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Laboratory intercomparison of Pleistocene bone radiocarbon dating protocols

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Abstract

Since its invention in the late 1940's, radiocarbon dating has become an important tool for absolute dating. A prerequisite for the acceptance of this method is consistency between, and compatibility of, radiocarbon dates from different laboratories. To meet these requirements, international laboratory intercomparison studies with different sample materials are frequently performed (e.g. TIRI, FIRI, VIRI and, most recently, SIRI).

Intercomparison is especially relevant and difficult for samples close to the dating limit of ~50 kBP, not least for bone samples. A radiocarbon intercomparison study between the Leibniz-Laboratory in Kiel (Germany), the Centre for Isotope Research (CIO) in Groningen (The Netherlands), and the Oxford Radiocarbon Accelerator Unit (ORAU; United Kingdom) was performed on three Pleistocene (MIS3) mammal bone samples from the Brick Quarry site Coenen (BQC) in Germany.

The comparison of individually prepared and measured bone collagen radiocarbon activities, results from shared collagen measurements, and respective background signatures and correction points to the latter as the main factor responsible for observed differences in final given radiocarbon estimates.

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Introduction

During the 1960's various large mammal remains were found in the Brick Quarry Site Coenen, Körrenzig, Germany (Figure 1). The inventory contains horse (*Equus* sp.), bos/bison, woolly rhino (*Coelodonta antiquitatis*), giant deer (*Megaloceros giganteus*), mammoth (*Mammuthus primigenius*), hyena (*Crocuta spelaea*), cave bear (ursus spelaeus), and lion (*Panthera spelaea*).

Nine radiocarbon measurements, performed in the Leibniz-Laboratory, Kiel, and the Centre for Isotope Research (CIO), Groningen, gave ^{14}C ages between 34,000 BP and > 45,000 BP, which place the find horizon into the Interpleniglacial (Marine Oxygen Isotope Stage MIS3) (Matzerath et al. 2012).

Radiocarbon dates on selected faunal samples point to a rather broad temporal deposition history, while the analysis of the stable isotope composition ($\delta^{13}C$, $\delta^{15}N$, $\delta^{34}S$) seems to indicate a comparably shorter deposition period (e.g. cold MIS3 stadial/ possibly Heinrich 5; Wißing et al. 2015).

Considering controversial discussions with respect to bone sample pretreatment for radiocarbon analysis, i.e. with or without ultrafiltration (e.g. Bronk Ramsey et al. 2004, Higham et al. 2006, Huels et al. 2007, 2009), a radiocarbon intercomparison study between the Leibniz-Laboratory in Kiel (Germany), the Centre for Isotope Research (CIO) in Groningen (The Netherlands), and the Oxford Radiocarbon Accelerator Unit (ORAU; United Kingdom) was performed on three Pleistocene (MIS3) mammal bone samples from the Brick Quarry site Coenen (BQC) in Germany:

- Sample BQC-101, *Equus* sp., right radius, seems to be intentionally fractured by humans, radiocarbon age GrA-53420: >45000 BP
- Sample BQC-55, *Equus* sp., right tibia,

- Sample BQC-78, *Bos/Bison*, distal piece of left femur, radiocarbon age KIA44874: 34190 +330/-320 BP

Methods

The three samples, each consisting of a single piece of bone, were individually sampled in the participating laboratories, prepared and subsequently measured using in-house AMS-systems. In addition to individual measurements, collagen extracted by each laboratory was also sent to the other participating laboratories for AMS measurements.

All three laboratories use a modified Longin-protocol (Longin 1971) for collagen extraction, i.e. an acid – base – acid (ABA) treatment for de-mineralization with small differences in concentration of chemicals used and temperatures for collagen dissolution during the gelatinization step (see Table 1; Grootes et al. 2004, Mook and Streurman 1983, Brock et al. 2010). An additional preparation / cleaning step after collagen gelatinization is applied by the Oxford laboratory using ultrafiltration (Bronk Ramsey et al. 2004a), which intends to remove lower molecular weight, (possible environmentally contaminated) protein molecules (Molecular Weight Cut off [MWCO] 30kDa).

