# Reference Material Data Sheet

# Koeln Loess (UoK)

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## Description of the reference material

*The UoK loess sample was collected and prepared as a candidate reference material under the direction of H.U. Kasper, University of Cologne, Germany. The sample was collected from Nussloch, 10 km South of Heidelberg and 3 km East of the upper Rhine Graben, Germany (49° 19' N, 8° 43' E) and 217 m above sea level. The basement of the loess consists of Middle Triassic limestone and dolomite ('Muschelkalk'). The main section comprises 16 m thick loess deposits from the Würmian. The sample was collected from the upper Würmian loess which was deposited as part of the last glacial - interglacial cycle, 15,000 - 20,000 a BP. Examination of this sample indicates that the main mineralogical components are quartz, feldspar, carbonate phases, mica, clay minerals and iron-rich minerals. The sample also contains accessory zircon, rutile, tourmaline, anatase, brookite, garnet, epidote and amphibole.*

*The first batch of this material was employed only as the test material for Round 13 of the GeoPT proficiency testing programme. A second batch of material from the same source was made available as a reference material and distributed with a data sheet (IAG UoK Loess version 1.00 – 26th February 2017) based on the data from Round 13. Material from the second batch was employed as the test material for round 50 of the GeoPT proficiency testing programme. The Proficiency Testing Committee for this round was P.C. Webb (administrator and results assessor), P.J. Potts (results reviewer), M. Thompson (statistical advisor) and C.J.B. Gowing (distribution manager). It would appear that small differences in the preparation procedures of the two batches resulted in small differences between the composition of the two batches. This version of the UoK data sheet is, therefore, a reassessment of composition of this reference material based on the GeoPT50 characterisation study.*

## Intended use

*This reference material is designed for use by laboratories undertaking the major and trace element mass fraction measurement of silicate rocks and equivalent matrices for the calibration of a measurement system, the assessment of a measurement procedure, assigning values to other materials, and quality control. Note that the material may be used only for a single purpose in the same measurement process.*

## Homogeneity assessment

*This second batch of Loess UoK was not conventionally tested for homogeneity. However, the earlier batch (used for the GeoPT13 proficiency testing round) was formally tested by selecting at random ten packets (16 for trace elements) of the sample prepared for distribution. Duplicate test portions from each packet were analysed by WDXRF at the Open University (Milton Keynes). For the elements for which values were assigned, homogeneity was considered to be satisfactory for use in the GeoPT13 round. For the GeoPT50 round, careful examination of data distributions was undertaken to check for any evidence of inhomogeneity. It is expected that*

*if present, inhomogeneity effects would significantly increase the uncertainty / target precision ratio (which is routinely used in the data assessment process) as well as degrading the identification of consensus values that complied with the strict criteria used designate assigned values. No such effects were detected.*

## Characterisation study

*Reference and indicative values were derived from a rigorous assessment of data submitted to round 50 of the IAG GeoPT proficiency testing programme in which 100 laboratories participated, contributing a total of 3614 individual measurement results.*

***Reference values** were GeoPT assigned values derived as consensus values from measurand data distributions that satisfied the following criteria:*

- *Sufficient laboratories had contributed data for estimating a clear consensus value (usually a minimum of 15).*
- *Visual assessment of the measurand data distribution gave confidence that a substantial proportion of the results distribution was symmetrically disposed about the consensus.*
- *The ratio of the uncertainty in the location estimate to the target precision for proficiency testing for that measurand was an acceptably small value.*
- *Where possible, an evaluation of measurement results by procedure was judged to provide no clear evidence of procedural bias among the measurement results from which the consensus was derived.*

*Uncertainties are the robust standard deviation of the mean, median or mode of the assigned value expanded by a coverage factor of two.*

*Further details of the assessment procedure can be found in the G-Probe 50 report (Webb et al., 2022).*

***Indicative values** are GeoPT provisional values derived from measurand data sets that nearly, but not fully, met the above criteria. Instances of provisional status were identified because one or more of the following criteria applied:*

- *A smaller number of results (less than 15 but more than 8) contributed to the consensus.*
- *The results were unduly dispersed in relation to the target precision for proficiency testing for that measurand.*
- *The distribution of results was significantly skewed (but not severely enough to preclude the recognition of a clear consensus).*
- *Procedural bias was identified but a target value could nevertheless be recognised based on the most coherent part of the overall data distribution conforming approximately to a random sample from a normal distribution.*

## Minimum sample size

*After taking account of the distribution of data reported from a range of measurement techniques submitted to the GeoPT50 round, the minimum size of test portion is recommended to be 0.2 g.*

## Period of validity

*Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. On exposure to air, the material may absorb moisture, and instructions for handling must be followed.*

