AN EXTRACTION SYSTEM TO MEASURE CARBON-14 TERRESTRIAL AGES OF METEORITES WITH A TANDETRON AMS AT NAGOYA UNIVERSITY

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ABSTRACT. We have constructed a system to extract carbon from meteorites using a vacuum-tight RF melting method in order to study radiocarbon activities in meteorites. The extraction system was examined using iron standards of known carbon content. The carbon extraction efficiencies and ^{14}C ages of the iron standards by this method were compared with the results obtained previously by our older melting system and a wet oxidation method. Higher collection efficiencies of about 90% for the iron samples of relatively high carbon content were achieved by the new system. The efficiency of extracting a small amount of carbon is also near 90% after improving the extraction procedure. The ^{14}C ages of the iron standards were compared to the ages by the wet method. The results indicate that contamination by modern carbon is negligible in the system. Furthermore, terrestrial ^{14}C ages of two Antarctic meteorites, Y-75102 and ALH-77294, from the Yamato and Allan Hills ice fields, respectively, were determined. The age of Y-75102 is estimated 4.0 ± 1.0 ka, and the age of ALH-77294 is 19.5 ± 1.2 ka. The ^{14}C ages on the meteorites roughly agree with the literature value. However, further study is needed in improvement on reducing a background value and of complete fusion of a meteorite in the extraction system.

INTRODUCTION

Terrestrial ages of Antarctic meteorites give us important information to estimate the terrestrial history of the meteorites. The radioisotopes of ^{36}Cl (e.g. Nishiizumi et al. 1979, 1981, 1983), ^{81}Kr (e.g. Freundel et al. 1986; Miura et al. 1993) and radiocarbon (e.g. Fireman 1978; Brown et al. 1984; Jull et al. 1984, 1989a, 1993) have been used in these studies. Since ^{14}C is the shortest half life, it is useful for determining older ages up to 50–60 ka such as are often observed in the Yamato ice field region of Antarctica site (Jull et al. 1993). The ^{14}C ages of meteorites were first measured using conventional counting techniques and large samples (e.g. Fireman 1978). With the advent of accelerator mass spectrometry (AMS), the required sample mass for measurement has been reduced. As a result, many more ^{14}C measurements have been performed intensively by the Toronto AMS group, Canada, the Arizona AMS group, USA, etc. (e.g. Brown et al. 1984; Jull et al. 1984; Beukens et al. 1988). In Japan, AMS ^{14}C measurements of meteorites have scarcely been performed, to our regret. We are now at the early stage of the program for studying ^{14}C activities in meteorites, and constructing a system to extract ^{14}C from meteorites using the RF melting method. We have already had a RF melting system for carbon extraction from iron artifacts (Nakamura et al. 1995).

In this system, an iron sample is combusted with O_2 gas in a RF furnace and combustion gas is collected in a gasbag, and then CO_2 is separated from O_2 in a vacuum line. The system, however, had some problems: possibility of a leak from the gasbag and contamination by modern carbon, and complicated handling. For a sample of low carbon content, especially for a meteorite, it is necessary to avoid contamination by foreign carbon. Therefore, we converted the extraction system; the section for combustion with a RF furnace is connected to the section for CO_2 separation from O_2 in a vacuum line, and the whole system is evacuated. Furthermore, we improved the glass-line to improve the separation of CO_2 from O_2 and to raise extraction efficiency. In this paper, we report results of carbon extraction from iron standards of known carbon content and their ^{14}C ages by the new extraction system. We also report the ^{14}C activities of two Antarctic meteorites, compared with the already reported ^{14}C terrestrial ages.

