



Project HORIZONTAL Validation Report on pH

Validation of a horizontal standard for the determination of pH in soils, sludges
and treated biowaste
in a European Intercomparison Exercise

E. Sobiecka, H. van der Sloot, I. Nilsson, P. Jennische, B. M. Gawlik



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Project HORIZONTAL Validation Report

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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 specifications 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 pH in soils, sludge and treated bio-waste. 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 | JRC | Joint Research Centre |
| CAS | Chemical Abstracts System | MILC | Measure Interlaboratory Comparison |
| CEN | Comité Européen de Normalisation | p | Number of labs |
| DG | Directorate General | r | Repeatability limit |
| ECN | Energy Research Centre for the Netherlands | R | Reproducibility limit |
| EU | European Union | s_r | Repeatability standard deviation |
| IES | Institute for Environment and Sustainability | s_R | Reproducibility standard deviation |
| IT | Information Technology | TC | Technical Committee |
| ISO | International Organization for Standardisation | | |

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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:

- The revision of the Sewage Sludge Directive 86/278/EEC.
- The Directive on the biological treatment of biodegradable waste.
- 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 pH 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:

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

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

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 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 pH draft standard.

Each laboratory received two bottles of each material and was requested to perform 6 independent analyses per material¹ (3 per bottle) using the respective draft standard methods. The 6 analyses per material should be carried out under repeatability conditions (i.e. same operator², 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 and 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 pH

1.2.1 Samples dispatched for the validation of pH

After a preliminary rough screening, the following materials were used for the validation round of pH.

- Compost 1 A pollutant loaded compost material from Vienna
- Compost 2 A pollutant loaded compost materia from Germany
- Sewage Sludge 1 A mixed sewage sludge from Essen, Germany
- Sewage Sludge 2 A mixed municipal WWTP sludge from North Rhine Westphalia, Germany
- Soil 4 A sludge amended soil from Hohenheim, Germany
- Soil 5 An agricultural soil from Reading, UK

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.

1.2.2 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/pH_standard_for_validation.pdf

1.2.3 Analytical program

Of each of the six materials 2 bottles had to be analyzed and each bottle had to analyze independently three times. 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.

1.2.4 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 pH to the 31st of January 2007. 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.

1.2.5 Participants

The following table lists the participating organizations and entities in the validation exercise for the horizontal pH standard;

- Austria
 - barbara - Engineering, Consulting, Research & Service GmbH
 - Federal Research and Training Centre for Forests, Natural Hazards and Landscape
 - NUA Umweltanalytik GmbH
 - Österreichische Agentur für Gesundheit und Ernährungssicherheit
 - Universität für Bodenkultur Wien
 - University of Natural resources and Applied Life Sciences
- Czech Republic
 - Central Institute for Supervising and Testing in Agriculture (UKZUZ)
- Finland
 - Finnish Food safety Authority Evira
 - VTT
- France
 - INERIS
 - INRA - Laboratoire d'Analyse des Sols
 - Institut Pasteur de Lille - Dep. Eaux et Environnement
 - SAS Laboratoire
- Germany
 - Umweltbundesamt
 - Fachhochschule Weihenstephan, Forschungsanstalt für Gartenbau
- Hungary
 - Chemical Laboratory Mertcontrol Ltd
- Italy
 - C.R.A. - Istituto Sperimentale per la Nutrizione delle Piante
 - European Commission DG JRC IES
- Sweden
 - ALCONTROL AB
- The Netherlands
 - ALCONTROL BV,
 - Analytico Milieu B. V.

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: $r_{\text{test}} = f \cdot \sqrt{2} \cdot s_{r,\text{test}}$ 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 pH in treated biowaste, sludge and soil. All concentrations are expressed pH unit.

| Matrix | Parameter | Mean | sr | sR | r | R | p | Outliers | Total number of data | No of LOD |
|---------------|------------------|-------------|-----------|-----------|----------|----------|----------|-----------------|-----------------------------|------------------|
| Sludge 1 | pH CaCl2 | 6.54 | 0.24% | 0.96% | 0.044 | 0.176 | 7 | 3 | 34 | 0 |
| Sludge 2 | pH CaCl2 | 7.09 | 0.39% | 3.17% | 0.077 | 0.629 | 11 | 0 | 57 | 0 |
| Compost 1 | pH CaCl2 | 7.68 | 0.26% | 1.90% | 0.057 | 0.409 | 13 | 0 | 64 | 0 |
| Compost 2 | pH CaCl2 | 7.68 | 0.24% | 2.36% | 0.051 | 0.506 | 9 | 3 | 46 | 0 |
| Soil 4 | pH CaCl2 | 6.48 | 0.60% | 2.27% | 0.109 | 0.412 | 12 | 0 | 58 | 0 |
| Soil 5 | pH CaCl2 | 6.59 | 0.47% | 1.20% | 0.087 | 0.221 | 8 | 1 | 43 | 0 |
| Sludge 1 | pH aq | 6.71 | 0.53% | 1.11% | 0.100 | 0.208 | 12 | 2 | 58 | 0 |
| Sludge 2 | pH aq | 7.51 | 0.33% | 1.43% | 0.069 | 0.301 | 13 | 2 | 61 | 0 |
| Compost 1 | pH aq | 8.13 | 0.50% | 0.94% | 0.115 | 0.215 | 14 | 2 | 70 | 0 |
| Compost 2 | pH aq | 8.37 | 0.49% | 1.73% | 0.115 | 0.405 | 13 | 3 | 67 | 0 |
| Soil 4 | pH aq | 7.05 | 0.59% | 2.58% | 0.117 | 0.509 | 14 | 1 | 66 | 0 |
| Soil 5 | pH aq | 7.10 | 0.32% | 2.10% | 0.064 | 0.418 | 10 | 3 | 47 | 0 |

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.

1.4 Annexes

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

Annex 2: Report on the validation materials used

Annex 3: Statistical calculations

Annex 4: Data submitted

Annex 1:

Model questionnaire to be filled by the participating laboratories

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)

| <i>Parameter</i> | <i>Concentration</i> |
|------------------|----------------------|
| 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

| | <i>Sewage sludge A Dusseldorf</i> | <i>sewage sludge D Dusseldorf</i> |
|-------------------------|---|---|
| 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)

| | <i>DiBP</i> | <i>DBP</i> | <i>DCHP</i> | <i>DEHP</i> | <i>Water</i> |
|--------------|-------------|------------|-------------|-------------|--------------|
| | 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)

| | <i>Sludge 2</i> (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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|--------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| | 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).

| <i>Sample</i> | <i>SiO2 (%)</i> | <i>Al2O3 (%)</i> | <i>CaO (%)</i> | <i>K2O (%)</i> | <i>Fe2O3 (%)</i> | <i>MgO (%)</i> | <i>TiO2 (PPM)</i> | <i>S (PPM)</i> | <i>P2O5 (PPM)</i> |
|---------------|-----------------|------------------|----------------|----------------|------------------|----------------|-------------------|----------------|-------------------|
| 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 |

| <i>Sample</i> | <i>Na2O (%)</i> | <i>Cl (PPM)</i> | <i>Pb (PPM)</i> | <i>Zn (PPM)</i> | <i>Cu (PPM)</i> | <i>Ni (PPM)</i> | <i>Mn (PPM)</i> | <i>Cr (PPM)</i> |
|---------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 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)

| | <i>DiBP</i> | <i>DBP</i> | <i>DCHP</i> | <i>DEHP</i> | <i>Water</i> |
|---------------------|-------------|------------|-------------|-------------|--------------|
| | 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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| | 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)

| <i>Parameter</i> | <i>Concentration</i> |
|-------------------|----------------------|
| 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 |

| <i>Parameter</i> | <i>Concentration</i> |
|-------------------------|----------------------|
| 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)

| | <i>Soil 3 (Reading)</i> |
|---------------|-----------------------------|
| 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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| 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).

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

| <i>Sample</i> | <i>Na2O (%)</i> | <i>Cl (PPM)</i> | <i>Pb (PPM)</i> | <i>Zn (PPM)</i> | <i>Cu (PPM)</i> | <i>Ni (PPM)</i> | <i>Mn (PPM)</i> | <i>Cr (PPM)</i> |
|------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 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).

| <i>Sample</i> | <i>Hg µg/g</i> |
|------------------|----------------|
| 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 intercomparison. 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 |
| NO ₃ -N | mg/kg F.M. | Fresh sample, <10mm | 3.5 |
| NH ₄ -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 |

| <i>PAH</i> | <i>Unit</i> | <i>Result</i> |
|-------------------------|-------------|---------------|
| 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

| <i>Parameter</i> | | | |
|------------------|-----------------|----------|------|
| Dioxine | TEQ (ITEF) | ng/kg DM | 7.3 |
| PCB | TEQ (WHO) | ng/kg DM | 3.5 |
| | Σ Ballschmitter | mg/kg DM | 0.05 |

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

| <i>Parameter</i> | <i>Result in ng/g</i> |
|-------------------------|-----------------------|
| PCB | |
| 28 | 2 |
| 52 | 2 |
| 101 | 4 |
| 118 | 3 |
| 153 | 10 |
| 105 | 1 |
| 138 | 8 |
| 156 | 1 |
| 180 | 5 |
| 170 | <1 |
| PAH | |
| Naphtalene | <10 |
| Acenaphthylene | <10 |
| Acenaphthene | <10 |
| Fluorene | <10 |
| Phenantrene | <10 |
| Anthracene | 26 |
| Fluoranthene | 611 |
| Pyrene | 510 |
| Benzo(a)anthracene | 888 |
| Chrysene | 957 |
| Benzo(b)fluoranthene | 1531 |
| Benzo(k)fluoranthene | 547 |
| Benzo(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 |

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 m².

Table 23 – Analytical data on Pavia soil

| <i>Parameter</i> | <i>Result</i> |
|---------------------|---------------|
| 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)

| | <i>DiBP</i> | <i>DBP</i> | <i>DCHP</i> | <i>DEHP</i> | <i>Water</i> |
|----------------|-------------|------------|-------------|-------------|--------------|
| | 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 microwave 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 | SiO ₂ (%) | Al ₂ O ₃ (%) | CaO (%) | K ₂ O (%) | Fe ₂ O ₃ (%) | MgO (%) | TiO ₂ (PPM) | S (PPM) | P ₂ O ₅ (PPM) |
|----------------|----------------------|------------------------------------|---------|----------------------|------------------------------------|---------|------------------------|---------|-------------------------------------|
| Soil 5 (Pavia) | 69.39 | 12.9 | 1.45 | 2.24 | 4.25 | 1.16 | 6118 | 255 | 1789 |

| Sample | Na ₂ O (%) | 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)

| | <i>DiBP</i> | <i>DBP</i> | <i>DCHP</i> | <i>DEHP</i> | <i>Water</i> |
|--------------------|-------------|------------|-------------|-------------|--------------|
| | 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)

| | <i>Soil 2</i> (<i>Lleida T.</i>) |
|---------------|---------------------------------------|
| 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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|--------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| | 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).

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

| <i>Sample</i> | <i>Na2O (%)</i> | <i>Cl (PPM)</i> | <i>Pb (PPM)</i> | <i>Zn (PPM)</i> | <i>Cu (PPM)</i> | <i>Ni (PPM)</i> | <i>Mn (PPM)</i> | <i>Cr (PPM)</i> |
|--------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 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).

| <i>Sample</i> | <i>Hg µg/g</i> |
|--------------------|----------------|
| 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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|----------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| | 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).

| <i>Sample</i> | <i>SiO2 (%)</i> | <i>Al2O3 (%)</i> | <i>CaO (%)</i> | <i>K2O (%)</i> | <i>Fe2O3 (%)</i> | <i>MgO (%)</i> | <i>TiO2 (PPM)</i> | <i>S (PPM)</i> | <i>P2O5 (PPM)</i> |
|----------------|-----------------|------------------|----------------|----------------|------------------|----------------|-------------------|----------------|-------------------|
| Soil 4 (Essen) | 79.47 | 4.42 | 0.85 | 0.6 | 0.86 | 0.07 | 2163 | 189 | 2019 |

| <i>Sample</i> | <i>Na2O (%)</i> | <i>Cl (PPM)</i> | <i>Pb (PPM)</i> | <i>Zn (PPM)</i> | <i>Cu (PPM)</i> | <i>Ni (PPM)</i> | <i>Mn (PPM)</i> | <i>Cr (PPM)</i> |
|----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 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).

| <i>Sample</i> | <i>Hg µg/g</i> |
|----------------|----------------|
| 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, where 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)

| | <i>DiBP</i> | <i>DBP</i> | <i>DCHP</i> | <i>DEHP</i> | <i>Water</i> |
|--------------------|-------------|------------|-------------|-------------|--------------|
| | 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)

| | <i>Soil 1</i> <i>(Stuttgart)</i> |
|---------------|-------------------------------------|
| 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!

| | <i>Cd</i> | <i>Co</i> | <i>Cr</i> | <i>Cu</i> | <i>Mn</i> | <i>Ni</i> | <i>Pb</i> | <i>Sb</i> | <i>Tl</i> | <i>V</i> | <i>Zn</i> |
|--------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|----------|-----------|
| | 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).

| <i>Sample</i> | <i>SiO2 (%)</i> | <i>Al2O3 (%)</i> | <i>CaO (%)</i> | <i>K2O (%)</i> | <i>Fe2O3 (%)</i> | <i>MgO (%)</i> | <i>TiO2 (PPM)</i> | <i>S (PPM)</i> | <i>P2O5 (PPM)</i> |
|--------------------|-----------------|------------------|----------------|----------------|------------------|----------------|-------------------|----------------|-------------------|
| Soil 1 (Stuttgart) | 71.94 | 10.06 | 1.33 | 1.86 | 3.66 | 0.88 | 7874 | 275 | 3571 |

| <i>Sample</i> | <i>Na2O (%)</i> | <i>Cl (PPM)</i> | <i>Pb (PPM)</i> | <i>Zn (PPM)</i> | <i>Cu (PPM)</i> | <i>Ni (PPM)</i> | <i>Mn (PPM)</i> | <i>Cr (PPM)</i> |
|--------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Soil 1 (Stuttgart) | 1.23 | 50 | 47 | 212 | 85 | 69 | 991 | 129 |

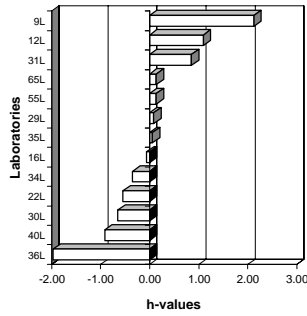
Table 44 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

| <i>Sample</i> | <i>Hg µg/g</i> |
|--------------------|----------------|
| Soil 1 (Stuttgart) | 1.77 |

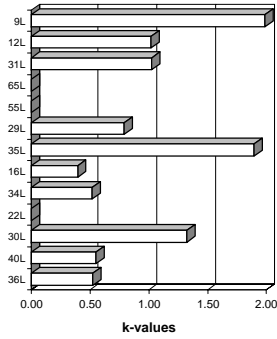
Annex 3:
Statistical calculations

Sample: **Compost 1**
 Element: **pH CaCl2**

Mandel's h statistics
 (Compost 1 - pH CaCl2)



Mandel's k statistics
 (Compost 1 - pH CaCl2)



Unit: pH unit

Mandel's k statistics (Compost 1 - pH CaCl2)
 Mandel's h statistics (Compost 1 - pH CaCl2)
 Compost 1 - pH CaCl2 -- Mean PARM = 7.68 [pH unit]