The conversion of extracted sample collagen to CO_2 was done either by closed quartz tube combustion (CTC; with CuO and Ag; Kiel) or using an elemental analyzer and cryogenic separation (Groningen, Oxford). The resulting sample CO_2 was graphitized by the Bosch reaction ($CO_2 + 2H_2 \xrightarrow{550-600°C;Fe\ catalyst} C + 2H_2O$) (Dee and Ramsey 2000, Nadeau et al. 1997, Nadeau et al. 1998, Aerts-Bijma et al. 1997).

Radiocarbon measurements were performed in each laboratory's own 3 MV HVEE AMS system, normalized to modern oxalic acid II standard (NBS SRM 4990C) and corrected for isotopic fractionation

$\{ F14C_{corrected} = F14C_{measured} * [\frac{0.975}{1+\frac{\delta^{13}C}{1000}}]^2 \}$ (van der Plicht and Hogg 2006).

Final conventional ^{14}C concentrations ($F^{14}C_{final}$) are calculated by subtracting background effects, i.e. apparent measured ^{14}C concentration measured in fossil sample material, which, according to its age, should not contain ^{14}C.

$$F14C_{final} = F14C_{corrected} - [F14C_{Background} * \left(1 - \frac{F14C_{sample}}{F14C_{standard}}\right)]$$

These corrections intend to remove — aside from material specific, intrinsic contamination — any possible effects occurring during the sample preparation, i.e. collagen extraction, CO_2 conversion, graphitization, and AMS measurement. The Kiel laboratory uses fossil bone samples as background material (Huels et al. 2014, Rakowski et al. 2014). For the measurements of shared collagen fractions, i.e. collagen extracted by another laboratory, background corrections in the Kiel AMS facility were performed using crude North Sea oil to correct for contamination effects occurring during CO_2 conversion, graphitization, and AMS-measurement. The Oxford laboratory uses CO_2 derived from anthracite to determine an AMS/graphitisation background; nylon to determine a combustion background; and fossil bone to determine a pretreatment background (Bronk Ramsey et al. 2004b, Wood et al. 2010). Due to concerns regarding the suitability of background bone standards to represent bone samples from different depositional environments, Groningen use a pragmatic approach by applying an upper limit of 45 ka BP ($F^{14}C$ concentration ~0.37) and using anthracite as background sample material (van der Plicht and Palstra 2014).

Final given uncertainties include variance observed during the measurement and the uncertainties in the background correction ($\sigma_{final} = \sqrt{{\sigma_{meas.}}^2 + {\sigma_{bg_corr}}^2}$). Kiel use for background correction uncertainty (σ_{bg_corr}) a factor of 1/3 of the applied background

correction (see Nadeau and Grootes 2013); Groningen and Oxford use the standard deviation of measured background signatures.

Results and discussion

In Table 2, collagen content (estimated in Kiel laboratory), C/N atomic weight ratios (measured by Groningen and Oxford laboratories), radiocarbon concentrations, and conventional radiocarbon ages for samples BQC-101, BQC-55, and BQC-78 are given.

All three bone samples are degraded with respect to organic preservation (collagen content < 10 wt% in comparison to > 20 wt% in fresh bone, Pasteris et al 2008). However, measured C/N ratios ~ 3.2 of extracted collagen and collagen contents above 1 wt% indicate reasonably preserved collagen with intact polypeptide properties (Dobberstein et al. 2009, van Klinken 1999).

For all three bones, final radiocarbon concentrations ≤ 0.011 $F^{14}C$ are measured. Comparatively large uncertainties seen for Kiel measurements stem from background correction uncertainties, i.e. using 1/3 of the background correction value as the background correction uncertainty. This factor is representative of the observed long-term scatter in bone background radiocarbon signature (Nadeau and Grootes 2013). Consequently, Kiel determined for all three bone samples infinite ages, i.e. final ^{14}C concentrations are smaller than two times the measurement uncertainty (so-called 2 σ criterion; Olsson 1989, van der Plicht and Hogg 2006). Groningen and Oxford, on the other hand, estimated finite ages. Inter laboratory estimated age differences for BQC-101 and BQC-55 are within given uncertainties, and a somewhat larger scatter is observed for results of BQC-78 (estimated age range from ~39– ~ 43 kyrs).

