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^{14}C TERRESTRIAL AGES OF NINE ANTARCTIC METEORITES USING CO AND CO_2 TEMPERATURE EXTRACTIONS

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Abstract: As a continuation of the work reported in R. P. BEUKENS *et al.* (Proc. NIPR Symp. Antarct. Meteorites, **1**, 224, 1988), a suite of Yamato and Trans-Antarctic Mountains meteorites have been analyzed for their ^{14}C content, to give an estimate of their terrestrial age. Further information on the samples' terrestrial history may be obtained from analysis of separate carbon species evolved at different temperatures. We report on data obtained from CO and CO_2 separations from a low temperature (500°C to 900°C) and high temperature ($\approx$1600°C) fraction from each sample. Three Allan Hills meteorites, ALH-77232 (H4), ALH-78112 (L6) and ALH-78130 (L6), give terrestrial ages >29000 years, and probably represent limit ages due to *in situ* production. Three other Trans-Antarctic meteorites, BTN-78002 (L6), MET-78028 (L6) and RKP-78002 (H4), give finite ^{14}C ages between 15000 and 27000 years. Three Yamato meteorites, Y-8011 (L6), Y-81132 (H5) and Y-82095 (L3), give ages between 16000 and 22000 years. The low temperature components indicate that recent weathering and atmospheric exchange has taken place for at least four (and possibly six) of the meteorites; ALH-78112, ALH-78130 and Y-81132 have low temperature activities that suggest an earlier exposure and weathering.

1. Introduction

The determination of a meteorite's terrestrial, or residence, age relies on an accurate assessment of the saturated state of a spallogenic radionuclide, and the ability to make a clean separation of that spallation component from any other sources of the radionuclide of interest. For ^{14}C ($T_{1/2}=5730$ years), which is useful for terrestrial ages up to 40000 years, sources other than from spallation are numerous (Table 1) and can be difficult to separate. While most of these other sources are treated as contamination, the possibility that further information on the meteorite's terrestrial history may be obtained from measurement of ^{14}C in any weathering components has garnered some interest (BROWN *et al.*, 1984; JULL *et al.*, 1992).

Mass pyrograms (*e.g.*, for Bruderheim, Fig. 1) are used to delimit the release patterns of carbon species, allowing an initial separation of components by temperature. Major release peaks occur at 200°C–400°C and 500°C–800°C, and the rise corresponding to breakdown of silicates begins at 1100°C. The lowest temperature release peak is dominated by atmospheric contaminants and is discarded. KOTRA *et al.* (1982) showed that carbonates will decompose between 400°C and 900°C, suggesting that this tem-

Table 1. Sources of radiocarbon in meteorites.

Source of ^{14}C	Conditions	^{14}C levels
1) Spallation: i) Cosmic rays	dependent on sample chemistry; shielding effects galactic origin	10^5–3×10^8 atoms/g (0.02–70 dpm/kg)
ii) *in situ*	dependent on location; shielding; fission products	10^3–10^7 atoms/g (0.01–1.0 dpm/kg)
2) Terrestrial contamination	recovery conditions, storage weathering, atmospheric exchange	up to 10^8 atoms/g (up to 20 dpm/kg)
3) Laboratory contamination	sample preparation, carrier gas, analysis	10^5–10^6 atoms (0.02–0.23 dpm)