General calc.parm.
 T1= 4.92720E+02
 T2= 3.79456E+03
 T3= 64
 T4= 338
 T5= 2.1112E-02
 n= variabel
 p= 13
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | | End Result: | | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|--------|---------------------|--------|-------------------------------|-----------------|--------|--------|------|-------|----------|-------|-----|----------|
| | | | | | h | k | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | | | | | | |
| 36L | 7.3900 | 0.010 | 3 | | -1.96 | 0.53 | | Fail | 7.3900 | 0.0100 | | 3 | 2 | -0.29 | | |
| 40L | 7.5450 | 0.010 | 6 | I | -0.92 | 0.55 | | Fail | 7.5450 | 0.0105 | | 6 | 5 | -0.14 | | |
| 30L | 7.5850 | 0.025 | 4 | | -0.64 | 1.32 | | Fail | 7.5850 | 0.0252 | | 4 | 3 | -0.10 | | |
| 22L | 7.6000 | | 2 | | -0.54 | | | Fail | 7.6000 | | | 2 | 1 | -0.08 | | |
| 34L | 7.6283 | 0.010 | 6 | | -0.35 | 0.52 | | Fail | 7.6283 | 0.0098 | | 6 | 5 | -0.05 | | |
| 16L | 7.6717 | 0.008 | 6 | | -0.06 | 0.40 | | Fail | 7.6717 | 0.0075 | | 6 | 5 | -0.01 | | |
| 35L | 7.6883 | 0.036 | 6 | | 0.06 | 1.89 | II | | 7.6883 | 0.0360 | | 6 | 5 | 0.01 | | |
| 29L | 7.6925 | 0.015 | 4 | | 0.08 | 0.79 | | | 7.6925 | 0.0150 | | 4 | 3 | 0.01 | | |
| 55L | 7.7000 | | 6 | | 0.13 | | | | 7.7000 | | | 6 | 5 | 0.02 | | |
| 65L | 7.7000 | | 4 | | 0.13 | | | | 7.7000 | | | 4 | 3 | 0.02 | | |
| 31L | 7.8060 | 0.019 | 5 | | 0.85 | 1.03 | | Fail | 7.8060 | 0.0195 | | 5 | 4 | 0.13 | | |
| 12L | 7.8417 | 0.019 | 6 | | 1.09 | 1.02 | | Fail | 7.8417 | 0.0194 | | 6 | 5 | 0.16 | | |
| 9L | 7.9933 | 0.038 | 6 | | 2.12 | 1.99 | II | Fail | 7.9933 | 0.0378 | | 6 | 5 | 0.31 | | |
| Tot.gem | 7.680 | 0.015 | | | 1%-level: 2.27 | (1.76) | | | 13 | 7.6801 | 0 | 13 | 12 | | | |
| Tot.std= | 0.148 | 0.013 | | | 5%-level: 1.84 | (1.51) | | | | | | | | | | |

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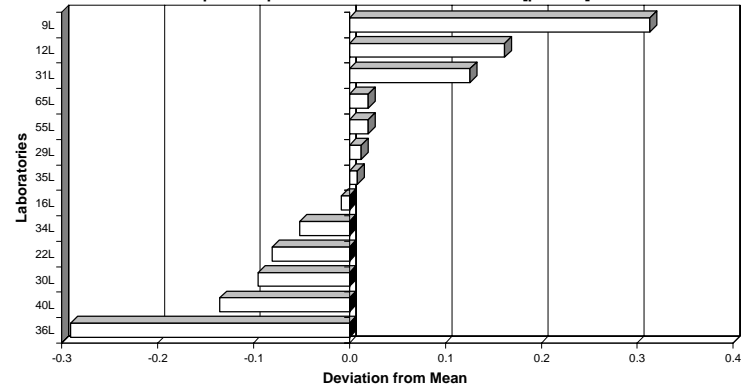
RESULTS: Mean = 7.68014 pH unit

Repeatability variance S2r = 0.00041
 Repeatability std. Sr = 0.02035 --> 0.26% r = 0.0570

Between lab variance S2L = 0.02094
 Reproducibility var. S2R = 0.02136
 Reproducibility std. SR = 0.14614 --> 1.90% R = 0.4092

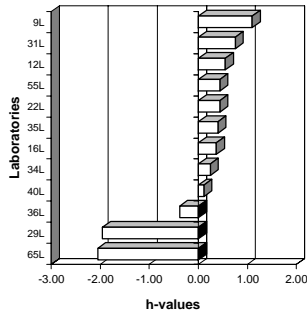
Remarks: none

Compost 1 - pH CaCl2 -- Mean PARM = 7.68 [pH unit]

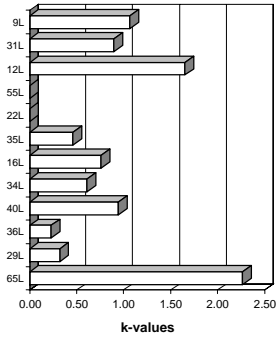


Sample: **Compost 2**
 Element: **pH CaCl2**

Mandel's h statistics
 (Compost 2 - pH CaCl2)



Mandel's k statistics
 (Compost 2 - pH CaCl2)



Unit: pH unit

Mandel's k statistics (Compost 2 - pH CaCl2)
 Mandel's h statistics (Compost 2 - pH CaCl2)
 Compost 2 - pH CaCl2 -- Mean PARM = 7.68 [pH unit]

General calc.parm.
 T1= 3.53890E+02
 T2= 2.72388E+03
 T3= 46
 T4= 254
 T5= 1.2480E-02
 n= variabel
 p= 9
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | h | k | k-mark 1xX > AvST+2std | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|---------------|---|--------|-------|--------|------------------------|-----------------|--------|--------|---------------|---|-----|----------|
| 65L | 6.5333 | 0.058 | 3 | I | -2.05 | 2.27 | !! | Fail | - | ,65L | - | - | - | -1.14 |
| 29L | 6.5800 | 0.008 | 4 | I | -1.95 | 0.32 | | Fail | - | ,29L | - | - | - | -1.10 |
| 36L | 7.3167 | 0.006 | 3 | | -0.38 | 0.23 | | Fail | 7.3167 | 0.0058 | | 3 | 2 | -0.36 |
| 40L | 7.5483 | 0.024 | 6 | | 0.11 | 0.94 | | Fail | 7.5483 | 0.0240 | | 6 | 5 | -0.13 |
| 34L | 7.6100 | 0.015 | 6 | | 0.24 | 0.61 | | Fail | 7.6100 | 0.0155 | | 6 | 5 | -0.07 |
| 16L | 7.6683 | 0.019 | 6 | | 0.37 | 0.76 | | Fail | 7.6683 | 0.0194 | | 6 | 5 | -0.01 |
| 35L | 7.6817 | 0.012 | 6 | | 0.40 | 0.46 | | Fail | 7.6817 | 0.0117 | | 6 | 5 | 0.01 |
| 22L | 7.7000 | | 2 | | 0.44 | | | Fail | 7.7000 | | | 2 | 1 | 0.02 |
| 55L | 7.7000 | | 6 | | 0.44 | | | Fail | 7.7000 | | | 6 | 5 | 0.02 |
| 12L | 7.7517 | 0.042 | 6 | | 0.55 | 1.65 | I | Fail | - | ,12L | - | - | - | 0.08 |
| 31L | 7.8480 | 0.023 | 5 | | 0.75 | 0.89 | | Fail | 7.8480 | 0.0228 | | 5 | 4 | 0.17 |
| 9L | 8.0083 | 0.027 | 6 | | 1.09 | 1.06 | | Fail | 8.0083 | 0.0271 | | 6 | 5 | 0.33 |
| Tot.gem | 7.496 | 0.020 pH unit | | | 2.25 | (1.75) | | | 9 | 7.6757 | (12L,29L,65L) | 9 | 8 | |
| Tot.std= | 0.468 | 0.017 | | | 1.83 | (1.51) | | | 3 | | | | | |

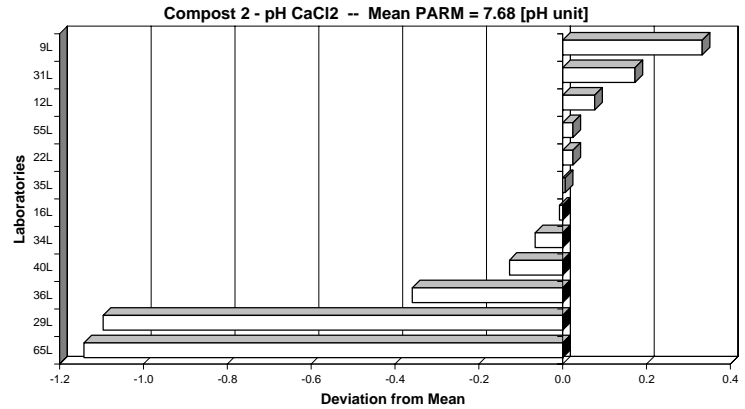
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RESULTS: Mean = 7.67570 pH unit

Repeatability variance S2r = 0.00034
 Repeatability std. Sr = 0.01837 --> 0.24% r = 0.0514

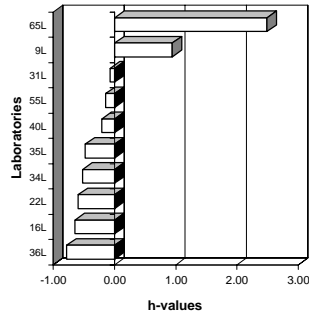
Between lab variance S2L = 0.03238
 Reproducibility var. S2R = 0.03272
 Reproducibility std. SR = 0.18088 --> 2.36% R = 0.5065

Remarks: 3 Labs rejected! (12L,29L,65L)

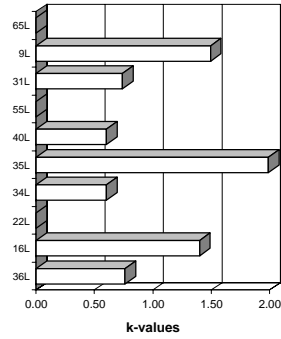


Sample: **Sludge 1**
 Element: **pH CaCl2**

**Mandel's h statistics
 (Sludge 1 - pH CaCl2)**



**Mandel's k statistics
 (Sludge 1 - pH CaCl2)**



Unit: pH unit

Mandel's k statistics (Sludge 1 - pH CaCl2)
Mandel's h statistics (Sludge 1 - pH CaCl2)
 Sludge 1 - pH CaCl2 -- Mean PARM = 6.54 [pH unit]

General calc.parm.
 T1= 2.22600E+02
 T2= 1.45748E+03
 T3= 34
 T4= 182
 T5= 6.7467E-03
 n= variabel 7
 p= 7
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | | End Result: | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|---------|----------|---------------|---|-----------|---------------------|--------|-------------------------------|-----------------|---|--------|--------|-----------------|---|-----|----------|
| | | | | | h | k | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | | | | | | | |
| 36L | 6.4567 | 0.015 | 3 | | -0.78 | 0.76 | | Fail | | 6.4567 | 0.0153 | | 3 | 2 | -0.08 |
| 16L | 6.4867 | 0.028 | 6 | | -0.65 | 1.40 | | Fail | | 6.4867 | 0.0280 | | 6 | 5 | -0.05 |
| 22L | 6.5000 | | 2 | | -0.59 | | | Fail | | 6.5000 | | | 2 | 1 | -0.04 |
| 34L | 6.5167 | 0.012 | 6 | | -0.51 | 0.60 | | Fail | | 6.5167 | 0.0121 | | 6 | 5 | -0.02 |
| 35L | 6.5250 | 0.040 | 6 | | -0.48 | 1.99 | II | Fail | | | | .35L | | | -0.01 |
| 40L | 6.5967 | 0.012 | 6 | | -0.21 | 0.60 | | Fail | | 6.5967 | 0.0121 | | 6 | 5 | 0.05 |
| 55L | 6.6000 | 0.000 | 6 | | -0.15 | 0.00 | | Fail | | 6.6000 | 0.0000 | | 6 | 5 | 0.06 |
| 31L | 6.6180 | 0.015 | 5 | | -0.07 | 0.74 | | Fail | | 6.6180 | 0.0148 | | 5 | 4 | 0.08 |
| 9L | 6.8483 | 0.030 | 6 | | 0.94 | 1.49 | I | Fail | | | | .9L | | | 0.31 |
| 65L | 7.2000 | | 2 | II | 2.49 | | | Fail | | | | .65L | | | 0.66 |
| Tot.gem | 6.634 | 0.015 pH unit | | 1%-level: | 2.18 | (1.74) | | | 7 | 6.5378 | | (.35L,.65L,.9L) | 7 | 6 | |
| Tot.std | 0.228 | 0.014 | | 5%-level: | 1.80 | (1.5) | | | 3 | | | | | | |

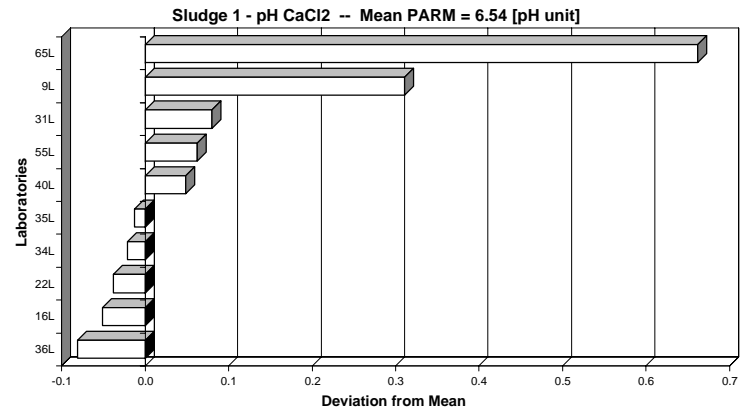
48

RESULTS: Mean = **6.53781** pH unit

Repeatability variance **S2r = 0.00025**
 Repeatability std. **Sr = 0.01581** --> 0.24% **r = 0.0443**

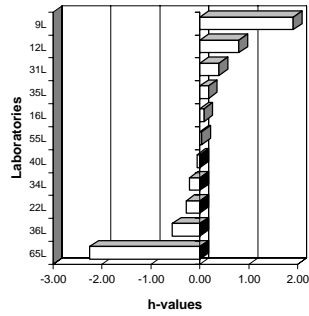
Between lab variance **S2L = 0.00371**
 Reproducibility var. **S2R = 0.00396**
 Reproducibility std. **SR = 0.06292** --> 0.96% **R = 0.1762**

Remarks: **3 Labs rejected! (35L,.65L,.9L)**

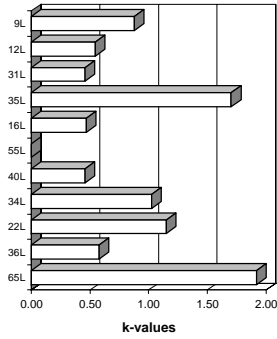


Sample: **Sludge 2**
 Element: **pH CaCl2**

Mandel's h statistics
 (Sludge 2 - pH CaCl2)



Mandel's k statistics
 (Sludge 2 - pH CaCl2)



Unit: pH unit

Mandel's k statistics (Sludge 2 - pH CaCl2)
 Mandel's h statistics (Sludge 2 - pH CaCl2)
 Sludge 2 - pH CaCl2 -- Mean PARM = 7.09 [pH unit]

General calc.parm.
 T1= 4.06450E+02
 T2= 2.90084E+03
 T3= 57
 T4= 315
 T5= 3.5233E-02
 n= variabel
 p= 11
 N-table= 5

| Mandel's statistics | | | | | | | | | | End Result: | | | | |
|---------------------|----------|---------------|---|--------|-----------|-------------|-------------------------------|-----------------|--------|-------------|----------|----|-----|----------|
| LAB | PARM-gem | Stdev | N | h-mark | h | k | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
| 65L | 6.5333 | 0.058 | 3 | II | -2.25 | 1.92 | I | Fail | 6.5333 | 0.0577 | | 3 | 2 | -0.56 |
| 36L | 6.9500 | 0.017 | 3 | | -0.57 | 0.58 | | Fail | 6.9500 | 0.0173 | | 3 | 2 | -0.14 |
| 22L | 7.0200 | 0.035 | 3 | | -0.28 | 1.15 | | Fail | 7.0200 | 0.0346 | | 3 | 2 | -0.07 |
| 34L | 7.0367 | 0.031 | 6 | | -0.22 | 1.02 | | Fail | 7.0367 | 0.0308 | | 6 | 5 | -0.05 |
| 40L | 7.0750 | 0.014 | 6 | | -0.06 | 0.46 | | | 7.0750 | 0.0138 | | 6 | 5 | -0.02 |
| 55L | 7.1000 | 0.000 | 6 | | 0.04 | 0.00 | | | 7.1000 | 0.0000 | | 6 | 5 | 0.01 |
| 16L | 7.1100 | 0.014 | 6 | | 0.08 | 0.47 | | | 7.1100 | 0.0141 | | 6 | 5 | 0.02 |
| 35L | 7.1333 | 0.051 | 6 | | 0.17 | 1.70 | II | Fail | 7.1333 | 0.0513 | | 6 | 5 | 0.04 |
| 31L | 7.1867 | 0.014 | 6 | | 0.39 | 0.45 | | Fail | 7.1867 | 0.0137 | | 6 | 5 | 0.10 |
| 12L | 7.2867 | 0.016 | 6 | | 0.79 | 0.54 | | Fail | 7.2867 | 0.0163 | | 6 | 5 | 0.20 |
| 9L | 7.5617 | 0.026 | 6 | I | 1.91 | 0.88 | | Fail | 7.5617 | 0.0264 | | 6 | 5 | 0.47 |
| Tot.gem | 7.090 | 0.025 pH unit | | | 1%-level: | 2.22 (1.74) | | | 11 | 7.0903 | () | 11 | 10 | |
| Tot.std= | 0.247 | 0.017 | | | 5%-level: | 1.82 (1.51) | | | | | | | | |