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SAMPLES

We used three iron standards of different carbon content: 4.67% C cast iron supplied by LECO Corporation, 0.196% C steel (part No. JSS 030-7) and 0.049% C steel (part No. JSS 201-12) supplied by the Japan Iron and Steel Federation. Two Antarctic meteorites of Y-75102 (L6) and ALH-77294 (H5) from the ice fields at Yamato and Allan Hills, respectively, were examined to measure ^{14}C concentrations. The terrestrial ages of Y-75102 are reported to be 4.2 ± 0.8 ka with conventional counting (Fireman 1983) and 1.7 ± 0.3 ka by accelerator mass spectrometry (Jull et al. 1984). The results of ALH-77294 are 30 ± 0.8 ka with conventional counting (Fireman and Norris 1981), and 9.5 ± 1.0 ka (Jull et al. 1989) and 16.5 ± 1.3 ka (Jull et al. 1998) by AMS.

EXPERIMENTS

The CO_2 extraction system from meteorites was modified after Jull et al. (1993). A sample was mixed with about 2 g of high purity Fe (LECO Corporation, part No. 502-231) and placed in an alumina crucible with a lid. The crucible and lid had been preheated at 1000 °C for 10 hr just before use. The crucible containing a sample was placed in a RF furnace (LECO HF-10) and then the closed glass-line system was evacuated. The sample was combusted in the RF furnace in a flow of purified carbon-free O_2. The meteorite samples were preheated in a muffle furnace at 500 °C prior to the combustion in the RF furnace to remove organic contamination and low-temperature weathering products, while iron standards were not preheated. The sample gases evolved were passed through a MnO_2 trap at room temperature and a Pt/CuO trap at 500 °C, and then the sample CO_2, together with water and liquid oxygen, was collected in several liquid N_2 traps (–196 °C). After combustion, the sample is allowed to cool, and then the sample CO_2 with water was separated in liquid N_2 traps by pumping out O_2 completely. The CO_2 was separated from water using ethanol slush traps (–100 °C). The amount of $^{14}CO_2$ was determined by a pressure transducer in a calibrated volume and diluted, if necessary, with a known amount of ^{14}C-free CO_2. The total CO_2 was graphitized by reducing with hydrogen in a Fe-powder catalyst and the produced graphite was measured for its ^{14}C concentration with a Tandetron AMS at the Center for Chronological Research, Nagoya University. Standards are graphite made from NBS oxalic acid (RM-49). The ^{13}C values were measured by a Finnigan MAT-252 mass spectrometry using an undiluted aliquot of the CO_2 gas.

RESULTS AND DISCUSSION

Iron Standards

The extraction efficiency and ^{14}C age of the iron standards by the new RF melting system are summarized in Table 1. The results by the old melting system (Yamada et al. 1997) and by the wet method (Oda et al. 1999) are also shown in Table 1. The wet method consists of dissolution of an iron sample in 2M $CuCl_2$ solution at 60 °C, dissolution of deposited Cu in 4M HCl at 60 °C, and collection of precipitated colloidal carbon on quartz wool by filtration (Oda et al. 1999). The ^{14}C ages of the iron standards by the new system are older than the ages of corresponding iron samples by the old melting system, and agree with those by the wet method. The result means organic contamination by modern carbon is decreased and negligible in the new melting system. The carbon collection efficiency is high for extracting a large amount of carbon, more than 1 mg (i.e. No. 1, 2 and 3 of 4.67% C iron standards; No. 1 and 2 of 0.196% C iron standards), whereas the efficiency is low for extracting the a small amount of carbon, less than 0.5 mg (i.e. No. 7 of 0.196% C iron standard; No. 1 and 2 of 0.049% C iron standards). The result indicates a fixed loss of carbon during combustion and/or CO_2 separation from O_2 in the system: probably during the latter, removing O_2 from mixed gas of CO_2 and O_2 collected in a liquid N_2 trap. The fixed loss of carbon by the new melting system

