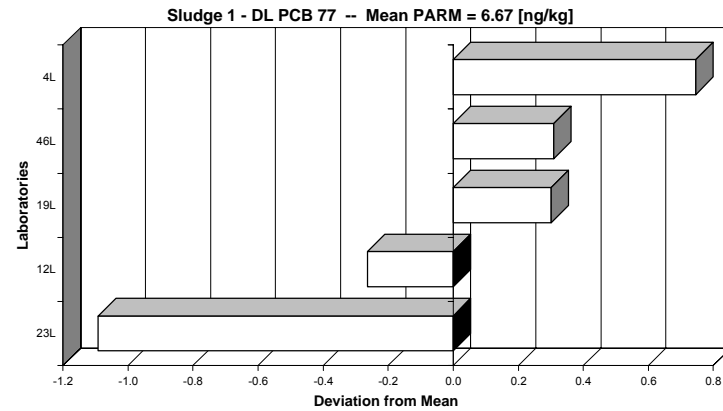
**Mandel's k statistics
(Sludge 1 - DL PCB 81)**
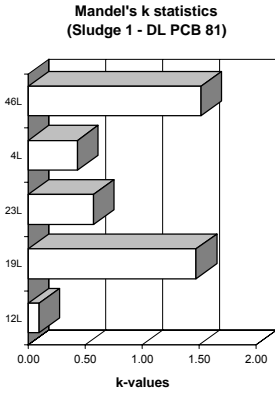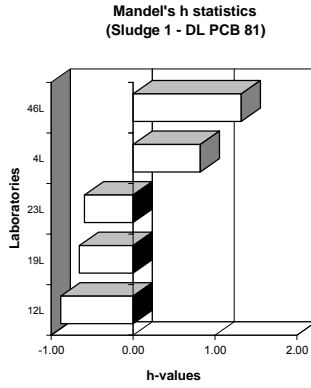
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 81)
Mandel's h statistics (Sludge 1 - DL PCB 81)
Sludge 1 - DL PCB 81 -- Mean PARM = 0.524 [ng/kg]

	General calc.parm.
T1=	1.01193E+01
T2=	6.31774E+00
T3=	19
T4=	73
T5=	4.2526E-02
n=	variabel
p=	5
N-table=	4

				Mandel's statistics				End Result:						
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
12L	0.3083	0.006	4		-0.88	0.10		Fail	0.3083	0.0055		4	3	-0.22
19L	0.3627	0.085	3		-0.66	1.47		Fail	0.3627	0.0849		3	2	-0.16
23L	0.3775	0.033	4		-0.60	0.57		Fail	0.3775	0.0330		4	3	-0.15
4L	0.7246	0.025	4		0.82	0.43	Fail		0.7246	0.0249		4	3	0.20
46L	0.8475	0.087	4		1.32	1.52	Fail		0.8475	0.0873		4	3	0.32
Tot.gem	0.524	0.047 ng/kg		1%-level:	1.72	(1.73)		5	0.5241	()		5	4	
Tot.std=	0.244	0.037		5%-level:	1.57	(1.53)								

RESULTS: Mean = **0.52410** ng/kg

Repeatability variance	S2r =	0.00304
Repeatability std.	Sr =	**0.05511** --> 10.52% r = 0.1543
Between lab variance	S2L =	0.06044
Reproducibility var.	S2R =	0.06347
Reproducibility std.	SR =	**0.25194** --> 48.07% R = 0.7054
Remarks:	none	

Sludge 1 - DL PCB 81 -- Mean PARM = 0.524 [ng/kg]

Sludge 1-DL PCB 81

Mandel's h statistics
(Sludge 1 - DL PCB 105)

Mandel's k statistics
(Sludge 1 - DL PCB 105)

Sample: **Sludge 1**

Element: **DL PCB 105**
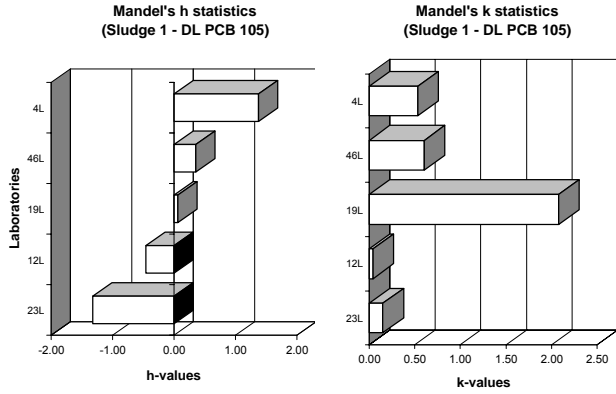
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 105)
Mandel's h statistics (Sludge 1 - DL PCB 105)
Sludge 1 - DL PCB 105 -- Mean PARM = 19.18 [ng/kg]

General calc.parm.

T1=	3.06948E+02
T2=	5.96805E+03
T3=	16
T4=	64
T5=	2.2105E+01
n=	variabel
p=	4
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	16.2500	0.500	4		-1.33	0.15			16.2500	0.5000		4	3	-2.93
12L	18.2000	0.141	4		-0.46	0.04			18.2000	0.1414		4	3	-0.98
19L	19.3500	6.859	2		0.06	2.08	!!		-		,19L	-		0.17
46L	20.0000	2.000	4		0.35	0.61			20.0000	2.0000		4	3	0.82
4L	22.2870	1.760	4		1.38	0.53			22.2870	1.7602		4	3	3.10
Tot.gem	19.217	2.252 ng/kg		1%-level:	1.72	(1.73)		4	19.1843	(19L)		4	3	
Tot.std=	2.230	2.695		5%-level:	1.57	(1.53)		1						

RESULTS: **Mean =**   **19.18425**   *ng/kg*

Repeatability variance	S2r =	1.84209			
Repeatability std.	Sr =	**1.35724**	-->	7.07%	r =	3.8003
Between lab variance	S2L =	6.16317			
Reproducibility var.	S2R =	8.00526			
Reproducibility std.	SR =	**2.82936**	-->	14.75%	R =	7.9222
Remarks:		1 Lab rejected! (19L)			

Sludge 1 - DL PCB 105 -- Mean PARM = 19.18 [ng/kg]

Sludge 1-DL PCB 105

Sample: **Sludge 1**

Element: **DL PCB 114**

Mandel's h statistics
(Sludge 1 - DL PCB 114)
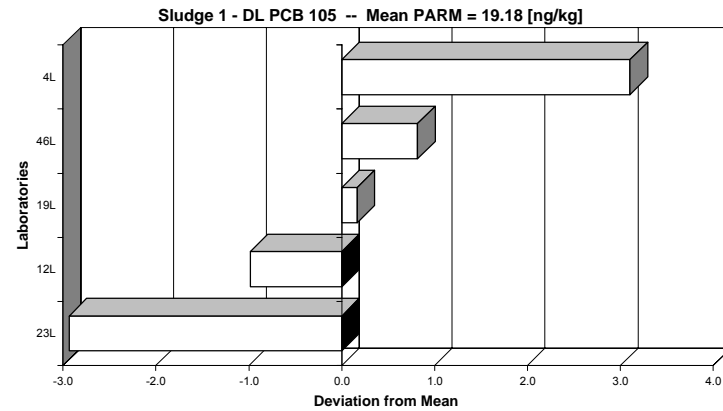
Mandel's k statistics
(Sludge 1 - DL PCB 114)
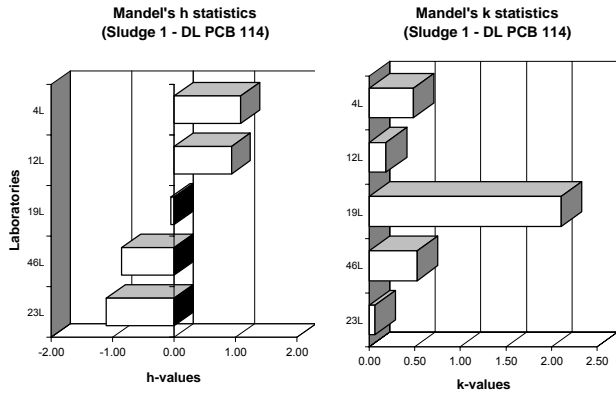
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 114)
Mandel's h statistics (Sludge 1 - DL PCB 114)
Sludge 1 - DL PCB 114 -- Mean PARM = 1.37 [ng/kg]

	General calc.parm.
T1=	2.18990E+01
T2=	3.18006E+01
T3=	16
T4=	64
T5=	5.4269E-02
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:						
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.9900	0.012	4		-1.11	0.06		Fail	0.9900	0.0115		4	3	-0.38
46L	1.0750	0.096	4		-0.85	0.53			1.0750	0.0957		4	3	-0.29
19L	1.3450	0.382	4		-0.06	2.11	!!		-		,19L	-		-0.02
12L	1.6800	0.034	4		0.93	0.19	Fail		1.6800	0.0337		4	3	0.31
4L	1.7298	0.087	4		1.08	0.48	Fail		1.7298	0.0875		4	3	0.36
Tot.gem	1.364	0.122 ng/kg		1%-level:	1.72	(1.73)		4	1.3687	(19L)		4	3	
Tot.std=	0.338	0.149		5%-level:	1.57	(1.53)		1						

RESULTS: **Mean =** **1.36869** ng/kg

Repeatability variance	**S2r =**	0.00452				
Repeatability std.	**Sr =**	**0.06725**	-->	4.91%	**r =**	0.1883
Between lab variance	**S2L =**	0.15118				
Reproducibility var.	**S2R =**	0.15570				
Reproducibility std.	**SR =**	**0.39459**	-->	28.83%	**R =**	1.1049
Remarks:	1 Lab rejected! (19L)					

Sludge 1 - DL PCB 114 -- Mean PARM = 1.37 [ng/kg]

Sludge 1-DL PCB 114

Sample: **Sludge 1**

Element: **DL PCB 118**

Mandel's h statistics
(Sludge 1 - DL PCB 118)

Laboratories: 4L, 46L, 12L, 23L, 19L — h-values (-2.00 to 2.00)

Mandel's k statistics
(Sludge 1 - DL PCB 118)

Laboratories: 4L, 46L, 12L, 23L, 19L — k-values (0.00 to 2.50)
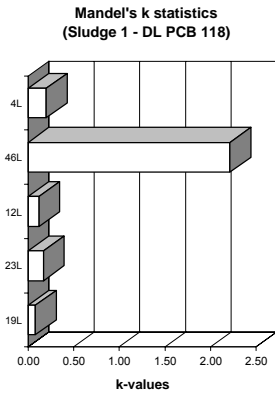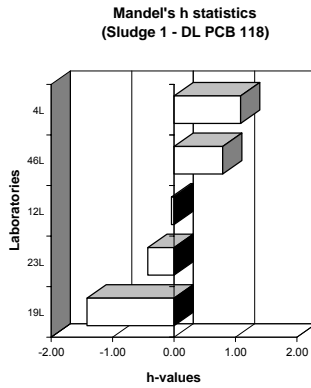
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 118)
Mandel's h statistics (Sludge 1 - DL PCB 118)
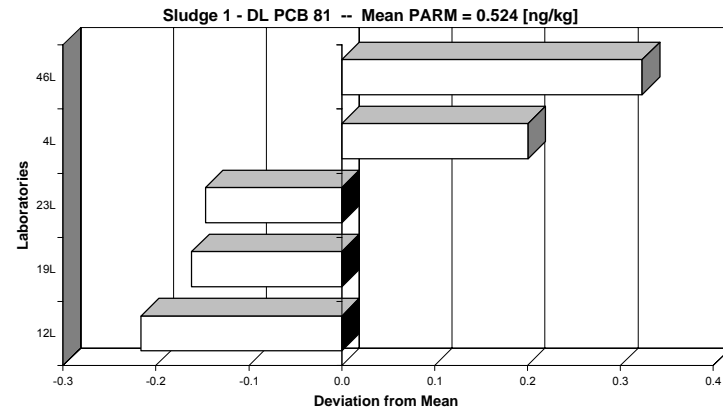Sludge 1 - DL PCB 118 -- Mean PARM = 32.59 [ng/kg]

	General calc.parm.
T1=	5.21365E+02
T2=	1.71649E+04
T3=	16
T4=	64
T5=	8.5628E+00
n=	variabel
p=	4
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	28.0750	0.457	4		-1.42	0.08			28.0750	0.4573		4	3	-4.51
23L	31.7500	0.957	4		-0.42	0.17			31.7500	0.9574		4	3	-0.84
12L	33.1750	0.670	4		-0.04	0.12			33.1750	0.6702		4	3	0.59
46L	36.2500	12.527	4		0.79	2.22	!!	-	-		,46L	-	-	3.66
4L	37.3413	1.131	4		1.09	0.20			37.3413	1.1310		4	3	4.76
Tot.gem	33.318	3.149 ng/kg		1%-level:	1.72	(1.73)		4	32.5853		(46L)	4	3	
Tot.std=	3.700	5.249		5%-level:	1.57	(1.53)		1						

RESULTS: **Mean =**   **32.58531**   *ng/kg*

Repeatability variance	**S2r =**	0.71356			
Repeatability std.	**Sr =**	**0.84473**	-->	2.59%	**r =** 2.3652
Between lab variance	**S2L =**	14.49072			
Reproducibility var.	**S2R =**	15.20429			
Reproducibility std.	**SR =**	**3.89927**	-->	11.97%	**R =** 10.9179
Remarks:	1 Lab rejected! (46L)				

