Inter-laboratory Calibration

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Inter-laboratory calibration of magnetic susceptibility and natural remanent magnetisation measurements of archaeomagnetic samples

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Introduction

AARCH is a Research Training Network which involves 12 laboratories throughout Europe that, apart from the creation of a skilled work force for efficient archaeomagnetic sampling and measuring, aims also to reconstruct the variations of the Earth's magnetic field during the past 10.000 years within Europe, based on common standards.
However, to obtain this aim through a fruitful collaboration of all the network laboratories, it is important that all of them "speak" the same language! The wide range of equipment used by each laboratory and the differences on measurement procedures, make difficult to compare the results obtained by the different laboratories. For this reason, the AARCH team has started an inter-laboratory calibration check in January 2004 involving all 12 AARCH member laboratories. Such inter-laboratory comparison has never been conducted previously.

In October 2003, the AARCH team of the University of Torino oversampled a Roman kiln at Canosa (southern Italy), in order to provide specimens for this inter-laboratory instrument check. Five cylindrical specimens of standard size (diameter = 25.4 mm, height = 22 mm) were prepared from five independently oriented bricks. Measurements involved bulk magnetic low field susceptibility (at room temperature) and natural remanent magnetisation (NRM). The specimens were first measured at the palaeomagnetic laboratory of the University of Torino. In order to check for any significant viscous magnetisation, the specimens were measured three times over a time span of one month before they were sent to the other laboratories. Each laboratory was asked to measure the NRM (inclination, declination and intensity) and the magnetic susceptibility, to specify the equipments used and to provide the results obtained. Responses were received by the Torino AARCH group that co-ordinated this cross-calibration exercise. All AARCH laboratories had received and measured the specimens by the end of February 2005.

Results

Viscosity test

The NRM of the calibration specimens was initially measured on December 2003 at the laboratory of Torino, using a JR-5 spinner magnetometer. The specimens were stored in the Earth magnetic field and were sequentially re-measured in one and two week time periods (see results at Table 1). After this initial viscosity test, the specimens were sent to all the other AARCH laboratories. One year later, in February 2005, the returned specimens were re-measured at the laboratory of Torino and finally they were again measured after the end of the measurements by all the 12 laboratories. Twin specimens were stepwise demagnetised, both thermally and in alternating field, revealing the presence of a very stable magnetic component and a very weak overprint (Fig.1).

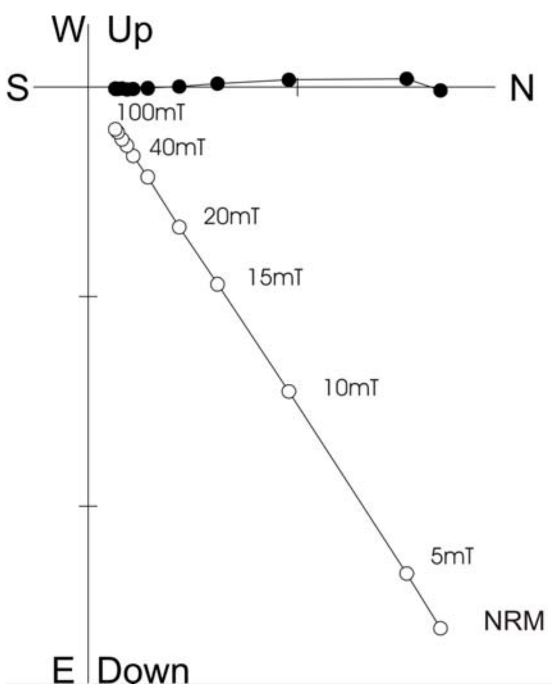

Fig.1: Zijderveld diagram for stepwise alternating field demagnetisation of specimen from Canosa kiln. Symbols: dark dots = declination, light dots = apparent inclination.

The results of these repetitive measurements (Table 1) show, that the viscous magnetisation acquired by the specimens during the one year is very weak. The small discrepancies are rather caused by experimental errors than by a secondary viscous component. For all specimens (except specimen C6) the intensity values measured in the different time periods remain the same, the inclination values vary not more than 2 degrees. Higher variations are only observed in declination values that, however, are more sensitive to the exact repositioning of the specimen in the instrument's holder.