Table 1 Extraction results of iron standards

Sample/ run number	Sample weight (g)	Carbon content (mg)	Yield of CO_2 (mgC[a])	Extraction efficiency (%)	Loss of carbon (mg)	$\delta^{13}C_{PDB}$ (%)	^{14}C age[b] (BP)
LECO (4.67% C) (Old melting method)							
1	0.095	4.44	4.25	96.2	0.19	–25.9	38,570 ± 420
2	0.091	4.25	4.07	96.2	0.18	–26.1	38,170 ± 580
3	0.033	1.54	1.36	87.0	0.18	–26.6	—
4	0.016	0.75	0.63	83.7	0.12	–26.5	—
5[c]	0.073	3.41	3.23	95.4	0.18	–26.2	—
6	0.008	0.37	0.34	93.7	0.03	–26.4	—
(Old melting method)[d]							
7	0.10	4.67	2.05	43.7	2.62	–26.1	30,150 ± 390
8	0.10	4.67	3.61	77.4	1.06	–26.4	31,230 ± 300
9	0.10	4.67	4.17	86.7	0.50	–25.9	32,400 ± 520
(Wet method)[e]							
10	0.159	7.43	6.15	83.0	1.28	25.2	37,150 ± 330
11	0.067	3.13	2.66	84.8	0.47	25.1	36,290 ± 330
JSS (0.196% C) (Old melting method)							
1	1.012	1.98	1.78	90.2	0.20	–23.5	25,710 ± 350
2	1.004	1.97	1.79	93.7	0.18	–24.9	27,380 ± 260
3	1.035	2.03	1.57	77.2	0.46	–23.8	26,340 ± 270
4	1.005	1.97	1.50	76.0	0.47	–24.2	25,630 ± 270
5	1.008	1.98	1.56	79.0	0.42	–24.2	25,080 ± 330
6	0.524	1.03	0.84	82.1	0.19	–24.4	—
7	0.217	0.43	0.24	56.9	0.19	–26.0	—
8	1.044	2.05	1.83	89.3	0.22	–24.4	—
9	0.233	0.46	0.40	87.7	0.06	—	—
(Old melting method)							
10	1.49	2.92	2.39	81.6	0.53	–23.3	25,560 ± 230
11	1.52	2.98	2.36	79.5	0.62	–23.5	25,550 ± 280
12	1.50	2.94	2.63	89.5	0.31	–23.3	25,100 ± 220
(Wet method)							
13	1.529	3.00	2.65	88.4	0.35	–23.0	25,980 ± 270
14	1.530	3.00	2.59	86.4	0.41	–23.3	25,330 ± 270
JSS (0.049% C) (New melting method)							
1	1.005	0.49	0.33	66.4	0.16	–24.7	—
2	/1.052	0.51	0.27	52.8	0.24	–26.4	—
3	1.034	0.51	0.45	88.5	0.06	–25.8	—
(Old melting method)							
4	2.49	1.22	0.99	81.1	0.23	–23.6	19,930 ± 290
5	2.00	1.96	0.54	55.5	1.42	—	20,750 ± 340
6	2.03	0.99	0.83	83.3	0.16	—	20,410 ± 830
(Wet method)							
7	6.004	2.94	2.47	84.1	0.47	–25.0	20,030 ± 190

[a]Converted weight as carbon
[b]Errors are 1σ
[c]Saturated activity used for estimation of terrestrial ^{14}C ages; these footnoted data were obtained after improving the carbon extraction method in our system.
[d]Data by the old melting method are from Yamada et al. (1997)
[e]Data by the wet method are from Oda et al. (1999)

is 0.12–0.47 mg, which is smaller than 0.16–2.62 mg using the old melting system and 0.35–1.28 mg using the wet method. To reduce the fixed loss of carbon and raise extraction efficiency for a small amount of carbon, the procedure of carbon extraction was improved. The improvements are (1) decrease in O_2 flow at combustion, (2) increase in the number of liquid N_2 traps for condensing CO_2, and (3) slowly pumping out O_2 to prevent sample CO_2 gas from flowing away together with O_2. The data marked with an asterisk are obtained by the improved extraction procedure. The efficiency of extracting a small amount of carbon was improved to 90% (i.e. No. 6 of 4.67% C iron standard; No. 9 of 0.196% C iron standard; No. 3 of 0.049% C iron standard).