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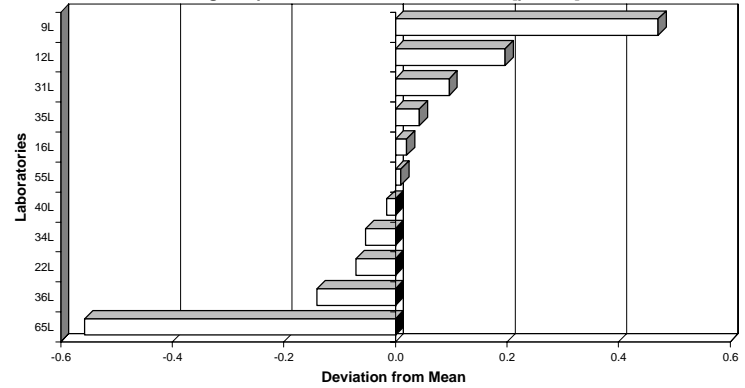
RESULTS: Mean = **7.09030** pH unit

Repeatability variance S2r = 0.00077
 Repeatability std. Sr = **0.02768** --> 0.39% r = 0.0775

Between lab variance S2L = 0.04967
 Reproducibility var. S2R = 0.05044
 Reproducibility std. SR = **0.22458** --> 3.17% R = 0.6288

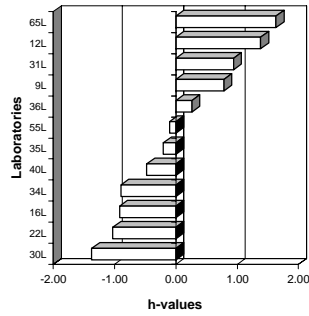
Remarks: none

Sludge 2 - pH CaCl2 -- Mean PARM = 7.09 [pH unit]

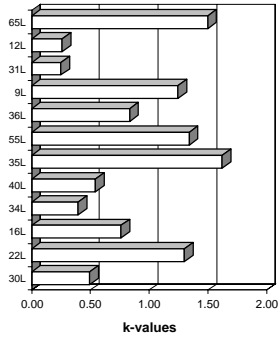


Sample: Soil 4
 Element: pH CaCl2

Mandel's h statistics
 (Soil 4 - pH CaCl2)



Mandel's k statistics
 (Soil 4 - pH CaCl2)



Unit: pH unit

Mandel's k statistics (Soil 4 - pH CaCl2)
 Mandel's h statistics (Soil 4 - pH CaCl2)
 Soil 4 - pH CaCl2 -- Mean PARM = 6.48 [pH unit]

General calc.parm.
 T1= 3.75160E+02
 T2= 2.42772E+03
 T3= 58
 T4= 298
 T5= 6.9867E-02
 n= variabel
 p= 12
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|---------------|---|-----------|---------------------|--------|-------------------------------|-----------------|--------|-------|----------|----|-------|----------|
| | | | | | h | k | | | | | | | | |
| 30L | 6.2725 | 0.019 | 4 | -1.37 | 0.49 | | Fail | 6.2725 | 0.0189 | | 4 | 3 | -0.21 | |
| 22L | 6.3250 | 0.050 | 4 | -1.03 | 1.30 | | Fail | 6.3250 | 0.0500 | | 4 | 3 | -0.16 | |
| 16L | 6.3417 | 0.029 | 6 | -0.92 | 0.76 | | Fail | 6.3417 | 0.0293 | | 6 | 5 | -0.14 | |
| 34L | 6.3450 | 0.015 | 6 | -0.90 | 0.39 | | Fail | 6.3450 | 0.0152 | | 6 | 5 | -0.14 | |
| 40L | 6.4100 | 0.021 | 6 | -0.47 | 0.54 | | Fail | 6.4100 | 0.0210 | | 6 | 5 | -0.07 | |
| 35L | 6.4517 | 0.062 | 6 | -0.20 | 1.62 | I | Fail | 6.4517 | 0.0624 | | 6 | 5 | -0.03 | |
| 55L | 6.4667 | 0.052 | 6 | -0.11 | 1.34 | | | 6.4667 | 0.0516 | | 6 | 5 | -0.02 | |
| 36L | 6.5233 | 0.032 | 3 | 0.26 | 0.83 | | Fail | 6.5233 | 0.0321 | | 3 | 2 | 0.04 | |
| 9L | 6.6033 | 0.048 | 6 | 0.78 | 1.25 | | Fail | 6.6033 | 0.0480 | | 6 | 5 | 0.12 | |
| 31L | 6.6275 | 0.010 | 4 | 0.94 | 0.25 | | Fail | 6.6275 | 0.0096 | | 4 | 3 | 0.14 | |
| 12L | 6.6950 | 0.010 | 4 | 1.38 | 0.26 | | Fail | 6.6950 | 0.0100 | | 4 | 3 | 0.21 | |
| 65L | 6.7333 | 0.058 | 3 | 1.63 | 1.50 | | Fail | 6.7333 | 0.0577 | | 3 | 2 | 0.25 | |
| Tot.gem | 6.483 | 0.034 pH unit | | 1%-level: | 2.25 | (1.75) | | 12 | 6.4829 | 0 | | 12 | 11 | |
| Tot.std= | 0.154 | 0.019 | | 5%-level: | 1.83 | (1.51) | | | | | | | | |

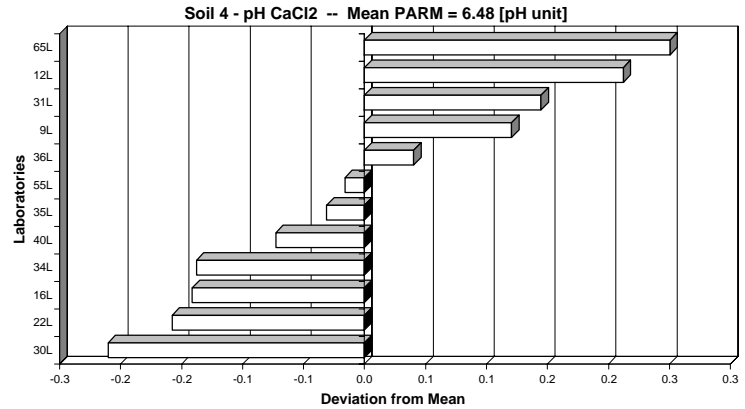
58

RESULTS: Mean = 6.48292 pH unit

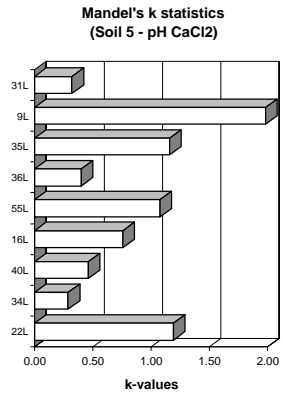
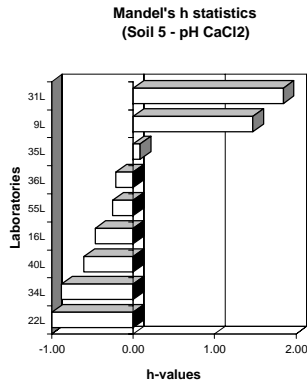
Repeatability variance S2r = 0.00152
 Repeatability std. Sr = 0.03897 --> 0.60% r = 0.1091

Between lab variance S2L = 0.02014
 Reproducibility var. S2R = 0.02166
 Reproducibility std. SR = 0.14716 --> 2.27% R = 0.4120

Remarks: none



Sample: Soil 5
 Element: pH CaCl2



Unit: pH unit

Mandel's k statistics (Soil 5 - pH CaCl2)
 Mandel's h statistics (Soil 5 - pH CaCl2)
 Soil 5 - pH CaCl2 -- Mean PARM = 6.59 [pH unit]

```

General calc.parm.
T1= 2.83220E+02
T2= 1.86564E+03
T3= 43
T4= 239
T5= 3.3567E-02
n= variabel
p= 8
N-table= 5
  
```

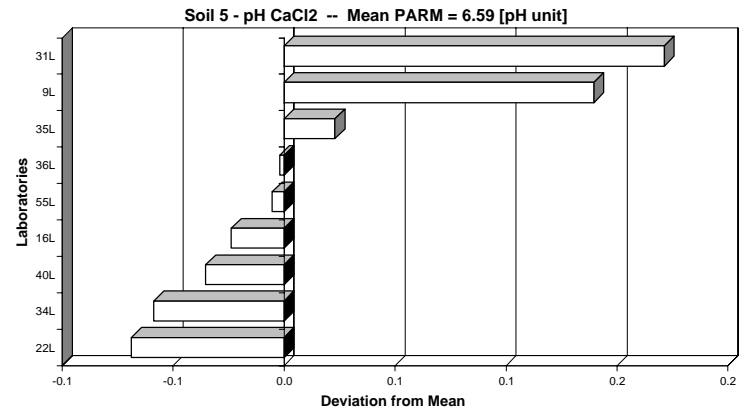
| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|--------|---------------------|------|-------------------------------|-----------------|--------|--------|----------|---|-----|----------|
| | | | | | h | k | | | | | | | | |
| 22L | 6.5200 | 0.045 | 5 | | -1.00 | 1.19 | | | 6.5200 | 0.0453 | | 5 | 4 | -0.07 |
| 34L | 6.5300 | 0.011 | 6 | | -0.88 | 0.29 | | Fail | 6.5300 | 0.0110 | | 6 | 5 | -0.06 |
| 40L | 6.5533 | 0.018 | 6 | | -0.60 | 0.46 | | Fail | 6.5533 | 0.0175 | | 6 | 5 | -0.04 |
| 16L | 6.5650 | 0.029 | 6 | | -0.46 | 0.76 | | | 6.5650 | 0.0288 | | 6 | 5 | -0.02 |
| 55L | 6.5833 | 0.041 | 6 | | -0.25 | 1.08 | | | 6.5833 | 0.0408 | | 6 | 5 | -0.01 |
| 36L | 6.5867 | 0.015 | 3 | | -0.21 | 0.40 | | | 6.5867 | 0.0153 | | 3 | 2 | 0.00 |
| 35L | 6.6117 | 0.044 | 6 | | 0.09 | 1.16 | | | 6.6117 | 0.0440 | | 6 | 5 | 0.02 |
| 9L | 6.7283 | 0.075 | 6 | | 1.47 | 1.98 | II | Fail | | | .9L | | | 0.14 |
| 31L | 6.7600 | 0.012 | 5 | I | 1.84 | 0.32 | | Fail | 6.7600 | 0.0122 | | 5 | 4 | 0.17 |
| Tot.gem | 6.604 | 0.032 | | | 1%-level: | 2.13 | (1.73) | | 8 | | | 8 | | |
| Tot.std= | 0.085 | 0.021 | | | 5%-level: | 1.78 | (1.5) | | 1 | | (9L) | 8 | 7 | |

49

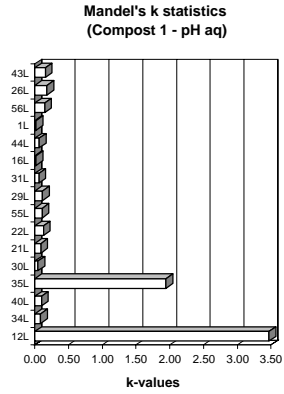
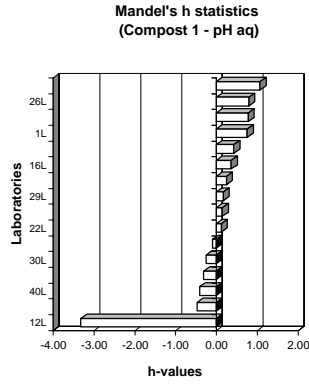
RESULTS: Mean = 6.58875 pH unit

Repeatability variance S2r = 0.00096
 Repeatability std. Sr = 0.03097 --> 0.47% r = 0.0867
 Between lab variance S2L = 0.00530
 Reproducibility var. S2R = 0.00626
 Reproducibility std. SR = 0.07909 --> 1.20% R = 0.2215

Remarks: 1 Lab rejected! (9L)



Sample: **Compost 1**
 Element: **pH aq**



Unit: pH unit

Mandel's k statistics (Compost 1 - pH aq)
 Mandel's h statistics (Compost 1 - pH aq)
 Compost 1 - pH aq -- Mean PARM = 8.13 [pH unit]

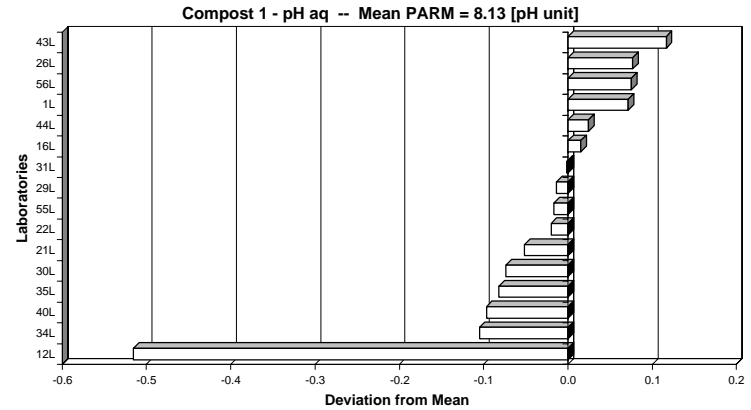
General calc.parm.
 T1= 5.69020E+02
 T2= 4.62578E+03
 T3= 70
 T4= 374
 T5= 9.3738E-02
 n= variabel
 p= 14
 N-table= 5

| LAB | PARM-gem | Stdev | N | Mandel's statistics | | h-mark | | k-mark | | End Result: | | Rej.labs | N | N-1 | dev_mean |
|----------|----------|---------------|---|---------------------|-------------|--------|----|--------|-------|-------------|--------|-----------|----|-----|----------|
| | | | | h | k | h | k | PARM | Stdev | | | | | | |
| 12L | 7.6183 | 1.273 | 6 | -3.32 | 3.47 | !! | !! | - | - | 8.0283 | 0.0313 | .12L | 6 | 5 | -0.52 |
| 34L | 8.0283 | 0.031 | 6 | -0.47 | 0.09 | | | | | 8.0367 | 0.0372 | | 6 | 5 | -0.10 |
| 40L | 8.0367 | 0.037 | 6 | -0.41 | 0.10 | | | | | | | | 6 | 5 | -0.10 |
| 35L | 8.0517 | 0.714 | 6 | -0.31 | 1.95 | | !! | | | | | .35L | 6 | 5 | -0.08 |
| 30L | 8.0600 | 0.017 | 3 | -0.25 | 0.05 | | | | | 8.0690 | 0.0173 | | 3 | 2 | -0.07 |
| 21L | 8.0817 | 0.034 | 6 | -0.10 | 0.09 | | | | | 8.0817 | 0.0343 | | 6 | 5 | -0.05 |
| 22L | 8.1133 | 0.046 | 6 | 0.12 | 0.13 | | | | | 8.1133 | 0.0463 | | 6 | 5 | -0.02 |
| 55L | 8.1167 | 0.041 | 6 | 0.14 | 0.11 | | | | | 8.1167 | 0.0408 | | 6 | 5 | -0.02 |
| 29L | 8.1200 | 0.042 | 4 | 0.17 | 0.12 | | | | | 8.1200 | 0.0424 | | 4 | 3 | -0.01 |
| 31L | 8.1320 | 0.024 | 5 | 0.25 | 0.07 | | | | | 8.1320 | 0.0239 | | 5 | 4 | 0.00 |
| 16L | 8.1483 | 0.008 | 6 | 0.36 | 0.02 | | | | | 8.1483 | 0.0075 | | 6 | 5 | 0.01 |
| 44L | 8.1575 | 0.025 | 4 | 0.43 | 0.07 | | | | | 8.1575 | 0.0250 | | 4 | 3 | 0.02 |
| 1L | 8.2050 | 0.007 | 2 | 0.76 | 0.02 | | | | | 8.2050 | 0.0071 | | 2 | 1 | 0.07 |
| 56L | 8.2083 | 0.054 | 6 | 0.78 | 0.15 | | | | | 8.2083 | 0.0538 | | 6 | 5 | 0.07 |
| 26L | 8.2100 | 0.066 | 6 | 0.79 | 0.18 | | | | | 8.2100 | 0.0663 | | 6 | 5 | 0.08 |
| 43L | 8.2500 | 0.058 | 4 | 1.07 | 0.16 | | | | | 8.2500 | 0.0577 | | 4 | 3 | 0.12 |
| Tot.gem | 8.096 | 0.155 pH unit | | 1%-level: | 2.33 (1.77) | | | | | 14 | 8.1334 | (12L,35L) | 14 | 13 | |
| Tot.stde | 0.144 | 0.343 | | 5%-level: | 1.86 (1.52) | | | | | 2 | | | | | |

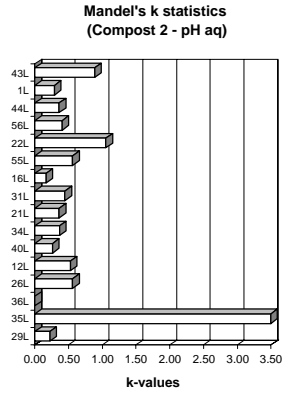
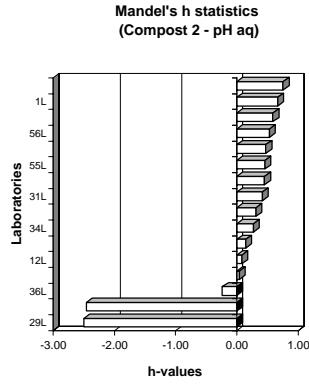
RESULTS: Mean = **8.13342** pH unit

