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CERTIFICATE OF ANALYSIS

ERM[®]-CC010**Adsorbable organically bound halogens (AOX) in soil**

| Certified Values | | |
|--|-------------------------------|---------------------------|
| | Certified value ¹⁾ | Uncertainty ²⁾ |
| Compound | Mass fraction in mg/kg | |
| Adsorbable organically bound halogen (AOX) | 1349 | ± 59 |
| ¹⁾ The certified value is the mean of 14 laboratory means (analytical procedure according to DIN 38414 Teil 18, Nov 1989). | | |
| ²⁾ Estimated expanded uncertainty <i>U</i> with a coverage factor of about <i>k</i> =2, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993. | | |

This certificate is valid for a period of 12 months beginning with the dispatch of the reference material from BAM.

The minimum sample intake for a single determination of AOX amounts to 20 mg.

NOTE

European Reference Material ERM[®]-CC010 was originally certified as BAM-U010. It was produced and certified under the responsibility of Bundesanstalt für Materialforschung und -prüfung (BAM) according to the principles laid down in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (<http://www.erm-crm.org>).

Accepted as an ERM[®], Berlin, 2004-04-14.

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DESCRIPTION OF THE SAMPLE

This reference material was prepared at BAM employing an air dried homogenised sieving fraction (< 0.063 mm) of a soil from a contaminated industrial site. The material comes in amber glass vials containing 5.7 g of material each. The screw caps with PTFE-inserts are sealed by shrinking foil.

The results of the homogeneity study were in accordance with the assumption of a homogenous distribution of the AOX content in the material.

The initial stability study after storage of selected units of the reference material at different temperatures revealed a shelf life of several years when kept at 4°C or lower. On storing the reference material at higher temperatures up to a maximum of 23°C, a deterioration of the content of AOX has to be taken into consideration. Therefore – starting with the date of sale of the reference material – the validity of the certificate expires after 12 months. Periodical investigations on the stability of this material will be carried out in order to keep this information up to date.

The tests for homogeneity and stability are described in detail in a technical report (see next page).

PARTICIPANTS

Fourteen laboratories whose proficiency has been established prior to this study by means of different materials participated in the certification study. All laboratories analysed the samples according to the German standard DIN 38414 Teil 18 (Nov. 1989) [1].

| Laboratory | City |
|--|---------------|
| Deutsche Steinkohle Aktiengesellschaft | Völklingen |
| Inst. f. Umweltanalytik u. Geotechnik GmbH | Wetzlar |
| wave GmbH Umweltlabor | Stuttgart |
| LUS GmbH Labor f. Umweltschutz und chemische Analyse | Magdeburg |
| Chemisches Untersuchungslabor Dr. Lörcher | Ludwigsburg |
| Dr.Graner & Partner GmbH | München |
| BEWAG Aktiengesellschaft | Berlin |
| DB-AG, Bahn-Umwelt-Zentrum, Umweltlabor und Messstelle | Brandenburg |
| TÜV Umwelt Berlin-Brandenburg GmbH | Berlin |
| Bundesanstalt für Materialforschung und -prüfung (BAM) | Berlin |
| Prüftechnik IFEP GmbH & Co. KG | Osnabrück |
| HWG Havelländische Wasser GmbH & Co. KG | Potsdam |
| GfA Gesellschaft für Arbeitsplatz- und Umweltanalytik | Münster-Roxel |
| ISEGA Forschungs- u. Untersuchungsgesellschaft | Aschaffenburg |

EVALUATION OF RESULTS

Organisation and evaluation of the certification study comply with the requirements of the „Guidelines for the production and certification of BAM reference materials“ [2]. Data processing and statistical tests were accomplished using the BCR programme SoftCRM V.1.1.0 [3] with manual re-evaluation of the erroneous Grubbs outlier indications of this programme. The certified mass fraction of AOX assigned to this reference material is the mean of laboratory means. Table 1 displays the laboratory means and the corresponding standard deviations calculated from 6 independent determinations.

Table 1: Means and standard deviations of the participating laboratories
 (Ordering does not correspond to the listing of participant addresses)

| No. | Mean [mg/kg] | Standard deviation [mg/kg] | Relative standard deviation [%] |
|-----|-----------------|-------------------------------|------------------------------------|
| 8 | 1351.2 | 67.9 | 5.0 |
| 10 | 1354.8 | 44.0 | 3.3 |
| 17 | 1198.0 | 42.9 | 3.6 |
| 19 | 1175.0 | 118.1 | 10.0 |
| 20 | 1522.3 | 15.4 | 1.0 |
| 26 | 1351.5 | 45.4 | 3.4 |
| 27 | 1412.0 | 21.5 | 1.5 |
| 28 | 1195.0 | 72.3 | 6.1 |
| 29 | 1363.8 | 24.9 | 1.8 |
| 30 | 1351.0 | 30.9 | 2.3 |
| 35 | 1329.5 | 122.0 | 9.2 |
| 36 | 1488.0 | 6.9 | 0.5 |
| 37 | 1398.7 | 29.0 | 2.1 |
| 38 | 1401.3 | 14.0 | 1.0 |

SAFETY INFORMATION

It should be noted that the source of AOX might be organochlorine pesticides. It is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal

INSTRUCTIONS FOR USE

Before withdrawing a sub-sample the bottle has to have reached room temperature. Thereafter, the bottle is to be closed tight and stored at 4°C or lower. The stability of the reference material is not affected by short periods of handling at ambient temperature during transport and use.

STORAGE

The sample is to be stored at a temperature of 4°C or lower.

TECHNICAL REPORT

A detailed technical report [4] (in German) describing the production, general characterisation as well as the analytical procedures applied and the treatment of the analytical data during certification of ERM[®]-CC010 is available on request from BAM.

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REFERENCES

- [1] DIN 38 414 Teil 18: Nov. 1989 Schlamm und Sedimente (Gruppe S) Bestimmung von adsorbierten organisch gebundenen Halogenen (AOX)
- [2] Guidelines for the production and certification of BAM reference materials, BAM 1997
- [3] SoftCRM V. 1.10, EU SM&T program, Contract No. SMT4-CT98-6533
- [4] Zertifizierungsbericht zur Herstellung der zertifizierten Referenzmaterialien BAM-U010, BAM-U011 und BAM-U012, BAM, 2002

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