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# Project HORIZONTAL Validation Report on total organic carbon

## Validation of a horizontal standard for the determination of total organic carbon (TOC) by dry combustion in soils, sludge and treated biowaste in a European Intercomparison Exercise

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EUR 23012 EN - 2007

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JRC 40201

EUR 23012 EN
ISBN 978-92-79-07412-7
ISSN 1018-5593
DOI 10.2788/47924

Luxembourg: Office for Official Publications of the European Communities



*Printed in Luxembourg*

# Project HORIZONTAL Validation Report

# Validation of a horizontal standard for the determination of total organic carbon (TOC) by dry combustion in soils, sludge and treated biowaste in a European Intercomparison Exercise

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Summary

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

This report summarises the work performed in a validation study for the draft standard for the determination of total organic carbon (TOC) in soil, sludge and treated bio-waste. It further explains the underlying statistical concept for the calculation of reproducibility and repeatability from intercomparison data. In addition all single values, results of the statistical evaluation as well as background information on the validation materials used are described and explained.

# Abbreviations

Throughout this report the following abbreviations are used:

| | |
|---|---|
| ANOVA | Analysis of variances |
| CAS | Chemical Abstracts System |
| CEN | Comitteé Européen de Normalisation |
| DG | Directorate General |
| ECN | Energy Research Centre for the Netherlands |
| EU | European Union |
| IES | Institute for Environment and Sustainability |
| IT | Information Technology |
| ISO | International Organization for Standardisation |
| JRC | Joint Research Centre |
| MILC | Measure Interlaboratory Comparison – (online data submission system) |
| p | Number of labs |
| r | Repeatability limit |
| R | Reproducibility limit |
| $s_r$ | Repeatability standard deviation |
| $s_R$ | Reproducibility standard deviation |
| TC | Technical Committee |
| TOC | Total Organic Carbon |

# Table of Contents

## *Introduction to the validation project*

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

- 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 total organic carbon (TOC) are described hereafter.

## 1.1. Statistical concept underlying the validation

According to the requirements of the work package concerning data handling and interpretation of the project HORIZONTAL the respective validation intercomparisons should be evaluated according to ISO 5725-2:1994. In particular repeatability and reproducibility of the draft standard should be determined. 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 work program the quantification of term *e* is explicitly asked for (i.e. repeatability and reproducibility). The repeatability variance is measured directly as the variance of the error term *e*, but the reproducibility depends on the sum of the repeatability variance and the between-laboratory variance:

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

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

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

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

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

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

## 1.1.2 Requirements for precision experiment

**Layout of the experiment**

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

Each laboratory received two bottles of each material and was requested to perform 6 independent analyses per material[1] (3 per bottle) using the draft standard method. The 6 analyses per material should be carried out under repeatability conditions (i.e. same operator[2], same equipment, within a short period of time). As far as possible, also the different materials should be measured under repeatability conditions; however, changes of e.g. operator or equipment was permitted, but had to be reported. Likewise, the different materials could 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 had 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 was set and should 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. The draft standard was made available to the laboratories on the Project Horizontal homepage. Each laboratory was requested to provide the signed statements on cooperation as given in Annex 1.

**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 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.

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[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.

### 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 TOC*

### 1.2.1 Samples dispatched for the validation of TOC

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

- Compost 1 — A compost material from Vienna
- Compost 2 — A compost material from Germany
- Sewage Sludge 1 — A mixed sewage sludge from Essen, Germany
- Sewage Sludge 2 — A mixed municipal 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/TOC_standard_for_validation.pdf

### 1.2.3 Analytical program

Two bottles had to be analysed for each of the six materials and each bottle had to be analysed 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 dry matter correction was free for each participant.

### 1.2.4 Timing and Submission of data

Dispatch of samples was done on the 18$^{th}$ of October 2006. For users of the online data submission system (MILC), user registration was possible from 14$^{th}$ of November 2006 with opening of the MILC Data Submission on 1$^{st}$ of December 2006. The deadline for submission of results has been set for TOC to the 31st of January 2007. Alternatively the participants were allowed to submit data electronically as Excel sheet using Email.

All data were treated in a confidential way. Any presentation hereafter refers 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 provided 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 TOC 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
  - Shimadzu Europa GmbH
  - Umweltbundesamt
  - University of Natural resources and Applied Life Sciences
- Belgium
  - VITO
- France
  - INERIS
  - CEMAGREF - UR QELY
  - INRA - Laboratoire d'Analyse des Sols
- Germany
  - ALBO-tec Technologiezentrum Für Analytik und Bodenmechanik GmbH
- Norway
  - Norwegian Institute for Water Research
- The Netherlands
  - ALCONTROL BV
  - Analytico Milieu B. V.

## 1.3 Summary results and derived performance characteristics

The result of the various statistical evaluation according to ISO 5725-2 including outlier tests, calculation of repeatability and reproducibility standard deviations can be found in Annex 3. 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.

The calculated average values, the repeatability standard deviation ($s_r$) and the reproducibility standard deviation ($s_R$) are given in Table 1.

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 the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures. The repeatability limit was calculated using the relationship: $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$ with the critical range factor f = 2.

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

**Table 1 - Results of the interlaboratory comparison studies of the determination of total organic carbon (TOC) by dry combustion in treated biowaste, sludge and soil. All concentrations are expressed in g/kg dry matter.**

| Matrix | Parameter | Mean | sr | sR | r | R | p | Outliers | Total number of data | No of LOD |
|---|---|---|---|---|---|---|---|---|---|---|
| Sludge 1 | TOC | 272 | 1.7% | 5.9% | 13 | 45 | 13 | 2 | 62 | 0 |
| Sludge 2 | TOC | 220 | 2.1% | 5.0% | 13 | 31 | 12 | 2 | 58 | 0 |
| Compost 1 | TOC | 246 | 2.5% | 4.3% | 17 | 30 | 13 | 2 | 58 | 0 |
| Compost 2 | TOC | 154 | 5.9% | 9.6% | 25 | 41 | 13 | 2 | 64 | 0 |
| Soil 4 | TOC | 16.4 | 2.6% | 5.2% | 1.2 | 2.4 | 12 | 3 | 62 | 0 |
| Soil 5 | TOC | 21.0 | 5.2% | 8.2% | 3.0 | 4.8 | 10 | 3 | 54 | 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

# Model questionnaire to be filled by the participating laboratories

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

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

Title of measurement method (copy attached):

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

Yes ☐ No ☐

As participant we understand that:

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

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

Comments:

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

## Annex 2:

## Report on the validation materials used

## Abstract

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

- Four sludge materials from Düsseldorf, Germany,
- An agricultural soil material from Reading, United Kingdom;
- A compost material from Vienna, Austria;
- A compost material from Korschenbroich, Germany;
- A sludge-amended, agricultural soil from Pavia Province, Italy;
- A sludge-amended soil from Barcelona, Spain
- A sludge-amended soil from Essen, Germany
- A long-term sludge exposed soil from Hohenheim, Germany

## List of Abbreviations

Throughout this report the following abbreviations are used.

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

# 1 Introduction

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

- Four sludge materials from Düsseldorf, Germany,
- An agricultural soil material from Reading, United Kingdom;
- A compost material from Vienna, Austria;
- A compost material from Korschenbroich, Germany;
- A sludge-amended, agricultural soil from Pavia Province, Italy;
- A sludge-amended soil from Barcelona, Spain
- A sludge-amended soil from Essen, Germany
- A long-term sludge exposed soil from Hohenheim, Germany

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

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

This work is gratefully acknowledged.

