**Supporting information**

**14C GIRI samples in AMS Golden Valley: graphite preparation using AGE-3 and Absorption-catalytic setup**

E.V. Parkhomchuk, A.V. Petrozhitskiy, M.M. Ignatov, D.V. Kuleshov, A.I. Lysikov, A.G. Okunev, K.A. Babina, V.V. Parkhomchuk

Table 1-SI. Results of GIRI samples graphitized by two devices, AGE-3 and ACS, and then measured on MICADAS-28 together with data on δ13C measured by MICADAS-28 and Delta-V-Advantage. F = the measured fraction modern with fractionation correction applied to both the sample and blank samples and corrected for background. Uncertainties represent 1 sigma.

|  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Name/  laboratory ID | Intersection (Scott et al. 2024) | Description | δ13C, ‰ | δ13C, ‰ MICADAS | | | F value | | Radiocarbon age (BP) | | | |
| This work | | | This work | | Preliminary consensus GIRI1 (Scott et al. 2024) |
| Set 1 | | | AGE-3 | ACS | AGE-3 | | ACS | | AGE-3 | ACS |
| A  GV-3799 | TIRI A | barley mash | -26.0 | -24.4 | -24.5 | 1.1644±0.0042 | | 1.1636±0.0076 | | - | - | 1.1643±0.00755 |
| -23.0 | 1.1633±0.0042 | |
| B  GV-3800 | VIRI U | humic acid2 | -29.7 | -28.9 | -29.2 | 0.2294±0.0012 | | 0.2240±0.0041 | | 11826±41 | 12018±148 | 11813±110 |
| -29.9 | -28.0 | 0.2291±0.0012 | | 0.2224±0.0041 | | 11837±41 | 12077±149 |
| -29.5 | 0.2280±0.0041 | | 11877±146 |
| C  GV-3801 |  | barley mash | -28.0 | -29.3 | -27,3 | 1.0075±0.0041 | | 1.0214±0.0070 | | - | - | 1.0227±0.00725 |
| -26,4 | 1.0151±0.0070 | |
| -15,8 | 1.0111±0.0070 | |
| -37,8 | 1.0168±0.0071 | |
| D  GV-3802 |  | humic acid2 | -28.5 | -26.7 | -24.2 | 0.6226±0.0025 | | 0.6114±0.0053 | | 3807±32 | 3952±70 | 3826±71 |
| -27.7 | -29.3 | 0.6227±0.0024 | | 0.6136±0.0053 | | 3805±31 | 3923±70 |
| -30.5 | 0.6141±0.0053 | | 3916±70 |
| E  GV-3803 | SIRI F,G,H | dendro-dated wood | -26.0 | -23.7 | -23,6 | 0.9569±0.0035 | | 0.9498±0.0066 | | 354±30 | 414±56 | 378±49 |
| -25.1 | -22,9 | 0.9558±0.0035 | | 0.9525±0.0066 | | 364±29 | 391±56 |
| F  GV-3804 |  | barley mash | -26.6 | -26.2 | -26.3 | 1.0185±0.0037 | | 1.0085±0.0069 | | - | - | 1.0162±0.01175 |
| -27.0 | 1.0162±0.0037 | |
| G  GV-3805 | TIRI B  FIRI D | dendro-dated wood | -23.4 | -22.0 | -25.6 | 0.5670±0.0023 | | 0.5650±0.0051 | | 4558±32 | 4587±72 | 4523±49 |
| -21.8 | -25.8 | 0.5710±0.0023 | | 0.5697±0.0051 | | 4501±32 | 4520±72 |
| H  GV-3806 |  | dendro-dated wood; single ring | -19.3 | -17.1 | - | 0.7590±0.0029 | | - | | 2215±30 | - | 2208±50 |
| -18.7 | 0.7595±0.0029 | | 2210±30 |
| I  GV-3807 |  | Kauri wood | -22.2 | -19.8 | - | 0.0534±0.0007 | | - | | 23531±104 | - | 23644±168 |
| -20.5 | 0.0543±0.0007 | | 23394±102 |
| -21.4 | 0.0534±0.0007 | | 23539±103 |
| -22.0 | 0.0529±0.0007 | | 23607±104 |
| J  GV-3808 |  | Kauri wood | -21.7 | -20.9 | -22.7 | 0.0085±0.0006 | | 0.0080±0.0038 | | 38265±545 | >35000 | 38571±886 |
| -20.3 | -22.6 | 0.0087±0.0006 | | 0.0064±0.0038 | | 38109±535 |
| -22.9 | 0.0084±0.0006 | | 38364±553 |
| K(1)3  GV-3809 | TIRI L | whalebone | -14.8 | -15.1 | -15.6 | 0.2029±0.0011 | | 0.1994±0.0040 | | 12814±44 | 12951±160 | 12780±114 |
| -12.2 | 0.2025±0.0011 | | 12827±45 |
| K(2)4  GV-3809 | -14.2 | -15.2 | 0.2047±0.0011 | | 0.1994±0.0040 | | 12743±44 | 12951±158 |
| -15.6 | 0.2019±0.0011 | | 12852±44 |
| L  GV-3810 |  | dendro-dated wood; single ring | -19.8 | -17.7 | - | 0.7454±0.0030 | | - | | 2360±32 | - | 2241±58 |
| -16.8 | 0.7352±0.0030 | | 2471±33 |
| M  GV-3811 | VIRI O | dendro-dated cellulose2 | -25.5 | -25.1 | -22.5 | 0.9828±0.0038 | | 0.9780±0.0068 | | 140±31 | 178±56 | 132±32 |
| -24.1 | -20.9 | 0.9813±0.0038 | | 0.9777±0.0068 | | 152±31 | 181±56 |
| N  GV-3812 |  | Kauri wood | -22.1 | -23.6 | -20.4 | 0.0018±0.0006 | | 0.0004±0.0038 | | >52000 | >35000 | 0.002146±0.001815 |
| -22.9 | -20.4 | 0.0024±0.0006 | | 0.0007±0.0038 | |
| Q  GV-3813 |  | dendro-dated wood; single ring | -23.4 | -25.6 | - | 0.9601±0.0038 | | - | | 327±31 | - | 336±46 |
| -24.6 | 0.9589±0.0037 | | 337±31 |
| O  GV-3814 | FIRI E | humic acid2 | -29.9 | -30.0 | -28.7 | 0.2301±0.0012 | | 0.2256±0.0041 | | 11802±41 | 11960±147 | 11826±153 |
| -29.7 | -28.7 | 0.2312±0.0012 | | 0.2257±0.0041 | | 11765±41 | 11959±147 |
| P  GV-3815 | FIRI H | dendro-dated wood | -24.4 | -23.5 | -22.8 | 0.7585±0.0030 | | 0.7529±0.0058 | | 2221±32 | 2280±62 | 2227±62 |
| -24.7 | -22.3 | 0.7582±0.0030 | | 0.7540±0.0058 | | 2223±32 | 2269±62 |