A comparison of the results of collagen extracted by one laboratory and measured by another, with in-house prepared and measured collagen offers a more detailed look into possible

causes for observed differences, since possible effects introduced by differences in sample preparation, e.g. with or without ultrafiltration or collagen gelatinization temperatures etc., will not be an issue.

In Figure 2, final ^{14}C concentrations ($F^{14}C$) are shown as stacked column graphs with respective background corrections.

Kiel Collagen

- Measurements made in Groningen and Oxford indicate comparable uncorrected ^{14}C-concentrations, suggesting comparable magnitudes of background contamination introduced by CO_2-conversion, graphitization, and AMS-measurement in comparison to Kiel measurements.

Groningen Collagen

- Measurements made in Kiel give slightly lower uncorrected ^{14}C-concentrations, indicating lower magnitudes of background introduced by combustion-graphitization-AMS procedures. The measurements at Oxford imply a comparable background to Groningen.

Oxford Collagen

- Measurements performed in Kiel give lower uncorrected ^{14}C-concentrations for BQC-101 and BQC-55, which seem to indicate a lower background from combustion-graphitization-AMS procedures, and a comparable magnitude of background in BQC-78. Measurements in Groningen indicate a similar size of background effects for BQC-101 and BQC-78 and a lower background effect for BQC-55.

Overall, the observed differences seem to have been caused largely by different apparent background signatures of the participating laboratories. For example, in sample BQC-101, the Leibniz-Laboratory in Kiel measured a $F^{14}C_{corrected}$ between 0.00411 – 0.00627. The same

collagen, measured in Groningen and Oxford, give $F^{14}C_{corrected}$=0.00660 and $F^{14}C_{corrected}$=0.00873, respectively. The ORAU, on the other hand, measures slightly more active, non-background corrected ^{14}C concentrations between $F^{14}C_{corrected}$=0.00775 and $F^{14}C_{corrected}$=0.00942 on ultrafiltered collagen. The apparent background of about $F^{14}C_{corrected}$=0.00455 – 0.00657 in Kiel and $F^{14}C_{corrected}$=0.00487 – 0.00742 in Oxford is comparable in magnitude, however, when background correction is applied, estimated $F^{14}C_{final}$ results in minimum ages of >43600 BP to >46500 BP for Kiel measurements and a finite age of 47000 ± 2900 BP and >44300 BP for Oxford measurements.

Observed inter- and intra-laboratory background signatures are greater, and mask, any differences in applied sample treatment (e.g. whether collagen is ultrafiltered or not). Limited in the number of samples measured, this intercomparison exercise is far away from being representative. However, the results shown indicate the necessity of a more detailed examination of background effects, that is, the measurement of background material, the procedures for correction, and the evaluation of the suitability of background material for a given set of sample materials, in particular for samples close to the limit of the radiocarbon dating method.

Conclusions

For three Pleistocene mammal bones samples, recovered from the Brick Quarry Site Coenen, Körrenzig (BQC), Germany, a radiocarbon intercomparison study was performed between the Leibniz-Laboratory in Kiel (Germany), the Centre for Isotope Research (CIO) in Groningen (The Netherlands), and the Oxford Radiocarbon Accelerator Unit (ORAU; United Kingdom).

The collagen contents of between 3wt% - 10wt% and C/N ratios ~3.2 indicates a reasonable preservation state of the three bones BQC-101, BQC-55, and BQC-78.

Estimated radiocarbon ages vary between minimum ages ~ >40000 BP to a finite age ~ 47000 BP.

The comparison of individually sampled, prepared, and measured bone collagen radiocarbon results as well as the results from shared collagen measurements points to the background correction (e.g. apparent measured background radiocarbon activity and correction applied) as the main factor responsible for observed differences in estimated radiocarbon ages.

With respect to the starting point, measured radiocarbon concentrations and radiocarbon ages are not in contradiction to the assumption of a short deposition period within a cold MIS3 stadial (Wißing et al. 2015).