Sludge 1 - DL PCB 118 -- Mean PARM = 32.59 [ng/kg]

Laboratories: 4L, 46L, 12L, 23L, 19L — Deviation from Mean (-5.0 to 5.0)

Sludge 1-DL PCB 118

Sample: **Sludge 1**

Element: **DL PCB 123**

Mandel's h statistics
(Sludge 1 - DL PCB 123)
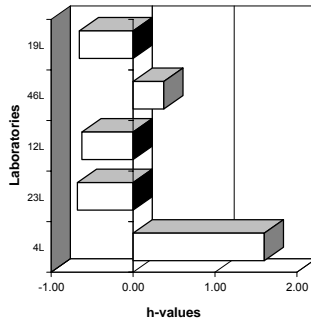
h-values

Mandel's k statistics
(Sludge 1 - DL PCB 123)

k-values
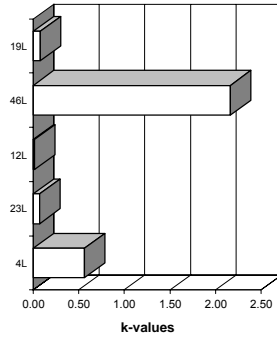
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 123)
Mandel's h statistics (Sludge 1 - DL PCB 123)
#NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
4L	4.6928	0.458	4	!	1.60	0.56	Fail	-	-	,4L	-	-		#NUM!
23L	0.7575	0.058	4		-0.68	0.07			#NUM!	,23L	-	-		#NUM!
12L	0.8498	0.013	4		-0.63	0.02			-	,12L	-	-		#NUM!
46L	2.5750	1.752	4		0.37	2.16	!!		-	,46L	-	-		#NUM!
19L	0.8003	0.065	4		-0.66	0.08			-	,19L	-	-		#NUM!
Tot.gem	1.935	0.469 ng/kg		1%-level:	1.72	(1.73)			#NUM!	(4L,23L,12L,46L,19L)			-1	
Tot.std=	1.722	0.739		5%-level:	1.57	(1.53)		5						

RESULTS: **Mean =** **#NUM!** *ng/kg*

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!
Remarks:		5 Labs rejected! (4L,23L,12L,46L,19L)				

#NUM!

Deviation from Mean

Sludge 1-DL PCB 123

**Mandel's h statistics
(Sludge 1 - DL PCB 126)**
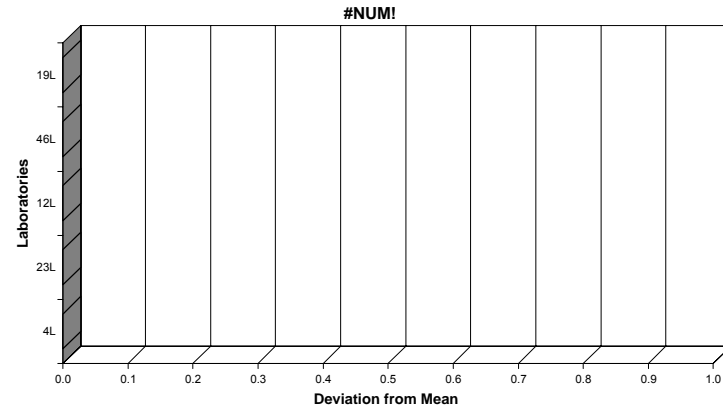
**Mandel's k statistics
(Sludge 1 - DL PCB 126)**
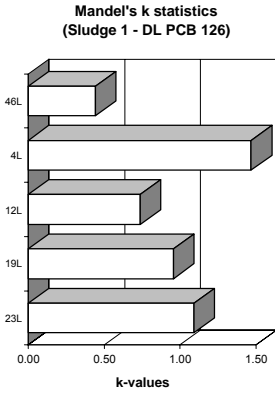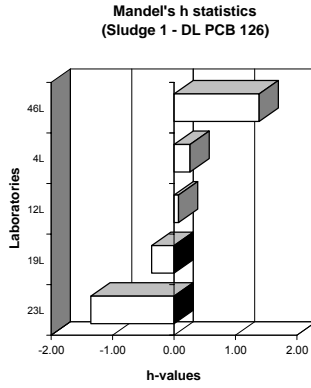
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 126)
Mandel's h statistics (Sludge 1 - DL PCB 126)
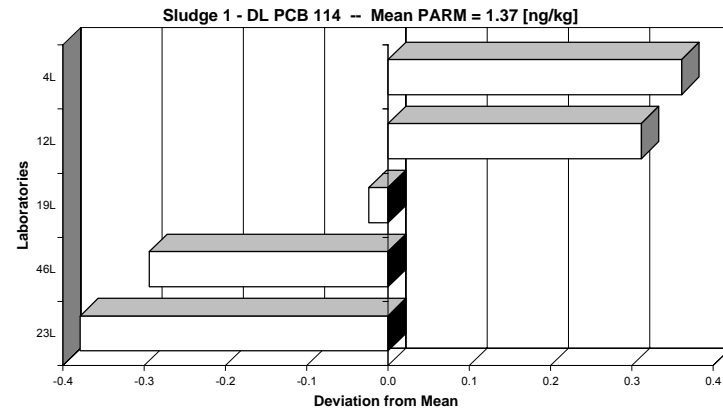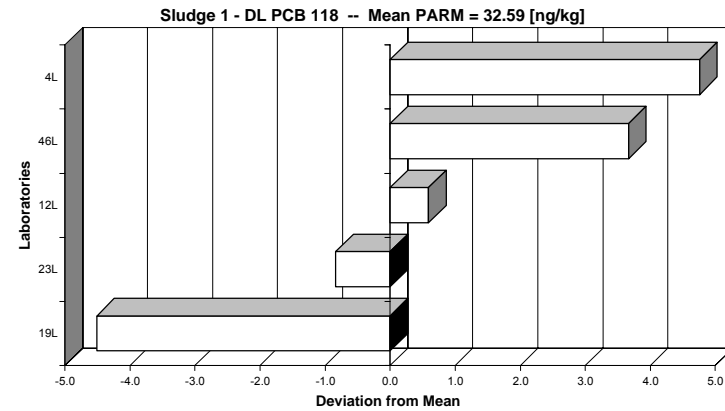Sludge 1 - DL PCB 126 -- Mean PARM = 0.25 [ng/kg]

	General calc.parm.
T1=	4.77910E+00
T2=	1.29646E+00
T3=	19
T4=	73
T5=	1.1401E-02
n=	variabel
p=	5
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.1450	0.031	4		-1.36	1.09		Fail	0.1450	0.0311		4	3	-0.11
19L	0.2220	0.027	3		-0.36	0.96		Fail	0.2220	0.0272		3	2	-0.03
12L	0.2558	0.021	4		0.07	0.74			0.2558	0.0209		4	3	0.01
4L	0.2700	0.042	4		0.26	1.47			0.2700	0.0418		4	3	0.02
46L	0.3575	0.013	4		1.39	0.44	Fail		0.3575	0.0126		4	3	0.11
Tot.gem	0.250	0.027 ng/kg		1%-level:	1.72	(1.73)		5	0.2501	()		5	4	
Tot.std=	0.077	0.011		5%-level:	1.57	(1.53)								

RESULTS: **Mean =**   **0.25006**   *ng/kg*

Repeatability variance	**S2r =**	0.00081				
Repeatability std.	**Sr =**	**0.02854**	-->	11.41%	**r =**	0.0799
Between lab variance	**S2L =**	0.00601				
Reproducibility var.	**S2R =**	0.00683				
Reproducibility std.	**SR =**	**0.08261**	-->	33.04%	**R =**	0.2313
Remarks:	**none**					

Sludge 1 - DL PCB 126 -- Mean PARM = 0.25 [ng/kg]

Sludge 1-DL PCB 126

Sample: **Sludge1**

Element: **DL PCB 156**

Unit: ng/kg

Mandel's h statistics
(Sludge1 - DL PCB 156)

Mandel's k statistics
(Sludge1 - DL PCB 156)
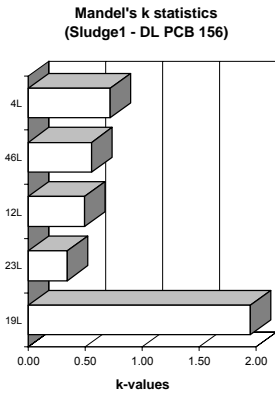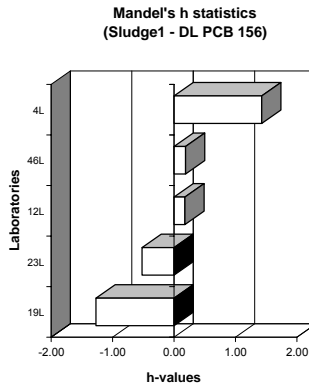
Mandel's k statistics (Sludge1 - DL PCB 156)
Mandel's h statistics (Sludge1 - DL PCB 156)

Sludge1 - DL PCB 156 -- Mean PARM = 7.31 [ng/kg]

	General calc.parm.
T1=	1.09765E+02
T2=	8.08898E+02
T3=	15
T4=	57
T5=	7.3657E-01
n=	variabel
p=	4
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	5.9575	0.927	4		-1.27	1.95	!!	Fail	-	-	,19L	-		-1.35
23L	6.6000	0.163	4		-0.52	0.34			6.6000	0.1633		4	3	-0.71
12L	7.1875	0.234	4		0.17	0.49			7.1875	0.2341		4	3	-0.12
46L	7.2000	0.265	3		0.19	0.56			7.2000	0.2646		3	2	-0.11
4L	8.2538	0.343	4		1.43	0.72		Fail	8.2538	0.3426		4	3	0.94
Tot.gem	7.040	0.386 ng/kg		1%-level:	1.72	(1.73)			4	7.3103	(19L)	4	3	
Tot.std=	0.849	0.309		5%-level:	1.57	(1.53)			1					

RESULTS: **Mean =** **7.31031** ng/kg

Repeatability variance	**S2r =**	0.06696				
Repeatability std.	**Sr =**	**0.25877**	-->	3.54%	**r =**	0.7246
Between lab variance	**S2L =**	0.48872				
Reproducibility var.	**S2R =**	0.55568				
Reproducibility std.	**SR =**	**0.74544**	-->	10.20%	**R =**	2.0872
Remarks:	1 Lab rejected! (19L)					

Sludge1 - DL PCB 156 -- Mean PARM = 7.31 [ng/kg]

Sludge1-DL PCB 156

Sample: **Sludge 1**

Element: **DL PCB 157**

Mandel's h statistics
(Sludge 1 - DL PCB 157)
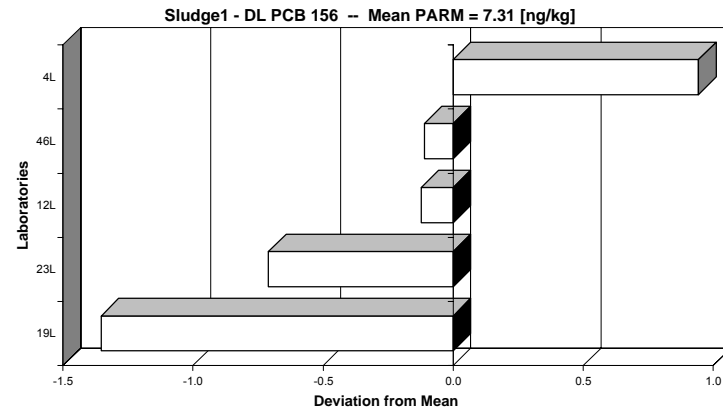
Mandel's k statistics
(Sludge 1 - DL PCB 157)
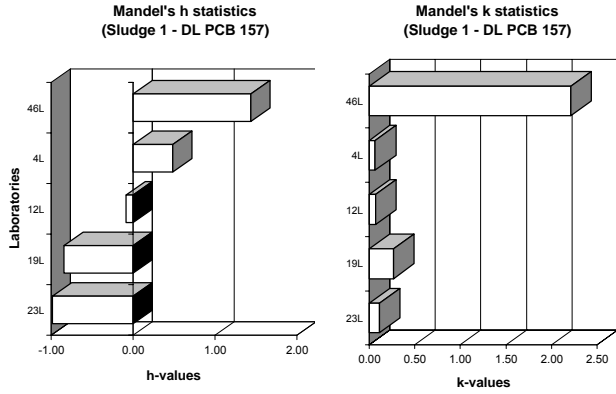
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 157)
Mandel's h statistics (Sludge 1 - DL PCB 157)
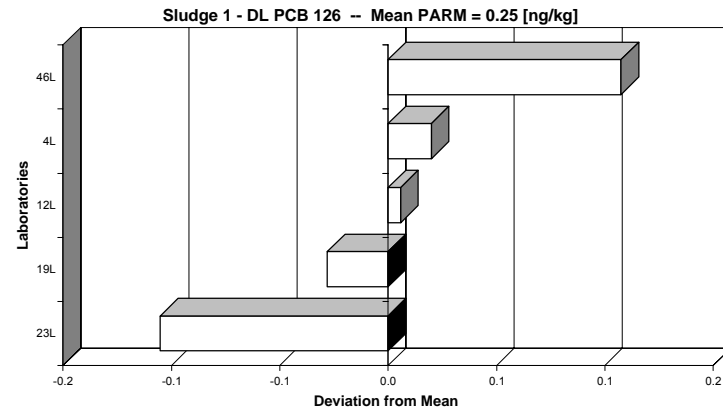Sludge 1 - DL PCB 157 -- Mean PARM = 0.864 [ng/kg]