Repeatability variance S2r = 0.00167
 Repeatability std. Sr = **0.04091** --> 0.50% r = 0.1146
 Between lab variance S2L = 0.00423
 Reproducibility var. S2R = 0.00590
 Reproducibility std. SR = **0.07683** --> 0.94% R = 0.2151

Remarks: 2 Labs rejected! (12L,35L)



Sample: **Compost 2**
 Element: **pH aq**



Unit: pH unit

Mandel's k statistics (Compost 2 - pH aq)
 Mandel's h statistics (Compost 2 - pH aq)
 Compost 2 - pH aq -- Mean PARM = 8.37 [pH unit]

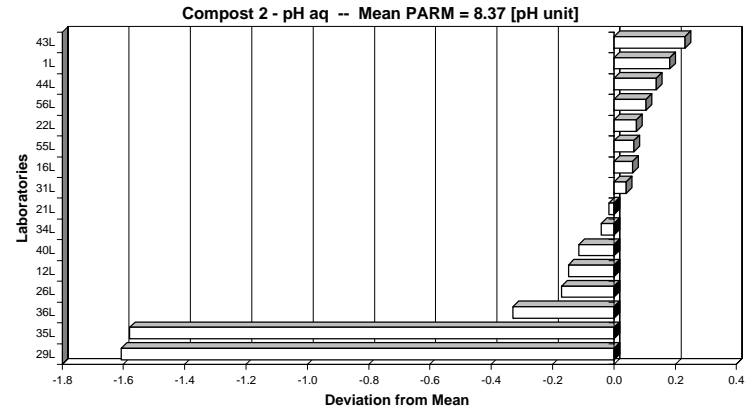
General calc.parm.
 T1= 5.60580E+02
 T2= 4.69150E+03
 T3= 67
 T4= 363
 T5= 9.1272E-02
 n= variabel
 p= 13
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | End Result: | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|-----------|----------|-------|---|-----------|---------------------|--------|-------------------------------|-----------------|-------------|--------|-------|-----------------|----|-----|----------|
| | | | | | h | k | | | | | | | | | |
| 29L | 6.7625 | 0.021 | 4 | !! | -2.50 | 0.22 | | | - | | ,29L | - | - | - | -1.61 |
| 35L | 6.7883 | 0.321 | 6 | !! | -2.46 | 3.49 | !! | Fail | - | | ,35L | - | - | - | -1.58 |
| 36L | 8.0400 | | 3 | | -0.24 | | | | 8.0400 | | | 3 | 2 | | -0.33 |
| 26L | 8.1980 | 0.052 | 5 | | 0.04 | 0.56 | | | 8.1980 | 0.0517 | | 5 | 4 | | -0.17 |
| 12L | 8.2200 | 0.049 | 6 | | 0.08 | 0.53 | | | 8.2200 | 0.0486 | | 6 | 5 | | -0.15 |
| 40L | 8.2550 | 0.024 | 6 | | 0.14 | 0.26 | | | 8.2550 | 0.0243 | | 6 | 5 | | -0.11 |
| 34L | 8.3267 | 0.034 | 6 | | 0.27 | 0.37 | | Fail | 8.3267 | 0.0339 | | 6 | 5 | | -0.04 |
| 21L | 8.3517 | 0.033 | 6 | | 0.31 | 0.36 | | Fail | 8.3517 | 0.0331 | | 6 | 5 | | -0.02 |
| 31L | 8.4083 | 0.041 | 6 | | 0.41 | 0.45 | | Fail | 8.4083 | 0.0412 | | 6 | 5 | | 0.04 |
| 16L | 8.4300 | 0.015 | 6 | | 0.45 | 0.17 | | Fail | 8.4300 | 0.0155 | | 6 | 5 | | 0.06 |
| 55L | 8.4333 | 0.052 | 6 | | 0.46 | 0.56 | | Fail | 8.4333 | 0.0516 | | 6 | 5 | | 0.06 |
| 22L | 8.4417 | 0.097 | 6 | | 0.47 | 1.05 | | Fail | | | ,22L | - | - | - | 0.07 |
| 56L | 8.4733 | 0.037 | 6 | | 0.53 | 0.41 | | Fail | 8.4733 | 0.0372 | | 6 | 5 | | 0.10 |
| 44L | 8.5075 | 0.033 | 4 | | 0.59 | 0.36 | | Fail | 8.5075 | 0.0330 | | 4 | 3 | | 0.14 |
| 1L | 8.5500 | 0.026 | 3 | | 0.67 | 0.29 | | Fail | 8.5500 | 0.0265 | | 3 | 2 | | 0.18 |
| 43L | 8.6000 | 0.082 | 4 | | 0.75 | 0.89 | | Fail | 8.6000 | 0.0816 | | 4 | 3 | | 0.23 |
| Tot.gem | 8.174 | 0.057 | | 1%-level: | 2.33 | (1.77) | | | 13 | 8.3688 | | (22L ,29L ,35L) | 13 | 12 | |
| Tot.stdev | 0.564 | 0.074 | | 5%-level: | 1.86 | (1.52) | | | 3 | | | | | | |

RESULTS: Mean = **8.36876** pH unit

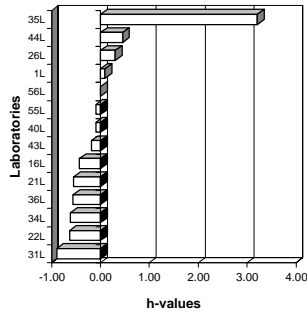
Repeatability variance S2r = 0.00169
 Repeatability std. Sr = **0.04111** --> 0.49% r = 0.1151
 Between lab variance S2L = 0.01923
 Reproducibility var. S2R = 0.02092
 Reproducibility std. SR = **0.14464** --> 1.73% R = 0.4050

Remarks: 3 Labs rejected! (22L ,29L ,35L)

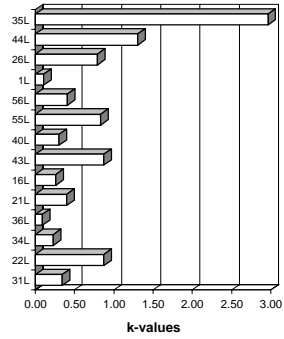


Sample: **Sludge 1**
 Element: **pH aq**

Mandel's h statistics
 (Sludge 1 - pH aq)



Mandel's k statistics
 (Sludge 1 - pH aq)



Unit: pH unit

Mandel's k statistics (Sludge 1 - pH aq)
 Mandel's h statistics (Sludge 1 - pH aq)
 Sludge 1 - pH aq -- Mean PARM = 6.71 [pH unit]

General calc.parm.
 T1= 3.89200E+02
 T2= 2.61190E+03
 T3= 58
 T4= 304
 T5= 5.8627E-02
 n= variabel
 p= 12
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|--------|---------------------|--------|-------------------------------|-----------------|--------|--------|-----------|---|-----|----------|
| | | | | | h | k | | | | | | | | |
| 31L | 6.6020 | 0.023 | 5 | | -0.89 | 0.35 | | | 6.6020 | 0.0228 | | 5 | 4 | -0.11 |
| 22L | 6.6500 | 0.058 | 4 | | -0.63 | 0.88 | | Fail | 6.6500 | 0.0577 | | 4 | 3 | -0.06 |
| 34L | 6.6533 | 0.015 | 6 | | -0.62 | 0.23 | | Fail | 6.6533 | 0.0151 | | 6 | 5 | -0.06 |
| 36L | 6.6633 | 0.006 | 3 | | -0.56 | 0.09 | | Fail | 6.6633 | 0.0058 | | 3 | 2 | -0.05 |
| 21L | 6.6667 | 0.027 | 6 | | -0.54 | 0.40 | | Fail | 6.6667 | 0.0266 | | 6 | 5 | -0.04 |
| 16L | 6.6883 | 0.017 | 6 | | -0.43 | 0.26 | | Fail | 6.6883 | 0.0172 | | 6 | 5 | -0.02 |
| 43L | 6.7333 | 0.058 | 3 | | -0.19 | 0.88 | | | 6.7333 | 0.0577 | | 3 | 2 | 0.02 |
| 40L | 6.7500 | 0.020 | 6 | | -0.10 | 0.30 | | | 6.7500 | 0.0200 | | 6 | 5 | 0.04 |
| 55L | 6.7500 | 0.055 | 6 | | -0.10 | 0.83 | | | 6.7500 | 0.0548 | | 6 | 5 | 0.04 |
| 56L | 6.7680 | 0.027 | 5 | | 0.00 | 0.41 | | | 6.7680 | 0.0268 | | 5 | 4 | 0.06 |
| 1L | 6.7850 | 0.007 | 2 | | 0.09 | 0.11 | | | 6.7850 | 0.0071 | | 2 | 1 | 0.07 |
| 26L | 6.8233 | 0.052 | 6 | | 0.30 | 0.79 | | | 6.8233 | 0.0524 | | 6 | 5 | 0.11 |
| 44L | 6.8520 | 0.086 | 5 | | 0.45 | 1.30 | | | | | .44L | | | 0.14 |
| 35L | 7.3633 | 0.195 | 6 | II | 3.21 | 2.96 | II | Fail | | | .35L | | | 0.65 |
| Tot.gem | 6.768 | 0.046 | | | 2.30 | (1.76) | | | 12 | 6.7111 | (35L,44L) | | | 11 |
| Tot.std= | 0.186 | 0.049 | | | 1.85 | (1.52) | | | 2 | | | | | |

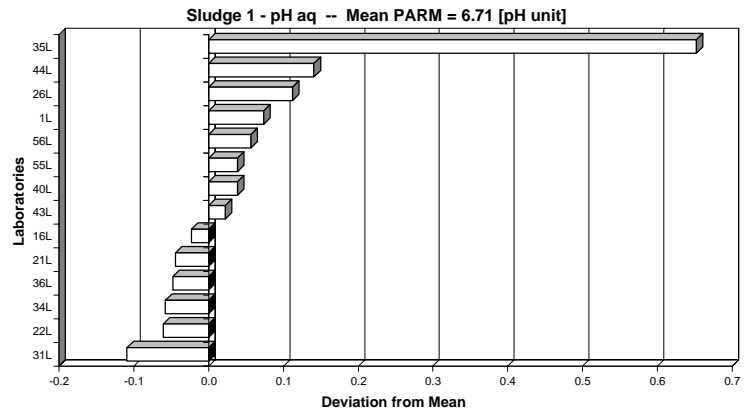
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RESULTS: Mean = 6.71111 pH unit

Repeatability variance S2r = 0.00127
 Repeatability std. Sr = 0.03570 --> 0.53% r = 0.1000

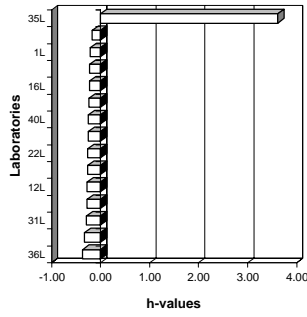
Between lab variance S2L = 0.00426
 Reproducibility var. S2R = 0.00553
 Reproducibility std. SR = 0.07437 --> 1.11% R = 0.2082

Remarks: 2 Labs rejected! (35L,44L)

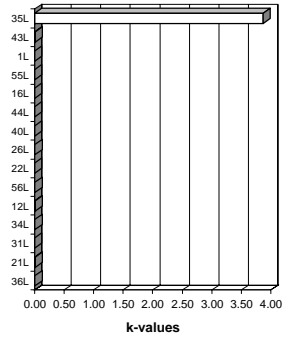


Sample: **Sludge 2**
 Element: **pH aq**

Mandel's h statistics
(Sludge 2 - pH aq)



Mandel's k statistics
(Sludge 2 - pH aq)



Unit: pH unit

Mandel's k statistics (Sludge 2 - pH aq)
 Mandel's h statistics (Sludge 2 - pH aq)
 Sludge 2 - pH aq -- Mean PARM = 7.51 [pH unit]

General calc.parm.
 T1= 4.58020E+02
 T2= 3.43967E+03
 T3= 61
 T4= 317
 T5= 2.8813E-02
 n= variabel
 p= 13
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | $AvX < AvST-2std$ | End Result: | | | | | | |
|----------|----------|---------------|---|--------|---------------------|------|-------------------------------|-------------------|-------------|--------|-----------|----|-----|----------|--|
| | | | | | h | k | | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean | |
| 36L | 7.2167 | 0.025 | 3 | | -0.37 | 0.00 | | | 7.2167 | 0.0252 | | 3 | 2 | -0.29 | |
| 21L | 7.3167 | 0.065 | 6 | | -0.33 | 0.01 | | | | | ,21L | | | -0.19 | |
| 31L | 7.4000 | 0.016 | 5 | | -0.29 | 0.00 | | | 7.4000 | 0.0158 | | 5 | 4 | -0.11 | |
| 34L | 7.4600 | 0.028 | 6 | | -0.27 | 0.00 | | | 7.4600 | 0.0283 | | 6 | 5 | -0.05 | |
| 12L | 7.4667 | 0.037 | 6 | | -0.27 | 0.01 | | | 7.4667 | 0.0372 | | 6 | 5 | -0.04 | |
| 56L | 7.4683 | 0.028 | 6 | | -0.27 | 0.00 | | | 7.4683 | 0.0279 | | 6 | 5 | -0.04 | |
| 22L | 7.5000 | | 2 | | -0.26 | | | | 7.5000 | | | 2 | 1 | -0.01 | |
| 26L | 7.5020 | 0.024 | 5 | | -0.26 | 0.00 | | | 7.5020 | 0.0239 | | 5 | 4 | -0.01 | |
| 40L | 7.5300 | 0.009 | 6 | | -0.24 | 0.00 | | | 7.5300 | 0.0089 | | 6 | 5 | 0.02 | |
| 44L | 7.5600 | 0.012 | 5 | | -0.23 | 0.00 | | | 7.5600 | 0.0122 | | 5 | 4 | 0.05 | |
| 16L | 7.5833 | 0.019 | 6 | | -0.22 | 0.00 | | | 7.5833 | 0.0186 | | 6 | 5 | 0.07 | |
| 55L | 7.6000 | 0.000 | 6 | | -0.22 | 0.00 | | | 7.6000 | 0.0000 | | 6 | 5 | 0.09 | |
| 1L | 7.6050 | 0.007 | 2 | | -0.22 | 0.00 | | | 7.6050 | 0.0071 | | 2 | 1 | 0.10 | |
| 43L | 7.7333 | 0.058 | 3 | | -0.17 | 0.01 | | | 7.7333 | 0.0577 | | 3 | 2 | 0.22 | |
| 35L | 17.5517 | 25.743 | 6 | II | 3.61 | 3.87 | II | | | | ,35L | | | 10.04 | |
| Tot.gem | 8.166 | 1.738 pH unit | | | 1%-level: | 2.32 | (1.76) | | 13 | 7.5096 | (21L,35L) | 13 | 12 | | |
| Tot.std= | 2.599 | 6.641 | | | 5%-level: | 1.86 | (1.52) | | 2 | | | | | | |

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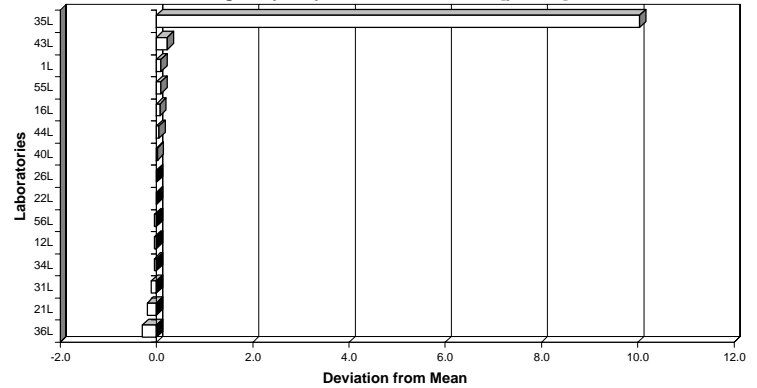
RESULTS: Mean = **7.50964** pH unit

Repeatability variance S2r = 0.00060
 Repeatability std. Sr = **0.02450** --> 0.33% r = 0.0686

Between lab variance S2L = 0.01097
 Reproducibility var. S2R = 0.01157
 Reproducibility std. SR = **0.10755** --> 1.43% R = 0.3011

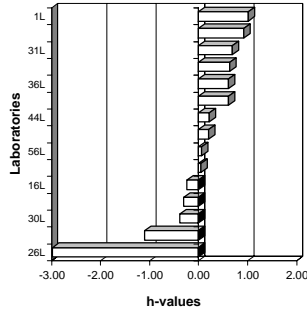
Remarks: 2 Labs rejected! (21L,35L)