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Tables

Table 1. Sample preparation: collagen extraction procedure, CO_2 conversion, graphitization, AMS-system, and applied measurement corrections.

	Leibniz-Laboratory, Kiel	**Centre for Isotope Research, Groningen**	**Oxford Radiocarbon Accelerator Unit**
Collagen extraction	Modified Longin: 0.3M-0.8M HCl, 0.3M NaOH, 0.3M HCl, room temperature; Gelatinization 85°C (pH3) 12-24h, filtration with 0.45µm Ag filter	Modified Longin (1-4% HCl,1% NaOH,1-4% HCl), room temperature, Gelatinization 90°C (pH3)	Modified Longin: 0.5M HCl, 0.1M NaOH, 0.5M HCl, room temperature; Gelatinization 75°C (pH3) 20h, filtration with 60-90µm Ezee™-filters
Ultrafiltration	no	no	30kDa MWCO, Vivaspin™15 (PES)
Lyophilization	yes	No, evaporation in hot stove	yes
CO_2-Conversion	CTC (900°C, 4h, CuO+Ag)	Elemental analyzer (Isocube, Elementar)	Elemental analyzer (Carbo-Erba NA 2000)
Graphitization	Bosch reaction: (10ml reactors, 0.9-1.1mgC/2mgFe)	Bosch reaction: (8ml reactors, 1.5 mgC/1.5mgFe)	Bosch reaction: (10ml reactors, 0.8-1.8mgC/2-2.5mgFe)
AMS-measurement	3 MV tandetron HVEE	3 MV tandetron HVEE	3 MV tandetron HVEE
Correction	• Calculating ^{13}C-corrected ^{14}C-concentrations • Background subtraction (for prepared bone sample using extracted background bone collagen; for combusted Groningen and Oxford collagen: combusted fossil oil) • Calculating conventional radiocarbon ages	• Calculating ^{13}C-corrected ^{14}C-concentrations • Background subtraction (anthracite) • Calculating conventional radiocarbon ages	• Subtract AMS/Graphitization background (fossil anthracite CO_2; all targets) • Calculating ^{13}C-corrected ^{14}C-concentrations • Background subtraction (combustion background correction all samples {nylon}; pretreatment background correction prepared bones {fossil bone}) (Wood et al. 2010) • Calculating conventional radiocarbon ages

Table 2. Radiocarbon concentrations ($F^{14}C_{corrected}$, background ^{14}C, and $F^{14}C_{final}$), $\delta^{13}C$ (AMS and IRMS), and conventional radiocarbon ages of samples BQC-101, -55, and -78.

Sample	Lab. ID	$F^{14}C_{corrected}$	Background ($F^{14}C_{corrected}$)	$F^{14}C_{final}$	$\delta^{13}C$ AMS (‰VPDB)	$\delta^{13}C$ IRMS (‰VPDB)	Age BP
				Kiel collagen			
BQC-101 wt%collagen (Leibniz-Laboratory Kiel): ~6-10 C/N-collagen (Groningen, Oxford): ~3.3	KIA50794	0.00613 ± 0.0002	0.00657 ± 0.0022	-0.00040 ± 0.0022	-20.9 ± 0.4		> 43600
	KIA50794	0.00627 ± 0.0002	0.00657 ± 0.0022	-0.00026 ± 0.0022	-20.6 ± 0.5		> 43600
	KIA50794	0.00486 ± 0.0002	0.00582 ± 0.0019	-0.00093 ± 0.0020	-19.8 ± 0.1		> 44550
	KIA50794	0.00411 ± 0.0002	0.00455 ± 0.0015	-0.00042 ± 0.0015	-18.9 ± 0.1		> 46500
	GrA62156	0.00660 ± 0.0003	0.00390 ± 0.0003	0.00273 ± 0.0004		-21.2 ± 0.2	>45000
	OxA_V-2616-52	0.00873 ± 0.0003	0.00492 ± 0.0005	0.00381 ± 0.0006		-20.7 ± 0.2	44700 ± 1200
				Groningen collagen			
	GrA59402	0.00600 ± 0.0002	0.00250 ± 0.0002	0.00351 ± 0.0003		-21.17 ± 0.2	> 45000
	GrA59403	0.00660 ± 0.0002	0.00250 ± 0.0003	0.00412 ± 0.0004		-21.15 ± 0.2	44100 +750/ -700
	KIA50797	0.00393 ± 0.0002	0.00093 ± 0.0003	0.00300 ± 0.00034	-20.2 ± 0.1		46700 +950/ -850
	KIA50797	0.00413 ± 0.0002	0.00120 ± 0.0004	0.00293 ± 0.0004	-21.0 ± 0.2		46900 +1300/ -1100
	OxA_V-2581-57	0.00571 ± 0.0002	0.00389 ± 0.0005	0.00182 ± 0.0005		-20.9 ± 0.2	50700 ± 2300
				Oxford collagen			
	OxA30241	0.00775 ± 0.0003	0.00487 ± 0.0010	0.00288 ± 0.0010		-20.9 ± 0.2	47000 ± 2900
	OxA30474	0.00942 ± 0.0004	0.00742 ± 0.0010	0.00200 ± 0.0010		-20.9 ± 0.2	> 44300
	KIA50800	0.00618 ± 0.0002	0.00093 ± 0.0003	0.00526 ± 0.0004	-20.7 ± 0.1		42200 +550/ -500
	KIA50800	0.00585 ± 0.0002	0.00120 ± 0.0004	0.00466 ± 0.0004	-21.0 ± 0.5		43100 +800/ -700
	GrA61745	0.00950 ± 0.0003	0.00410 ± 0.0003	0.00540 ± 0.0004		-21.0 ± 0.2	41900 +650/ -600