	General calc.parm.
T1=	1.38191E+01
T2=	1.22413E+01
T3=	16
T4=	64
T5=	3.8332E-02
n=	variabel
p=	4
N-table=	4

LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
									End Result:					
						Mandel's statistics								
23L	0.7175	0.042	4		-0.99	0.11			0.7175	0.0419		4	3	-0.15
19L	0.7510	0.099	4		-0.84	0.27			0.7510	0.0988		4	3	-0.11
12L	0.9265	0.026	4		-0.09	0.07			0.9265	0.0258		4	3	0.06
4L	1.0598	0.024	4		0.49	0.07			1.0598	0.0244		4	3	0.20
46L	1.2800	0.814	4		1.44	2.21	!!		-	-	,46L	-	-	0.42
Tot.gem	0.947	0.201 ng/kg		1%-level:	1.72	(1.73)		4	0.8637		(46L)	4	3	
Tot.std=	0.232	0.344		5%-level:	1.57	(1.53)		1						

RESULTS: **Mean =**   **0.86369**   *ng/kg*

Repeatability variance	**S2r =**	0.00319				
Repeatability std.	**Sr =**	**0.05652**	-->	6.54%	**r =**	0.1583
Between lab variance	**S2L =**	0.02469				
Reproducibility var.	**S2R =**	0.02788				
Reproducibility std.	**SR =**	**0.16699**	-->	19.33%	**R =**	0.4676
Remarks:		1 Lab rejected! (46L)				

Sludge 1 - DL PCB 157 -- Mean PARM = 0.864 [ng/kg]
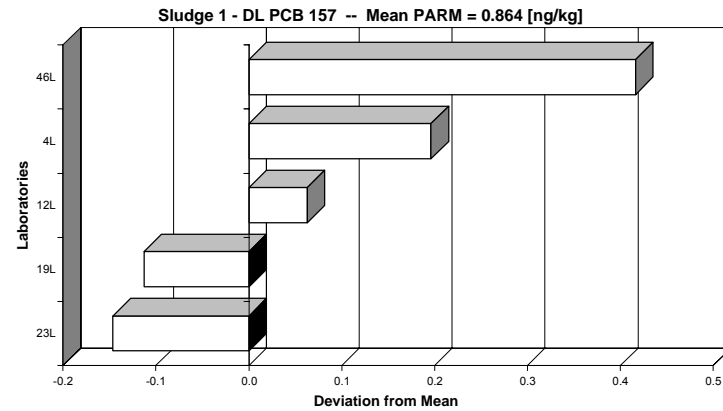
Sludge 1-DL PCB 157

Sample: **Sludge 1**

Element: **DL PCB 167**

Mandel's h statistics
(Sludge 1 - DL PCB 167)
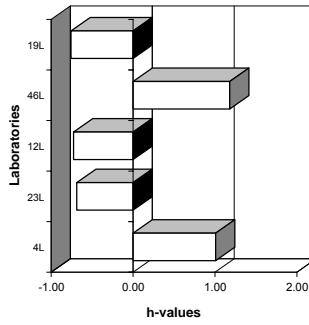
Mandel's k statistics
(Sludge 1 - DL PCB 167)
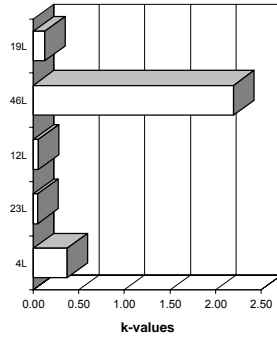
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 167)
Mandel's h statistics (Sludge 1 - DL PCB 167)
  #NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	4

					Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
4L	5.1284	0.758	4		1.01	0.37		-	-	,4L	-	-		#NUM!
23L	3.2250	0.096	4		-0.69	0.05		-	-	,23L	-	-		#NUM!
12L	3.1775	0.108	4		-0.73	0.05		-	-	,12L	-	-		#NUM!
46L	5.3250	4.451	4		1.18	2.20	!!	-	-	,46L	-	-		#NUM!
19L	3.1450	0.254	4		-0.76	0.13			#NUM!	-	,19L	-	-	#NUM!
Tot.gem	4.000	1.133 ng/kg		1%-level:	1.72	(1.73)			#NUM!	(4L,23L,12L,46L,19L)			-1	
Tot.std=	1.122	1.874		5%-level:	1.57	(1.53)		5						

RESULTS: **Mean =**   **#NUM!**   *ng/kg*

Repeatability variance	**S2r =**	**#DIV/0!**					
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!	
Between lab variance	**S2L =**	**#NUM!**					
Reproducibility var.	**S2R =**	**#NUM!**					
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!	

Remarks: 5 Labs rejected! (4L,23L,12L,46L,19L)

#NUM!
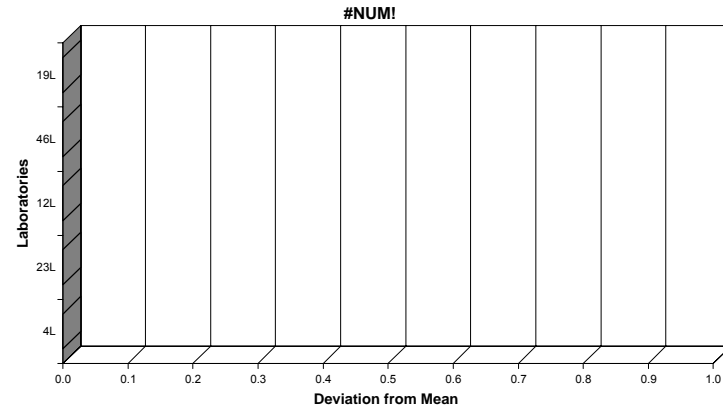
Sludge 1-DL PCB 167

Sample: **Sludge 1**
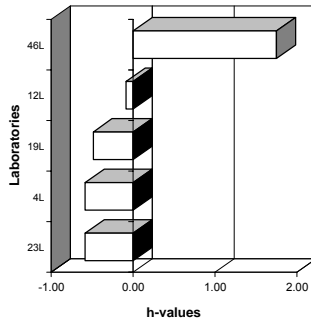
Element: **DL PCB 189**
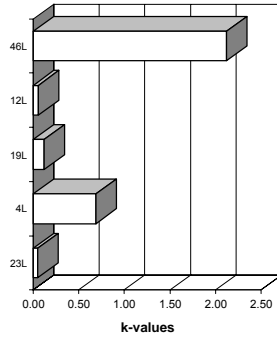
Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 189)
Mandel's h statistics (Sludge 1 - DL PCB 189)

#NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	4

Mandel's h statistics chart: Laboratories (46L, 12L, 19L, 4L, 23L) vs h-values axis from -1.00 to 2.00

Mandel's k statistics chart: Laboratories (46L, 12L, 19L, 4L, 23L) vs k-values axis from 0.00 to 2.50

				Mandel's statistics					End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.8900	0.038	4		-0.59	0.05			#NUM! -	,23L	-	-		#NUM!
4L	0.8909	0.506	4		-0.59	0.69			-	,4L	-	-		#NUM!
19L	0.9340	0.089	4		-0.49	0.12			-	,19L	-	-		#NUM!
12L	1.1175	0.043	4		-0.09	0.06			-	,12L	-	-		#NUM!
46L	1.9500	1.567	4	!!	1.75	2.12	!!		-	,46L	-	-		#NUM!
Tot.gem	1.156	0.449 ng/kg		1%-level:	1.72	(1.73)			#NUM!	(4L,23L,12L,46L,19L)			-1	
Tot.std=	0.453	0.655		5%-level:	1.57	(1.53)		5						

RESULTS: **Mean =** **#NUM!**   ng/kg

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!
Remarks:	5 Labs rejected! (4L,23L,12L,46L,19L)					

#NUM!

Chart: Laboratories (46L, 12L, 19L, 4L, 23L) vs Deviation from Mean axis from 0.0 to 1.0

Sludge 1-DL PCB 189

Mandel's h statistics
(Sludge 1 - Total DL PCB)

Mandel's k statistics
(Sludge 1 - Total DL PCB)
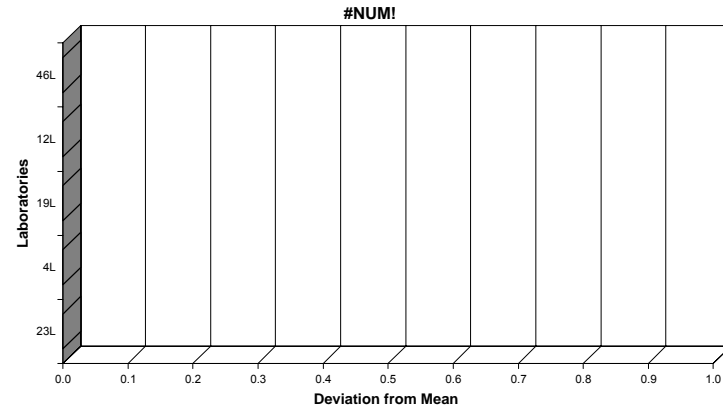
Sample: **Sludge 1**

Element: **Total DL PCB**
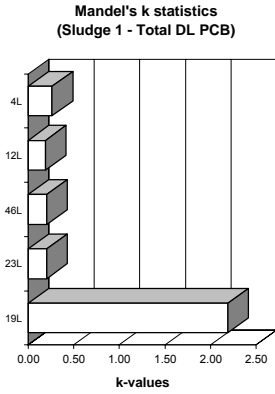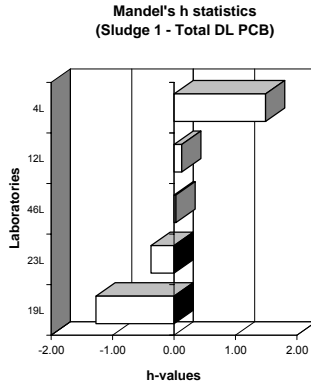
Unit: ng/kg

Mandel's k statistics (Sludge 1 - Total DL PCB)
Mandel's h statistics (Sludge 1 - Total DL PCB)
Sludge 1 - Total DL PCB -- Mean PARM = 75.64 [ng/kg]

	General calc.parm.
T1=	1.13809E+03
T2=	8.74948E+04
T3=	15
T4=	57
T5=	2.3213E+01
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
19L	56.3732	14.578	4		-1.27	2.19	!!	Fail	-	-	,19L	-		-19.27
23L	67.2775	1.357	4		-0.37	0.20			67.2775	1.3569		4	3	-8.36
46L	72.1303	1.363	3		0.03	0.21			72.1303	1.3629		3	2	-3.51
12L	73.3050	1.258	4		0.13	0.19			73.3050	1.2580		4	3	-2.33
4L	89.8429	1.754	4		1.49	0.26	Fail		89.8429	1.7537		4	3	14.20
Tot.gem	71.786	4.062 ng/kg		1%-level:	1.72	(1.73)		4	75.6389	(19L)		4	3	
Tot.std=	12.107	5.882		5%-level:	1.57	(1.53)		1						

RESULTS: Mean = **75.63891** ng/kg

Repeatability variance	S2r =	2.11024				
Repeatability std.	Sr =	**1.45267**	-->	1.92%	r =	4.0675
Between lab variance	S2L =	101.62752				
Reproducibility var.	S2R =	103.73776				
Reproducibility std.	SR =	**10.18517**	-->	13.47%	R =	28.5185
Remarks:	1 Lab rejected! (19L)					

Sludge 1 - Total DL PCB -- Mean PARM = 75.64 [ng/kg]

Sludge 1-Total DL PCB

Sample: **Compost 1**

Element: **PCB 77**

**Mandel's h statistics
(Compost 1 - PCB 77)**
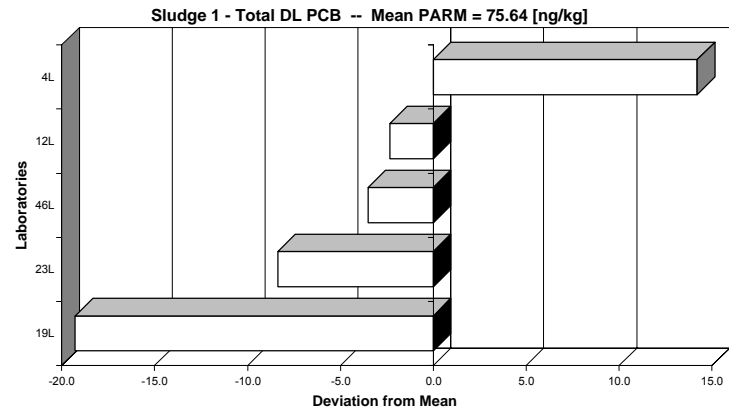
**Mandel's k statistics
(Compost 1 - PCB 77)**
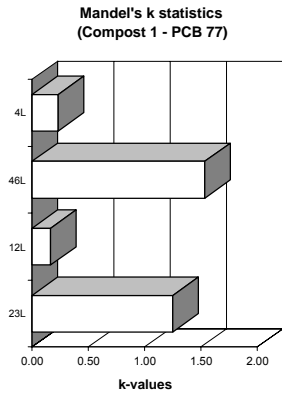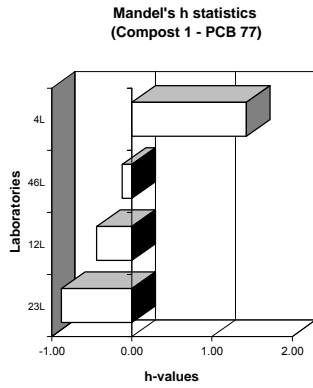
Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 77)
Mandel's h statistics (Compost 1 - PCB 77)
Compost 1 - PCB 77 -- Mean PARM = 0.132 [ng/kg]