Sludge 2 - pH aq -- Mean PARM = 7.51 [pH unit]

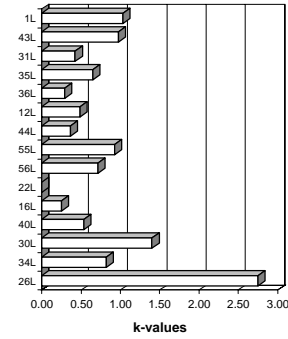


Sample: Soil 4
Element: pH aq

Mandel's h statistics
(Soil 4 - pH aq)



Mandel's k statistics
(Soil 4 - pH aq)



Unit: pH unit

Mandel's k statistics (Soil 4 - pH aq)
Mandel's h statistics (Soil 4 - pH aq)

Soil 4 - pH aq -- Mean PARM = 7.05 [pH unit]

General calc.parm.
T1= 4.63770E+02
T2= 3.26075E+03
T3= 66
T4= 340
T5= 9.1397E-02
n= variabel
p= 14
N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|--------|---------------------|------|-------------------------------|-----------------|--------|--------|----------|----|-----|----------|
| | | | | | h | k | | | | | | | | |
| 26L | 6.0800 | 0.162 | 5 | !! | -2.99 | 2.75 | !! | | | | | | | -0.97 |
| 34L | 6.6533 | 0.048 | 6 | | -1.10 | 0.82 | | Fail | 6.6533 | 0.0484 | | 6 | 5 | -0.40 |
| 30L | 6.8700 | 0.083 | 4 | | -0.38 | 1.40 | | Fail | 6.8700 | 0.0829 | | 4 | 3 | -0.18 |
| 40L | 6.8950 | 0.031 | 6 | | -0.30 | 0.53 | | Fail | 6.8950 | 0.0315 | | 6 | 5 | -0.16 |
| 16L | 6.9133 | 0.015 | 6 | | -0.24 | 0.25 | | | 6.9133 | 0.0151 | | 6 | 5 | -0.14 |
| 22L | 7.0000 | | 2 | | 0.05 | | | | 7.0000 | | | 2 | 1 | -0.05 |
| 56L | 7.0033 | 0.042 | 6 | | 0.06 | 0.72 | | | 7.0033 | 0.0423 | | 6 | 5 | -0.05 |
| 55L | 7.0500 | 0.055 | 6 | | 0.21 | 0.93 | | | 7.0500 | 0.0548 | | 6 | 5 | 0.00 |
| 44L | 7.0533 | 0.022 | 6 | | 0.22 | 0.37 | | | 7.0533 | 0.0216 | | 6 | 5 | 0.00 |
| 12L | 7.1700 | 0.029 | 6 | | 0.61 | 0.49 | Fail | | 7.1700 | 0.0290 | | 6 | 5 | 0.12 |
| 36L | 7.1700 | 0.017 | 3 | | 0.61 | 0.29 | Fail | | 7.1700 | 0.0173 | | 3 | 2 | 0.12 |
| 35L | 7.1780 | 0.038 | 5 | | 0.63 | 0.65 | Fail | | 7.1780 | 0.0383 | | 5 | 4 | 0.13 |
| 31L | 7.1950 | 0.025 | 4 | | 0.69 | 0.43 | Fail | | 7.1950 | 0.0252 | | 4 | 3 | 0.14 |
| 43L | 7.2667 | 0.058 | 3 | | 0.92 | 0.98 | Fail | | 7.2667 | 0.0577 | | 3 | 2 | 0.22 |
| 1L | 7.2933 | 0.061 | 3 | | 1.01 | 1.03 | Fail | | 7.2933 | 0.0611 | | 3 | 2 | 0.24 |
| Tot.gem | 6.986 | 0.046 | | | 1%-level: | 2.32 | | | 14 | 7.0508 | | 14 | 13 | |
| Tot.std= | 0.303 | 0.039 | | | 5%-level: | 1.86 | | | 1 | | (26L) | | | |

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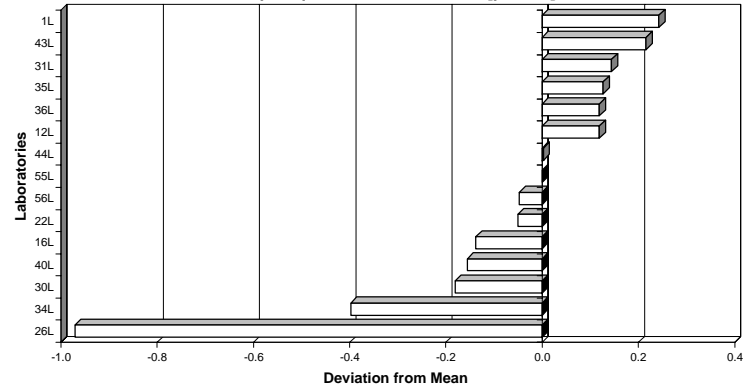
RESULTS: Mean = 7.05081 pH unit

Repeatability variance S2r = 0.00176
Repeatability std. Sr = 0.04192 --> 0.59% r = 0.1174

Between lab variance S2L = 0.03129
Reproducibility var. S2R = 0.03304
Reproducibility std. SR = 0.18178 --> 2.58% R = 0.5090

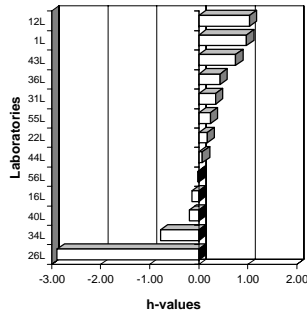
Remarks: 1 Lab rejected! (26L)

Soil 4 - pH aq -- Mean PARM = 7.05 [pH unit]

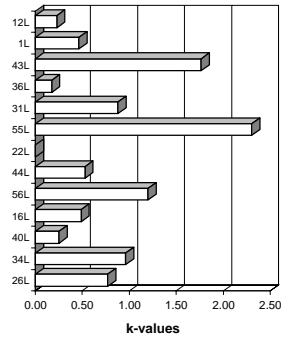


Sample: Soil 5
Element: pH aq

Mandel's h statistics
(Soil 5 - pH aq)



Mandel's k statistics
(Soil 5 - pH aq)



Unit: pH unit

Mandel's k statistics (Soil 5 - pH aq)
Mandel's h statistics (Soil 5 - pH aq)
Soil 5 - pH aq -- Mean PARM = 7.1 [pH unit]

General calc.parm.
T1= 3.33140E+02
T2= 2.36224E+03
T3= 47
T4= 243
T5= 1.9287E-02
n= variabel
p= 10
N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|-----------|---------------------|--------|-------------------------------|-----------------|--------|--------|-----------------|----|-----|----------|
| | | | | | h | k | | | | | | | | |
| 26L | 6.3100 | 0.025 | 6 | II | -2.89 | 0.77 | | | | | | | | -0.79 |
| 34L | 6.8533 | 0.031 | 6 | | -0.78 | 0.96 | | Fail | 6.8533 | 0.0314 | | 6 | 5 | -0.25 |
| 40L | 7.0050 | 0.008 | 6 | | -0.20 | 0.26 | | Fail | 7.0050 | 0.0084 | | 6 | 5 | -0.10 |
| 16L | 7.0183 | 0.016 | 6 | | -0.14 | 0.49 | | Fail | 7.0183 | 0.0160 | | 6 | 5 | -0.09 |
| 56L | 7.0517 | 0.039 | 6 | | -0.01 | 1.20 | | | 7.0517 | 0.0392 | | 6 | 5 | -0.05 |
| 44L | 7.0750 | 0.017 | 4 | | 0.08 | 0.53 | | | 7.0750 | 0.0173 | | 4 | 3 | -0.03 |
| 22L | 7.1000 | | 2 | | 0.17 | | Fail | | 7.1000 | | | 2 | 1 | 0.00 |
| 55L | 7.1167 | 0.075 | 6 | | 0.24 | 2.30 | II | Fail | | | | | | 0.01 |
| 31L | 7.1440 | 0.029 | 5 | | 0.34 | 0.88 | | Fail | 7.1440 | 0.0288 | | 5 | 4 | 0.04 |
| 36L | 7.1667 | 0.006 | 3 | | 0.43 | 0.18 | | Fail | 7.1667 | 0.0058 | | 3 | 2 | 0.06 |
| 43L | 7.2500 | 0.058 | 4 | | 0.75 | 1.76 | I | Fail | | | .43L | | | 0.15 |
| 1L | 7.3067 | 0.015 | 3 | | 0.97 | 0.47 | | Fail | 7.3067 | 0.0153 | | 3 | 2 | 0.20 |
| 12L | 7.3217 | 0.008 | 6 | | 1.03 | 0.23 | | Fail | 7.3217 | 0.0075 | | 6 | 5 | 0.22 |
| Tot.gem | 7.055 | 0.025 | | 1%-level: | 2.27 | (1.76) | | | 10 | | | 10 | 9 | |
| Tot.std= | 0.258 | 0.022 | | 5%-level: | 1.84 | (1.51) | | | 3 | | (26L, 43L, 55L) | | | |

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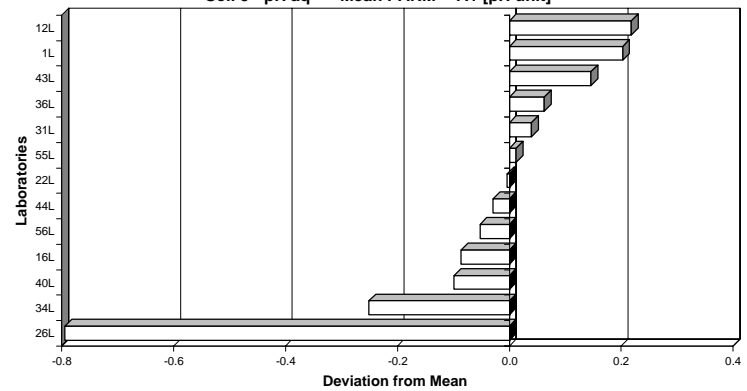
RESULTS: Mean = 7.10423 pH unit

Repeatability variance S2r = 0.00052
Repeatability std. Sr = 0.02283 --> 0.32% r = 0.0639

Between lab variance S2L = 0.02176
Reproducibility var. S2R = 0.02228
Reproducibility std. SR = 0.14928 --> 2.10% R = 0.4180

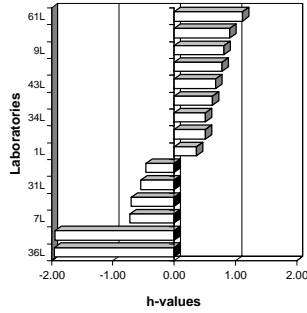
Remarks: 3 Labs rejected! (26L, 43L, 55L)

Soil 5 - pH aq -- Mean PARM = 7.1 [pH unit]

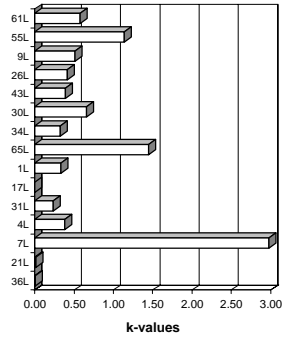


Sample: **Compost 1**
 Element: **EC**

Mandel's h statistics
(Compost 1 - EC)



Mandel's k statistics
(Compost 1 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Compost 1 - EC)
 Mandel's h statistics (Compost 1 - EC)
 Compost 1 - EC -- Mean PARM = 237.2 [mS/m at 20 deg]

General calc.parm.
 T1= 1.59678E+04
 T2= 4.90039E+06
 T3= 64
 T4= 348
 T5= 6.4446E+02
 n= variabel
 p= 13
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark | k-mark > AvST+2std | AvX < AvST-2std | End Result: | | Rej.labs | N | N-1 | dev_mean |
|----------|----------|--------|---|--------|---------------------|------|--------|--------------------|-----------------|-------------|----------|----------|----|---------|----------|
| | | | | | h | k | | | | PARM | Stdev | | | | |
| 36L | 1.8933 | 0.032 | 3 | I | -1.96 | 0.00 | 1.8933 | Fail | 1.8933 | 0.0321 | | 3 | 2 | -235.33 | |
| 21L | 3.2517 | 0.087 | 6 | I | -1.95 | 0.01 | 3.2517 | Fail | 3.2517 | 0.0873 | | 6 | 5 | -233.97 | |
| 7L | 149.5667 | 20.142 | 6 | | -0.72 | 2.97 | II | Fail | | | .7L | | | -87.66 | |
| 4L | 151.3000 | 2.574 | 6 | | -0.70 | 0.38 | | Fail | 151.3000 | 2.5745 | | 6 | 5 | -85.93 | |
| 31L | 171.0000 | 1.581 | 5 | | -0.54 | 0.23 | | Fail | 171.0000 | 1.5811 | | 5 | 4 | -66.23 | |
| 17L | 180.0000 | - | 1 | | -0.46 | - | | Fail | 180.0000 | - | | 1 | | -57.23 | |
| 1L | 279.3667 | 2.250 | 3 | | 0.37 | 0.33 | | Fail | 279.3667 | 2.2502 | | 3 | 2 | 42.14 | |
| 65L | 295.8250 | 9.798 | 4 | | 0.51 | 1.45 | | Fail | | | .65L | | | 58.60 | |
| 34L | 296.4667 | 2.203 | 6 | | 0.51 | 0.33 | | Fail | 296.4667 | 2.2033 | | 6 | 5 | 59.24 | |
| 30L | 310.5000 | 4.435 | 4 | | 0.63 | 0.65 | | Fail | 310.5000 | 4.4347 | | 4 | 3 | 73.27 | |
| 43L | 316.6500 | 2.660 | 6 | | 0.68 | 0.39 | | Fail | 316.6500 | 2.6599 | | 6 | 5 | 79.42 | |
| 26L | 328.8333 | 2.787 | 6 | | 0.78 | 0.41 | | Fail | 328.8333 | 2.7869 | | 6 | 5 | 91.61 | |
| 9L | 332.5000 | 3.450 | 6 | | 0.81 | 0.51 | | Fail | 332.5000 | 3.4496 | | 6 | 5 | 95.27 | |
| 55L | 343.3333 | 7.711 | 6 | | 0.91 | 1.14 | | Fail | 343.3333 | 7.7115 | | 6 | 5 | 106.11 | |
| 61L | 368.8333 | 3.920 | 6 | | 1.12 | 0.58 | | Fail | 368.8333 | 3.9200 | | 6 | 5 | 131.61 | |
| Tot.gem | 235.288 | 4.545 | | | 1%-level: | 2.32 | (1.76) | | 13 | 237.2253 | (65L,7L) | 13 | 12 | | |
| Tot.std= | 119.296 | 5.209 | | | 5%-level: | 1.86 | (1.52) | | 2 | | | | | | |

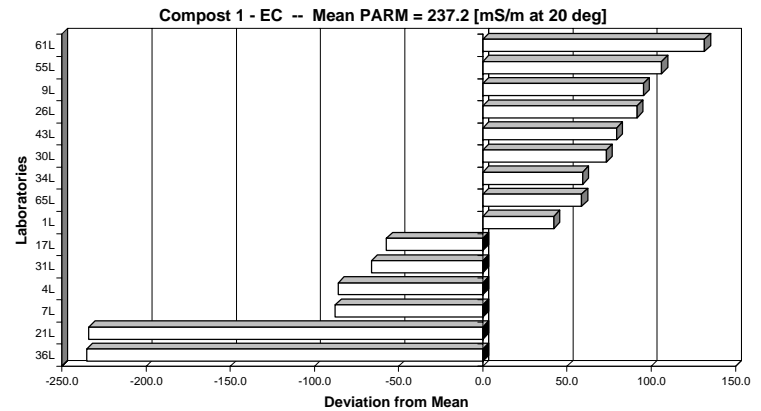
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RESULTS: Mean = 237.22526 nS/m at 20 deg

Repeatability variance S2r = 12.63638
 Repeatability std. Sr = 3.55477 --> 1.50% r = 9.9534

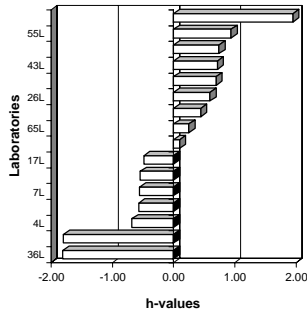
Between lab variance S2L = 15646.93160
 Reproducibility var. S2R = 15659.56798
 Reproducibility std. SR = 125.13820 --> 52.75% R = 350.3869

Remarks: 2 Labs rejected! (65L,7L)