# 2 Overview on property values

## *2.1 Sludge materials from Düsseldorf, Germany*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

### 2.2 Agricultural soil material from Reading, United Kingdom

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

| ***Sample*** | ***Hg*** *µg/g* |
|---|---|
| Soil 3 (Reading) | 0.12 |

### 2.3 Compost from Vienna, Austria

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

| ***Sample*** | ***Hg*** *µg/g* |
|---|---|
| Compost 1 (Vienna) | 0.17 |

### 2.4 Agricultural soil, sludge amended soil from Pavia, Italy

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

*Table 23 – Analytical data on Pavia soil*

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

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

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

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

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

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

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

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

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

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

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

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

| ***Sample*** | ***Hg*** *µg/g* |
|---|---|
| Soil 5 (Pavia) | 0.06 |

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

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

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

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

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

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

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

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

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

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

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

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

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

### 2.6 Sludge amended soil from Essen, Germany

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

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

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

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

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

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

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

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

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

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

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

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

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

| Sample | Hg µg/g |
|---|---|
| Soil 4 (Essen) | 0.04 |

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

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

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

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

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

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

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

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

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

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

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

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

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

## Annex 3:

## Statistical calculations

Unit: g/kg

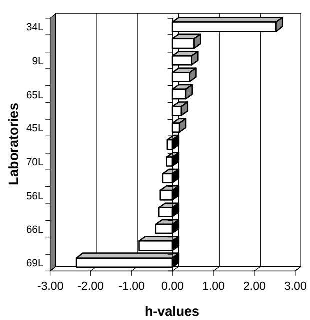
**Mandel's k statistics (Compost 1 - TOC)**
**Mandel's h statistics (Compost 1 - TOC)**

Compost 1 - TOC -- Mean PARM = 246.3 [g/kg]

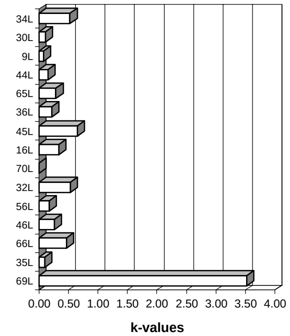
*Sample:* **Compost 1**

*Element:* **TOC**

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




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




| | *General calc.parm.* |
|---|---|
| T1= | 1.42643E+04 |
| T2= | 3.51264E+06 |
| T3= | 58 |
| T4= | 292 |
| T5= | 1.6501E+03 |
| n= | variabel |
| p= | 13 |
| N-table= | *5* |

Mandel's statistics — End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *\vX > AvST+2std* | *AvX < AvST-2std* | *PARM* | *Stdev* | | *Rej.labs* | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 69L | 196.0000 | 64.265 | 4 | !! | -2.35 | 3.52 | !! | Fail | - | - | | ,69L | - | - | | -50.27 |
| 35L | 229.1667 | 1.674 | 6 | | -0.81 | 0.09 | | | | 229.1667 | 1.6741 | | | 6 | 5 | -17.10 |
| 66L | 237.7425 | 8.552 | 4 | | -0.41 | 0.47 | | | | 237.7425 | 8.5518 | | | 4 | 3 | -8.53 |
| 46L | 239.5000 | 4.796 | 4 | | -0.33 | 0.26 | | | | 239.5000 | 4.7958 | | | 4 | 3 | -6.77 |
| 56L | 240.1227 | 3.133 | 6 | | -0.30 | 0.17 | | | | 240.1227 | 3.1330 | | | 6 | 5 | -6.14 |
| 32L | 241.5000 | 9.713 | 4 | | -0.24 | 0.53 | | | | 241.5000 | 9.7125 | | | 4 | 3 | -4.77 |
| 70L | 243.5100 | - | 1 | | -0.14 | | | | | 243.5100 | - | | | 1 | | -2.76 |
| 16L | 244.0000 | 6.033 | 6 | | -0.12 | 0.33 | | | | 244.0000 | 6.0332 | | | 6 | 5 | -2.27 |
| 45L | 250.3800 | 11.759 | 6 | | 0.18 | 0.64 | | | | 250.3800 | 11.7589 | | | 6 | 5 | 4.11 |
| 36L | 251.4416 | 3.994 | 3 | | 0.23 | 0.22 | | | | 251.4416 | 3.9937 | | | 3 | 2 | 5.17 |
| 65L | 253.7333 | 5.216 | 3 | | 0.33 | 0.29 | | | | 253.7333 | 5.2157 | | | 3 | 2 | 7.47 |
| 44L | 255.5667 | 2.783 | 6 | | 0.42 | 0.15 | | | | 255.5667 | 2.7826 | | | 6 | 5 | 9.30 |
| 9L | 256.8167 | 1.356 | 6 | | 0.48 | 0.07 | | | | 256.8167 | 1.3556 | | | 6 | 5 | 10.55 |
| 30L | 258.0000 | 2.000 | 3 | | 0.53 | 0.11 | | | | 258.0000 | 2.0000 | | | 3 | 2 | 11.73 |
| 34L | 301.3269 | 9.580 | 6 | !! | 2.55 | 0.53 | | Fail | - | - | | ,34L | - | - | | 55.06 |
| *Tot.gem* | 246.587 | 9.632 g/kg | | *1%-level:* | 2.32 | (1.76) | | | 13 | *246.2677* | | (34L ,69L) | | 13 | 12 | |
| *Tot.std=* | 21.507 | 16.070 | | *5%-level:* | 1.86 | (1.52) | | | 2 | | | | | | | |

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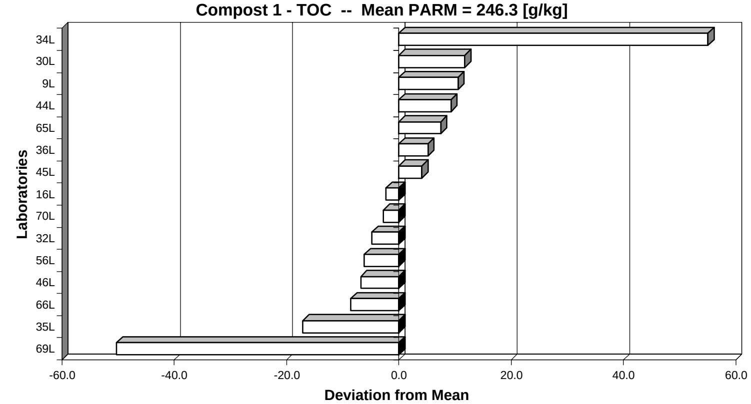
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **246.26770** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 36.66797 | | | | |
| **Repeatability std.** | **Sr =** | **6.05541** | --> | 2.46% | **r =** | 16.9551 |
| **Between lab variance** | **S2L =** | 77.15586 | | | | |
| **Reproducibility var.** | **S2R =** | 113.82383 | | | | |
| **Reproducibility std.** | **SR =** | **10.66882** | --> | 4.33% | **R =** | 29.8727 |
| **Remarks:** | **2 Labs rejected! (34L ,69L)** | | | | | |

**Compost 1 - TOC -- Mean PARM = 246.3 [g/kg]**



Unit: g/kg

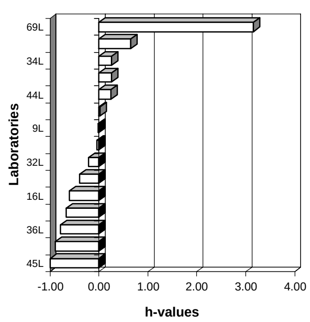
**Mandel's k statistics (Compost 2 - TOC)**
**Mandel's h statistics (Compost 2 - TOC)**