The $\delta^{13}C_{PDB}$ values for each run of three iron standards are in good agreement. The $\delta^{13}C_{PDB}$ for 4.67% C and 0.049% C iron standards are around –26‰, lower by 2‰ than those for 0.196% C iron standard. The difference of $\delta^{13}C_{PDB}$ value indicates that the sources of carbon in the iron standards are different. The different ^{14}C ages of the three iron standards support this thought.

Antarctic Meteorites

Table 2 shows the ^{14}C concentrations and the resulting ^{14}C terrestrial ages of two Antarctic meteorites. The fourth column gives the $^{14}C/^{12}C$ ratio for the sample divided by the $^{14}C/^{12}C$ ratio for the NBS oxalic acid standard (RM-49). The measurement error is given as one standard deviation. The fifth column gives the disintegration rate of cosmogenic ^{14}C of the meteorites. The terrestrial age is calculated by the following equation:

$$\text{Terrestrial age} = \tau \ln (A_{saturated} / A_{sample}) \qquad (1)$$

where τ is the mean life of the radionuclide, $A_{saturated}$ is the saturated activity and A_{sample} is the activity in the sample. The τ is 8268 years in the case of ^{14}C. The saturated activities for some recently fallen L-chondrites were measured: Bruderheim (49.8 ± 1.8 dpm/kg, Brown et al. 1984; 54.6 ± 0.5 dpm/kg, Cresswell et al. 1993; 51.9 ± 0.3 dpm/kg, Jull et al. 1993; 47.6 ± 2.0 dpm/kg, Knauer et al. 1995), Peace River (55.1 ± 1.0 dpm/kg, Cresswell et al. 1993), Peekskill (51.1 ± 0.4 dpm/kg, Graf et al. 1996) and Mbale (58.1 ± 0.4 dpm/kg, Jull et al. 1998). The recently fallen H-chondrites have the saturated activities of 44 ± 1 dpm/kg for Holbrook (Jull et al. 1998) and 42 ± 2 dpm/kg for Torino (Wieler et al. 1996). The averages of them are 53 dpm/kg for the L-chondrites and 43 dpm/kg for the H-chondrites. We do not have any data on saturated activities for recently fallen chondrites in our laboratory, so that 53 dpm/kg and 43 dpm/kg were used as the saturated activities of Y-75102 (L6) and ALH-77294 (H5), respectively. The errors of terrestrial ages were determined only by the experimental errors in the ^{14}C determinations.

To measure the background on ^{14}C measurements of meteorites, blank samples of crucibles with 2 g high purity iron chips were studied. The samples were preheated to 500 °C in the muffle furnace as above described. The blank gives 0.27 ± 0.03 dpm/kg. In Table 2, the data of chondrites are shown with correction for this blank value. The dilution factor of $^{14}CO_2$ in a sample with ^{14}C-free CO_2 is between 20 and 50.

Our terrestrial ages of Y-75102 almost agree with the values of Fireman (1983), though a little younger than the age of Jull et al. (1984). Jull et al. (1984) reported that the weathering age of Y-75102, which is calculated from ^{14}C released from the sample at 500 °C, is 5.1 ± 0.5 ka, slightly older than its terrestrial age. They explain the older age with weathering of the meteorite by slightly older melt water. The age of Y-75102 as a whole considered from the terrestrial and the weathering ages is 2.8 ± 0.5 ka, consistent with our result within the error. The ^{14}C terrestrial ages of ALH-77294 are between the value of Fireman and Norris (1981) and the value of Jull et al. (1989b), and