	Specimen C1f			Specimen C3f			Specimen C4f			Specimen C6f			Specimen C8f		
TORINO	D(º)	I(º)	J(A/m)	D(º)	I(º)	J(A/m)	D(º)	I(º)	J(A/m)	D(º)	I(º)	J(A/m)	D(º)	I(º)	J(A/m)
23/12/03	0.7	54.6	4.1	359.2	59.5	12.0	357.8	59.3	8.1	359.8	67.7	0.92	5.6	60.9	2.1
6/01/04	360	54.3	4.1	358.6	58.5	12.0	356.7	58.2	7.9	1.5	66.8	0.84	3.9	58.9	2.0
15/01/04	0.4	54.4	4.0	358.4	58.8	12.0	356.7	58.6	7.9	1.5	67.1	0.85	1.5	59.5	2.0
4/02/05	1.8	54.1	4.1	358.8	58.6	12.0	358.3	58.2	8.0	358.2	65.1	0.87	5.1	59.9	2.1
1/04/05	357.9	54.2	4.0	358.5	59.1	11.7	357.1	57.2	7.9	355.5	65.1	0.84	4.5	60.1	2.1

Table 1: NRM results of the inter-calibration specimens measured at the laboratory of Torino in different time periods.

Magnetic susceptibility results

Bulk magnetic susceptibility at room temperature was measured by 10 of the 12 laboratories. The majority of the laboratories measured the magnetic susceptibility in axial position, while in a few cases systematic measurements of the anisotropy of magnetic susceptibility were taken into account, in order to calculate the bulk susceptibility value. This could be one reason for the small variations observed (Table 2). All susceptibility values are given in 10^{-3} SI, referring to a specimen volume of 11cm³.

Instrument	C1f	C3f	C4f	C6f	C8f
KLY-3	3.120	9.755	12.71	3.947	8.084
Bartington	3.106	9.782	13.18	3.957	7.810
Bartington	2.995	9.515	12.79	3.821	7.294
Bartington	3.064	9.627	12.81	3.981	7.782
KLY-3S	3.258	10.05	13.45	4.071	9.213
KLY-2	3.210	9.860	12.72	3.980	8.100
KLY-2	3.160	9.980	13.01	4.000	8.220
KLY-3	3.193	10.10	13.14	3.990	8.240
Bartington style	2.000*	6.40*	8.90*	2.600*	5.500*
Bartington	3.045	9.600	12.95	3.900	7.509
Mean value:	3.128	9.808	12.973	3.961	8.028
Stand.Dev:	0.09	0.21	0.25	0.07	0.55
Mean value:					
Bartington	3.053	9.631	12.930	3.915	7.599
KLY-2/-3	3.188	9.865	13.006	4.010	8.443

Table 2: Bulk magnetic susceptibility results. All susceptibility values are in 10^{-3} SI.
*: values not taken in consideration for the calculation of the mean value.

Bartington or Kappabridge magnetic susceptibility meters (KLY-2 and KLY-3 models) were used to measure the susceptibility. In one case, a hand made Bartington-style susceptibility meter was used. Having calculated the mean values for both instruments (Table 2), it results that Bartington meters measure slightly lower susceptibility values than Kappabridge. However, this difference is always less than $0.5*10^{-3}$SI unit (with only exception the specimen C8f).

Natural remanent magnetisation results

The natural remanent magnetisation has been measured by all the 12 AARCH laboratories. Together with the specimens, the field orientation values of each brick (strike and dip) have been provided to the participants, in order to transform the measured values into the geographical co-ordinates system. The results are summarised in Table 3; two of the 12 laboratories measured the specimens with two different instruments and therefore the total number of results presented in Table 3 is 14. The majority of the laboratories have used Molspin magnetometers, while in two cases a JR-5 spinner magnetometer was used. Other two laboratories performed their measurements on 2G cryogenic magnetometers. Mean NRM intensity and its standard deviation for each specimen is calculated in Table 3 (bottom).