Compost 2 - TOC -- Mean PARM = 154.2 [g/kg]

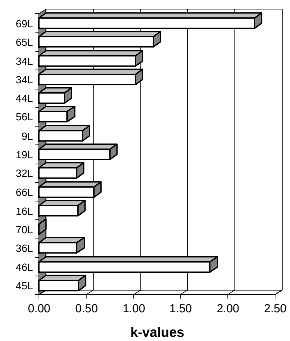
*Sample:* **Compost 2**

*Element:* **TOC**

**Mandel's h statistics (Compost 2 - TOC)**




**Mandel's k statistics (Compost 2 - TOC)**




| | *General calc.parm.* |
|---|---|
| T1= | 9.94897E+03 |
| T2= | 1.55556E+06 |
| T3= | 64 |
| T4= | 346 |
| T5= | 4.1738E+03 |
| n= | variabel |
| p= | 13 |
| N-table= | *5* |

Mandel's statistics / End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *\vX > AvST+2std* | *AvX < AvST-2std* | | *PARM* | *Stdev* | *Rej.labs* | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 45L | 132.6733 | 5.665 | 6 | | -1.00 | 0.42 | | | Fail | | 132.6733 | 5.6646 | | 6 | 5 | -21.50 |
| 46L | 135.5000 | 24.365 | 4 | | -0.89 | 1.81 | ! | | Fail | - | - | ,46L | - | - | | -18.67 |
| 36L | 138.1949 | 5.407 | 3 | | -0.78 | 0.40 | | | Fail | | 138.1949 | 5.4070 | | 3 | 2 | -15.98 |
| 70L | 141.4120 | - | 1 | | -0.66 | | | | Fail | | 141.4120 - | | | 1 | | -12.76 |
| 16L | 142.8333 | 5.565 | 6 | | -0.61 | 0.41 | | | | | 142.8333 | 5.5648 | | 6 | 5 | -11.34 |
| 66L | 148.5550 | 7.805 | 4 | | -0.39 | 0.58 | | | | | 148.5550 | 7.8047 | | 4 | 3 | -5.62 |
| 32L | 153.2500 | 5.377 | 4 | | -0.21 | 0.40 | | | | | 153.2500 | 5.3774 | | 4 | 3 | -0.92 |
| 19L | 157.6833 | 10.138 | 6 | | -0.04 | 0.75 | | | | | 157.6833 | 10.1381 | | 6 | 5 | 3.51 |
| 9L | 158.2333 | 6.166 | 6 | | -0.02 | 0.46 | | | | | 158.2333 | 6.1659 | | 6 | 5 | 4.06 |
| 56L | 159.4432 | 4.046 | 6 | | 0.03 | 0.30 | | | | | 159.4432 | 4.0463 | | 6 | 5 | 5.27 |
| 44L | 165.3000 | 3.651 | 6 | | 0.25 | 0.27 | | | | | 165.3000 | 3.6508 | | 6 | 5 | 11.13 |
| 34L | 165.5127 | 13.717 | 6 | | 0.26 | 1.02 | | | | | 165.5127 | 13.7168 | | 6 | 5 | 11.34 |
| 34L | 165.5127 | 13.717 | 6 | | 0.26 | 1.02 | | | | | 165.5127 | 13.7168 | | 6 | 5 | 11.34 |
| 65L | 175.6500 | 16.295 | 4 | | 0.65 | 1.21 | | Fail | | | 175.6500 | 16.2949 | | 4 | 3 | 21.48 |
| 69L | 241.5000 | 30.687 | 4 | !! | 3.16 | 2.28 | !! | Fail | | - | - | ,69L | - | - | | 87.33 |
| *Tot.gem* | 158.750 | 10.900 g/kg | | *1%-level:* | 2.32 | (1.76) | | | | 13 | *154.1734* | | (46L ,69L) | 13 | 12 | |
| *Tot.std=* | 26.187 | 8.164 | | *5%-level:* | 1.86 | (1.52) | | | | 2 | | | | | | |

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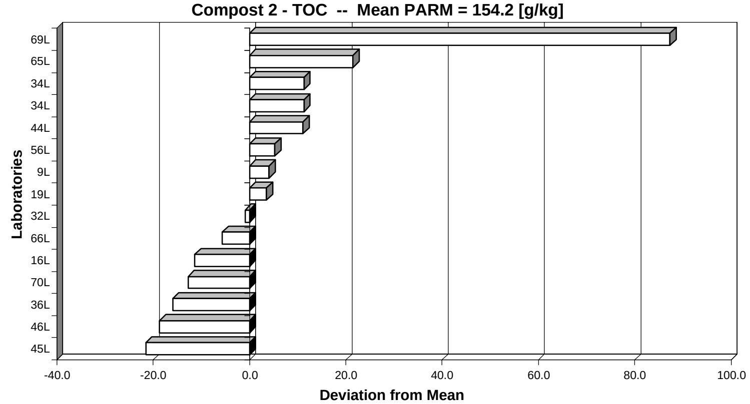
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **154.17337** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 81.83950 | | | | |
| **Repeatability std.** | **Sr =** | **9.04652** | --> | 5.87% | **r =** | 25.3303 |
| **Between lab variance** | **S2L =** | 136.31046 | | | | |
| **Reproducibility var.** | **S2R =** | 218.14995 | | | | |
| **Reproducibility std.** | **SR =** | **14.76990** | --> | 9.58% | **R =** | 41.3557 |
| **Remarks:** | **2 Labs rejected! (46L ,69L)** | | | | | |

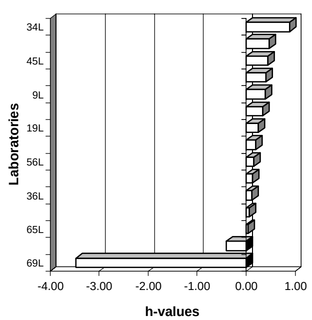
**Compost 2 - TOC -- Mean PARM = 154.2 [g/kg]**



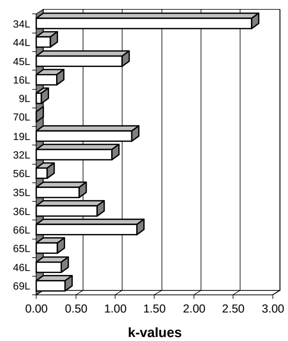
*Sample:* **Sludge 1**

*Element:* **TOC**

**Mandel's h statistics (Sludge 1 - TOC)**




**Mandel's k statistics (Sludge 1 - TOC)**




Unit: g/kg

**Mandel's k statistics (Sludge 1 - TOC)**
**Mandel's h statistics (Sludge 1 - TOC)**

Sludge 1 - TOC -- Mean PARM = 272.1 [g/kg]

| | *General calc.parm.* |
|---|---|
| T1= | 1.69626E+04 |
| T2= | 4.65445E+06 |
| T3= | 62 |
| T4= | 326 |
| T5= | 1.0312E+03 |
| n= | variabel |
| p= | 13 |
| N-table= | *5* |