Sample	Lab. ID	$F^{14}C_{corrected}$	Background ($F^{14}C_{corrected}$)	$F^{14}C_{final}$	$\delta^{13}C$ AMS (‰VPDB)	$\delta^{13}C$ IRMS (‰VPDB)	Age BP
				Kiel collagen			
BQC-55 wt%collagen (Leibniz-Laboratory Kiel): ~3-5 C/N-collagen (Groningen, Oxford): ~3.2	KIA50795	0.00868 ± 0.0002	0.00657 ± 0.0022	0.00217 ± 0.0022	-20.6 ± 0.2		> 40400
	KIA50795	0.00788 ± 0.0002	0.00657 ± 0.0022	0.00136 ± 0.0022	-20.6 ± 0.2		> 41400
	KIA50795	0.00477 ± 0.0001	0.00582 ± 0.0019	-0.00102 ± 0.0019	-19.8 ± 0.1		> 44600
	KIA50795	0.00446 ± 0.0002	0.00453 ± 0.0015	-0.00005 ± 0.0015	-20.7 ± 0.2		> 46600
	GrA62158	0.00820 ± 0.0003	0.00390 ± 0.0003	0.00433 ± 0.00042		-21.1 ± 0.2	43700 +800/ -750
	Ox_V-2616-53	0.01027 ± 0.0003	0.00495 ± 0.0005	0.00532 ± 0.0006		-20.6 ± 0.2	42100 ± 900
				Groningen collagen			
	GrA59397	0.00620 ± 0.0002	0.00250 ± 0.0003	0.00372 ± 0.0004		-20.8 ± 0.2	> 45000
	GrA59400	0.00580 ± 0.0003	0.00250 ± 0.0003	0.00331 ± 0.0004		-20.5 ± 0.2	> 45000
	KIA50798	0.00425 ± 0.0001	0.00093 ± 0.0003	0.00332 ± 0.0003	-20.1 ± 0.1		45900 + 850/ -800
	KIA50798	0.00384 ± 0.0002	0.00119 ± 0.0004	0.00265 ± 0.0004	-20.1 ± 0.2		47700 + 1450/ -1250
	Ox_V-2580-38	0.00682 ± 0.0003	0.00366 ± 0.0005	0.00316 ± 0.0006		-20.8 ± 0.2	46300 ± 1500
				Oxford collagen			
	OxA30472	0.00878 ± 0.0004	0.00739 ± 0.0009	0.00139 ± 0.0010		-20.8 ± 0.2	>45700
	OxA30700	0.00891 ± 0.0003	0.00578 ± 0.0009	0.00313 ± 0.0010		-20.5 ± 0.2	46300 ± 2500
	KIA50801	0.00451 ± 0.0001	0.00093 ± 0.0003	0.00358 ± 0.0003	-20.2 ± 0.1		45200 + 800/ -750
	KIA50801	0.00470 ± 0.0002	0.00120 ± 0.0004	0.00351 ± 0.0004	-20.4 ± 0.6		45400 + 1050/ -950
	GrA61775	0.00710 ± 0.0002	0.00410 ± 0.0003	0.00303 ± 0.0004		-20.9 ± 0.2	>45000