	General calc.parm.
T1=	2.10450E+00
T2=	2.78903E-01
T3=	16
T4=	64
T5=	1.0204E-03
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
23L	0.1200	0.012	4		-0.87	1.25			0.1200	0.0115		4	3	-0.01
12L	0.1258	0.002	4		-0.44	0.16			0.1258	0.0015		4	3	-0.01
46L	0.1300	0.014	4		-0.12	1.53	!		0.1300	0.0141		4	3	0.00
4L	0.1504	0.002	4	!	1.43	0.23	Fail		0.1504	0.0021		4	3	0.02
Tot.gem	0.132	0.007 ng/kg		*1%-level:*	1.49	(1.67)		4	*0.1315*	()		4	3	
Tot.std=	0.013	0.006		*5%-level:*	1.42	(1.5)								

RESULTS: **Mean =** **0.13153** *ng/kg*

Repeatability variance	**S2r =**	0.00009			
Repeatability std.	**Sr =**	**0.00922**	-->	7.01%	**r =**	0.0258
Between lab variance	**S2L =**	0.00015			
Reproducibility var.	**S2R =**	0.00024			
Reproducibility std.	**SR =**	**0.01544**	-->	11.74%	**R =**	0.0432
Remarks:	**none**				

Compost 1 - PCB 77 -- Mean PARM = 0.132 [ng/kg]

Compost 1-PCB 77

Sample: **Compost 1**

Element: **PCB 105**

Mandel's h statistics
(Compost 1 - PCB 105)

Mandel's k statistics
(Compost 1 - PCB 105)
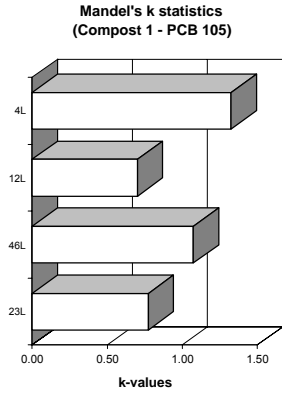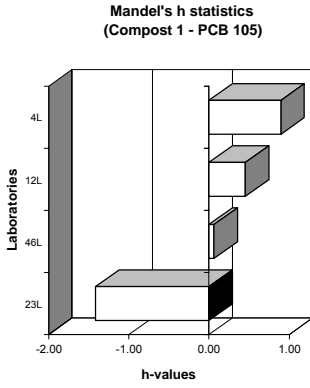
Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 105)
Mandel's h statistics (Compost 1 - PCB 105)
Compost 1 - PCB 105 -- Mean PARM = 0.683 [ng/kg]

	General calc.parm.
T1=	1.09233E+01
T2=	7.63227E+00
T3=	16
T4=	64
T5=	4.7234E-02
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.5125	0.049	4		-1.41	0.77		Fail	0.5125	0.0486		4	3	-0.17
46L	0.6900	0.067	4		0.06	1.07			0.6900	0.0673		4	3	0.01
12L	0.7375	0.044	4		0.45	0.70	Fail		0.7375	0.0439		4	3	0.05
4L	0.7908	0.083	4		0.90	1.33	Fail		0.7908	0.0832		4	3	0.11
Tot.gem	0.683	0.061 ng/kg		1%-level:	1.49	(1.67)		4	0.6827	()		4	3	
Tot.std=	0.121	0.018		5%-level:	1.42	(1.5)								

RESULTS: **Mean =** **0.68271** ng/kg

Repeatability variance	**S2r =**	0.00394			
Repeatability std.	**Sr =**	**0.06274**	-->	9.19%	**r =** 0.1757
Between lab variance	**S2L =**	0.01359			
Reproducibility var.	**S2R =**	0.01752			
Reproducibility std.	**SR =**	**0.13238**	-->	19.39%	**R =** 0.3707
Remarks:	**none**				

Compost 1 - PCB 105 -- Mean PARM = 0.683 [ng/kg]

Compost 1-PCB 105

Sample: **Compost 1**

Element: **PCB 118**

**Mandel's h statistics
(Compost 1 - PCB 118)**

**Mandel's k statistics
(Compost 1 - PCB 118)**
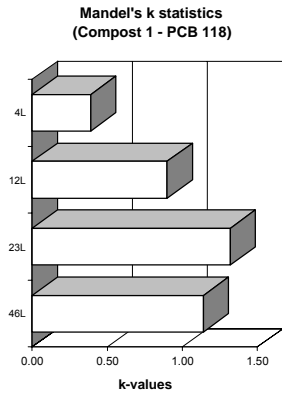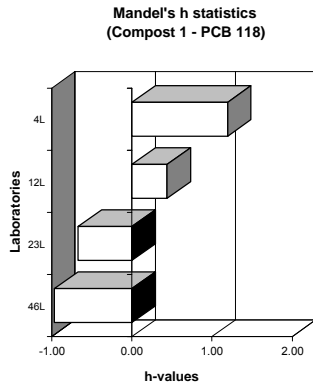
Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 118)
Mandel's h statistics (Compost 1 - PCB 118)
Compost 1 - PCB 118 -- Mean PARM = 1.95 [ng/kg]

General calc.parm.
T1=	3.11260E+01
T2=	6.13277E+01
T3=	16
T4=	64
T5=	4.3094E-01
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	1.7000	0.216	4		-0.96	1.14		Fail	1.7000	0.2160		4	3	-0.25
23L	1.7750	0.250	4		-0.67	1.32		Fail	1.7750	0.2500		4	3	-0.17
12L	2.0575	0.170	4		0.44	0.90			2.0575	0.1704		4	3	0.11
4L	2.2490	0.074	4		1.19	0.39	Fail		2.2490	0.0739		4	3	0.30
Tot.gem	1.945	0.178 ng/kg		1%-level:	1.49	(1.67)			4	1.9454	()		4	3
Tot.std=	0.254	0.076		5%-level:	1.42	(1.5)								

RESULTS: **Mean =** **1.94538** ng/kg

Repeatability variance	**S2r =**	0.03591			
Repeatability std.	**Sr =**	**0.18950**	-->	9.74%	**r =** 0.5306
Between lab variance	**S2L =**	0.05569			
Reproducibility var.	**S2R =**	0.09160			
Reproducibility std.	**SR =**	**0.30265**	-->	15.56%	**R =** 0.8474
Remarks:	none				
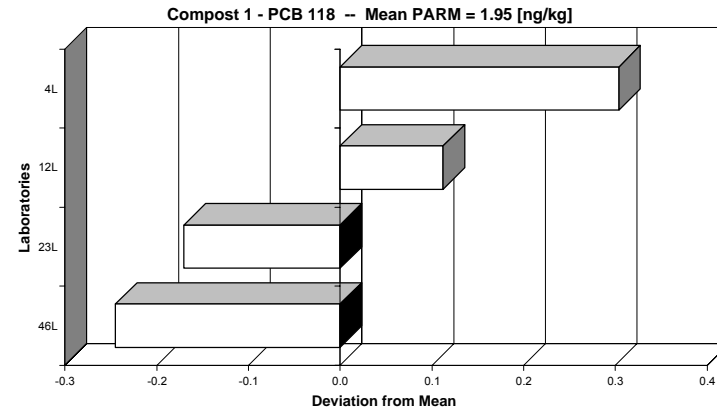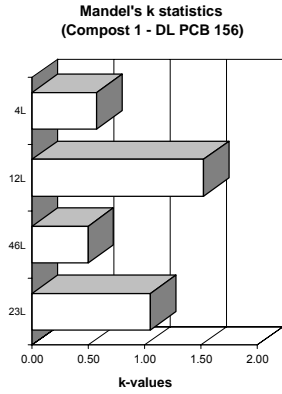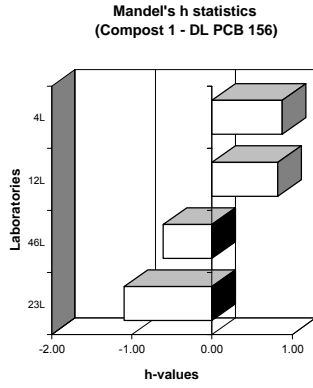
Compost 1 - PCB 118 -- Mean PARM = 1.95 [ng/kg]

Compost 1-PCB 118

Mandel's h statistics
(Compost 1 - DL PCB 156)

Mandel's k statistics
(Compost 1 - DL PCB 156)

Unit: ng/kg

Mandel's k statistics (Compost 1 - DL PCB 156)
Mandel's h statistics (Compost 1 - DL PCB 156)
Compost 1 - DL PCB 156 -- Mean PARM = 0.882 [ng/kg]

	General calc.parm.
T1=	1.41164E+01
T2=	1.27037E+01
T3=	16
T4=	64
T5=	6.9603E-01
n=	variabel
p=	4
N-table=	4

						Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.7250	0.254	4		-1.09	1.05			0.7250	0.2536		4	3	-0.16
46L	0.7950	0.120	4		-0.61	0.50			0.7950	0.1196		4	3	-0.09
12L	1.0005	0.366	4		0.82	1.52	!		1.0005	0.3664		4	3	0.12
4L	1.0086	0.138	4		0.88	0.57			1.0086	0.1383		4	3	0.13
Tot.gem	0.882	0.219 ng/kg		1%-level:	1.49	(1.67)		4	0.8823	()		4	3	
Tot.std=	0.144	0.115		5%-level:	1.42	(1.5)								

RESULTS:	Mean =	**0.88228**	ng/kg			
Repeatability variance	S2r =	0.05800				
Repeatability std.	Sr =	**0.24084**	-->	27.30%	r =	0.6743
Between lab variance	S2L =	0.00626				
Reproducibility var.	S2R =	0.06426				
Reproducibility std.	SR =	**0.25350**	-->	28.73%	R =	0.7098
Remarks:	none					

Compost 1 - DL PCB 156 -- Mean PARM = 0.882 [ng/kg]

Mandel's h statistics
(Compost 1 - PCB 157)

Mandel's k statistics
(Compost 1 - PCB 157)

Sample: **Compost 1**

Element: **PCB 157**
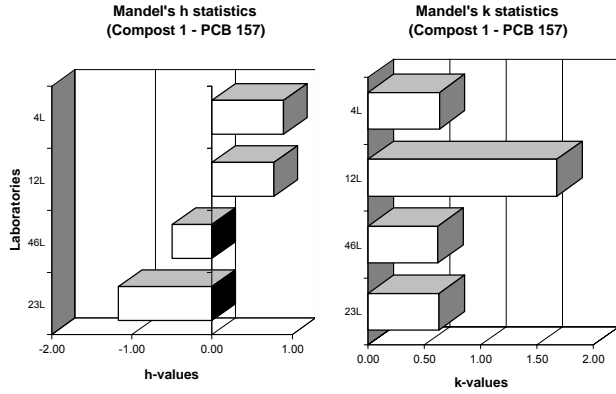
Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 157)
Mandel's h statistics (Compost 1 - PCB 157)
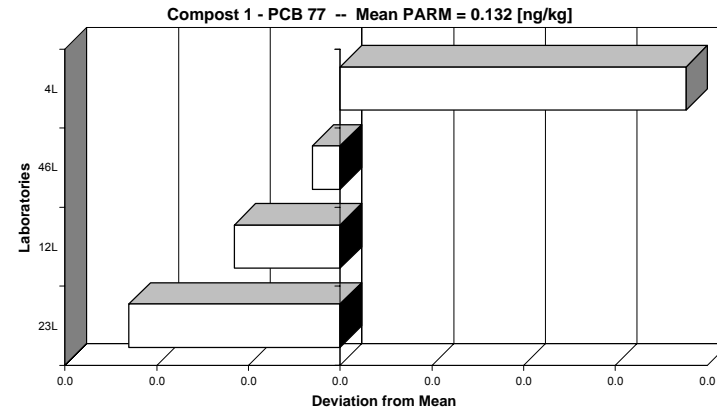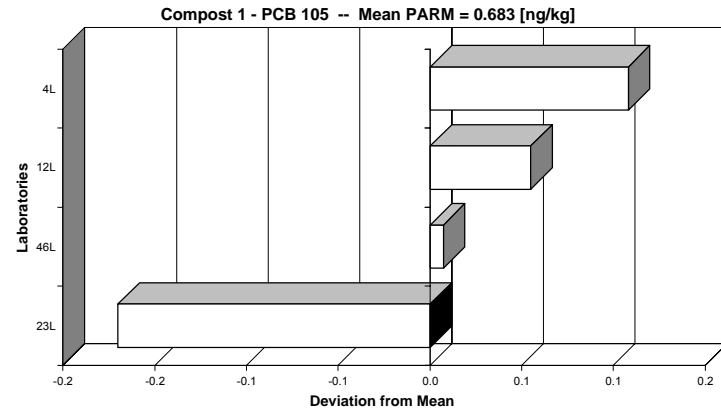Compost 1 - PCB 157 -- Mean PARM = 0.102 [ng/kg]