Mandel's statistics — End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *\vX > AvST+2std* | *AvX < AvST-2std* | *PARM* | *Stdev* | | *Rej.labs* | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 69L | 23.3000 | 2.362 | 4 | !! | -3.47 | 0.37 | | | Fail | - | - | ,69L | - | - | | -248.80 |
| 46L | 231.2500 | 2.062 | 4 | | -0.40 | 0.32 | | | Fail | 231.2500 | 2.0616 | | | 4 | 3 | -40.85 |
| 65L | 261.5500 | 1.708 | 4 | | 0.04 | 0.26 | | | | 261.5500 | 1.7078 | | | 4 | 3 | -10.55 |
| 66L | 262.7200 | 8.268 | 4 | | 0.06 | 1.28 | | | | 262.7200 | 8.2678 | | | 4 | 3 | -9.38 |
| 36L | 266.4041 | 4.976 | 3 | | 0.11 | 0.77 | | | | 266.4041 | 4.9758 | | | 3 | 2 | -5.69 |
| 35L | 267.1500 | 3.533 | 6 | | 0.13 | 0.55 | | | | 267.1500 | 3.5331 | | | 6 | 5 | -4.95 |
| 56L | 269.2298 | 0.874 | 6 | | 0.16 | 0.14 | | Fail | | 269.2298 | 0.8738 | | | 6 | 5 | -2.87 |
| 32L | 271.7500 | 6.185 | 4 | | 0.19 | 0.96 | | Fail | | 271.7500 | 6.1847 | | | 4 | 3 | -0.35 |
| 19L | 275.3333 | 7.819 | 6 | | 0.25 | 1.21 | | Fail | | 275.3333 | 7.8189 | | | 6 | 5 | 3.24 |
| 70L | 281.4600 | - | 1 | | 0.34 | | | Fail | | 281.4600 | - | | | 1 | | 9.36 |
| 9L | 285.3667 | 0.398 | 6 | | 0.39 | 0.06 | | Fail | | 285.3667 | 0.3983 | | | 6 | 5 | 13.27 |
| 16L | 286.0000 | 1.673 | 6 | | 0.40 | 0.26 | | Fail | | 286.0000 | 1.6733 | | | 6 | 5 | 13.90 |
| 45L | 288.4433 | 7.033 | 6 | | 0.44 | 1.09 | | Fail | | 288.4433 | 7.0331 | | | 6 | 5 | 16.35 |
| 44L | 290.6167 | 1.129 | 6 | | 0.47 | 0.17 | | Fail | | 290.6167 | 1.1286 | | | 6 | 5 | 18.52 |
| 34L | 318.6874 | 17.674 | 6 | | 0.89 | 2.74 | !! | Fail | | - | - | ,34L | - | - | | 46.59 |
| *Tot.gem* | 258.617 | 4.692 g/kg | | *1%-level:* | 2.32 | (1.76) | | | 13 | *272.0980* | | (34L ,69L) | | 13 | 12 | |
| *Tot.std=* | 67.800 | 4.610 | | *5%-level:* | 1.86 | (1.52) | | | 2 | | | | | | | |

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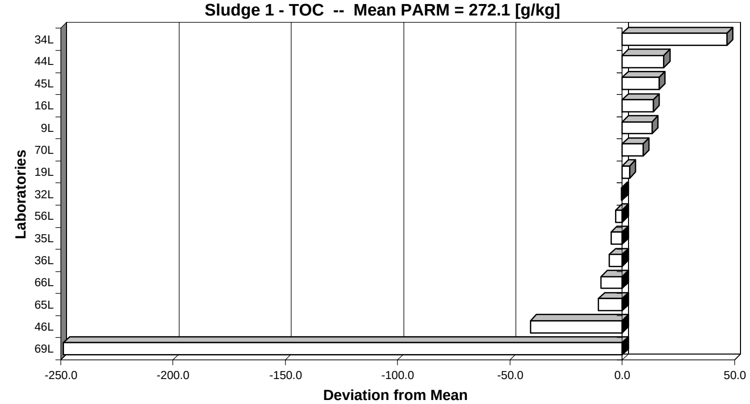
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **272.09799** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 21.04538 | | | | |
| **Repeatability std.** | **Sr =** | **4.58752** | --> | 1.69% | **r =** | 12.8451 |
| **Between lab variance** | **S2L =** | 236.18387 | | | | |
| **Reproducibility var.** | **S2R =** | 257.22925 | | | | |
| **Reproducibility std.** | **SR =** | **16.03837** | --> | 5.89% | **R =** | 44.9074 |
| **Remarks:** | **2 Labs rejected! (34L ,69L)** | | | | | |

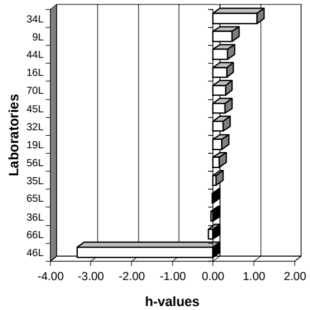
**Sludge 1 - TOC -- Mean PARM = 272.1 [g/kg]**



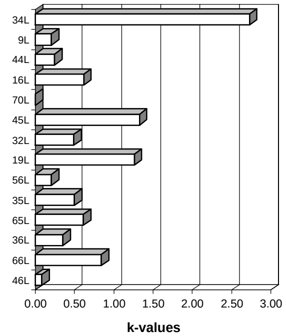
*Sample:* **Sludge 2**

*Element:* **TOC**

**Mandel's h statistics (Sludge 2 - TOC)**




**Mandel's k statistics (Sludge 2 - TOC)**




Unit: g/kg

**Mandel's k statistics (Sludge 2 - TOC)**
**Mandel's h statistics (Sludge 2 - TOC)**

Sludge 2 - TOC -- Mean PARM = 220.7 [g/kg]

| | *General calc.parm.* |
|---|---|
| T1= | 1.28601E+04 |
| T2= | 2.85689E+06 |
| T3= | 58 |
| T4= | 310 |
| T5= | 9.7826E+02 |
| n= | variabel |
| p= | 12 |
| N-table= | *5* |

Mandel's statistics / End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *AvX > AvST+2std* | *AvX < AvST-2std* | *PARM* | *Stdev* | *Rej.labs* | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 46L | 18.2500 | 0.500 | 4 | !! | -3.33 | 0.08 | | | Fail | - | - | ,46L | - | - | -202.44 |
| 66L | 202.9475 | 5.267 | 4 | | -0.12 | 0.84 | | | | 202.9475 | 5.2674 | | 4 | 3 | -17.74 |
| 36L | 207.2609 | 2.222 | 3 | | -0.05 | 0.35 | | | | 207.2609 | 2.2222 | | 3 | 2 | -13.43 |
| 65L | 208.4750 | 3.865 | 4 | | -0.02 | 0.62 | | | | 208.4750 | 3.8647 | | 4 | 3 | -12.21 |
| 35L | 214.0333 | 3.119 | 6 | | 0.07 | 0.50 | | | | 214.0333 | 3.1194 | | 6 | 5 | -6.65 |
| 56L | 218.8021 | 1.282 | 6 | | 0.15 | 0.20 | | | | 218.8021 | 1.2822 | | 6 | 5 | -1.88 |
| 19L | 222.3667 | 7.916 | 6 | | 0.22 | 1.26 | | Fail | | 222.3667 | 7.9157 | | 6 | 5 | 1.68 |
| 32L | 224.2500 | 3.096 | 4 | | 0.25 | 0.49 | | Fail | | 224.2500 | 3.0957 | | 4 | 3 | 3.56 |
| 45L | 226.6600 | 8.322 | 6 | | 0.29 | 1.32 | | Fail | | 226.6600 | 8.3216 | | 6 | 5 | 5.97 |
| 70L | 227.2460 | - | 1 | | 0.30 | | | Fail | | 227.2460 - | | | 1 | | 6.56 |
| 16L | 229.1667 | 3.869 | 6 | | 0.33 | 0.62 | | Fail | | 229.1667 | 3.8687 | | 6 | 5 | 8.48 |
| 44L | 230.2667 | 1.546 | 6 | | 0.35 | 0.25 | | Fail | | 230.2667 | 1.5462 | | 6 | 5 | 9.58 |
| 9L | 236.7667 | 1.296 | 6 | | 0.47 | 0.21 | | Fail | | 236.7667 | 1.2956 | | 6 | 5 | 16.08 |
| 34L | 271.8591 | 17.124 | 6 | | 1.08 | 2.73 | !! | Fail | - | - | | ,34L | - | - | 51.17 |
| *Tot.gem* | 209.882 | 4.571 g/kg | | *1%-level:* | 2.30 | (1.76) | | | 12 | *220.6868* | | (34L ,46L) | 12 | 11 | |
| *Tot.std=* | 57.628 | 4.485 | | *5%-level:* | 1.85 | (1.51) | | | 2 | | | | | | |