In order to concisely show differences between the laboratories, the directional results of each specimen are plotted in an equal-area projection (Fig. 2). Mean direction, confidence factor α_{95} and concentration parameter k are given in Table 4. Moreover, the results obtained by each laboratory for the 5 specimens have been used in order to calculate a 'site' mean value (Table 5).

	Specimen C1f			Specimen C3f			Specimen C4f			Specimen C6f			Specimen C8f		
INSTRUMENT	D(°)	I(°)	J(A/m)	D(°)	I(°)	J(A/m)	D(°)	I(°)	J(A/m)	D(°)	I(°)	J(A/m)	D(°)	I(°)	J(A/m)
JR-5	0.4	54.4	4.00	358.4	58.8	12.0	356.7	58.6	7.90	1.5	67.1	0.85	3.7	59.5	2.00
Molspin	352.3	58.2	3.70	356.0	57.5	10.6	348.7	57.6	7.20	357.8	64.8	0.83	352.2	52.8	2.08
Spinner	359.4	55.2	3.45	358.6	59.3	10.03	357.1	58.8	6.90	2.65	65.4	0.76	1.8	60.6	1.83
Molspin	0.39	55.1	3.35	357.6	60.3	9.9	359.3	61.8	6.80	1.94	67.0	0.72	4.2	58.7	1.76
Molspin	350.6	58.4	3.61	355.0	57.9	10.45	354.5	59.8	7.19	351.3	67.3	0.79	353.5	56.1	1.94
2G Cryogenic	1.2	55.3	3.95	356.9	55.1	11.54	358.4	58.5	7.73	355.9	66.0	0.83	353.0	56.0	2.04
2G Cryogenic	354.0	55.2	3.94	354.0	57.6	11.48	356.3	57.6	7.68	354.7	65.0	0.84	356.5	59.5	2.06
Molspin	1.58	54.8	3.13	0.56	60.4	9.15	358.8	59.2	6.27	3.73	66.4	0.67	3.3	61.4	1.63
JR-5	350.7	57.3	4.00	357.9	56.1	11.0	353.6	57.7	8.00	350.2	65.0	0.80	351.4	54.7	2.00
Molspin	353.0	59.1	3.67	1.28	58.4	12.53	355.5	57.0	8.63	353.0	63.1	0.79	6.37	54.9	1.82
Parastatic	351.6	54.7	3.87	2.30	56.6	11.26	357.5	55.0	7.51	358.0	63.6	0.80	7.96	52.2	1.93
Molspin	357.9	57.5	3.39	356.3	58.1	9.86	355.7	59.5	6.42	354.5	65.4	0.70	356.0	56.6	1.81
Molspin	344.7	62.7	4.21	350.0	58.0	13.2	346.0	64.6	8.76	335.5	67.8	0.98	354.2	57.1	2.26
Molspin	344.9	57.6	3.34	359.7	53.9	12.26	352.8	56.4	9.71	356.4	61.7	0.78	5.09	50.3	1.92
Mean value			3.686			11.09			7.623			0.796			1.934
Standard deviation			0.32			1.16			0.95			0.08			0.16

Table 3: Directional results of the 5 calibration specimens together with the calculated mean values and standard deviations for the intensity measurements.