1 – mean value ± stdev

2 – pretreatment not required

3 – δ15N = 12.8 ‰; C/N = 3.2

4 – δ15N = 12.7 ‰; C/N = 3.2

5 – given as F

Let us apply the t-test to the data shown in Table 1-SI. The populations for averaging were very small, from 2 to 4 graphites, making it necessary to use the t-test and any other statistics with a certain degree of caution and awareness that the n=2 test has low significance. Alternatively, the preliminary consensus mean values were calculated from a rather large population, from 48 to 112 measurements, implying that to assess the statistical significance, we should consider the preliminary consensus data as the assumed true value and our measurements as the population mean. Then we compared both series of measurements: from AGE-3 and ACS with the true values (one-sample t-test), as well as both populations of measurements from two series (independent two-sample t-test) (Table 2). However, due to high differences in the populations’ sizes, we did not compare all three populations using any statistics, such as ANOVA.

The calculation of the one-sample t-test was as follows:

, (1)

For an independent two-sample t-test, the following values were calculated to assess the statistical significance (p-value) between AGE-3 and ACS:

, (2)

, (3)

(4)

with “1” and “2” referring to the AGE-3 and ACS series., respectively.

The results of these manipulations are shown in Table 1-SI. Statistical significance was lower than 0.05 for sample **D** in the ACS series, with the other samples having no or minor differences.

Table 2-SI. Student’s t-test comparing the mean values (F or radiocarbon age) of GIRI samples graphitized by AGE-3 and ACS and then measured on MICADAS-28, with the preliminary consensus mean values (Scott et al. 2023) as specified ones (true) and t-test of the values from AGE-3 and ACS.