	General calc.parm.
T1=	1.63670E+00
T2=	1.70543E-01
T3=	16
T4=	64
T5=	3.7135E-03
n=	variabel
p=	4
N-table=	4

				Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	0.0835	0.011	4		-1.17	0.63		Fail	0.0835	0.0111		4	3	-0.02
46L	0.0943	0.011	4		-0.50	0.62			0.0943	0.0109		4	3	-0.01
12L	0.1148	0.030	4		0.77	1.68	!!		0.1148	0.0295		4	3	0.01
4L	0.1167	0.011	4		0.89	0.63			0.1167	0.0111		4	3	0.01
Tot.gem	0.102	0.016 ng/kg		1%-level:	1.49	(1.67)		4	0.1023	()		4	3	
Tot.std=	0.016	0.009		5%-level:	1.42	(1.5)								

RESULTS: **Mean =** **0.10229** ng/kg

Repeatability variance	**S2r =**	0.00031					
Repeatability std.	**Sr =**	**0.01759**	-->	17.20%	**r =**	0.0493	
Between lab variance	**S2L =**	0.00018					
Reproducibility var.	**S2R =**	0.00049					
Reproducibility std.	**SR =**	**0.02218**	-->	21.68%	**R =**	0.0621	
Remarks:	none						

Compost 1 - PCB 157 -- Mean PARM = 0.102 [ng/kg]

Compost 1-PCB 157

Mandel's h statistics
(Compost 1 - 167)
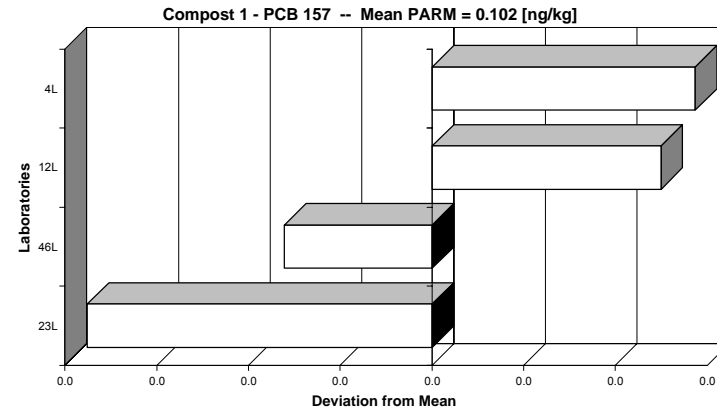
Mandel's k statistics
(Compost 1 - 167)
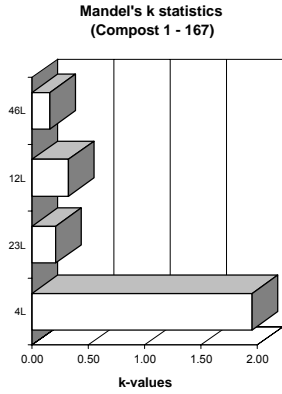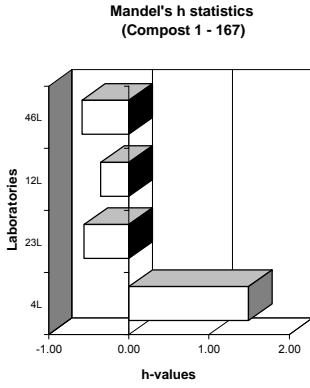
Unit: ng/kg

Mandel's k statistics (Compost 1 - 167)
Mandel's h statistics (Compost 1 - 167)
#NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	4

				Mandel's statistics			End Result:							
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
4L	1.0995	0.941	4	!!	1.49	1.96	!!		-	-	,4L	-	-	#NUM!
23L	0.4000	0.101	4		-0.56	0.21			-	-	,23L	-	-	#NUM!
12L	0.4700	0.155	4		-0.35	0.32			-	-	,12L	-	-	#NUM!
46L	0.3900	0.075	4		-0.59	0.16			#NUM!	-	,46L	-	-	#NUM!
Tot.gem	0.590	0.318 ng/kg		1%-level:	1.49	(1.67)			#NUM!		(4L,23L,12L,46L)		-1	
Tot.std=	0.342	0.417		5%-level:	1.42	(1.5)								

RESULTS: **Mean =**   **#NUM!**   ng/kg

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

Remarks: 4 Labs rejected! (4L,23L,12L,46L)

#NUM!
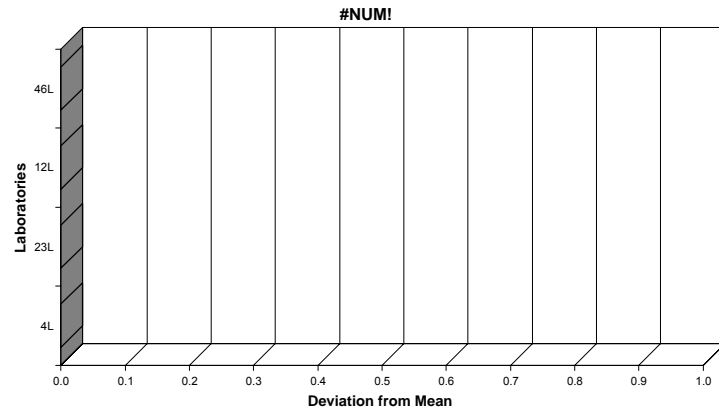
Compost 1-167

Sample: **Compost 1**

Element: **PCB 189**

Mandel's h statistics (Compost 1 - PCB 189)

Mandel's k statistics (Compost 1 - PCB 189)
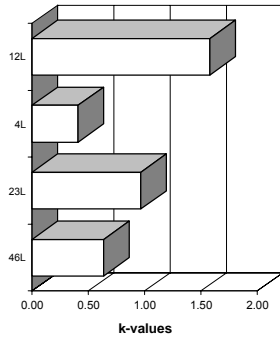
Mandel's k statistics (Compost 1 - PCB 189)
Mandel's h statistics (Compost 1 - PCB 189)
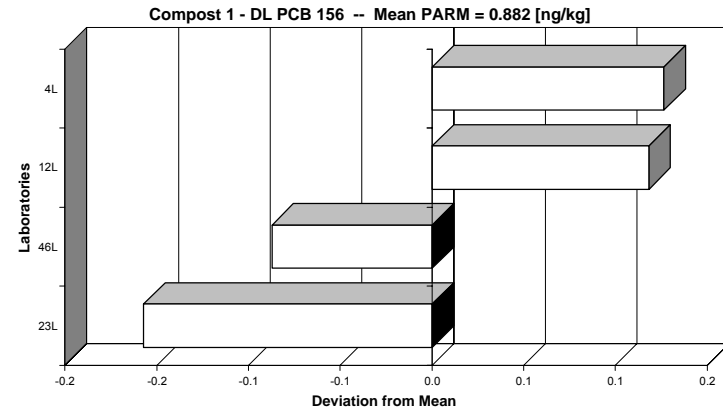Compost 1 - PCB 189 -- Mean PARM = 0.177 [ng/kg]

	General calc.parm.
T1=	2.82910E+00
T2=	5.03453E-01
T3=	16
T4=	64
T5=	5.2370E-02
n=	variabel
p=	4
N-table=	4

LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	0.1575	0.042	4		-1.18	0.63			0.1575	0.0419		4	3	-0.02
23L	0.1700	0.064	4		-0.42	0.97			0.1700	0.0638		4	3	-0.01
4L	0.1853	0.027	4		0.52	0.41			0.1853	0.0271		4	3	0.01
12L	0.1945	0.104	4		1.08	1.58	!		0.1945	0.1044		4	3	0.02
Tot.gem	0.177	0.059 ng/kg		1%-level:	1.49	(1.67)		4	0.1768	()		4	3	
Tot.std=	0.016	0.034		5%-level:	1.42	(1.5)								

Mandel's statistics / End Result:

RESULTS:	**Mean =**	**0.17682**	ng/kg
Repeatability variance	**S2r =**	0.00436	
Repeatability std.	**Sr =**	**0.06606**	--> 37.36% **r =** 0.1850
Between lab variance	**S2L =**	-0.00082	
Reproducibility var.	**S2R =**	0.00436	
Reproducibility std.	**SR =**	**0.06606**	--> 37.36% **R =** 0.1850
Remarks:	none		

Compost 1 - PCB 189 -- Mean PARM = 0.177 [ng/kg]

Compost 1-PCB 189

Unit: ng/kg

Mandel's h statistics
(Compost 1 - Total DL PCB)

Mandel's k statistics
(Compost 1 - Total DL PCB)

Mandel's k statistics (Compost 1 - Total DL PCB)
Mandel's h statistics (Compost 1 - Total DL PCB)
Compost 1 - Total DL PCB -- Mean PARM = 4.66 [ng/kg]
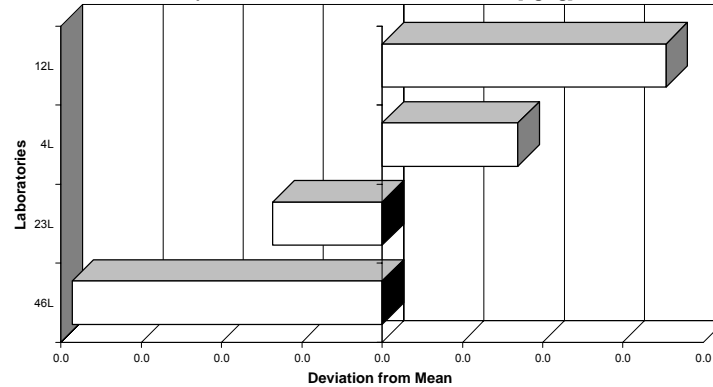
Sample: **Compost 1**

Element: **Total DL PCB**

	General calc.parm.
T1=	7.46177E+01
T2=	3.57297E+02
T3=	16
T4=	64
T5=	8.1859E+00
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
23L	3.7860	0.716	4		-1.00	0.87		Fail	3.7860	0.7161		4	3	-0.88
46L	4.2123	0.564	4		-0.51	0.68		Fail	4.2123	0.5635		4	3	-0.45
12L	4.8432	0.874	4		0.20	1.06			4.8432	0.8741		4	3	0.18
4L	5.8130	1.065	4		1.30	1.29	Fail		5.8130	1.0650		4	3	1.15
Tot.gem	4.664	0.805 ng/kg		*1%-level:*	1.49	(1.67)		4	*4.6636*	*()*		4	3	
Tot.std=	0.881	0.215		*5%-level:*	1.42	(1.5)								

RESULTS:	**Mean =**	**4.66361**	*ng/kg*		
Repeatability variance	**S2r =**	0.68216			
Repeatability std.	**Sr =**	**0.82593**	--> 17.71%	**r =**	2.3126
Between lab variance	**S2L =**	0.60520			
Reproducibility var.	**S2R =**	1.28736			
Reproducibility std.	**SR =**	**1.13462**	--> 24.33%	**R =**	3.1769
Remarks:	**none**				
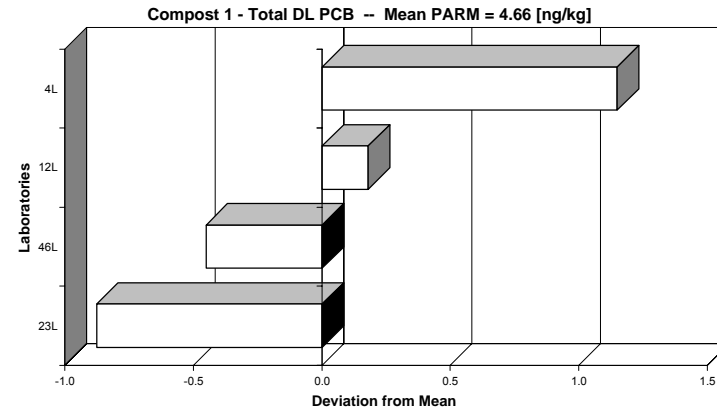
Compost 1 - Total DL PCB -- Mean PARM = 4.66 [ng/kg]

Compost 1-Total DL PCB

Sample: **Soil 3**

Element: **DL PCB 105**

Mandel's h statistics
(Soil 3 - DL PCB 105)

Mandel's k statistics
(Soil 3 - DL PCB 105)
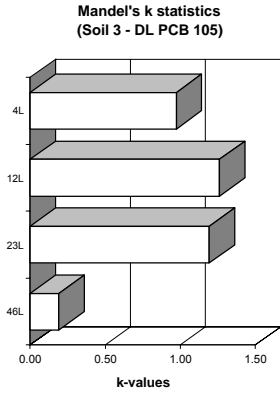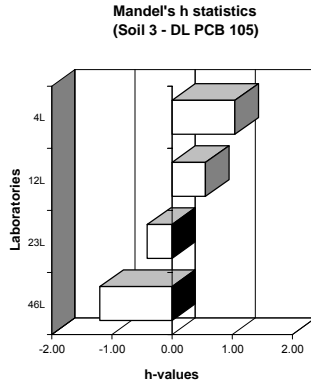
Unit: ng/kg

Mandel's k statistics (Soil 3 - DL PCB 105)
Mandel's h statistics (Soil 3 - DL PCB 105)
Soil 3 - DL PCB 105 -- Mean PARM = 0.096 [ng/kg]