Table 2 Terrestrial ^{14}C ages of two Antarctic meteorites

	Weight (g)	CO_2 (cm^3 STP)	$(^{14}C/^{12}C)$sam / $(^{14}C/^{12}C)$std	^{14}C (dpm/kg)	Saturated activity[a]	Terrestrial age (ka)	Reference
Y-75102	0.982	0.015	74.6 ± 0.8	35.3 ± 1.1	57	3.0 ± 0.7	This work
	0.680	0.014	44.9 ± 0.3	30.1 ± 1.5	57	4.2 ± 0.7	This work
	—	—	—	34.1 ± 2.7	57	4.2 ± 0.8	Fireman (1983)
	5.0	7.6	—	46.3 ± 1.4	57	1.7 ± 0.3	Jull et al. (1984)
ALH-77294	0.832	0.147	2.0 ± 0.1	4.2 ± 0.2	44	20.1 ± 0.8	This work
	0.831	0.048	4.1 ± 0.2	3.9 ± 0.2	44	20.7 ± 0.9	This work
	10.5	1.32	—	1.6 ± 0.3	61	30 ± 2	Fireman and Norris (1981)
	0.42	—	—	13.9 ± 0.3	44	9.5 ± 1.0	Jull et al. (1989)

[a]Saturated activity used for estimation of terrestrial ^{14}C ages; these footnoted data were obtained after improving the carbon extraction method in our system.

close to the value of Jull et al. (1998). In experimental procedure for ^{14}C determinations, Fireman (1983) and Fireman and Norris (1981) use stepwise heating and small-counter technique, and Jull et al. (1984, 1989b, 1998) use RF melting and AMS technique. We used the latter technique. The result of the small-counter method tends to be oldest, the result of the AMS method by Jull et al. is youngest, and our result is intermediate. The difference would be caused by analytical uncertainties, contamination in samples, incomplete fusion of samples and background value in the extraction system. For small sample size used, an amount of CO_2 extracted is closed to blank level, so that the blank-corrected data have large errors. In Table 2, a very small quantity of CO_2 is extracted from the meteorite samples. This means incomplete extraction and/or conversion of CO to CO_2, although samples can be actually have very low carbon content.

The saturated activity for ^{14}C varies with the depth and size of the meteorite (Reedy 1985; Graf et al. 1990; Jull et al. 1993; Wieler et al. 1996). Wieler et al. (1996) reported that chondrites of pre-atmospheric radii from 20 to 45 cm have the saturated activity varying from about 40 to 52 dpm/kg and that smaller chondrites have very lower saturated activities, from the measured and calculated ^{14}C activities in the Torino fragment (H6). The additional error is not included in estimating of terrestrial ages. The ages, therefore, might have more uncertainly.

Further studies are needed to improve analytical technique: reducing background value of the extraction system and complete combustion of meteorites by such as longer heating time in RF furnace and use of much more combustion accelerator. It is indispensable to measure saturated ^{14}C activity of recently fallen meteorites with our extraction system. Furthermore, shielding or depth corrections are needed for ^{14}C terrestrial age determination of a meteorite sample if the meteoroid was very large or very small. We intend to obtain the other radioisotope data such as ^{10}Be to estimate the shielding effect. By normalizing the saturated activity of ^{14}C to that of ^{10}Be in a meteorite, more correct terrestrial age for the meteorite could be obtained.

CONCLUSION

An extraction procedure for ^{14}C from meteorites was constructed. The ^{14}C age measurements by the RF melting method from several iron standards indicate good agreement with earlier values by the wet method. The RF melting procedure gives high carbon extraction efficiency of around 90% for low carbon content samples as well as for high carbon content samples. The contamination by modern carbon is negligible in the system, from the result of ^{14}C ages for the iron standards. Furthermore, terrestrial ^{14}C ages of two Antarctic meteorites, Y-75102 and ALH-77294, from the Yamato and Allan Hills ice fields, respectively, were determined. The age of Y-75102 is estimated 4.0 ± 1.0 ka, and the age of ALH-77294 is 19.5 ± 1.2 ka. The ^{14}C ages on the meteorites roughly consistent with the earlier literature values. However, further study for the problems of incomplete combustion and considerable background in the system is needed on meteorite samples.

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