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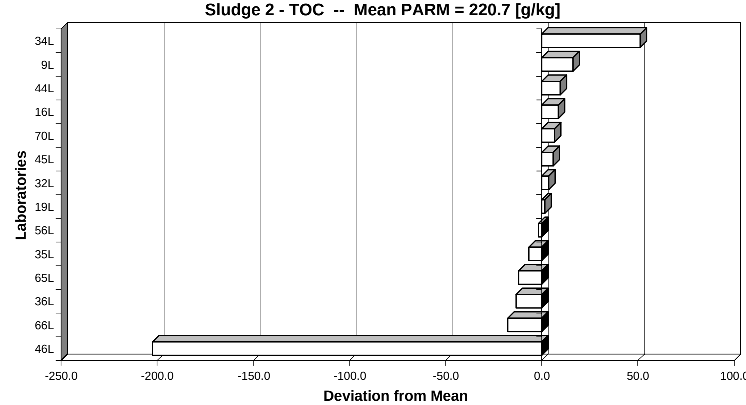
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **220.68679** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 21.26659 | | | | |
| **Repeatability std.** | **Sr =** | **4.61157** | --> | 2.09% | **r =** | 12.9124 |
| **Between lab variance** | **S2L =** | 99.60432 | | | | |
| **Reproducibility var.** | **S2R =** | 120.87091 | | | | |
| **Reproducibility std.** | **SR =** | **10.99413** | --> | 4.98% | **R =** | 30.7836 |
| **Remarks:** | **2 Labs rejected! (34L ,46L)** | | | | | |

**Sludge 2 - TOC -- Mean PARM = 220.7 [g/kg]**



Unit: g/kg

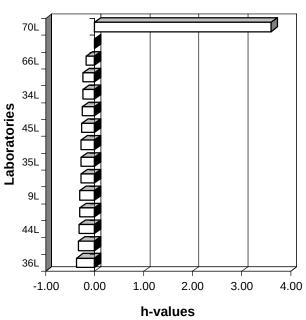
**Mandel's k statistics (Soil 4 - TOC)**
**Mandel's h statistics (Soil 4 - TOC)**

Soil 4 - TOC -- Mean PARM = 16.41 [g/kg]

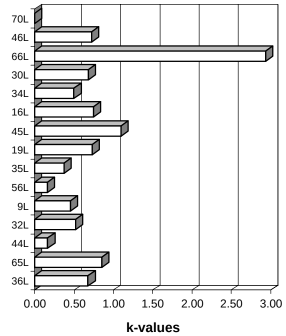
*Sample:* **Soil 4**

*Element:* **TOC**

**Mandel's h statistics (Soil 4 - TOC)**




**Mandel's k statistics (Soil 4 - TOC)**



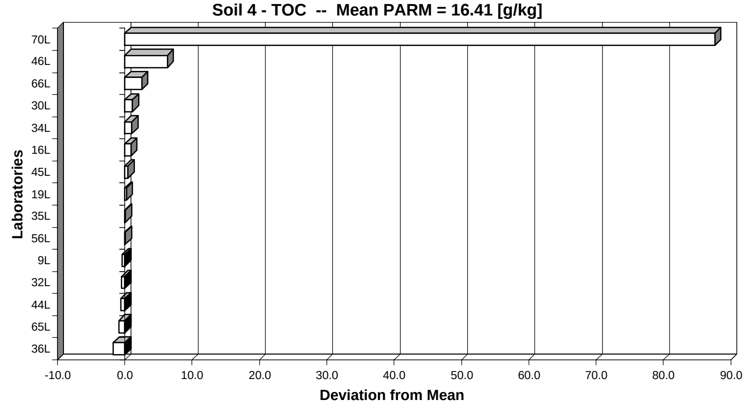

| | |
|---|---|
| | *General calc.parm.* |
| T1= | 1.02171E+03 |
| T2= | 1.68700E+04 |
| T3= | 62 |
| T4= | 338 |
| T5= | 9.4040E+00 |
| n= | variabel |
| p= | 12 |
| N-table= | *5* |

Mandel's statistics — End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *\vX > AvST+2std* | *AvX < AvST-2std* | *PARM* | *Stdev* | *Rej.labs* | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 36L | 14.7161 | 0.470 | 3 | | -0.36 | 0.68 | | Fail | | 14.7161 | 0.4702 | | 3 | 2 | -1.69 |
| 65L | 15.5250 | 0.591 | 4 | | -0.32 | 0.85 | | Fail | | 15.5250 | 0.5909 | | 4 | 3 | -0.88 |
| 44L | 15.7900 | 0.115 | 6 | | -0.31 | 0.17 | | Fail | | 15.7900 | 0.1152 | | 6 | 5 | -0.62 |
| 32L | 15.9250 | 0.359 | 4 | | -0.31 | 0.52 | | Fail | | 15.9250 | 0.3594 | | 4 | 3 | -0.48 |
| 9L | 16.0500 | 0.315 | 6 | | -0.30 | 0.45 | | Fail | | 16.0500 | 0.3146 | | 6 | 5 | -0.36 |
| 56L | 16.4927 | 0.113 | 6 | | -0.28 | 0.16 | | Fail | | 16.4927 | 0.1127 | | 6 | 5 | 0.08 |
| 35L | 16.5000 | 0.261 | 6 | | -0.28 | 0.38 | | Fail | | 16.5000 | 0.2608 | | 6 | 5 | 0.09 |
| 19L | 16.6667 | 0.505 | 6 | | -0.27 | 0.73 | | Fail | | 16.6667 | 0.5046 | | 6 | 5 | 0.26 |
| 45L | 16.8850 | 0.763 | 6 | | -0.26 | 1.10 | | Fail | | 16.8850 | 0.7628 | | 6 | 5 | 0.48 |
| 16L | 17.3333 | 0.516 | 6 | | -0.24 | 0.74 | | Fail | | 17.3333 | 0.5164 | | 6 | 5 | 0.93 |
| 34L | 17.4761 | 0.346 | 6 | | -0.24 | 0.50 | | Fail | | 17.4761 | 0.3462 | | 6 | 5 | 1.07 |
| 30L | 17.5333 | 0.473 | 3 | | -0.24 | 0.68 | | Fail | | 17.5333 | 0.4726 | | 3 | 2 | 1.13 |
| 66L | 18.9375 | 2.035 | 4 | | -0.17 | 2.94 | !! | Fail | | - | - | ,66L | - | - | 2.53 |
| 46L | 22.7500 | 0.500 | 4 | | 0.00 | 0.72 | | | | - | - | ,46L | - | - | 6.34 |
| 70L | 104.0400 | - | 1 | !! | 3.60 | | | Fail | | - | - | ,70L | - | - | 87.63 |
| *Tot.gem* | 22.841 | 0.526 g/kg | | *1%-level:* | 2.32 | (1.76) | | | 12 | *16.4078* | | (46L ,66L,70L) | 12 | 11 | |
| *Tot.std=* | 22.541 | 0.469 | | *5%-level:* | 1.86 | (1.52) | | | 3 | | | | | | |
| | | | | | | | | | 71 | | | | | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **16.40777** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 0.18808 | | | | |
| **Repeatability std.** | **Sr =** | **0.43368** | --> | 2.64% | **r =** | 1.2143 |
| **Between lab variance** | **S2L =** | 0.54747 | | | | |
| **Reproducibility var.** | **S2R =** | 0.73555 | | | | |
| **Reproducibility std.** | **SR =** | **0.85764** | --> | 5.23% | **R =** | 2.4014 |
| **Remarks:** | **3 Labs rejected! (46L ,66L,70L)** | | | | | |