	General calc.parm.
T1=	1.42530E+00
T2=	1.36772E-01
T3=	15
T4=	57
T5=	3.0222E-04
n=	variabel
p=	4
N-table=	4

					Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	0.0825	0.001	4		-1.20	0.19		Fail	0.0825	0.0010		4	3	-0.01
23L	0.0913	0.006	4		-0.41	1.19			0.0913	0.0062		4	3	0.00
12L	0.1020	0.007	4		0.56	1.26	Fail		0.1020	0.0066		4	3	0.01
4L	0.1074	0.005	3		1.04	0.97	Fail		0.1074	0.0051		3	2	0.01
Tot.gem	0.096	0.005 ng/kg		1%-level:	1.49	(1.67)			4	0.0958	()		4	3	
Tot.std=	0.011	0.003		5%-level:	1.42	(1.5)								

RESULTS:	Mean =	**0.09579**	ng/kg			
Repeatability variance	S2r =	0.00003				
Repeatability std.	Sr =	**0.00524**	-->	5.47%	r =	0.0147
Between lab variance	S2L =	0.00011				
Reproducibility var.	S2R =	0.00014				
Reproducibility std.	SR =	**0.01182**	-->	12.34%	R =	0.0331
Remarks:	none					

Soil 3 - DL PCB 105 -- Mean PARM = 0.096 [ng/kg]

Soil 3-DL PCB 105

Sample: **Soil 3**

Element: **DL PCB 118**

**Mandel's h statistics
(Soil 3 - DL PCB 118)**
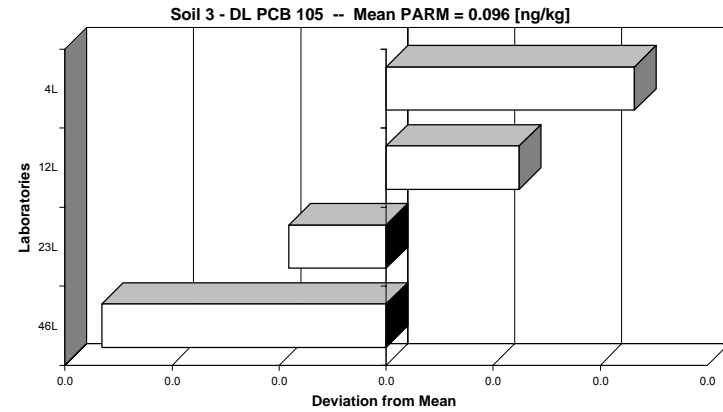
**Mandel's k statistics
(Soil 3 - DL PCB 118)**
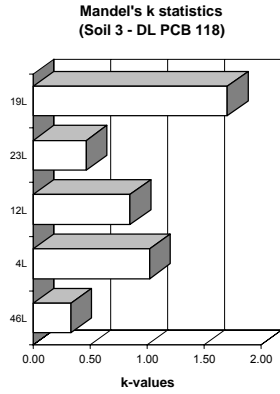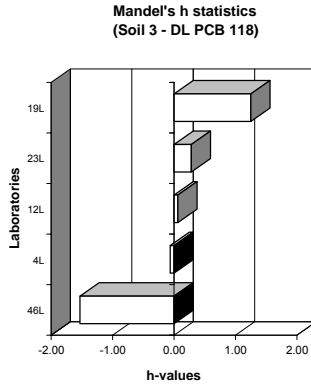
Unit: ng/kg

Mandel's k statistics (Soil 3 - DL PCB 118)
Mandel's h statistics (Soil 3 - DL PCB 118)
Soil 3 - DL PCB 118 -- Mean PARM = 0.218 [ng/kg]

	General calc.parm.
T1=	4.09500E+00
T2=	9.07083E-01
T3=	19
T4=	73
T5=	3.7118E-03
n=	variabel
p=	5
N-table=	4

				Mandel's statistics				End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
46L	0.1550	0.006	4		-1.53	0.33		Fail	0.1550	0.0058		4	3	-0.06
4L	0.2155	0.018	4		-0.07	1.02			0.2155	0.0179		4	3	0.00
12L	0.2208	0.015	4		0.06	0.85			0.2208	0.0149		4	3	0.00
23L	0.2300	0.008	4		0.28	0.47			0.2300	0.0082		4	3	0.01
19L	0.2700	0.030	3		1.25	1.70	! Fail		0.2700	0.0299		3	2	0.05
Tot.gem	0.218	0.015 ng/kg		1%-level:	1.72	(1.73)		5	0.2183	()		5	4	
Tot.std=	0.041	0.009		5%-level:	1.57	(1.53)								

RESULTS: Mean =   **0.21825**   ng/kg

Repeatability variance	**S2r =**	0.00027				
Repeatability std.	**Sr =**	**0.01628**	-->	7.46%	**r =**	0.0456
Between lab variance	**S2L =**	0.00155				
Reproducibility var.	**S2R =**	0.00181				
Reproducibility std.	**SR =**	**0.04256**	-->	19.50%	**R =**	0.1192
Remarks:	**none**					

Soil 3 - DL PCB 118 -- Mean PARM = 0.218 [ng/kg]

Soil 3-DL PCB 118
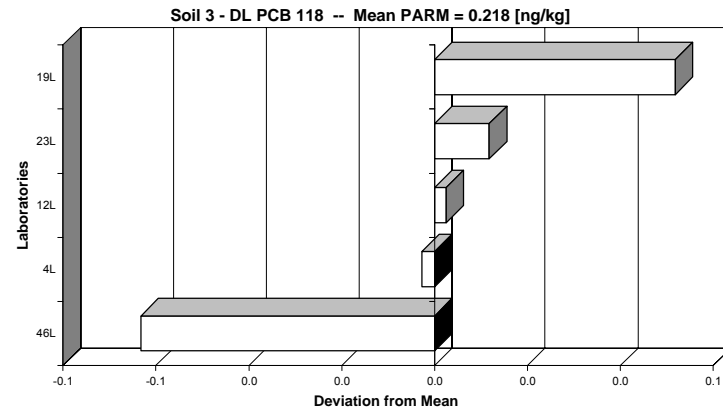
Sample: **Soil 3**

Element: **Total DL PCB**

Mandel's h statistics
(Soil 3 - Total DL PCB)

Mandel's k statistics
(Soil 3 - Total DL PCB)
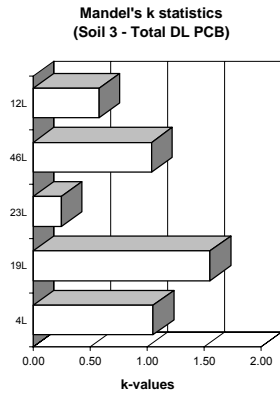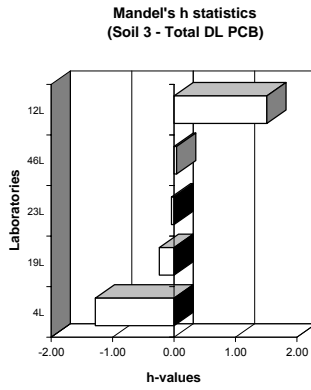
Unit: ng/kg

Mandel's k statistics (Soil 3 - Total DL PCB)
Mandel's h statistics (Soil 3 - Total DL PCB)
Soil 3 - Total DL PCB -- Mean PARM = 0.37 [ng/kg]

	General calc.parm.
T1=	7.03950E+00
T2=	2.66077E+00
T3=	19
T4=	73
T5=	2.7290E-02
n=	variabel
p=	5
N-table=	4

					Mandel's statistics			End Result:					
LAB	PARM-gem	Stdev	N	h-mark	h	k	k-mark \vX > AvST+2std	AvX < AvST-2std	PARM	Stdev	Rej.labs	N	N-1	dev_mean
4L	0.2961	0.049	4		-1.28	1.05	Fail		0.2961	0.0491		4	3	-0.07
19L	0.3562	0.072	3		-0.24	1.55			0.3562	0.0724		3	2	-0.01
23L	0.3675	0.012	4		-0.04	0.25			0.3675	0.0116		4	3	0.00
46L	0.3724	0.048	4		0.04	1.04			0.3724	0.0484		4	3	0.00
12L	0.4568	0.027	4		1.51	0.58	Fail		0.4568	0.0270		4	3	0.09
Tot.gem	0.370	0.042 ng/kg		1%-level:	1.72	(1.73)		5	0.3698	()		5	4	
Tot.std=	0.057	0.023		5%-level:	1.57	(1.53)								

RESULTS: Mean = **0.36979**   ng/kg

Repeatability variance	**S2r =**	0.00195			
Repeatability std.	**Sr =**	**0.04415**	--> 11.94%	**r =**	0.1236
Between lab variance	**S2L =**	0.00296			
Reproducibility var.	**S2R =**	0.00491			
Reproducibility std.	**SR =**	**0.07005**	--> 18.94%	**R =**	0.1962
Remarks:	none				

Soil 3 - Total DL PCB -- Mean PARM = 0.37 [ng/kg]

Soil 3-Total DL PCB
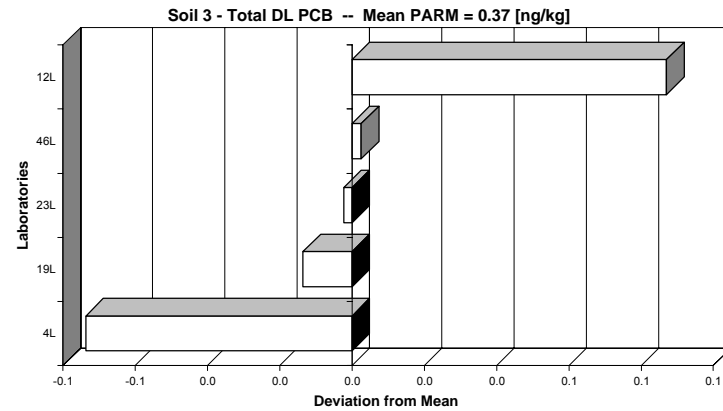
Annex 4:

Raw data submitted

| Sample: | Compost 1 | | Sample: | Compost 1 | | Sample: | Compost 1 | |
| Element: | 3,4,6,7,8-HpCDD | | Element: | 2,3,4,7,8-HxCDD | | Element: | ,3,6,7,8-HxCDD | |
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
4L	244.90		12L	0.77		4L	5.10	
4L	231.00		12L	0.84		4L	4.10	
4L	240.00		12L	0.88		4L	4.00	
4L	233.20		12L	0.67		4L	5.20	
23L	230.00		46L	0.85		12L	3.29	
23L	220.00		46L	0.48		12L	3.20	
23L	270.00		46L	0.72		12L	3.00	
23L	230.00		46L	0.69		12L	3.32	
12L	172.26					46L	3.40	
12L	172.19					46L	2.40	
12L	170.11					46L	3.10	
12L	182.67					46L	3.20	
56L	75.70							
56L	53.00							
56L	67.50							
56L	64.60							
46L	180.00							
46L	140.00							
46L	170.00							
46L	170.00							

Sample:	Compost 1			Sample:	Compost 1			Sample:	Compost 1	
Element:	,3,7,8,9-HxCDD			Element:	I,2,3,7,8-PeCD			Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]
4L	2.60			12L	0.55			12L	0.14	
4L	2.10			12L	0.48			12L	0.18	
4L	2.30			12L	0.59			12L	0.19	
4L	2.40			12L	0.66			12L	0.18	
12L	1.53			46L	0.56					
12L	1.49			46L	0.54					
12L	1.49			46L	0.47					
12L	1.67			46L	0.46					
56L	2.70									
56L	3.10									
46L	1.60									
46L	1.20									
46L	1.60									
46L	1.80									

Sample:	Compost 1	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	1502.90	
4L	1248.80	
4L	1377.20	
4L	1257.30	
23L	1700.00	
23L	1400.00	
23L	1600.00	
23L	1600.00	
12L	924.48	
12L	861.05	
12L	890.34	
12L	1149.65	
56L	564.70	
56L	453.40	
56L	569.30	
56L	453.70	
46L	880.00	
46L	710.00	
46L	620.00	
46L	850.00	

Sample:	Sludge 1	
Element:	3,4,6,7,8-HpCDD	
LAB	**PARM**	[ng/kg]
4L	236.70	
4L	232.50	
4L	246.10	
4L	231.60	
23L	160.00	
23L	160.00	
23L	180.00	
23L	210.00	
12L	170.66	
12L	170.64	
12L	162.66	
12L	162.18	
56L	170.10	
56L	115.60	
56L	109.50	
56L	178.00	
46L	190.00	
46L	200.00	
46L	210.00	
46L	210.00	
19L	174.00	
19L	208.00	
19L	150.00	
19L	208.00	

Sample:	Sludge 1	
Element:	2,3,4,7,8-HxCDD	
LAB	**PARM**	[ng/kg]
4L	4.60	
4L	4.00	
4L	4.00	
4L	3.80	
12L	2.87	
12L	2.97	
12L	3.32	
12L	3.13	
56L	16.50	
46L	3.30	
46L	3.40	
46L	3.60	
19L	3.42	
19L	3.53	
19L	3.10	
19L	3.49	