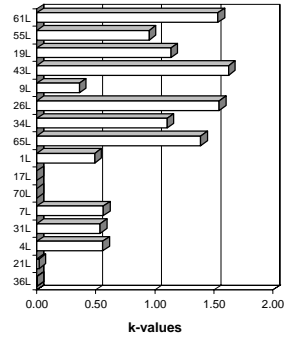


Sample: **Compost 2**
 Element: **EC**

Mandel's h statistics
(Compost 2 - EC)



Mandel's k statistics
(Compost 2 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Compost 2 - EC)
 Mandel's h statistics (Compost 2 - EC)
 Compost 2 - EC -- Mean PARM = 206.8 [mS/m at 20 deg]

General calc.parm.
 T1= 1.72362E+04
 T2= 4.78781E+06
 T3= 78
 T4= 432
 T5= 1.9510E+03
 n= variabel 16
 N-table= 5

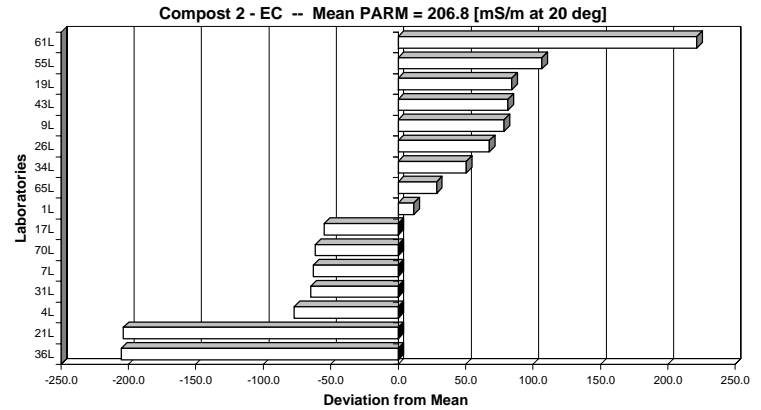
| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark | k-mark > AvST+2std | AvX < AvST-2std | End Result: | | Rej.labs | N | N-1 | dev_mean |
|-----------|----------|-------|---|--------|---------------------|------|--------|--------------------|-----------------|-------------|--------|----------|----|-----|----------|
| | | | | | h | k | | | | PARM | Stdev | | | | |
| 36L | 1.6700 | 0.020 | 3 | | -1.81 | 0.00 | | | | 1.6700 | 0.0200 | | 3 | 2 | -205.13 |
| 21L | 2.9017 | 0.105 | 6 | | -1.80 | 0.02 | | | | 2.9017 | 0.1050 | | 6 | 5 | -203.90 |
| 4L | 129.5667 | 3.049 | 6 | | -0.68 | 0.56 | | | | 129.5667 | 3.0487 | | 6 | 5 | -77.24 |
| 31L | 142.1667 | 2.927 | 6 | | -0.57 | 0.54 | | | | 142.1667 | 2.9269 | | 6 | 5 | -64.64 |
| 7L | 144.0500 | 3.074 | 6 | | -0.55 | 0.56 | | | | 144.0500 | 3.0742 | | 6 | 5 | -62.75 |
| 70L | 145.3000 | - | 1 | | -0.54 | | | | | 145.3000 | - | | 1 | | -61.50 |
| 17L | 152.0000 | - | 1 | | -0.48 | | | | | 152.0000 | - | | 1 | | -54.80 |
| 1L | 218.2333 | 2.684 | 3 | | 0.10 | 0.49 | | Fail | | 218.2333 | 2.6839 | | 3 | 2 | 11.43 |
| 65L | 235.2750 | 7.575 | 4 | | 0.25 | 1.39 | | Fail | | 235.2750 | 7.5751 | | 4 | 3 | 28.47 |
| 34L | 257.1667 | 6.022 | 6 | | 0.44 | 1.10 | | Fail | | 257.1667 | 6.0215 | | 6 | 5 | 50.36 |
| 26L | 274.5000 | 8.432 | 6 | | 0.60 | 1.54 | ! | Fail | | 274.5000 | 8.4321 | | 6 | 5 | 67.70 |
| 9L | 285.3333 | 1.966 | 6 | | 0.69 | 0.36 | | Fail | | 285.3333 | 1.9664 | | 6 | 5 | 78.53 |
| 43L | 288.3000 | 8.871 | 6 | | 0.72 | 1.62 | ! | Fail | | 288.3000 | 8.8706 | | 6 | 5 | 81.50 |
| 19L | 290.8667 | 6.198 | 6 | | 0.74 | 1.13 | | Fail | | 290.8667 | 6.1976 | | 6 | 5 | 84.06 |
| 55L | 313.3333 | 5.203 | 6 | | 0.94 | 0.95 | | Fail | | 313.3333 | 5.2026 | | 6 | 5 | 106.53 |
| 61L | 428.1667 | 8.377 | 6 | ! | 1.95 | 1.53 | ! | Fail | | 428.1667 | 8.3766 | | 6 | 5 | 221.36 |
| Tot.gem | 206.802 | 4.607 | | | | | | | 16 | 206.8019 | () | | 16 | 15 | |
| Tot.stdev | 113.375 | 3.042 | | | 1%-level: | 2.33 | (1.76) | | | | | | | | |
| | | | | | 5%-level: | 1.86 | (1.52) | | | | | | | | |

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RESULTS: Mean = **206.80188** mS/m at 20 deg

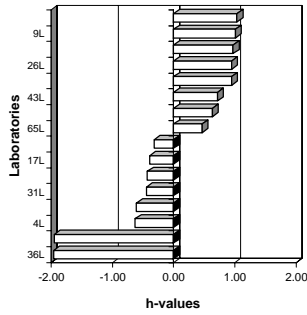
Repeatability variance **S2r = 31.46707**
 Repeatability std. **Sr = 5.60955** --> 2.71% **r = 15.7067**
 Between lab variance **S2L = 13504.14435**
 Reproducibility var. **S2R = 13535.61143**
 Reproducibility std. **SR = 116.34265** --> 56.26% **R = 325.7594**

Remarks: none

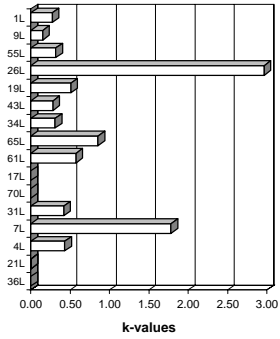


Sample: **Sludge 1**
 Element: **EC**

Mandel's h statistics
 (Sludge 1 - EC)



Mandel's k statistics
 (Sludge 1 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Sludge 1 - EC)
 Mandel's h statistics (Sludge 1 - EC)

Sludge 1 - EC -- Mean PARM = 319.6 [mS/m at 20 deg]

General calc.parm.
 T1= 2.19933E+04
 T2= 9.07225E+06
 T3= 66
 T4= 360
 T5= 1.3933E+03
 n= variabel
 p= 14
 N-table= 5

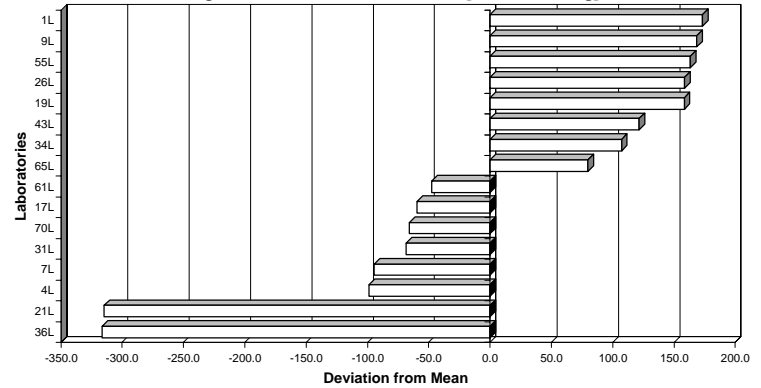
| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark | AvX > AvST+2std | AvX < AvST-2std | End Result: | | | | | |
|-----------|----------|--------|----------------|--------|---------------------|------|--------|-----------------|-----------------|-------------|-----------|----------|----|-----|----------|
| | | | | | h | k | | | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
| 36L | 3.2067 | 0.023 | 3 | I | -1.96 | 0.00 | | Fail | | 3.2067 | 0.0231 | | 3 | 2 | -316.42 |
| 21L | 4.9567 | 0.048 | 6 | I | -1.95 | 0.00 | | Fail | | 4.9567 | 0.0480 | | 6 | 5 | -314.67 |
| 4L | 221.0667 | 5.481 | 6 | | -0.63 | 0.43 | | Fail | | 221.0667 | 5.4815 | | 6 | 5 | -98.56 |
| 7L | 224.8500 | 22.690 | 6 | | -0.60 | 1.78 | II | Fail | | | | .7L | | | -94.78 |
| 31L | 251.1667 | 5.345 | 6 | | -0.44 | 0.42 | | Fail | | 251.1667 | 5.3448 | | 6 | 5 | -69.46 |
| 70L | 253.6500 | - | 1 | | -0.43 | | | Fail | | 253.6500 | | | 1 | | -65.98 |
| 17L | 260.0000 | - | 1 | | -0.39 | | | Fail | | 260.0000 | | | 1 | | -59.63 |
| 61L | 271.8333 | 7.333 | 6 | | -0.32 | 0.58 | | Fail | | 271.8333 | 7.3326 | | 6 | 5 | -47.80 |
| 65L | 399.5500 | 10.889 | 4 | | 0.46 | 0.85 | | Fail | | 399.5500 | 10.8890 | | 4 | 3 | 79.92 |
| 34L | 426.7667 | 3.959 | 6 | | 0.63 | 0.31 | | Fail | | 426.7667 | 3.9591 | | 6 | 5 | 107.14 |
| 43L | 441.0000 | 3.578 | 6 | | 0.72 | 0.28 | | Fail | | 441.0000 | 3.5777 | | 6 | 5 | 121.37 |
| 19L | 477.9333 | 6.465 | 6 | | 0.94 | 0.51 | | Fail | | 477.9333 | 6.4646 | | 6 | 5 | 158.31 |
| 26L | 478.1667 | 37.775 | 6 | | 0.95 | 2.96 | II | Fail | | | | .26L | | | 158.54 |
| 55L | 482.6667 | 4.033 | 6 | | 0.97 | 0.32 | | Fail | | 482.6667 | 4.0332 | | 6 | 5 | 163.04 |
| 9L | 488.1667 | 1.941 | 6 | | 1.01 | 0.15 | | Fail | | 488.1667 | 1.9408 | | 6 | 5 | 168.54 |
| 1L | 492.8333 | 3.479 | 3 | | 1.04 | 0.27 | | Fail | | 492.8333 | 3.4790 | | 3 | 2 | 173.21 |
| Tot.gem | 323.613 | 8.074 | mS/m at 20 deg | | 1% level: | 2.33 | (1.76) | | 14 | 319.6283 | (26L, 7L) | | 14 | 13 | |
| Tot.stdev | 163.349 | 10.237 | | | 5% level: | 1.86 | (1.52) | | 2 | | | | | | |

RESULTS: Mean = 319.62833 nS/m at 20 deg

Repeatability variance S2r = 26.79467
 Repeatability std. Sr = 5.17636 --> 1.62% r = 14.4938
 Between lab variance S2L = 28788.77519
 Reproducibility var. S2R = 28815.56986
 Reproducibility std. SR = 169.75149 --> 53.11% R = 475.3042

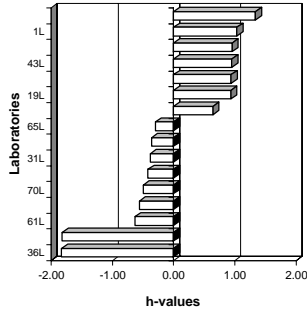
Remarks: 2 Labs rejected! (26L, 7L)

Sludge 1 - EC -- Mean PARM = 319.6 [mS/m at 20 deg]

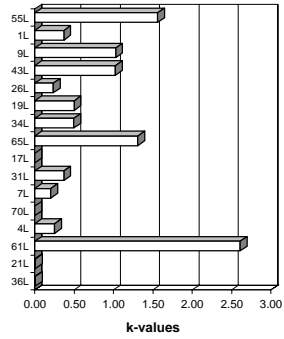


Sample: **Sludge 2**
 Element: **EC**

Mandel's h statistics
 (Sludge 2 - EC)



Mandel's k statistics
 (Sludge 2 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Sludge 2 - EC)
 Mandel's h statistics (Sludge 2 - EC)

Sludge 2 - EC -- Mean PARM = 358.2 [mS/m at 20 deg]

General calc.parm.
 T1= 2.21369E+04
 T2= 1.03049E+07
 T3= 61
 T4= 317
 T5= 1.6848E+03
 n= variabel
 p= 14
 N-table= 5

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark | k-mark > AvST+2std | AvX < AvST-2std | End Result: | | | | | | |
|----------|----------|--------|----------------|-----------|---------------------|--------|--------|--------------------|-----------------|-------------|-----------|----------|----|---------|----------|--|
| | | | | | h | k | | | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean | |
| 36L | 3.3667 | 0.084 | 3 | -1.83 | 0.01 | -1.83 | 0.01 | Fail | 3.3667 | 0.0839 | | 3 | 2 | -354.88 | | |
| 21L | 5.6483 | 0.092 | 6 | -1.82 | 0.01 | -1.82 | 0.01 | Fail | 5.6483 | 0.0924 | | 6 | 5 | -352.60 | | |
| 61L | 242.5000 | 24.656 | 6 | -0.63 | 2.61 | -0.63 | 2.61 | Fail | | | 61L | 6 | 5 | -115.75 | | |
| 4L | 257.1167 | 2.399 | 6 | -0.56 | 0.25 | -0.56 | 0.25 | Fail | 257.1167 | 2.3987 | | 6 | 5 | -101.13 | | |
| 70L | 269.8500 | - | 1 | -0.49 | - | -0.49 | - | Fail | 269.8500 | - | | 1 | 0 | -88.40 | | |
| 7L | 285.1667 | 1.941 | 6 | -0.42 | 0.21 | -0.42 | 0.21 | Fail | 285.1667 | 1.9408 | | 6 | 5 | -73.08 | | |
| 31L | 291.6667 | 3.502 | 6 | -0.38 | 0.37 | -0.38 | 0.37 | Fail | 291.6667 | 3.5024 | | 6 | 5 | -66.58 | | |
| 17L | 296.0000 | - | 1 | -0.36 | - | -0.36 | - | Fail | 296.0000 | - | | 1 | 0 | -62.25 | | |
| 65L | 308.6250 | 12.367 | 4 | -0.30 | 1.31 | -0.30 | 1.31 | Fail | 308.6250 | 12.3670 | | 4 | 3 | -49.62 | | |
| 34L | 495.9667 | 4.694 | 6 | 0.64 | 0.50 | 0.64 | 0.50 | Fail | 495.9667 | 4.6941 | | 6 | 5 | 137.72 | | |
| 19L | 554.6500 | 4.738 | 2 | 0.93 | 0.50 | 0.93 | 0.50 | Fail | 554.6500 | 4.7376 | | 2 | 1 | 196.40 | | |
| 26L | 556.0000 | 2.236 | 5 | 0.94 | 0.24 | 0.94 | 0.24 | Fail | 556.0000 | 2.2361 | | 5 | 4 | 197.75 | | |
| 43L | 557.6667 | 9.688 | 6 | 0.95 | 1.03 | 0.95 | 1.03 | Fail | 557.6667 | 9.6885 | | 6 | 5 | 195.42 | | |
| 9L | 558.8333 | 9.704 | 6 | 0.96 | 1.03 | 0.96 | 1.03 | Fail | 558.8333 | 9.7040 | | 6 | 5 | 200.58 | | |
| 1L | 574.9333 | 3.479 | 3 | 1.04 | 0.37 | 1.04 | 0.37 | Fail | 574.9333 | 3.4790 | | 3 | 2 | 216.68 | | |
| 55L | 634.8333 | 14.757 | 6 | 1.34 | 1.56 | 1.34 | 1.56 | Fail | | | 55L | 6 | 5 | 276.58 | | |
| Tot.gem | 368.301 | 6.738 | mS/m at 20 deg | 1%-level: | 2.33 | (1.76) | | | 14 | 358.2493 | (55L,61L) | 14 | 13 | | | |
| Tot.std= | 199.383 | 6.874 | | 5%-level: | 1.86 | (1.52) | | | 2 | | | | | | | |

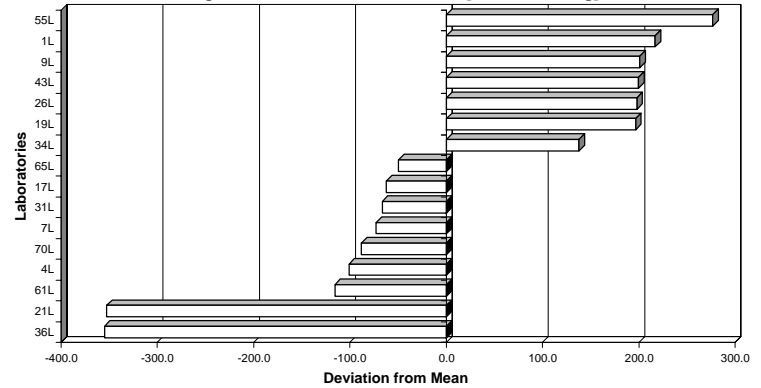
RESULTS: Mean = 358.24929 nS/m at 20 deg