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Name** | **Laboratory ID** | **Preliminary consensus values** | | | **AGE-3 ⋛ true** | | | | | **ACS ⋛ true** | | | | | **AGE-3 ⋛ ACS** | | | |
| **True** | | | **One-sample t-test** | | | | | **One-sample t-test** | | | | | **Two-sample t-test** | | | |
| **Mean F** | **stdev** | **N** | **Mean F** | **stdev** | **N** | **t** | **p-value** | **Mean F** | **stdev** | **N** | **t** | **p-value** | **n** | **s** | **t** | **p-value** |
| **A** | GV-3799 | 1.1643 | 0.0075 | 99 | 1.1638 | 0.0030 | 2 | 0.22 | 0.86 | 1.1636 | 0.0076 | 1 | 0.10 |  | 1 | 0.008 | 0.04 | 0.98 |
| **C** | GV-3801 | 1.0227 | 0.0072 | 98 | 1.0075 | 0.0041 | 1 | 3.76 |  | 1.0161 | 0.0043 | 4 | 3.11 | 0.053 | 3 | 0.005 | 1.88 | 0.16 |
| **F** | GV-3804 | 1.0162 | 0.0117 | 96 | 1.0174 | 0.0026 | 2 | 0.63 | 0.64 | 1.0085 | 0.0069 | 1 | 1.11 |  | 1 | 0.007 | 1.23 | 0.43 |
| **N** | GV-3813 | 0.002146 | 0.00181 | 104 | 0.0021 | 0.0004 | 2 | 0.17 | 0.89 | 0.0005 | 0.0027 | 2 | 0.85 | 0.55 | 2 | 0.002 | 0.81 | 0.50 |
|  |  | **Mean age** |  |  | **Mean age** |  |  |  |  | **Mean age** |  |  |  |  |  |  |  |  |
| **B** | GV-3800 | 11813 | 110 | 98 | 11832 | 29 | 2 | 0.93 | 0.52 | 11991 | 102 | 3 | 3.01 | 0.095 | 3 | 63 | 2.54 | 0.09 |
| **D** | GV-3802 | 3826 | 71 | 98 | 3806 | 22 | 2 | 1.28 | 0.42 | 3930 | 40 | 3 | 4.48 | **0.046** | 3 | 28 | 4.43 | **0.02** |
| **E** | GV-3803 | 378 | 49 | 113 | 359 | 21 | 2 | 1.30 | 0.42 | 402 | 40 | 2 | 0.86 | 0.55 | 2 | 32 | 1.37 | 0.31 |
| **G** | GV-3805 | 4523 | 49 | 112 | 4530 | 41 | 2 | 0.22 | 0.86 | 4553 | 51 | 2 | 0.84 | 0.56 | 2 | 46 | 0.51 | 0.66 |
| **H** | GV-3806 | 2208 | 50 | 50 | 2213 | 21 | 2 | 0.31 | 0.81 |  |  |  |  |  |  |  |  |  |
| **I** | GV-3807 | 23644 | 168 | 95 | 23518 | 89 | 4 | 2.83 | 0.07 |  |  |  |  |  |  |  |  |  |
| **J** | GV-3808 | 38571 | 886 | 99 | 38245 | 314 | 3 | 1.80 | 0.21 |  |  |  |  |  |  |  |  |  |
| **K** | GV-3809 | 12780 | 114 | 94 | 12810 | 47 | 4 | 1.28 | 0.29 | 12894 | 113 | 2 | 1.43 | 0.39 | 4 | 83 | 1.01 | 0.37 |
| **L** | GV-3810 | 2241 | 58 | 48 | 2416 | 78 | 2 | 3.15 | 0.20 |  |  |  |  |  |  |  |  |  |
| **M** | GV-3811 | 132 | 32 | 91 | 146 | 22 | 2 | 0.87 | 0.54 | 180 | 39 | 2 | 1.72 | 0.34 | 2 | 32 | 1.07 | 0.40 |
| **O** | GV-3814 | 11826 | 153 | 106 | 11783 | 29 | 2 | 2.08 | 0.29 | 11960 | 104 | 2 | 1.83 | 0.32 | 2 | 76 | 2.32 | 0.15 |
| **P** | GV-3815 | 2227 | 62 | 112 | 2221 | 23 | 2 | 0.37 | 0.77 | 2274 | 44 | 2 | 1.54 | 0.37 | 2 | 35 | 1.53 | 0.27 |
| **Q** | GV-3813 | 336 | 46 | 83 | 332 | 22 | 2 | 0.24 | 0.85 |  |  |  |  |  |  |  |  |  |

|  |  |
| --- | --- |
|  |  |