**Soil 4 - TOC -- Mean PARM = 16.41 [g/kg]**



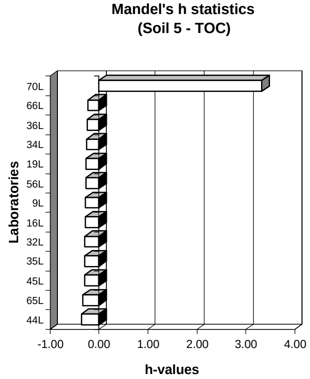
Unit: g/kg

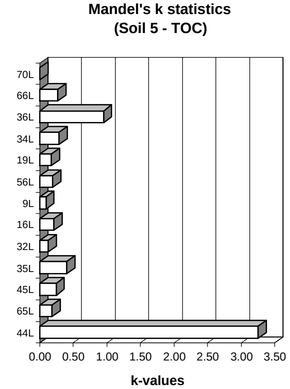
**Mandel's k statistics (Soil 5 - TOC)**
**Mandel's h statistics (Soil 5 - TOC)**

Soil 5 - TOC -- Mean PARM = 21.02 [g/kg]

*Sample:* **Soil 5**

*Element:* **TOC**



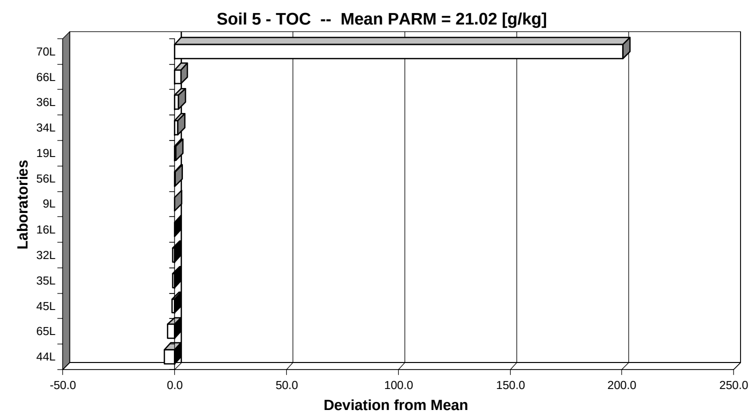




| | *General calc.parm.* |
|---|---|
| T1= | 1.13661E+03 |
| T2= | 2.40201E+04 |
| T3= | 54 |
| T4= | 300 |
| T5= | 5.1756E+01 |
| n= | variabel |
| p= | 10 |
| N-table= | *5* |

Mandel's statistics | End Result:

| *LAB* | *PARM-gem* | *Stdev* | *N* | *h-mark* | *h* | *k* | *k-mark* | *\vX > AvST+2std* | *AvX < AvST-2std* | | *PARM* | *Stdev* | *Rej.labs* | | *N* | *N-1* | *dev_mean* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 44L | 16.4383 | 14.708 | 6 | | -0.36 | 3.25 | !! | | Fail | - | - | | ,44L | - | - | | -4.58 |
| 65L | 18.1250 | 0.830 | 4 | | -0.33 | 0.18 | | | Fail | | 18.1250 | 0.8302 | | | 4 | 3 | -2.90 |
| 45L | 20.0317 | 1.116 | 6 | | -0.29 | 0.25 | | | Fail | | 20.0317 | 1.1163 | | | 6 | 5 | -0.99 |
| 35L | 20.1833 | 1.828 | 6 | | -0.29 | 0.40 | | | Fail | | 20.1833 | 1.8280 | | | 6 | 5 | -0.84 |
| 32L | 20.3250 | 0.585 | 4 | | -0.29 | 0.13 | | | Fail | | 20.3250 | 0.5852 | | | 4 | 3 | -0.70 |
| 16L | 20.9000 | 0.959 | 6 | | -0.28 | 0.21 | | | Fail | | 20.9000 | 0.9592 | | | 6 | 5 | -0.12 |
| 9L | 21.1667 | 0.427 | 6 | | -0.27 | 0.09 | | | Fail | | 21.1667 | 0.4274 | | | 6 | 5 | 0.15 |
| 56L | 21.4030 | 0.887 | 6 | | -0.27 | 0.20 | | | Fail | | 21.4030 | 0.8868 | | | 6 | 5 | 0.38 |
| 19L | 21.6000 | 0.795 | 6 | | -0.26 | 0.18 | | | Fail | | 21.6000 | 0.7950 | | | 6 | 5 | 0.58 |
| 34L | 22.6009 | 1.308 | 6 | | -0.24 | 0.29 | | | Fail | | 22.6009 | 1.3076 | | | 6 | 5 | 1.58 |
| 36L | 22.8783 | 4.310 | 3 | | -0.24 | 0.95 | | | Fail | - | - | | ,36L | - | - | | 1.86 |
| 66L | 23.8750 | 1.234 | 4 | | -0.22 | 0.27 | | | Fail | | 23.8750 | 1.2340 | | | 4 | 3 | 2.85 |
| 70L | 221.3900 | - | 1 | !! | 3.33 | | | Fail | | - | - | | ,70L | - | - | | 200.37 |
| *Tot.gem* | 36.224 | 2.416 g/kg | | *1%-level:* | 2.27 | (1.75) | | | | 10 | *21.0211* | | (36L,44L ,70L) | | 10 | 9 | |
| *Tot.std=* | 55.669 | 4.003 | | *5%-level:* | 1.84 | (1.51) | | | | 3 | | | | | | | |
| | | | | | | | | | | 64 | | | | | | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **RESULTS:** | **Mean =** | **21.02106** | *g/kg* | | | |
| **Repeatability variance** | **S2r =** | 1.17627 | | | | |
| **Repeatability std.** | **Sr =** | **1.08456** | --> | 5.16% | **r =** | 3.0368 |
| **Between lab variance** | **S2L =** | 1.76699 | | | | |
| **Reproducibility var.** | **S2R =** | 2.94326 | | | | |
| **Reproducibility std.** | **SR =** | **1.71559** | --> | 8.16% | **R =** | 4.8037 |
| **Remarks:** | **3 Labs rejected! (36L,44L ,70L)** | | | | | |