## Storage information

*Store in a sealed container in a cool dry environment.*

## Instructions for handling

*Before any measurements are made, every portion of the test sample must be dried at 105 ± 5°C for at least 2 hours. Avoid contamination and cross-contamination of the test material.*

| Reference values for IAG UoK Loess | | | | | | |
|---|---|---|---|---|---|---|
| **Oxide / element** | **Reference value** | **Expanded uncertainty** | | **Element** | **Reference value** | **Expanded uncertainty** |
| | **g /100g** | **g /100g** | | | **mg/kg** | **mg/kg** |
| **$SiO_2$** | 52.92 | 0.14 | | **Li** | 21.6 | 0.6 |
| **$TiO_2$** | 0.420 | 0.004 | | **Lu** | 0.39 | 0.02 |
| **$Al_2O_3$** | 6.09 | 0.04 | | **Mo** | 0.82 | 0.06 |
| **$Fe_2O_3T$** | 2.08 | 0.02 | | **Nb** | 8.28 | 0.29 |
| **MnO** | 0.064 | 0.001 | | **Nd** | 24.3 | 0.4 |
| **MgO** | 2.91 | 0.03 | | **Ni** | 31.0 | 1.0 |
| **CaO** | 16.53 | 0.11 | | **Pb** | 11.1 | 0.5 |
| **$Na_2O$** | 1.04 | 0.02 | | **Pr** | 6.36 | 0.16 |
| **$K_2O$** | 1.34 | 0.01 | | **Rb** | 50.9 | 0.7 |
| **$P_2O_5$** | 0.130 | 0.002 | | **Sb** | 0.51 | 0.05 |
| **LOI** | 16.22 | 0.06 | | **Sm** | 5.01 | 0.09 |
| | **mg/kg** | **mg/kg** | | **Sn** | 1.58 | 0.12 |
| **Ba** | 197 | 3 | | **Sr** | 280 | 5 |
| **Be** | 0.97 | 0.04 | | **Ta** | 0.73 | 0.04 |
| **Ce** | 52.5 | 1.5 | | **Tb** | 0.71 | 0.05 |
| **Co** | 5.53 | 0.39 | | **Th** | 8.27 | 0.21 |
| **Cs** | 2.63 | 0.07 | | **Tl** | 0.33 | 0.03 |
| **Dy** | 4.30 | 0.06 | | **Tm** | 0.390 | 0.012 |
| **Er** | 2.53 | 0.09 | | **U** | 2.75 | 0.09 |
| **Eu** | 0.88 | 0.03 | | **V** | 35.2 | 1.0 |
| **Ga** | 6.95 | 0.15 | | **Y** | 24.0 | 2.4 |
| **Gd** | 4.60 | 0.22 | | **Yb** | 2.52 | 0.03 |
| **Hf** | 9.26 | 0.37 | | **Zn** | 32.0 | 0.9 |
| **Ho** | 0.86 | 0.05 | | **Zr** | 343 | 6 |
| **La** | 26.1 | 0.6 | | | | |

*The expanded uncertainty represents the 95% confidence limit*
*$Fe_2O_3T$ is the total iron expressed as the oxide*

| Indicative values for IAG UoK Loess | | | | | | |
|---|---|---|---|---|---|---|
| **Element** | **Indicative value** | **Expanded uncertainty** | | **Element** | **Indicative value** | **Expanded uncertainty** |
| | **mg/kg** | **mg/kg** | | | **mg/kg** | **mg/kg** |
| **As** | 6.4 | 0.4 | | **Cu** | 9.8 | 0.5 |
| **Bi** | 0.13 | 0.02 | | **In** | 0.026 | 0.004 |
| **C(tot)** | 40470 | 850 | | **Sc** | 5.6 | 0.5 |
| **Cd** | 0.114 | 0.009 | | **W** | 1.3 | 0.2 |
| **Cr** | 83 | 3 | | | | |

*The expanded uncertainty represents the 95% confidence limit*
*C(tot) is the total elemental carbon.*

## Metrological traceability

*Traceability was not formally demonstrated for this reference material. However, traceability can be inferred from the clear consensus values derived from results contributed by the 100 participating laboratories. Additional evidence of traceability could be demonstrated through laboratories participating in this round using certified reference materials as calibrators or for data assessment (although this information is not currently recorded by the GeoPT programme). On occasions when certified reference materials have been used as test materials in the GeoPT programme, no significant difference has been found between assigned and certified values (see Potts et al., 2015).*

## Reference material characterisation report

*Full details of the measurement methods used, the results, their statistical analysis and assessment, on which the property values listed in this data sheet are based, can be found in the GeoPT50 report (Webb et al. 2022). This report can be freely downloaded for personal use from the International Association of Geoanalysts website (http://www.geoanalyst.org/index.php).*

## Safety information

*Silicate rock powders can cause harm especially if ingested, inhaled or in contact with the skin. User organisations must undertake a health and safety risk assessment and ensure that the appropriate procedures are followed in the handling and use of this material. A material safety data sheet is available from the supplier, on request.*

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## References

***Potts P.J., Thompson M., Chenery S.R., Webb, P.C. and Kasper H.U.*** *(2003) GEOPT13 - an international proficiency test for analytical geochemistry laboratories - report on round 13 / July 2003 (Köln Loess). International Association of Geoanalysts*

***Potts P.J., Thompson M. and Webb, P.C.*** *(2015) The reliability of assigned values from the GeoPT proficiency testing programme from an evaluation of data for six test materials that have been characterised as certified reference materials. Geostandards and Geoanalytical Research, 39, 407–417.*

***Webb, P.C., Potts, P.J.. , Thompson, M. and Gowing, C.J.B.*** *(2022). GeoPT50 – An international proficiency test for analytical geochemistry laboratories – report on round 50 (Calcified sediment CSd-1 / January 2022). International Association of Geoanalysts: Unpublished report, 47pp. (http://www.geoanalyst.org/geopt/GeoPT50Report.pdf).*

## Approvals

*This reference material information sheet was approved by the Reference Material and Certification Committee of the International Association of Geoanalysts.*

**Names: Philip J. Potts** **Position:** ***IAG Reference Material*** **Date: 22nd February 2022**
**Peter C. Webb** ***and Certification Committee***
**Jennifer M. Cook**

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