Sample:	Sludge 1	
Element:	2,3,6,7,8-HxCDD	
LAB	**PARM**	[ng/kg]
4L	10.90	
4L	11.90	
4L	11.80	
4L	10.00	
12L	7.14	
12L	7.47	
12L	7.73	
12L	7.64	
56L	6.60	
56L	5.30	
56L	8.70	
56L	9.80	
46L	8.40	
46L	7.50	
46L	8.10	
46L	8.80	
19L	7.72	
19L	7.69	
19L	5.40	
19L	6.97	

Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1	
Element:	2,3,7,8,9-HxCDD		Element:	2,3,7,8-PeCDD		Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
4L	6.00		4L	3.60		12L	0.88	
4L	7.60		4L	4.90		12L	0.91	
4L	6.70		4L	4.20		12L	0.88	
4L	5.90		4L	3.50		12L	0.91	
12L	4.04		12L	1.66		46L	0.49	
12L	3.81		12L	1.95		46L	0.54	
12L	3.80		12L	1.53		46L	0.37	
12L	4.08		12L	1.84		19L	1.16	
56L	10.50		46L	2.10		19L	0.75	
56L	8.90		46L	1.70				
56L	12.40		46L	1.70				
56L	10.80		46L	1.70				
46L	4.90		19L	1.66				
46L	4.90		19L	1.62				
46L	3.30		19L	0.94				
46L	5.20		19L	1.97				
19L	13.00							
19L	4.40							
19L	16.30							
19L	4.35							

Sample:	Sludge 1	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	1840.70	
4L	1823.30	
4L	1995.70	
4L	1923.90	
23L	1500.00	
23L	1700.00	
23L	2000.00	
23L	1800.00	
12L	1165.99	
12L	1167.30	
12L	1130.76	
12L	1132.28	
56L	1514.50	
56L	884.40	
56L	1029.50	
56L	1698.60	
46L	1400.00	
46L	1400.00	
46L	1400.00	
46L	1500.00	
19L	1230.00	
19L	1470.00	
19L	1150.00	
19L	1630.00	

Sample:	Soil 3		Sample:	Soil 3		Sample:	Soil 3	
Element:	3,4,6,7,8-HpCDD		Element:	2,3,4,7,8-HxCDD		Element:	2,3,6,7,8-HxCDD	
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
4L	7.50		12L	0.05		12L	0.29	
4L	9.30		12L	0.06		12L	0.33	
4L	7.50		12L	0.08		12L	0.34	
4L	8.00		12L	0.06		12L	0.32	
23L	10.00		19L	0.13		46L	0.29	
23L	8.50					46L	0.31	
23L	7.30					46L	0.23	
23L	12.00					19L	0.53	
12L	5.63							
12L	5.33							
12L	5.61							
12L	5.55							
56L	13.90							
56L	10.50							
56L	16.60							
56L	9.70							
46L	6.50							
46L	5.90							
46L	5.90							
46L	5.70							
19L	7.10							
19L	6.07							
19L	6.47							

Sample:	Soil 3	
Element:	2,3,7,8,9-HxCDD	
LAB	PARM	[ng/kg]
12L	0.19	
12L	0.11	
12L	0.14	
12L	0.14	
56L	2.30	
46L	0.21	
46L	0.21	
46L	0.18	

Sample:	Spoil 3	
Element:	2,3,7,8-PeCDD	
LAB	PARM	[ng/kg]
12L	0.09	
12L	0.11	
12L	0.08	
12L	0.11	

Sample:	Soil 3	
Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]
19L	0.84	
19L	0.26	

Sample:	Soil 3	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	71.30	
4L	69.30	
4L	64.90	
4L	66.60	
23L	72.00	
23L	81.00	
23L	70.00	
23L	74.00	
12L	45.30	
12L	47.03	
12L	44.04	
12L	43.64	
56L	141.50	
56L	95.70	
56L	181.50	
56L	66.00	
46L	46.00	
46L	44.00	
46L	46.00	
46L	44.00	
19L	51.00	
19L	55.20	
19L	50.30	

Sample:	Compost 1			Sample:	Compost 1			Sample:	Compost 1	
Element:	3,4,6,7,8-HpCDF			Element:	3,4,7,8,9-HpCDF			Element:	2,3,4,7,8-HxCDF	
LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]
4L	28.80			4L	5.60			4L	2.90	
4L	21.20			4L	2.70			4L	2.60	
4L	18.90			4L	2.60			4L	2.50	
4L	19.10			12L	1.16			4L	2.70	
23L	17.00			12L	1.15			12L	1.81	
23L	25.00			12L	1.22			12L	1.69	
23L	12.00			12L	1.30			12L	1.79	
23L	39.00			56L	3.79			12L	1.47	
12L	14.04			46L	1.10			56L	2.60	
12L	12.20			46L	0.52			56L	4.50	
12L	12.78			46L	1.40			56L	3.00	
12L	14.10			46L	1.20			56L	4.60	
56L	9.56							46L	1.70	
56L	7.86							46L	1.20	
56L	7.30							46L	1.90	
56L	8.83							46L	1.70	
46L	12.00									
46L	10.00									
46L	11.00									
46L	13.00									

Sample:	Compost 1			Sample:	Compost 1			Sample:	Compost 1	
Element:	2,3,6,7,8-HxCDF			Element:	2,3,7,8,9-HxCDF			Element:	1,2,3,7,8-PeCDF	
LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]		LAB	PARM	[ng/kg]
4L	2.90			12L	0.27			4L	2.30	
4L	2.50			12L	0.26			4L	2.30	
4L	2.50			12L	0.20			4L	2.20	
4L	2.50			12L	0.32			12L	1.26	
23L	10.00			46L	0.15			12L	1.29	
12L	1.76			46L	0.11			12L	1.31	
12L	1.67							12L	1.47	
12L	1.62							56L	1.60	
12L	1.69							46L	0.97	
46L	1.40							46L	0.66	
46L	0.84							46L	1.40	
46L	1.50							46L	1.10	
46L	1.60									

Sample:	Compost 1		Sample:	Compost 1		Sample:	Compost 1
Element:	,4,6,7,8-HxCDF		Element:	,3,4,7,8-PeCDF		Element:	2,3,7,8-TCDF
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
4L	3.70		4L	3.00		4L	3.50
4L	3.30		4L	2.60		4L	3.20
4L	3.20		4L	2.80		4L	3.00
4L	3.30		4L	2.70		4L	3.10
12L	1.32		12L	3.10		12L	2.85
12L	1.86		12L	1.90		12L	2.67
12L	1.84		56L	1.50		12L	2.65
12L	1.83		56L	1.50		12L	2.76
56L	2.60		56L	3.07		56L	1.70
56L	3.40		56L	1.90		56L	2.20
56L	3.50		46L	1.40		56L	2.20
46L	1.40		46L	0.87		46L	1.80
46L	1.10		46L	1.70		46L	1.30
46L	1.40		46L	1.30		46L	2.10
46L	1.50					46L	1.80

Sample:	Compost 1	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	31.40	
4L	28.50	
4L	29.30	
4L	31.30	
23L	40.00	
23L	29.00	
23L	35.00	
23L	320.00	
12L	24.79	
12L	22.72	
12L	22.62	
12L	44.17	
56L	31.14	
56L	34.57	
56L	19.14	
56L	24.67	
46L	21.00	
46L	16.00	
46L	21.00	
46L	20.00	

Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1	
Element:	3,4,6,7,8-HpCDF		Element:	,3,4,7,8,9-HpCDF		Element:	2,3,4,7,8-HxCDF	
LAB	**PARM**	[ng/kg]	**LAB**	**PARM**	[ng/kg]	**LAB**	**PARM**	[ng/kg]
4L	127.60		4L	7.20		4L	12.30	
4L	125.60		4L	8.20		4L	12.30	
4L	114.30		4L	7.50		4L	13.00	
4L	128.50		4L	7.90		4L	12.70	
23L	100.00		12L	5.43		12L	9.89	
23L	110.00		12L	5.36		12L	9.98	
23L	110.00		12L	5.41		12L	9.85	
23L	98.00		12L	5.38		12L	9.52	
12L	85.25		56L	10.75		56L	51.00	
12L	87.47		56L	8.28		56L	15.20	
12L	86.06		56L	15.22		56L	18.40	
12L	87.37		56L	9.05		56L	20.50	
56L	87.02		46L	6.10		46L	11.00	
56L	61.01		46L	6.20		46L	11.00	
56L	64.35		46L	4.10		46L	11.00	
56L	93.84		46L	9.70		46L	18.00	
46L	89.00		19L	6.25		19L	8.30	
46L	89.00		19L	6.69		19L	10.40	
46L	96.00		19L	6.84		19L	7.03	
46L	98.00		19L	6.58		19L	10.50	
19L	83.20							
19L	95.60							
19L	78.90							
19L	93.80							

Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1	
Element:	2,3,6,7,8-HxCDF		Element:	2,3,7,8,9-HxCDF		Element:	,2,3,7,8-PeCDF	
LAB	**PARM**	[ng/kg]	**LAB**	**PARM**	[ng/kg]	**LAB**	**PARM**	[ng/kg]
4L	9.70		4L	3.20		4L	7.50	
4L	9.40		4L	3.10		4L	6.60	
4L	9.50		4L	2.70		4L	6.70	
4L	10.20		4L	3.10		4L	6.90	
12L	6.51		12L	0.87		12L	4.62	
12L	6.69		12L	0.69		12L	4.45	
12L	7.12		12L	0.84		12L	4.39	
12L	6.43		12L	0.70		12L	4.61	
56L	7.40		56L	7.60		56L	8.70	
56L	3.90		56L	4.30		56L	2.10	
56L	6.40		46L	0.62		56L	3.10	
56L	6.40		19L	0.95		56L	5.50	
46L	6.60		19L	2.60		46L	4.50	
46L	6.60		19L	0.82		46L	3.60	
46L	7.20		19L	2.71		46L	5.30	
46L	7.40					19L	4.75	
19L	7.01					19L	4.82	
19L	7.41					19L	5.14	
19L	8.77					19L	4.90	
19L	7.31							

Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1	
Element:	3,4,6,7,8-HxCDF		Element:	3,4,7,8-PeCDF		Element:	2,3,7,8-TCDF	
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
4L	12.40		4L	12.70		4L	10.60	
4L	11.40		4L	11.60		4L	12.00	
4L	13.00		4L	12.90		4L	11.90	
4L	12.10		4L	12.50		4L	12.30	
12L	7.01		12L	9.60		12L	10.40	
12L	7.50		12L	6.40		12L	11.26	
12L	7.58		12L	9.10		12L	10.50	
12L	7.39		12L	14.20		12L	9.77	
56L	15.20		56L	9.65		56L	21.10	
56L	8.90		56L	6.35		56L	10.90	
56L	9.50		56L	9.14		56L	11.70	
46L	7.20		56L	14.20		56L	19.80	
46L	7.50		46L	8.40		46L	9.30	
46L	7.20		46L	8.10		46L	8.60	
46L	8.00		46L	8.70		46L	8.80	
19L	12.20		46L	13.00		46L	10.00	
19L	8.87		19L	6.24		19L	8.45	
19L	11.30		19L	10.50		19L	8.99	
19L	8.89		19L	1.10		19L	8.09	
			19L	11.30		19L	9.56	

Sample:	Sludge 1	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	237.10	
4L	248.10	
4L	261.90	
4L	272.70	
23L	160.00	
23L	200.00	
23L	200.00	
23L	160.00	
12L	188.57	
12L	191.70	
12L	180.37	
12L	180.96	
56L	164.48	
56L	129.08	
56L	144.56	
56L	207.68	
46L	170.00	
46L	170.00	
46L	160.00	
46L	190.00	
19L	224.00	
19L	267.00	
19L	160.00	
19L	259.00	

Sample:	Soil 3	
Element:	3,4,6,7,8-HpCDF	
LAB	PARM	[ng/kg]
4L	14.30	
4L	16.50	
4L	12.70	
4L	12.30	
23L	15.00	
23L	12.00	
23L	13.00	
23L	18.00	
12L	12.55	
12L	11.89	
12L	11.53	
12L	12.56	
56L	7.69	
56L	13.28	
56L	18.33	
56L	11.29	
46L	8.60	
46L	11.00	
46L	11.00	
46L	11.00	
19L	13.20	
19L	10.60	
19L	8.94	

Sample:	Soil 3	
Element:	3,4,7,8,9-HpCDF	
LAB	PARM	[ng/kg]
12L	0.21	
12L	0.15	
12L	0.18	
12L	0.18	
46L	0.27	
46L	0.25	

Sample:	Soil 3	
Element:	2,3,4,7,8-HxCDF	
LAB	PARM	[ng/kg]
12L	0.21	
12L	0.24	
12L	0.29	
56L	0.29	
56L	2.60	
46L	0.23	
46L	0.15	
46L	0.20	
19L	0.24	
19L	0.13	

Sample:	Soil 3	
Element:	2,3,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
12L	0.23	
12L	0.22	
12L	0.21	
12L	0.21	
46L	0.16	
46L	0.19	
46L	0.13	

Sample:	Soil 3	
Element:	2,3,7,8,9-HxCDF	
LAB	PARM	[ng/kg]
12L	0.04	
12L	0.03	
12L	0.09	
12L	0.05	

Sample:	Soil 3	
Element:	2,3,7,8-PeCDF	
LAB	PARM	[ng/kg]
12L	0.15	
12L	0.18	
12L	0.16	
12L	0.19	
19L	0.18	