Repeatability variance S2r = 35.84704
 Repeatability std. Sr = 5.98724 --> 1.67% r = 16.7643

Between lab variance S2L = 40694.55099
 Reproducibility var. S2R = 40730.39803
 Reproducibility std. SR = 201.81773 --> 56.33% R = 565.0897

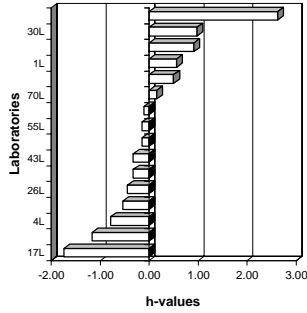
Remarks: 2 Labs rejected! (55L,61L)

Sludge 2 - EC -- Mean PARM = 358.2 [mS/m at 20 deg]

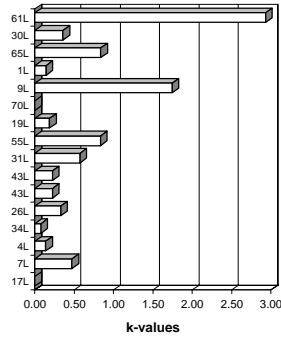


Sample: Soil 4
Element: EC

Mandel's h statistics
(Soil 4 - EC)



Mandel's k statistics
(Soil 4 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Soil 4 - EC)
Mandel's h statistics (Soil 4 - EC)

Soil 4 - EC -- Mean PARM = 10.29 [mS/m at 20 deg]

General calc.parm.
T1= 6.32135E+02
T2= 6.62727E+03
T3= 62
T4= 320
T5= 9.4118E+00
n= variabel
p= 14
N-table= 5

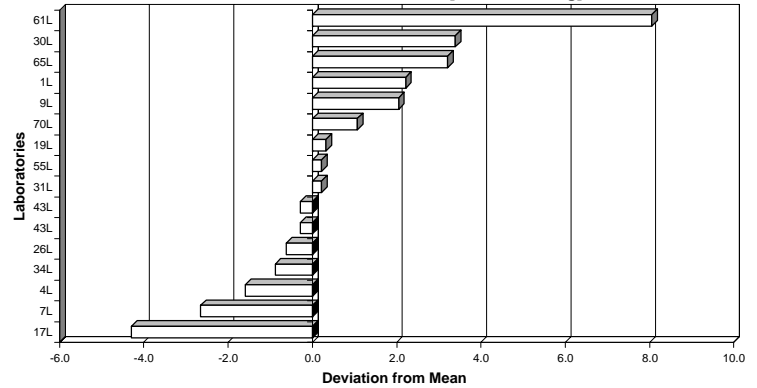
| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{k} > \text{AvST} + 2\text{std}$ | AvX < AvST - 2std | End Result: | | | | | | |
|----------|----------|-------|---|--------|---------------------|------|---|-------------------|-------------|---------|-----------|----|-----|----------|-------|
| | | | | | h | k | | | PARM | Stdev | Rej.labs | N | N-1 | dev_mean | |
| 17L | 6.0000 | - | 1 | | -1.74 | | | | 6.0000 | - | | 1 | | | -4.29 |
| 7L | 7.6350 | 0.474 | 6 | | -1.16 | 0.47 | | Fail | 7.6350 | 0.4741 | | 6 | 5 | | -2.65 |
| 4L | 8.6958 | 0.140 | 6 | | -0.79 | 0.14 | | Fail | 8.6958 | 0.1398 | | 6 | 5 | | -1.59 |
| 34L | 9.4050 | 0.077 | 6 | | -0.54 | 0.08 | | Fail | 9.4050 | 0.0774 | | 6 | 5 | | -0.88 |
| 26L | 9.6733 | 0.333 | 6 | | -0.44 | 0.33 | | | 9.6733 | 0.3327 | | 6 | 5 | | -0.62 |
| 43L | 10.0000 | 0.224 | 5 | | -0.33 | 0.22 | | | 10.0000 | 0.2236 | | 5 | 4 | | -0.29 |
| 43L | 10.0000 | 0.224 | 5 | | -0.33 | 0.22 | | | 10.0000 | 0.2236 | | 5 | 4 | | -0.29 |
| 31L | 10.5000 | 0.577 | 4 | | -0.15 | 0.57 | | | 10.5000 | 0.5774 | | 4 | 3 | | 0.21 |
| 55L | 10.5000 | 0.837 | 6 | | -0.15 | 0.83 | | | 10.5000 | 0.8367 | | 6 | 5 | | 0.21 |
| 19L | 10.6050 | 0.189 | 6 | | -0.11 | 0.19 | | | 10.6050 | 0.1893 | | 6 | 5 | | 0.32 |
| 70L | 11.3500 | - | 1 | | 0.15 | | | | 11.3500 | - | | 1 | | | 1.06 |
| 9L | 12.3333 | 1.751 | 6 | | 0.50 | 1.74 | II | | - | .9L | | - | | | 2.04 |
| 1L | 12.5000 | 0.141 | 2 | | 0.56 | 0.14 | | | 12.5000 | 0.1414 | | 2 | 1 | | 2.21 |
| 65L | 13.5000 | 0.841 | 4 | | 0.91 | 0.84 | | Fail | 13.5000 | 0.8406 | | 4 | 3 | | 3.21 |
| 30L | 13.6750 | 0.359 | 4 | | 0.98 | 0.36 | | Fail | 13.6750 | 0.3594 | | 4 | 3 | | 3.39 |
| 61L | 18.3333 | 2.944 | 6 | II | 2.62 | 2.93 | II | Fail | - | .61L | | - | | | 8.04 |
| Tot.gem | 10.919 | 0.651 | | | 1%-level: | 2.33 | (1.76) | | 14 | 10.2885 | (61L, 9L) | 14 | 13 | | |
| Tot.std= | 2.826 | 0.794 | | | 5%-level: | 1.86 | (1.52) | | 2 | | | | | | |

RESULTS: Mean = 10.28851 mS/m at 20 deg

Repeatability variance S2r = 0.19608
 Repeatability std. Sr = 0.44281 --> 4.30% r = 1.2399
 Between lab variance S2L = 3.16063
 Reproducibility var. S2R = 3.35670
 Reproducibility std. SR = 1.83213 --> 17.81% R = 5.1300

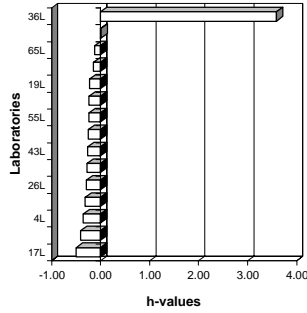
Remarks: 2 Labs rejected! (61L, 9L)

Soil 4 - EC -- Mean PARM = 10.29 [mS/m at 20 deg]

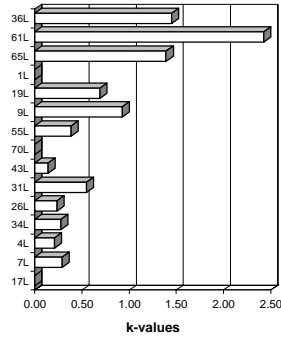


Sample: Soil 5
Element: EC

Mandel's h statistics
(Soil 5 - EC)



Mandel's k statistics
(Soil 5 - EC)



Unit: mS/m at 20 deg

Mandel's k statistics (Soil 5 - EC)
Mandel's h statistics (Soil 5 - EC)

Soil 5 - EC -- Mean PARM = 10.26 [mS/m at 20 deg]

```

General calc.parm.
T1= 5.95699E+02
T2= 6.24720E+03
T3= 58
T4= 316
T5= 1.7046E+01
n= variabel
p= 13
N-table= 4
  
```

| LAB | PARM-gem | Stdev | N | h-mark | Mandel's statistics | | k-mark $\sqrt{X} > AvST+2std$ | AvX < AvST-2std | PARM | Stdev | Rej.labs | N | N-1 | dev_mean |
|----------|----------|-------|---|--------|---------------------|------|-------------------------------|-----------------|---------|---------|------------|----|-----|----------|
| | | | | | h | k | | | | | | | | |
| 17L | 6.0000 | - | 1 | | -0.50 | | | Fail | 6.0000 | - | | 1 | | -4.26 |
| 7L | 7.8600 | 0.306 | 6 | | -0.40 | 0.29 | | Fail | 7.8600 | 0.3056 | | 6 | 5 | -2.40 |
| 4L | 8.8432 | 0.220 | 6 | | -0.35 | 0.21 | | Fail | 8.8432 | 0.2197 | | 6 | 5 | -1.42 |
| 34L | 9.4783 | 0.294 | 6 | | -0.32 | 0.28 | | Fail | 9.4783 | 0.2936 | | 6 | 5 | -0.78 |
| 26L | 10.0050 | 0.250 | 6 | | -0.29 | 0.24 | | Fail | 10.0050 | 0.2495 | | 6 | 5 | -0.25 |
| 31L | 10.3333 | 0.577 | 3 | | -0.27 | 0.55 | | Fail | 10.3333 | 0.5774 | | 3 | 2 | 0.07 |
| 43L | 10.4667 | 0.151 | 6 | | -0.27 | 0.14 | | Fail | 10.4667 | 0.1506 | | 6 | 5 | 0.21 |
| 70L | 10.7000 | - | 1 | | -0.25 | | | Fail | 10.7000 | - | | 1 | | 0.44 |
| 55L | 11.1667 | 0.408 | 6 | | -0.23 | 0.39 | | Fail | 11.1667 | 0.4082 | | 6 | 5 | 0.91 |
| 9L | 11.1667 | 0.983 | 6 | | -0.23 | 0.93 | | Fail | 11.1667 | 0.9832 | | 6 | 5 | 0.91 |
| 19L | 11.3800 | 0.730 | 6 | | -0.22 | 0.69 | | Fail | 11.3800 | 0.7296 | | 6 | 5 | 1.12 |
| 1L | 12.7000 | - | 1 | | -0.15 | | | Fail | 12.7000 | - | | 1 | | 2.44 |
| 65L | 13.2750 | 1.471 | 4 | | -0.12 | 1.39 | | Fail | 13.2750 | 1.4705 | | 4 | 3 | 3.02 |
| 61L | 15.8333 | 2.563 | 6 | | 0.01 | 2.43 | | Fail | - | - | .61L | - | - | 5.57 |
| 36L | 84.6667 | 1.531 | 3 | II | 3.59 | 1.45 | II | Fail | - | - | .36L | - | - | 74.41 |
| Tot.gem | 15.592 | 0.790 | | | 1% -level: | 2.32 | (1.85) | | 13 | 10.2596 | | 13 | 12 | |
| Tot.std= | 19.246 | 0.732 | | | 5% -level: | 1.86 | (1.58) | | 2 | | (36L, 61L) | | | |

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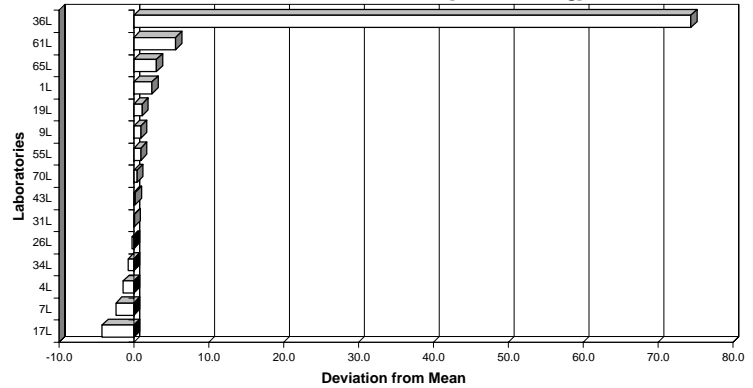
RESULTS: Mean = 10.25960 mS/m at 20 deg

Repeatability variance S2r = 0.37881
 Repeatability std. Sr = 0.61547 --> 6.00% r = 1.7233

Between lab variance S2L = 2.36771
 Reproducibility var. S2R = 2.74652
 Reproducibility std. SR = 1.65726 --> 16.15% R = 4.6403

Remarks: 2 Labs rejected! (36L, 61L)

Soil 5 - EC -- Mean PARM = 10.26 [mS/m at 20 deg]



Annex 4:

Raw data submitted

| Sample: | Sewage Sludge 2 | |
|----------|---------------------|--|
| Element: | pH _{CaCl2} | |
| LAB | PARM | |
| 12L | 7.3 | |
| 12L | 7.3 | |
| 12L | 7.3 | |
| 12L | 7.28 | |
| 12L | 7.28 | |
| 12L | 7.26 | |
| 16L | 7.13 | |
| 16L | 7.12 | |
| 16L | 7.11 | |
| 16L | 7.11 | |
| 16L | 7.1 | |
| 16L | 7.09 | |
| 22L | 7.06 | |
| 22L | 7 | |
| 22L | 7 | |
| 31L | 7.21 | |
| 31L | 7.19 | |
| 31L | 7.19 | |
| 31L | 7.18 | |
| 31L | 7.18 | |
| 31L | 7.17 | |
| 34L | 7.07 | |
| 34L | 7.06 | |
| 34L | 7.05 | |
| 34L | 7.04 | |
| 34L | 7.01 | |
| 34L | 6.99 | |
| 35L | 7.23 | |
| 35L | 7.15 | |
| 35L | 7.12 | |
| 35L | 7.1 | |
| 35L | 7.1 | |
| 35L | 7.1 | |
| 36L | 6.96 | |
| 36L | 6.96 | |
| 36L | 6.93 | |
| 40L | 7.09 | |
| 40L | 7.09 | |
| 40L | 7.08 | |
| 40L | 7.07 | |
| 40L | 7.06 | |
| 40L | 7.06 | |
| 55L | 7.1 | |
| 55L | 7.1 | |
| 55L | 7.1 | |
| 55L | 7.1 | |
| 55L | 7.1 | |
| 55L | 7.1 | |
| 65L | 6.6 | |
| 65L | 6.5 | |
| 65L | 6.5 | |
| 9L | 7.59 | |
| 9L | 7.58 | |
| 9L | 7.58 | |
| 9L | 7.55 | |
| 9L | 7.55 | |
| 9L | 7.52 | |

| Sample: | Soil 4 | |
|----------|---------------------|--|
| Element: | pH _{CaCl2} | |
| LAB | PARM | |
| 12L | 6.71 | |
| 12L | 6.69 | |
| 12L | 6.69 | |
| 12L | 6.69 | |
| 16L | 6.37 | |
| 16L | 6.36 | |
| 16L | 6.36 | |
| 16L | 6.35 | |
| 16L | 6.31 | |
| 16L | 6.3 | |
| 22L | 6.4 | |
| 22L | 6.3 | |
| 22L | 6.3 | |
| 30L | 6.3 | |
| 30L | 6.27 | |
| 30L | 6.26 | |
| 30L | 6.26 | |
| 31L | 6.64 | |
| 31L | 6.63 | |
| 31L | 6.62 | |
| 31L | 6.62 | |
| 31L | 6.62 | |
| 34L | 6.37 | |
| 34L | 6.35 | |
| 34L | 6.35 | |
| 34L | 6.35 | |
| 34L | 6.34 | |
| 34L | 6.33 | |
| 34L | 6.33 | |
| 35L | 6.54 | |
| 35L | 6.52 | |
| 35L | 6.43 | |
| 35L | 6.42 | |
| 35L | 6.41 | |
| 35L | 6.39 | |
| 36L | 6.56 | |
| 36L | 6.51 | |
| 36L | 6.5 | |
| 40L | 6.45 | |
| 40L | 6.41 | |
| 40L | 6.41 | |
| 40L | 6.4 | |
| 40L | 6.4 | |
| 40L | 6.4 | |
| 40L | 6.39 | |
| 55L | 6.5 | |
| 55L | 6.5 | |
| 55L | 6.5 | |
| 55L | 6.5 | |
| 55L | 6.5 | |
| 55L | 6.4 | |
| 55L | 6.4 | |
| 65L | 6.8 | |
| 65L | 6.7 | |
| 65L | 6.7 | |
| 9L | 6.67 | |
| 9L | 6.63 | |
| 9L | 6.63 | |
| 9L | 6.58 | |
| 9L | 6.57 | |
| 9L | 6.54 | |