Sample	Lab. ID	$F^{14}C_{corrected}$	Background ($F^{14}C_{corrected}$)	$F^{14}C_{final}$	$\delta^{13}C$ AMS (‰VPDB)	$\delta^{13}C$ IRMS (‰VPDB)	Age BP
				Kiel collagen			
	KIA50793	0.00899 ± 0.0002	0.00657 ± 0.0022	0.00248 ± 0.0022	-20.4 ± 0.3		> 40000
	KIA50793	0.00878 ± 0.0002	0.00657 ± 0.0022	0.00227 ± 0.0022	-20.8 ± 0.3		> 40200
	KIA50793	0.00689 ± 0.0002	0.00582 ± 0.0019	0.00111 ± 0.0020	-19.2 ± 0.3		> 42500
	KIA50793	0.00650 ± 0.0002	0.00483 ± 0.0016	0.00170 ± 0.0016	-20.2 ± 0.2		> 42700
	GrA62155	0.00920 ± 0.0003	0.00390 ± 0.0003	0.00534 ± 0.0004		-20.3 ± 0.2	42000 + 650/ -600
BQC-78 wt%collagen (Leibniz-Laboratory Kiel): ~4-10 C/N-collagen (Groningen, Oxford): ~3.2	OxA_V-2616-51	0.01096 ± 0.0003	0.00493 ± 0.0005	0.00603 ± 0.0006		-19.8 ± 0.2	41050 ± 750
				Groningen collagen			
	GrA59398	0.00960 ± 0.0003	0.00250 ± 0.0003	0.00712 ± 0.0004		-20.13 ± 0.2	39700 + 500/ -450
	GrA59401	0.01150 ± 0.0003	0.00250 ± 0.0003	0.00903 ± 0.0004		-20.26 ± 0.2	37800 + 400/ -350
	KIA50796	0.00683 ± 0.0002	0.00093 ± 0.0003	0.00591 ± 0.0004	-20.0 ± 0.1		41200 + 500/ -450
	KIA50796	0.00659 ± 0.0002	0.00120 ± 0.0004	0.00540 ± 0.0004	-19.9 ± 0.4		41900 + 700/ -650
	OxA_V-2580-57	0.00947 ± 0.0003	0.00364 ± 0.0005	0.00583 ± 0.0006		-20.1 ± 0.2	41300 ± 800
	OxA_V-2581-55	0.01020 ± 0.0003	0.00387 ± 0.0004	0.00633 ± 0.0005		-20 ± 0.2	40650 ± 650
				Oxford collagen			
	OxA30216	0.00918 ± 0.0004	0.00466 ± 0.0010	0.00452 ± 0.0010		-20.0 ± 0.2	43400 ± 1800
	OxA30473	0.01300 ± 0.0004	0.00739 ± 0.0009	0.00561 ± 0.0010		-20.2 ± 0.2	41600 ± 1500
	KIA50799	0.01165 ± 0.0002	0.00093 ± 0.0003	0.01073 ± 0.0004	-19.4 ± 0.1		36400 ± 300
	KIA50799	0.00852 ± 0.0003	0.00120 ± 0.0004	0.00733 ± 0.0005	-20.3 ± 0.8		39500 + 550/ -500
	GrA61777	0.01110 ± 0.0003	0.00410 ± 0.0003	0.00705 ± 0.0004		-20.5 ± 0.2	39800 + 500/ -450

Figures

Figure 1. Locality (black triangle) of the Brick Quarry site Coenen, Körrenzig, Germany, and Loess distribution along the left lower Rhine valley (Lehmkuhl et al. 2016)