## Annex 4:

## Raw data submitted

| Sample: | Compost 1 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 30L | 260.0 | |
| 30L | 258.0 | |
| 30L | 256.0 | |
| 34L | 296.9 | |
| 34L | 293.6 | |
| 34L | 295.8 | |
| 34L | 299.4 | |
| 34L | 319.9 | |
| 34L | 302.4 | |
| 35L | 230.6 | |
| 35L | 230.0 | |
| 35L | 229.8 | |
| 35L | 229.6 | |
| 35L | 229.1 | |
| 35L | 225.9 | |
| 36L | 254.6 | |
| 36L | 252.7 | |
| 36L | 247.0 | |
| 44L | 258.3 | |
| 44L | 258.1 | |
| 44L | 257.8 | |
| 44L | 253.9 | |
| 44L | 252.8 | |
| 44L | 252.5 | |
| 45L | 265.8 | |
| 45L | 260.0 | |
| 45L | 254.6 | |
| 45L | 246.1 | |
| 45L | 240.9 | |
| 45L | 235.0 | |
| 46L | 246.0 | |
| 46L | 240.0 | |
| 46L | 237.0 | |
| 46L | 235.0 | |
| 56L | 243.6 | |
| 56L | 243.5 | |
| 56L | 240.5 | |
| 56L | 239.7 | |
| 56L | 237.5 | |
| 56L | 235.8 | |
| 65L | 258.7 | |
| 65L | 254.2 | |
| 65L | 248.3 | |
| 66L | 245.4 | |
| 66L | 242.9 | |
| 66L | 236.5 | |
| 66L | 226.2 | |
| 69L | 273.0 | |
| 69L | 225.0 | |
| 69L | 144.0 | |
| 69L | 142.0 | |
| 70L | 243.5 | |
| 9L | 259.4 | |
| 9L | 257.0 | |
| 9L | 256.5 | |
| 9L | 256.3 | |
| 9L | 256.2 | |
| 9L | 255.5 | |
| 32L | 229.0 | |
| 32L | 239.0 | |
| 32L | 251.0 | |
| 32L | 247.0 | |
| 16L | 238.0 | |
| 16L | 253.0 | |
| 16L | 240.0 | |
| 16L | 250.0 | |
| 16L | 242.0 | |
| 16L | 241.0 | |

| Sample: | Compost 2 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 19L | 169.1 | |
| 19L | 163.4 | |
| 19L | 161.9 | |
| 19L | 160.4 | |
| 19L | 149.9 | |
| 19L | 141.4 | |
| 34L | 152.7 | |
| 34L | 162.2 | |
| 34L | 151.9 | |
| 34L | 175.3 | |
| 34L | 163.6 | |
| 34L | 187.4 | |
| 35L | 150.6 | |
| 35L | 131.2 | |
| 35L | 129.4 | |
| 35L | 127.9 | |
| 35L | 119.1 | |
| 35L | 117.7 | |
| 36L | 141.8 | |
| 36L | 140.8 | |
| 36L | 132.0 | |
| 44L | 171.9 | |
| 44L | 166.4 | |
| 44L | 165.4 | |
| 44L | 163.7 | |
| 44L | 162.4 | |
| 44L | 162.0 | |
| 45L | 142.8 | |
| 45L | 132.5 | |
| 45L | 132.3 | |
| 45L | 132.1 | |
| 45L | 130.9 | |
| 45L | 125.3 | |
| 46L | 158.0 | |
| 46L | 155.0 | |
| 46L | 117.0 | |
| 46L | 112.0 | |
| 56L | 164.2 | |
| 56L | 162.6 | |
| 56L | 162.2 | |
| 56L | 157.1 | |
| 56L | 156.1 | |
| 56L | 154.5 | |
| 65L | 193.3 | |
| 65L | 183.8 | |
| 65L | 169.3 | |
| 65L | 156.2 | |
| 66L | 156.1 | |
| 66L | 154.5 | |
| 66L | 142.0 | |
| 66L | 141.7 | |
| 69L | 270.0 | |
| 69L | 266.0 | |
| 69L | 217.0 | |
| 69L | 213.0 | |
| 70L | 141.4 | |
| 9L | 164.4 | |
| 9L | 164.1 | |
| 9L | 160.2 | |
| 9L | 158.9 | |
| 9L | 152.6 | |
| 9L | 149.2 | |
| 32L | 147.0 | |
| 32L | 152.0 | |
| 32L | 154.0 | |
| 32L | 160.0 | |
| 16L | 140.0 | |
| 16L | 144.0 | |
| 16L | 147.0 | |
| 16L | 151.0 | |
| 16L | 136.0 | |
| 16L | 139.0 | |

| Sample: | Sewage Sludge 1 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 19L | 286.7 | |
| 19L | 281.4 | |
| 19L | 276.9 | |
| 19L | 272.0 | |
| 19L | 268.8 | |
| 19L | 266.2 | |
| 34L | 315.2 | |
| 34L | 314.9 | |
| 34L | 343.0 | |
| 34L | 336.5 | |
| 34L | 304.2 | |
| 34L | 298.3 | |
| 35L | 270.7 | |
| 35L | 270.3 | |
| 35L | 268.8 | |
| 35L | 267.4 | |
| 35L | 263.1 | |
| 35L | 262.6 | |
| 36L | 271.3 | |
| 36L | 266.5 | |
| 36L | 261.4 | |
| 44L | 292.0 | |
| 44L | 291.6 | |
| 44L | 291.2 | |
| 44L | 290.0 | |
| 44L | 289.6 | |
| 44L | 289.3 | |
| 45L | 297.6 | |
| 45L | 295.2 | |
| 45L | 288.5 | |
| 45L | 286.6 | |
| 45L | 284.0 | |
| 45L | 278.7 | |
| 46L | 233.0 | |
| 46L | 233.0 | |
| 46L | 230.0 | |
| 46L | 229.0 | |
| 56L | 270.2 | |
| 56L | 270.1 | |
| 56L | 269.4 | |
| 56L | 269.3 | |
| 56L | 268.4 | |
| 56L | 268.0 | |
| 65L | 264.1 | |
| 65L | 260.9 | |
| 65L | 260.7 | |
| 65L | 260.5 | |
| 66L | 270.1 | |
| 66L | 267.4 | |
| 66L | 261.9 | |
| 66L | 251.4 | |
| 69L | 26.3 | |
| 69L | 24.0 | |
| 69L | 21.9 | |
| 69L | 21.0 | |
| 70L | 281.5 | |
| 9L | 285.7 | |
| 9L | 285.6 | |
| 9L | 285.5 | |
| 9L | 285.5 | |
| 9L | 285.3 | |
| 9L | 284.6 | |
| 32L | 265.0 | |
| 32L | 271.0 | |
| 32L | 271.0 | |
| 32L | 280.0 | |
| 16L | 284.0 | |
| 16L | 284.0 | |
| 16L | 286.0 | |
| 16L | 287.0 | |
| 16L | 287.0 | |
| 16L | 288.0 | |

