

CERTIFICATE OF ANALYSIS

ERM[®]-CC007a

| Organochlorine pesticides in soil | | |
|-----------------------------------|-------------------------------|---------------------------|
| Certified Values | | |
| | Certified value ¹⁾ | Uncertainty ²⁾ |
| Compound | Mass fraction in µg/kg | |
| α-HCH | 219 | ± 23 |
| β-HCH | 1570 | ± 210 |
| γ-HCH | 21.4 | ± 2.6 |
| p,p'-DDE | 380 | ± 60 |
| o,p'-DDT | 340 | ± 50 |
| p,p'-DDT | 960 | ± 140 |

¹⁾ The certified value is the mean of 7-8 laboratory means using GC-ECD and GC-MS including IDMS. The values are traceable to the SI (Système International d'Unités) via calibration using substances with certified purity.

²⁾ Estimated expanded uncertainty *U* with a coverage factor of *k*=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 size for one determination is 5 g.

NOTE

The European Reference Material ERM[®]-CC007a replaces ERM[®]-CC007 and was produced and certified under the responsibility of BAM Bundesanstalt für Materialforschung und –prüfung according to the principles laid down in ISO guide 35:2006 and in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available via the internet (<http://www.erm-crm.org>).

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INDICATIVE VALUES

The mass fractions of δ -HCH ($16.5 \pm 2.5 \mu\text{g/kg}$) and o,p'-DDD ($18 \pm 4 \mu\text{g/kg}$) are given as not certified indicative values.

DESCRIPTION OF THE SAMPLE

The reference material ERM[®]-CC007a can be used for method validation, to confirm the correct implementation of ISO 10382 [1] or to confirm the acceptability of method modifications.

ERM[®]-CC007a is a natural matrix reference material taken from a contaminated industrial site near Berlin. The material was dried, classified by sieving and homogenised. It is provided in 100 ml amber glass bottles each containing approximately 103 grams of the soil material. The screw caps with PTFE-inserts are sealed with shrinking foil.

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 $-20 \text{ }^\circ\text{C}$. On storing the reference material at temperatures higher than $+4 \text{ }^\circ\text{C}$, a deterioration of the content of the organochlorine pesticides has to be taken into consideration. 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 AND ANALYTICAL METHOD USED FOR CERTIFICATION

The BAM in-house certification study involved eight different combinations of extraction and instrumental quantification as shown in the following table:

| Entry | Analytical method | Extraction method | Extraction solvent |
|-------|-------------------|------------------------------|----------------------------|
| 1 | GC/IDMS | Shaking | Cyclohexane; acetone (1:1) |
| 2 | GC/IDMS | Fluidised bed extraction | Toluene |
| 3 | GC/ECD | Sonication | Cyclohexane/acetone (1:1) |
| 4 | GC/ECD | Pressurised fluid extraction | Cyclohexane/acetone (1:1) |
| 5 | GC/ECD | Shaking | Cyclohexane; acetone (1:1) |
| 6 | GC/ECD | Fluidised bed extraction | Toluene |
| 7 | GC/IDMS | Sonication | Cyclohexane/acetone (1:1) |
| 8 | GC/IDMS | Pressurised fluid extraction | Cyclohexane/acetone (1:1) |

Specific remarks on the procedures for extraction and quantification can be found in the technical report.

SAFETY INFORMATION

This reference material contains hazardous compounds (e.g. DDT) in very low mass fractions between 16 µg/kg and 1600 µg/kg.

Proper use of the reference material is essential for avoiding potential harm to the user.

It is strongly recommended to handle and to dispose of 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 and is to be mixed thoroughly. Thereafter, the bottle is to be closed tightly and stored at (-20 ± 5) °C. The stability of the reference material is not affected by short periods of handling at ambient temperature during transport and use.

STORAGE

The reference material must be stored in its original bottles at (-20 ± 5) °C.

TECHNICAL REPORT

A detailed technical report describing the analytical procedures and the treatment of the analytical data used to certify the reference material ERM[®]-CC007a is available on request from BAM.

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REFERENCES

- [1] ISO 10382:2002-10-15 "Soil quality - Determination of organochlorine pesticides and polychlorinated biphenyls – Gas-chromatographic method with electron capture detection"

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