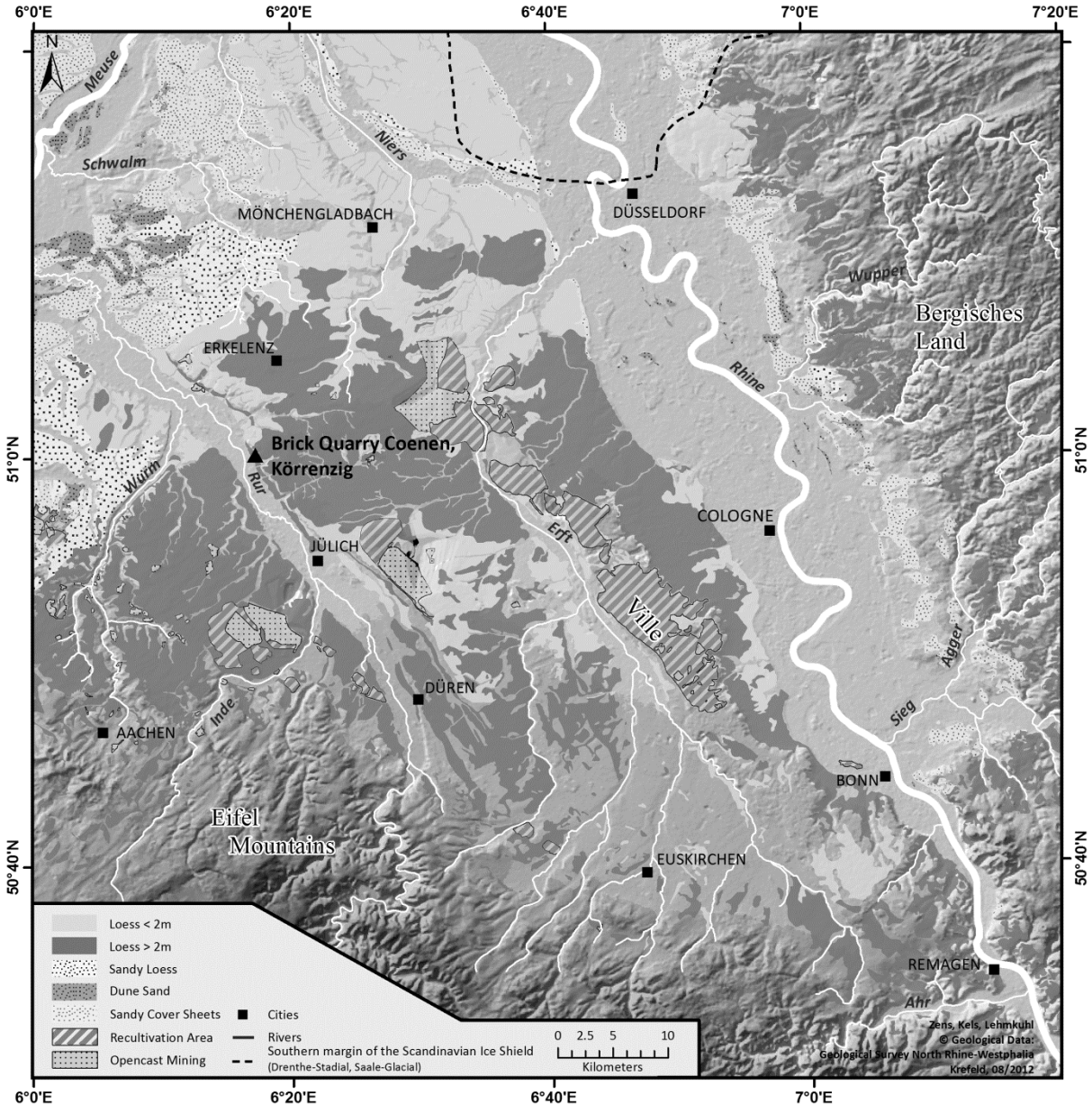
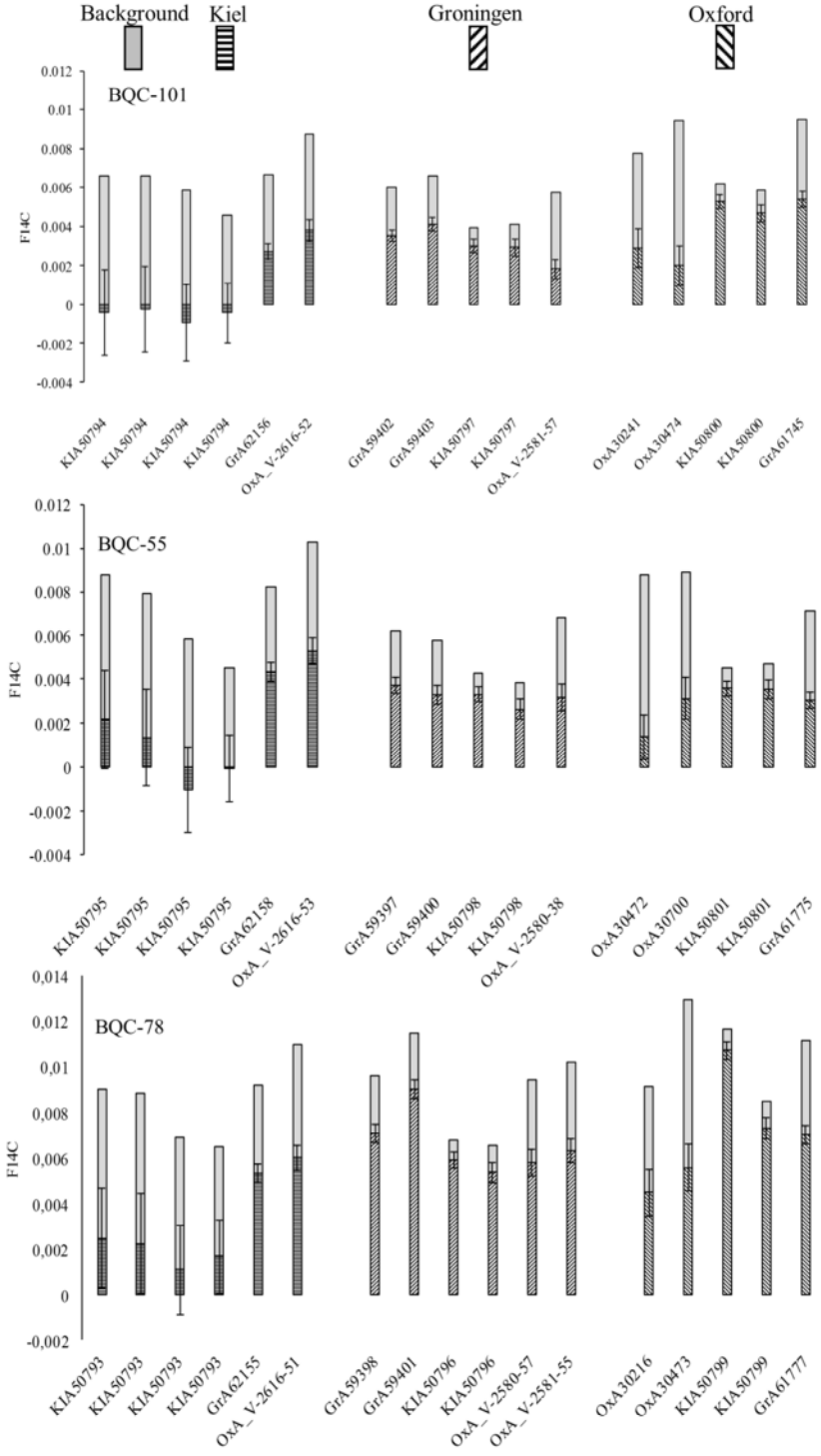


Figure 2. Measured ^{14}C-concentrations of BQC-101, BQC-55, and BQC-78. Patterned columns represent Kiel, Groningen, and Oxford (from left to right) extracted collagen $F^{14}C_{final}$ values. The magnitude of applied background correction is shown in grey columns.





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Laboratory intercomparison of Pleistocene bone radiocarbon dating protocols

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