| Sample: | Sewage Sludge 2 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 19L | 230.8 | |
| 19L | 230.3 | |
| 19L | 223.9 | |
| 19L | 220.4 | |
| 19L | 219.2 | |
| 19L | 209.6 | |
| 34L | 263.1 | |
| 34L | 247.1 | |
| 34L | 297.7 | |
| 34L | 278.5 | |
| 34L | 278.0 | |
| 34L | 266.8 | |
| 35L | 217.9 | |
| 35L | 216.0 | |
| 35L | 215.8 | |
| 35L | 213.2 | |
| 35L | 211.9 | |
| 35L | 209.4 | |
| 36L | 209.6 | |
| 36L | 206.9 | |
| 36L | 205.2 | |
| 44L | 232.5 | |
| 44L | 230.9 | |
| 44L | 230.7 | |
| 44L | 230.5 | |
| 44L | 228.7 | |
| 44L | 228.3 | |
| 45L | 233.1 | |
| 45L | 232.7 | |
| 45L | 230.8 | |
| 45L | 228.7 | |
| 45L | 223.4 | |
| 45L | 211.3 | |
| 46L | 19.0 | |
| 46L | 18.0 | |
| 46L | 18.0 | |
| 46L | 18.0 | |
| 56L | 220.4 | |
| 56L | 219.7 | |
| 56L | 219.3 | |
| 56L | 218.5 | |
| 56L | 218.3 | |
| 56L | 216.7 | |
| 65L | 213.6 | |
| 65L | 209.1 | |
| 65L | 206.5 | |
| 65L | 204.7 | |
| 66L | 210.5 | |
| 66L | 202.2 | |
| 66L | 200.9 | |
| 66L | 198.3 | |
| 70L | 227.2 | |
| 9L | 238.5 | |
| 9L | 237.7 | |
| 9L | 237.5 | |
| 9L | 235.9 | |
| 9L | 235.6 | |
| 9L | 235.4 | |
| 32L | 224.0 | |
| 32L | 226.0 | |
| 32L | 220.0 | |
| 32L | 227.0 | |
| 16L | 230.0 | |
| 16L | 231.0 | |
| 16L | 235.0 | |
| 16L | 226.0 | |
| 16L | 229.0 | |
| 16L | 224.0 | |

| Sample: | Soil 4 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 19L | 17.20 | |
| 19L | 17.00 | |
| 19L | 16.90 | |
| 19L | 16.70 | |
| 19L | 16.40 | |
| 19L | 15.80 | |
| 30L | 17.90 | |
| 30L | 17.70 | |
| 30L | 17.00 | |
| 34L | 17.40 | |
| 34L | 16.92 | |
| 34L | 17.27 | |
| 34L | 17.67 | |
| 34L | 17.77 | |
| 34L | 17.83 | |
| 35L | 17.00 | |
| 35L | 16.50 | |
| 35L | 16.50 | |
| 35L | 16.40 | |
| 35L | 16.30 | |
| 35L | 16.30 | |
| 36L | 15.00 | |
| 36L | 14.97 | |
| 36L | 14.17 | |
| 44L | 15.94 | |
| 44L | 15.87 | |
| 44L | 15.84 | |
| 44L | 15.74 | |
| 44L | 15.73 | |
| 44L | 15.62 | |
| 45L | 18.27 | |
| 45L | 17.09 | |
| 45L | 16.66 | |
| 45L | 16.65 | |
| 45L | 16.64 | |
| 45L | 16.00 | |
| 46L | 23.00 | |
| 46L | 23.00 | |
| 46L | 23.00 | |
| 46L | 22.00 | |
| 56L | 16.68 | |
| 56L | 16.54 | |
| 56L | 16.53 | |
| 56L | 16.43 | |
| 56L | 16.39 | |
| 56L | 16.38 | |
| 65L | 16.30 | |
| 65L | 15.60 | |
| 65L | 15.30 | |
| 65L | 14.90 | |
| 66L | 21.79 | |
| 66L | 19.00 | |
| 66L | 17.62 | |
| 66L | 17.34 | |
| 70L | 104.04 | |
| 9L | 16.40 | |
| 9L | 16.40 | |
| 9L | 16.10 | |
| 9L | 15.90 | |
| 9L | 15.90 | |
| 9L | 15.60 | |
| 32L | 16.40 | |
| 32L | 16.00 | |
| 32L | 15.60 | |
| 32L | 15.70 | |
| 16L | 17.00 | |
| 16L | 18.00 | |
| 16L | 17.00 | |
| 16L | 18.00 | |
| 16L | 17.00 | |
| 16L | 17.00 | |

| Sample: | Soil 5 | |
|---|---|---|
| Element: | TOC | [g/kg] |
| LAB | PARM | |
| 19L | 22.50 | |
| 19L | 22.30 | |
| 19L | 22.10 | |
| 19L | 21.10 | |
| 19L | 21.00 | |
| 19L | 20.60 | |
| 34L | 24.43 | |
| 34L | 23.34 | |
| 34L | 23.15 | |
| 34L | 22.21 | |
| 34L | 21.76 | |
| 34L | 20.73 | |
| 35L | 23.30 | |
| 35L | 20.80 | |
| 35L | 20.10 | |
| 35L | 19.80 | |
| 35L | 19.30 | |
| 35L | 17.80 | |
| 36L | 25.40 | |
| 36L | 25.34 | |
| 36L | 17.90 | |
| 44L | 30.21 | |
| 44L | 29.82 | |
| 44L | 29.56 | |
| 44L | 3.07 | |
| 44L | 3.00 | |
| 44L | 2.97 | |
| 45L | 21.49 | |
| 45L | 20.92 | |
| 45L | 20.33 | |
| 45L | 19.71 | |
| 45L | 19.33 | |
| 45L | 18.41 | |
| 56L | 22.37 | |
| 56L | 22.22 | |
| 56L | 22.03 | |
| 56L | 20.69 | |
| 56L | 20.68 | |
| 56L | 20.44 | |
| 65L | 19.20 | |
| 65L | 18.20 | |
| 65L | 17.90 | |
| 65L | 17.20 | |
| 66L | 25.43 | |
| 66L | 24.24 | |
| 66L | 23.21 | |
| 66L | 22.62 | |
| 70L | 221.39 | |
| 9L | 21.80 | |
| 9L | 21.40 | |
| 9L | 21.30 | |
| 9L | 21.10 | |
| 9L | 20.70 | |
| 9L | 20.70 | |
| 32L | 20.50 | |
| 32L | 21.00 | |
| 32L | 20.20 | |
| 32L | 19.60 | |
| 16L | 21.00 | |
| 16L | 22.00 | |
| 16L | 22.00 | |
| 16L | 20.60 | |
| 16L | 19.70 | |
| 16L | 20.10 | |

European Commission

**EUR 23012 EN – Joint Research Centre – Institute for Environment and Sustainability**
Title: Project HORIZONTAL Validation Report on total organic carbon
Author(s): E. Sobiecka, H. van der Sloot, K. Andersen, B. M. Gawlik
Luxembourg: Office for Official Publications of the European Communities
2007 – 50 pp. – 21.0 x 29.7 cm
EUR – Scientific and Technical Research series – ISSN 1018-5593
ISBN 978-92-79-07412-7

Abstract
Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. In the context of this standardization project, a series of draft technical specification were designed upon an extensive desk study, fine-tuned after expert consultations and finally validated in international intercomparisons exercise.
This report summarises the work performed within the validation study of the draft standard for the determination of total organic carbon (TOC) 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.

LB-NA-23012-EN-C

EUROPEAN COMMISSION

Publications Office
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ISBN 978-92-79-07412-7