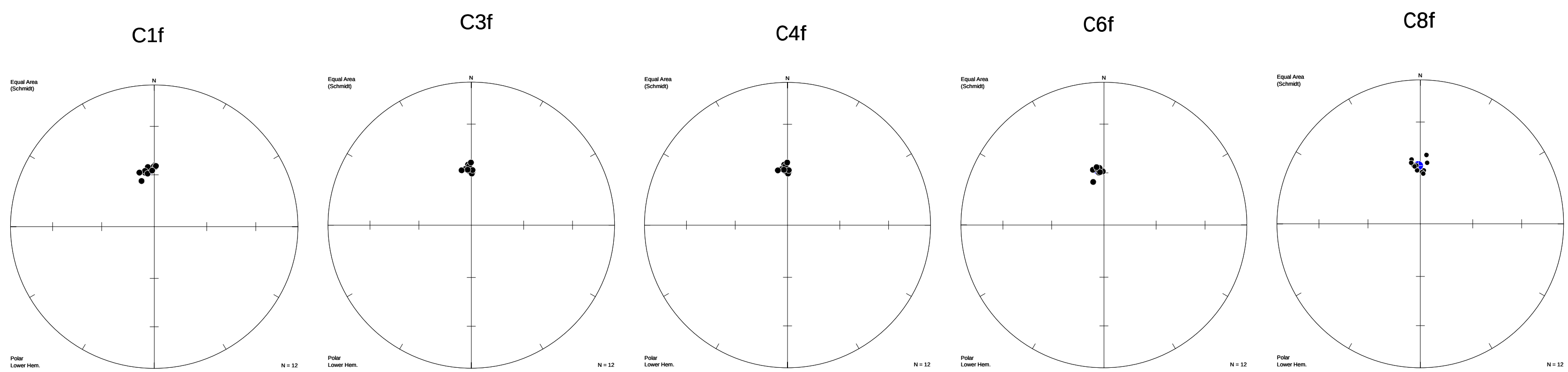

Fig.2: Equal area projections of each specimen, using the NRM directions obtained by all laboratories.

	C1f	C3f	C4f	C6f	C8f
N=12					
Mean D (°)	354.5	357.0	354.6	354.9	358.0
Mean I (°)	57.3	57.6	58.8	65.6	56.7
Alpha-95 (°)	2.1	1.3	1.5	1.8	2.4
k	415	1097	816	552	325

Table 4: Mean direction for each specimen using the results of the 12 AARCH laboratories.

INSTRUMENT	Mean D (°)	Mean I (°)	Alpha-95 (°)	k
JR-5	0.1	59.7	4.5	284
Molspin	353.2	58.2	4.4	303
Spinner	359.8	59.9	3.6	443
Molspin	0.7	60.6	4.3	318
Molspin	351.3	59.9	4.5	295
2G Cryogenic	357.1	58.2	4.6	278
2G Cryogenic	355.1	59.0	3.5	467
Molspin	1.5	60.5	4.1	357
JR-5	352.9	58.2	4.1	350
Molspin	358.1	58.6	4.1	350
Parastatic	359.6	56.5	5.2	214
Molspin	356.2	59.4	3.4	519
Molspin	346.9	62.2	5.2	217
Molspin	356.2	56.2	5.7	180

Table 5: Site mean NRM direction, confidence factor α_{95} and concentration parameter k calculated using the results of the 5 specimens obtained by each laboratory.

Conclusions

Despite different instrumentation and programs used by the participating laboratories, no significant discrepancies on the results are noticed. In most cases, magnetic susceptibility and NRM intensity values show a good agreement among the different laboratories. The NRM intensity standard deviation is around 10%. This error may be also reflected in palaeointensity determinations. Undoubtedly, a calibration of intensity is difficult, as it concerns an absolute value. However, it is important to draw the attention to the maximum precision of measurements and instrumental standards, mainly for the laboratories that carry out palaeointensity determinations.
As far as directional results are concerned, they are well concentrated and the alpha-95 angle is in all cases smaller than 2.5°, when plotting the results of all laboratories for each specimen. The site mean values obtained by each laboratory (Table 5) using the 5 specimens, are statistically indistinguishable within the error limits (confidence circles). It worth to point that these mean directions are obtained from NRM results, only, without any demagnetisation cleaning. An alpha-95 confidence factor of around 4 to 5 degrees could therefore be normally expected. These outcomes give confidence in the results obtained from each laboratory in the Network. However, if single specimen measurements from each laboratory are compared, differences of more than 10° in

declination and inclination may be noticed in some cases. As for archaeomagnetic dating maximum precision is required, it is necessary to further investigate and understand the cause of these differences. The inter-laboratory calibration results can provide a useful base for a thorough control that each laboratory can do at its own measurement system.
This inter-laboratory calibration study is open to other laboratories, for checking their instruments and methods against those used in this study. The calibration specimens will be preserved at the laboratory of Torino, periodically re-measured and in disposition of any laboratory that would be interested to take part at this inter-calibration test.

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