| Sample: | Soil 5 | |
|----------|---------------------|--|
| Element: | pH _{CaCl2} | |
| LAB | PARM | |
| 12L | 7 | |
| 12L | 7 | |
| 12L | 6.99 | |
| 12L | 6.95 | |
| 12L | 6.89 | |
| 12L | 6.81 | |
| 16L | 6.6 | |
| 16L | 6.59 | |
| 16L | 6.57 | |
| 16L | 6.56 | |
| 16L | 6.55 | |
| 16L | 6.52 | |
| 22L | 6.6 | |
| 22L | 6.51 | |
| 22L | 6.5 | |
| 22L | 6.5 | |
| 22L | 6.49 | |
| 31L | 6.77 | |
| 31L | 6.77 | |
| 31L | 6.76 | |
| 31L | 6.76 | |
| 31L | 6.74 | |
| 34L | 6.54 | |
| 34L | 6.54 | |
| 34L | 6.54 | |
| 34L | 6.52 | |
| 34L | 6.52 | |
| 34L | 6.52 | |
| 35L | 6.67 | |
| 35L | 6.64 | |
| 35L | 6.62 | |
| 35L | 6.6 | |
| 35L | 6.6 | |
| 35L | 6.54 | |
| 36L | 6.6 | |
| 36L | 6.59 | |
| 36L | 6.57 | |
| 40L | 6.58 | |
| 40L | 6.56 | |
| 40L | 6.56 | |
| 40L | 6.55 | |
| 40L | 6.54 | |
| 40L | 6.53 | |
| 55L | 6.6 | |
| 55L | 6.6 | |
| 55L | 6.6 | |
| 55L | 6.6 | |
| 55L | 6.6 | |
| 55L | 6.6 | |
| 55L | 6.5 | |
| 9L | 6.82 | |
| 9L | 6.78 | |
| 9L | 6.78 | |
| 9L | 6.69 | |
| 9L | 6.67 | |
| 9L | 6.63 | |

| Sample: Element: | <i>Compost 1</i> <i>pH aq</i> | |
|---------------------|----------------------------------|-------------|
| | <i>LAB</i> | <i>PARM</i> |
| | 12L | 8.17 |
| | 12L | 8.17 |
| | 12L | 8.16 |
| | 12L | 8.1 |
| | 12L | 8.09 |
| | 12L | 5.02 |
| | 16L | 8.16 |
| | 16L | 8.15 |
| | 16L | 8.15 |
| | 16L | 8.15 |
| | 16L | 8.14 |
| | 16L | 8.14 |
| | 1L | 8.21 |
| | 1L | 8.2 |
| | 21L | 8.13 |
| | 21L | 8.11 |
| | 21L | 8.09 |
| | 21L | 8.06 |
| | 21L | 8.06 |
| | 21L | 8.04 |
| | 22L | 8.18 |
| | 22L | 8.16 |
| | 22L | 8.1 |
| | 22L | 8.09 |
| | 22L | 8.09 |
| | 22L | 8.06 |
| | 26L | 8.28 |
| | 26L | 8.26 |
| | 26L | 8.24 |
| | 26L | 8.21 |
| | 26L | 8.17 |
| | 26L | 8.1 |
| | 29L | 8.16 |
| | 29L | 8.15 |
| | 29L | 8.1 |
| | 29L | 8.07 |
| | 30L | 8.07 |
| | 30L | 8.07 |
| | 30L | 8.04 |

| Sample: Element: | <i>Compost 1</i> <i>pH aq</i> | |
|---------------------|----------------------------------|-------------|
| | <i>LAB</i> | <i>PARM</i> |
| | 31L | 8.16 |
| | 31L | 8.15 |
| | 31L | 8.13 |
| | 31L | 8.12 |
| | 31L | 8.1 |
| | 34L | 8.05 |
| | 34L | 8.05 |
| | 34L | 8.03 |
| | 34L | 8.02 |
| | 34L | 7.97 |
| | 35L | 8.45 |
| | 35L | 8.34 |
| | 35L | 8.34 |
| | 35L | 8.32 |
| | 35L | 8.26 |
| | 35L | 6.6 |
| | 36L | 7.96 |
| | 36L | 7.95 |
| | 36L | 7.93 |
| | 40L | 8.09 |
| | 40L | 8.07 |
| | 40L | 8.04 |
| | 40L | 8.02 |
| | 40L | 8 |
| | 40L | 8 |
| | 43L | 8.3 |
| | 43L | 8.3 |
| | 43L | 8.2 |
| | 43L | 8.2 |
| | 44L | 8.19 |
| | 44L | 8.16 |
| | 44L | 8.15 |
| | 44L | 8.13 |
| | 55L | 8.2 |
| | 55L | 8.1 |
| | 55L | 8.1 |
| | 55L | 8.1 |
| | 55L | 8.1 |
| | 55L | 8.1 |
| | 56L | 8.28 |
| | 56L | 8.25 |
| | 56L | 8.23 |
| | 56L | 8.19 |
| | 56L | 8.15 |
| | 56L | 8.15 |

| Sample: | Compost 2 | |
|----------|-----------|------|
| Element: | pH aq | |
| | LAB | PARM |
| 12L | | 8.27 |
| 12L | | 8.25 |
| 12L | | 8.23 |
| 12L | | 8.23 |
| 12L | | 8.21 |
| 12L | | 8.13 |
| 16L | | 8.45 |
| 16L | | 8.44 |
| 16L | | 8.44 |
| 16L | | 8.42 |
| 16L | | 8.42 |
| 16L | | 8.41 |
| 1L | | 8.58 |
| 1L | | 8.54 |
| 1L | | 8.53 |
| 21L | | 8.4 |
| 21L | | 8.37 |
| 21L | | 8.37 |
| 21L | | 8.33 |
| 21L | | 8.32 |
| 21L | | 8.32 |
| 22L | | 8.6 |
| 22L | | 8.49 |
| 22L | | 8.45 |
| 22L | | 8.42 |
| 22L | | 8.35 |
| 22L | | 8.34 |
| 26L | | 8.26 |
| 26L | | 8.23 |
| 26L | | 8.21 |
| 26L | | 8.15 |
| 26L | | 8.14 |
| 29L | | 6.79 |
| 29L | | 6.76 |
| 29L | | 6.76 |
| 29L | | 6.74 |

| Sample: | Compost 2 | |
|----------|-----------|------|
| Element: | pH aq | |
| | LAB | PARM |
| 31L | | 8.47 |
| 31L | | 8.43 |
| 31L | | 8.41 |
| 31L | | 8.41 |
| 31L | | 8.38 |
| 31L | | 8.35 |
| 34L | | 8.36 |
| 34L | | 8.36 |
| 34L | | 8.33 |
| 34L | | 8.33 |
| 34L | | 8.31 |
| 34L | | 8.27 |
| 35L | | 7.44 |
| 35L | | 6.71 |
| 35L | | 6.68 |
| 35L | | 6.65 |
| 35L | | 6.64 |
| 35L | | 6.61 |
| 36L | | 8.04 |
| 36L | | 8.04 |
| 36L | | 8.04 |
| 40L | | 8.29 |
| 40L | | 8.27 |
| 40L | | 8.26 |
| 40L | | 8.25 |
| 40L | | 8.24 |
| 40L | | 8.22 |
| 43L | | 8.7 |
| 43L | | 8.6 |
| 43L | | 8.6 |
| 43L | | 8.5 |
| 44L | | 8.55 |
| 44L | | 8.51 |
| 44L | | 8.5 |
| 44L | | 8.47 |
| 55L | | 8.5 |
| 55L | | 8.5 |
| 55L | | 8.4 |
| 55L | | 8.4 |
| 55L | | 8.4 |
| 55L | | 8.4 |
| 56L | | 8.51 |
| 56L | | 8.5 |
| 56L | | 8.49 |
| 56L | | 8.48 |
| 56L | | 8.45 |
| 56L | | 8.41 |

| Sample: Element: | Sewage Sludge 1 pH aq | |
|---------------------|--------------------------|------|
| | LAB | PARM |
| 16L | | 6.71 |
| 16L | | 6.7 |
| 16L | | 6.69 |
| 16L | | 6.69 |
| 16L | | 6.68 |
| 16L | | 6.66 |
| 1L | | 6.79 |
| 1L | | 6.78 |
| 21L | | 6.71 |
| 21L | | 6.68 |
| 21L | | 6.67 |
| 21L | | 6.66 |
| 21L | | 6.64 |
| 21L | | 6.64 |
| 22L | | 6.7 |
| 22L | | 6.7 |
| 22L | | 6.6 |
| 22L | | 6.6 |
| 26L | | 6.86 |
| 26L | | 6.85 |
| 26L | | 6.85 |
| 26L | | 6.84 |
| 26L | | 6.82 |
| 26L | | 6.72 |

| Sample: Element: | Sewage Sludge 1 pH aq | |
|---------------------|--------------------------|------|
| | LAB | PARM |
| 31L | | 6.64 |
| 31L | | 6.6 |
| 31L | | 6.6 |
| 31L | | 6.59 |
| 31L | | 6.58 |
| 34L | | 6.67 |
| 34L | | 6.66 |
| 34L | | 6.66 |
| 34L | | 6.66 |
| 34L | | 6.64 |
| 34L | | 6.63 |
| 35L | | 7.5 |
| 35L | | 7.47 |
| 35L | | 7.45 |
| 35L | | 7.44 |
| 35L | | 7.34 |
| 35L | | 6.98 |
| 36L | | 6.67 |
| 36L | | 6.66 |
| 36L | | 6.66 |
| 40L | | 6.78 |
| 40L | | 6.77 |
| 40L | | 6.74 |
| 40L | | 6.74 |
| 40L | | 6.74 |
| 40L | | 6.73 |
| 43L | | 6.8 |
| 43L | | 6.7 |
| 43L | | 6.7 |
| 44L | | 6.96 |
| 44L | | 6.93 |
| 44L | | 6.8 |
| 44L | | 6.79 |
| 44L | | 6.78 |
| 55L | | 6.8 |
| 55L | | 6.8 |
| 55L | | 6.8 |
| 55L | | 6.7 |
| 55L | | 6.7 |
| 55L | | 6.7 |
| 56L | | 6.81 |
| 56L | | 6.77 |
| 56L | | 6.77 |
| 56L | | 6.75 |
| 56L | | 6.74 |

| Sample: Element: | Sewage Sludge 2 pH aq | |
|---------------------|--------------------------|------|
| | LAB | PARM |
| 12L | | 7.5 |
| 12L | | 7.49 |
| 12L | | 7.49 |
| 12L | | 7.47 |
| 12L | | 7.45 |
| 12L | | 7.4 |
| 16L | | 7.61 |
| 16L | | 7.6 |
| 16L | | 7.58 |
| 16L | | 7.58 |
| 16L | | 7.57 |
| 16L | | 7.56 |
| 1L | | 7.61 |
| 1L | | 7.6 |
| 21L | | 7.4 |
| 21L | | 7.37 |
| 21L | | 7.34 |
| 21L | | 7.3 |
| 21L | | 7.25 |
| 21L | | 7.24 |
| 22L | | 7.5 |
| 22L | | 7.5 |
| 26L | | 7.53 |
| 26L | | 7.52 |
| 26L | | 7.5 |
| 26L | | 7.49 |
| 26L | | 7.47 |

| Sample: Element: | Sewage Sludge 2 pH aq | |
|---------------------|--------------------------|------|
| | LAB | PARM |
| 31L | | 7.42 |
| 31L | | 7.41 |
| 31L | | 7.4 |
| 31L | | 7.39 |
| 31L | | 7.38 |
| 34L | | 7.49 |
| 34L | | 7.48 |
| 34L | | 7.47 |
| 34L | | 7.47 |
| 34L | | 7.43 |
| 34L | | 7.42 |
| 35L | | 70.1 |
| 35L | | 7.12 |
| 35L | | 7.04 |
| 35L | | 7.02 |
| 35L | | 7.02 |
| 35L | | 7.01 |
| 36L | | 7.24 |
| 36L | | 7.22 |
| 36L | | 7.19 |
| 40L | | 7.54 |
| 40L | | 7.54 |
| 40L | | 7.53 |
| 40L | | 7.53 |
| 40L | | 7.52 |
| 40L | | 7.52 |
| 43L | | 7.8 |
| 43L | | 7.7 |
| 43L | | 7.7 |
| 44L | | 7.58 |
| 44L | | 7.56 |
| 44L | | 7.56 |
| 44L | | 7.55 |
| 44L | | 7.55 |
| 55L | | 7.6 |
| 55L | | 7.6 |
| 55L | | 7.6 |
| 55L | | 7.6 |
| 55L | | 7.6 |
| 55L | | 7.6 |
| 56L | | 7.5 |
| 56L | | 7.5 |
| 56L | | 7.47 |
| 56L | | 7.46 |
| 56L | | 7.45 |
| 56L | | 7.43 |

| Sample: Element: | Soil 4 pH aq |
|---------------------|-----------------|
| LAB | PARM |
| 12L | 7.21 |
| 12L | 7.2 |
| 12L | 7.17 |
| 12L | 7.15 |
| 12L | 7.15 |
| 12L | 7.14 |
| 16L | 6.94 |
| 16L | 6.92 |
| 16L | 6.91 |
| 16L | 6.91 |
| 16L | 6.9 |
| 16L | 6.9 |
| 1L | 7.36 |
| 1L | 7.28 |
| 1L | 7.24 |
| 22L | 7.00 |
| 22L | 7.00 |
| 26L | 6.17 |
| 26L | 6.15 |
| 26L | 6.15 |
| 26L | 6.14 |
| 26L | 5.79 |
| 30L | 6.98 |
| 30L | 6.87 |
| 30L | 6.85 |
| 30L | 6.78 |

| Sample: Element: | Soil 4 pH aq |
|---------------------|-----------------|
| LAB | PARM |
| 31L | 7.22 |
| 31L | 7.2 |
| 31L | 7.2 |
| 31L | 7.16 |
| 34L | 6.7 |
| 34L | 6.68 |
| 34L | 6.66 |
| 34L | 6.66 |
| 34L | 6.66 |
| 34L | 6.56 |
| 35L | 7.22 |
| 35L | 7.21 |
| 35L | 7.18 |
| 35L | 7.15 |
| 35L | 7.13 |
| 36L | 7.19 |
| 36L | 7.16 |
| 36L | 7.16 |
| 40L | 6.92 |
| 40L | 6.92 |
| 40L | 6.91 |
| 40L | 6.91 |
| 40L | 6.86 |
| 40L | 6.85 |
| 43L | 7.3 |
| 43L | 7.3 |
| 43L | 7.2 |
| 44L | 7.08 |
| 44L | 7.07 |
| 44L | 7.06 |
| 44L | 7.05 |
| 44L | 7.04 |
| 44L | 7.02 |
| 55L | 7.1 |
| 55L | 7.1 |
| 55L | 7.1 |
| 55L | 7.00 |
| 55L | 7.00 |
| 55L | 7.00 |
| 56L | 7.04 |
| 56L | 7.04 |
| 56L | 7.02 |
| 56L | 7.01 |
| 56L | 6.98 |
| 56L | 6.93 |

| Sample: Element: | Soil 5 pH aq |
|---------------------|-----------------|
| LAB | PARM |
| 12L | 7.33 |
| 12L | 7.33 |
| 12L | 7.32 |
| 12L | 7.32 |
| 12L | 7.32 |
| 12L | 7.31 |
| 16L | 7.04 |
| 16L | 7.02 |
| 16L | 7.02 |
| 16L | 7.02 |
| 16L | 7.02 |
| 16L | 6.99 |
| 1L | 7.32 |
| 1L | 7.31 |
| 1L | 7.29 |
| 22L | 7.1 |
| 22L | 7.1 |
| 26L | 6.34 |
| 26L | 6.33 |
| 26L | 6.32 |
| 26L | 6.3 |
| 26L | 6.3 |
| 26L | 6.27 |

| Sample: Element: | Soil 5 pH aq |
|---------------------|-----------------|
| LAB | PARM |
| 31L | 7.18 |
| 31L | 7.17 |
| 31L | 7.13 |
| 31L | 7.12 |
| 31L | 7.12 |
| 34L | 6.91 |
| 34L | 6.87 |
| 34L | 6.84 |
| 34L | 6.84 |
| 34L | 6.83 |
| 34L | 6.83 |
| 36L | 7.17 |
| 36L | 7.17 |
| 36L | 7.16 |
| 40L | 7.01 |
| 40L | 7.01 |
| 40L | 7.01 |
| 40L | 7.01 |
| 40L | 7 |
| 40L | 6.99 |
| 43L | 7.3 |
| 43L | 7.3 |
| 43L | 7.2 |
| 43L | 7.2 |
| 44L | 7.09 |
| 44L | 7.09 |
| 44L | 7.06 |
| 44L | 7.06 |
| 55L | 7.2 |
| 55L | 7.2 |
| 55L | 7.1 |
| 55L | 7.1 |
| 55L | 7.1 |
| 55L | 7 |
| 56L | 7.1 |
| 56L | 7.1 |
| 56L | 7.04 |
| 56L | 7.04 |
| 56L | 7.02 |
| 56L | 7.01 |

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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 specifications 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 pH in soils, sludge and treated bio-waste. 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.

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