Sample:	Soil 3		Sample:	Soil 3		Sample:	Soil 3	
Element:	3,4,6,7,8-HxCDF		Element:	,3,4,7,8-PeCDF		Element:	2,3,7,8-TCDF	
LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]	LAB	PARM	[ng/kg]
12L	0.47		56L	1.50		12L	0.34	
12L	0.38		56L	1.50		12L	0.31	
12L	0.48		56L	1.50		12L	0.37	
12L	0.50		56L	1.50		12L	0.33	
56L	2.80		19L	0.18		56L	1.20	
56L	15.20					56L	1.30	
46L	0.42					19L	0.23	
46L	0.36							
46L	0.30							
19L	0.48							
19L	0.55							

Sample:	Soil 3	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	43.80	
4L	53.20	
4L	46.40	
4L	48.90	
23L	51.00	
23L	50.00	
23L	42.00	
23L	57.00	
12L	37.42	
12L	35.26	
12L	35.06	
12L	40.62	
56L	58.23	
56L	55.23	
56L	81.70	
56L	42.36	
46L	30.00	
46L	31.00	
46L	31.00	
46L	30.00	
19L	49.80	
19L	45.90	
19L	41.40	

Unit ng/kg **DL PCB**

Sample: Compost 1 — Element: 77

LAB	PARM
4L	0.1495
4L	0.1530
4L	0.1480
4L	0.1510
23L	0.1300
23L	0.1100
23L	0.1300
23L	0.1100
12L	0.1280
12L	0.1250
12L	0.1250
12L	0.1250
46L	0.1400
46L	0.1100
46L	0.1300
46L	0.1400

Sample: Compost 1 — Element: 81

LAB	PARM
12L	0.0051
12L	0.0051
12L	0.0051
12L	0.0046
46L	0.0310
46L	0.0300
46L	0.0370
46L	0.0340

Sample: Compost 1 — Element: 105

LAB	PARM
4L	0.7074
4L	0.7893
4L	0.7619
4L	0.9047
23L	0.5600
23L	0.4500
23L	0.5400
23L	0.5000
12L	0.7330
12L	0.7080
12L	0.7080
12L	0.8010
46L	0.6700
46L	0.6100
46L	0.7700
46L	0.7100

Sample: Compost 1 — Element: 114

LAB	PARM
12L	0.0686
12L	0.0678
12L	0.0691
12L	0.1020
46L	0.0250
46L	0.0210
46L	0.0270
46L	0.0230

Sample: Compost 1 — Element: 118

LAB	PARM
4L	2.3080
4L	2.3140
4L	2.1650
4L	2.2090
23L	2.1000
23L	1.5000
23L	1.8000
23L	1.7000
12L	2.0800
12L	1.9300
12L	1.9300
12L	2.2900
46L	1.6000
46L	1.5000
46L	2.0000
46L	1.7000

Sample: Compost 1 — Element: 123

LAB	PARM
4L	0.1967
4L	0.1952
4L	0.2178
4L	0.2413
12L	0.0265
12L	0.0254
12L	0.0253
12L	0.0253
46L	0.1400
46L	0.1300
46L	0.1900
46L	0.1600

Sample: Compost 1 — Element: 126

LAB	PARM
12L	0.0311
12L	0.0315
12L	0.0317
12L	0.0310
46L	0.0440
46L	0.0330
46L	0.0470
46L	0.0380

Sample: Compost 1 — Element: 156

LAB	PARM
4L	1.1580
4L	1.0920
4L	0.8698
4L	0.9146
23L	1.1000
23L	0.5400
23L	0.6300
23L	0.6300
12L	0.8300
12L	0.8090
12L	0.8130
12L	1.5500
46L	0.7600
46L	0.7000
46L	0.9700
46L	0.7500

Sample: Compost 1 — Element: 157

LAB	PARM
4L	0.1255
4L	0.1242
4L	0.1013
4L	0.1156
23L	0.1000
23L	0.0800
23L	0.0760
23L	0.0780
12L	0.1020
12L	0.0993
12L	0.0988
12L	0.1590
46L	0.0920
46L	0.0850
46L	0.1100
46L	0.0900

Sample: Compost 1 — Element: 167

LAB	PARM
4L	2.5050
4L	0.7580
4L	0.5583
4L	0.5767
23L	0.5500
23L	0.3300
23L	0.3500
23L	0.3700
12L	0.3970
12L	0.3900
12L	0.3900
12L	0.7030
46L	0.3700
46L	0.3300
46L	0.5000
46L	0.3600

Sample: Compost 1 — Element: 169

LAB	PARM
12L	0.0038
12L	0.0038
12L	0.0041
12L	0.0039
46L	0.0026
46L	0.0028
46L	0.0038
46L	0.0028

Sample: Compost 1 — Element: 189

LAB	PARM
4L	0.2089
4L	0.2069
4L	0.1541
4L	0.1712
23L	0.2600
23L	0.1200
23L	0.1700
23L	0.1300
12L	0.1450
12L	0.1370
12L	0.1450
12L	0.3510
46L	0.1400
46L	0.1300
46L	0.2200
46L	0.1400

Unit ng/kg DL PCB

Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1
Element:	77		Element:	81		Element:	105		Element:	114		Element:	118		Element:	123
LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM
4L	7.4230		4L	0.7550		4L	23.0150		4L	1.6650		4L	37.5510		4L	5.2690
4L	6.9340		4L	0.6950		4L	21.5180		4L	1.8480		4L	38.1470		4L	4.2430
4L	7.6600		4L	0.7185		4L	20.2860		4L	1.7440		4L	37.9800		4L	4.8370
4L	7.6320		4L	0.7298		4L	24.3290		4L	1.6620		4L	35.6870		4L	4.4220
23L	5.7000		23L	0.3800		23L	16.0000		23L	0.9800		23L	31.0000		23L	0.7700
23L	5.2000		23L	0.4000		23L	16.0000		23L	1.0000		23L	32.0000		23L	0.8200
23L	5.7000		23L	0.4000		23L	16.0000		23L	0.9800		23L	31.0000		23L	0.6800
23L	5.7000		23L	0.3300		23L	17.0000		23L	1.0000		23L	33.0000		23L	0.7600
12L	6.4500		12L	0.3130		12L	18.3000		12L	1.6600		12L	33.4000		12L	0.8390
12L	6.6100		12L	0.3120		12L	18.2000		12L	1.7300		12L	33.9000		12L	0.8550
12L	6.2700		12L	0.3010		12L	18.3000		12L	1.6600		12L	33.1000		12L	0.8660
12L	6.2800		12L	0.3070		12L	18.0000		12L	1.6700		12L	32.3000		12L	0.8390
46L	6.9000		46L	0.8900		46L	19.0000		46L	1.0000		46L	29.0000		46L	1.7000
46L	7.0000		46L	0.7800		46L	19.0000		46L	1.1000		46L	31.0000		46L	1.6000
46L	7.3000		46L	0.9500		46L	23.0000		46L	1.2000		46L	55.0000		46L	5.2000
46L	6.7000		46L	0.7700		46L	19.0000		46L	1.0000		46L	30.0000		46L	1.8000
19L	7.5100		19L	0.4040		19L	24.2000		19L	1.1900		19L	28.0000		19L	0.7550
19L	7.1000		19L	0.2650		19L	14.5000		19L	1.4100		19L	27.6000		19L	0.8220
19L	6.2900		19L	0.4190					19L	0.9400		19L	28.7000		19L	0.7420
									19L	1.8400		19L	28.0000		19L	0.8820

Sample:	Sludge 1		Sample:	Sludge1		Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1		Sample:	Sludge 1
Element:	126		Element:	156		Element:	157		Element:	167		Element:	169		Element:	189
LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM
4L	0.2905		4L	7.9900		4L	1.0654		4L	4.6320		4L	0.1008		4L	1.1490
4L	0.2836		4L	7.9280		4L	1.0352		4L	5.0117		4L	0.1089		4L	1.1580
4L	0.2080		4L	8.5850		4L	1.0913		4L	4.6360		12L	0.0273		4L	0.1320
4L	0.2980		4L	8.5120		4L	1.0472		4L	6.2340		12L	0.0245		4L	1.1245
23L	0.1700		23L	6.6000		23L	0.6600		23L	3.3000		12L	0.0237		23L	0.9200
23L	0.1600		23L	6.6000		23L	0.7300		23L	3.2000		12L	0.0234		23L	0.8400
23L	0.1000		23L	6.8000		23L	0.7200		23L	3.1000		46L	0.0190		23L	0.9200
23L	0.1500		23L	6.4000		23L	0.7600		23L	3.3000		46L	0.0190		23L	0.8800
12L	0.2870		12L	7.4500		12L	0.9650		12L	3.3100		46L	0.0200		12L	1.1800
12L	0.2480		12L	7.3200		12L	0.9170		12L	3.2000		46L	0.0230		12L	1.1100
12L	0.2450		12L	6.9900		12L	0.9130		12L	3.1500		19L	0.0799		12L	1.0900
12L	0.2430		12L	6.9900		12L	0.9110		12L	3.0500		19L	0.0280		12L	1.0900
46L	0.3600		46L	6.9000		46L	0.8500		46L	3.0000					46L	1.1000
46L	0.3700		46L	7.3000		46L	0.8700		46L	3.2000					46L	1.2000
46L	0.3600		46L	28.0000		46L	2.5000		46L	12.0000					46L	4.3000
46L	0.3400		46L	7.4000		46L	0.9000		46L	3.1000					46L	1.2000
19L	0.1920		19L	6.2200		19L	0.8850		19L	3.4000					19L	0.9990
19L	0.2290		19L	5.5500		19L	0.7020		19L	2.8200					19L	0.8780
19L	0.2450		19L	7.1100		19L	0.6570		19L	3.2800					19L	0.8390
			19L	4.9500		19L	0.7600		19L	3.0800					19L	1.0200

UNIT ng/kg **DL PCB**

Sample: Element:	Soil 3 77		Sample: Element:	Soil 3 81		Sample: Element:	Soil 3 105		Sample: Element:	Soil 3 114		Sample: Element:	Soil 3 118		Sample: Element:	Soil 3 123
LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM
12L	0.0261		12L	0.0010		4L	0.1115		12L	0.0109		4L	0.1920		12L	0.0056
12L	0.0248		12L	0.0009		4L	0.1017		12L	0.0090		4L	0.2330		12L	0.0056
12L	0.0252		12L	0.0008		4L	0.1090		12L	0.0100		4L	0.2120		12L	0.0053
12L	0.0242		12L	0.0009		23L	0.0850		12L	0.0093		4L	0.2250		12L	0.0048
46L	0.0240		46L	0.0043		23L	0.0870		46L	0.0040		23L	0.2200		46L	0.0110
46L	0.0220		46L	0.0035		23L	0.0980		46L	0.0039		23L	0.2400		46L	0.0093
46L	0.0260		46L	0.0032		23L	0.0950		46L	0.0043		23L	0.2300		46L	0.0120
46L	0.0230		46L	0.0030		12L	0.1110		46L	0.0039		23L	0.2300		46L	0.0110
19L	0.0330					12L	0.0951		19L	0.1000		12L	0.2420			
19L	0.0200					12L	0.1010		19L	0.1000		12L	0.2090			
19L	0.0350					12L	0.1010					12L	0.2200			
						46L	0.0830					12L	0.2120			
						46L	0.0810					46L	0.1500			
						46L	0.0830					46L	0.1500			
						46L	0.0830					46L	0.1600			
												46L	0.1600			
												19L	0.2920			
												19L	0.2360			
												19L	0.2820			

Sample: Element:	Soil 3 126		Sample: Element:	Soil 3 156		Sample: Element:	Soil 3 157		Sample: Element:	Soil 3 167		Sample: Element:	Soil 3 169		Sample: Element:	Soil 3 189
LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM		LAB	PARM
12L	0.0020		23L	0.0460		12L	0.0104		12L	0.0224		12L	0.0005		12L	0.0083
12L	0.0019		23L	0.0490		12L	0.0083		12L	0.0243		12L	0.0005		12L	0.0095
12L	0.0020		23L	0.0470		12L	0.0087		12L	0.0207		12L	0.0005		12L	0.0074
12L	0.0021		23L	0.0430		12L	0.0090		12L	0.0216		12L	0.0005		12L	0.0086
46L	0.0037		12L	0.0560		46L	0.0065		46L	0.0170		46L	0.0004		46L	0.0065
46L	0.0022		12L	0.0466		46L	0.0069		46L	0.0160		46L	0.0003		46L	0.0066
46L	0.0031		12L	0.0496		46L	0.0069		46L	0.0180		46L	0.0826		46L	0.0076
46L	0.0019		12L	0.0506		46L	0.0064		46L	0.0160					46L	0.0064
19L	0.0016		46L	0.0390												
19L	0.0041		46L	0.0420												
			46L	0.0380												
			46L	0.0370												

Abstract
Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. In the context of this standardization project, a series of draft technical specification were designed upon an extensive desk study, fine-tuned after expert consultations and finally validated in international intercomparisons exercise.
This report summarises the work performed within the validation study of the draft standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludge and treated bio-waste using high resolution mass selective detection (HR GC-MS). It further explains the underlying statistical concept for the calculation of reproducibility and repeatability from intercomparisons data. In addition all single values, results of the statistical evaluation as well as background information on the validation materials used are described and explained.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

JRC
EUROPEAN COMMISSION

9 789279 071225

Publications Office
Publications.eu.int