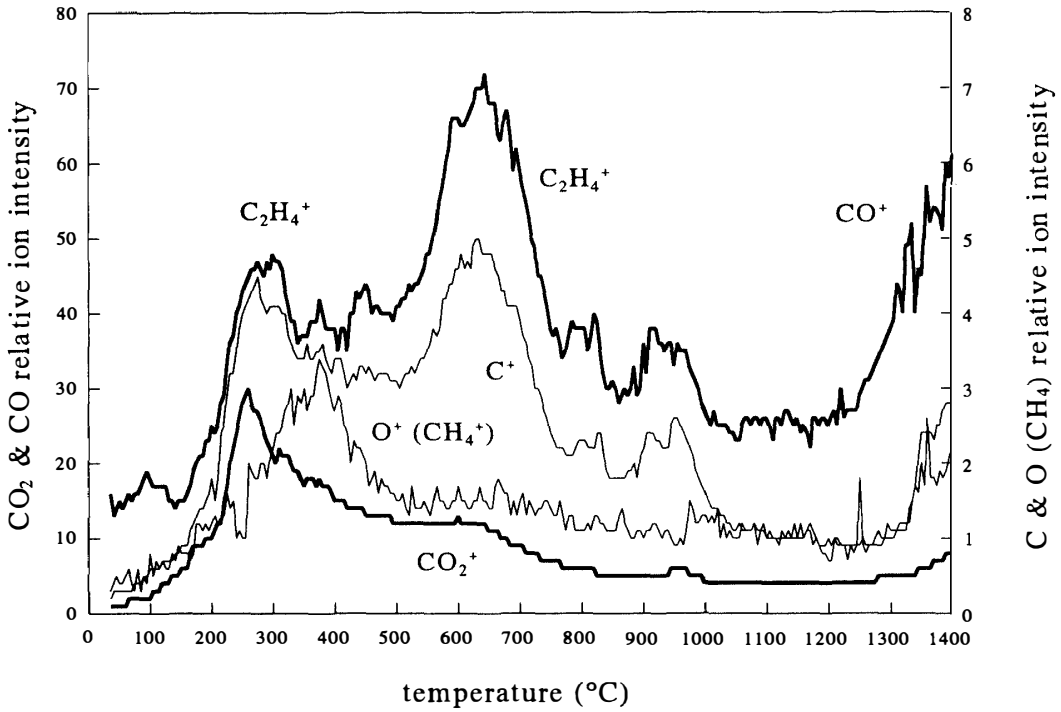

Fig. 1. Mass pyrogram for Bruderheim (L6) for masses: 12 (C^+), 16 (O^+ and CH_4^+), 28 (CO^+) and 44 (CO_2^+). 76.8 mg of finely crushed meteorite was heated at 10°C/min from 35°C to 1400°C.

perature fraction seen in meteorites is due to the presence of weathering efflorescences that have been described from many samples (*e.g.*, VELBEL *et al.*, 1991).

Spallation ^{14}C is generated predominantly through the reaction: $^{16}O(p, 3p)^{14}C$ (REEDY and ARNOLD, 1972), and thus is found mainly in the silicate phases of meteorites. This is only released at high temperatures, through diffusion of the carbon out of the crystal lattice.

2. Procedure

An extraction system for the pyrolysis of small (200–500 mg) samples has been

Table 2. Comparison of saturated values for recent falls.

Meteorite	Class	Weight (g)	CO_2		CO		CO_2+CO	
			ppm C	dpm/kg	ppm C	dpm/kg	ppm C	dpm/kg
Bruderheim	L6	0.430	214	8.85 (0.51)[1)]	682	45.72 (1.67)	896	54.57 (0.53)
Peace River	L6	0.434	802	20.62 (0.53)	975	34.47 (0.53)	1777	55.09 (1.06)
Abee	EH4	0.356	253	5.30 (0.32)	8296[2)]	47.97 (0.64)[2)]	8549[2)]	53.27 (0.97)[2)]

[1)] 1σ errors (dpm/kg).
[2)] Includes a contribution of 215 ppm (yielding 4.04 dpm/kg) from the re-melt.

designed and built at the IsoTrace Laboratory. Gas and temperature separations are possible, using the procedure developed by CRESSWELL (in prep.). As well as carrying out temperature separations, evolved gases are separated into fractions based on their freezing points. Thus, gases with freezing points above -150°C (123 K), predominantly CO_2, are separated from non-condensibles, largely CO, and both fractions are measured for their ^{14}C content. The Bruderheim meteorite (L6: fall date, March 4th, 1960) has been used as the saturated standard, cross-checked using Peace River (L6: fall date, March 31st, 1963) and Abee (EH4: fall date, June 9th, 1952)(Table 2). As a comparison, the value adopted here for the spallation component in Bruderheim (54.57 ± 0.53 dpm/kg) compares to 52.9 ± 2.3 measured by KIGOSHI and MATSUDA (1986), 51.1 ± 1.5 adopted by MIURA *et al.* (1992) and 50.1 ± 0.3 taken as the reference level for terrestrial age determinations by BEUKENS *et al.* (1988).

Samples of 300–500 mg are lightly crushed to less than 18 mesh (1 mm) and loaded in a molybdenum crucible in a zirconia boat inside a quartz pyrolysis tube. The crucible and boat were previously cleaned by baking to 1100°C under a vacuum of $<10^{-3}$ torr for 1 hour, followed by heating the molybdenum crucible to white heat (>1300°C) using the induction furnace. Once the sample is loaded, the crucible and boat are pre-heated to 500°C in air and pumped to remove any adsorbed atmospheric CO_2 and hydrocarbons (SWART *et al.*, 1983), then slowly cooled to room temperature. After 8 hours, the sample is re-heated under vacuum to 500°C, and any evolved gases are pumped away. Approximately 1 *l* of ultra-pure helium is then added to the system, bringing the internal pressure to about 500 torr, and is cycled, using a peristaltic pump, over the sample and liquid-nitrogen-cooled traps. The temperature of the sample is raised to ≈ 900°C, and held for 1 hour to decompose carbonates, and any secondary-products present on the meteorite's surface. Evolved CO_2 is trapped in a variable temperature trap (modified after DESMARAIS, 1978), while non-condensibles are oxidized over CuO at 650°C and trapped using liquid-nitrogen-cooled pyrex traps.

Three m*l* of ^{14}C-free CO_2 is added to each aliquot, and the gas is converted to a graphitic material, as described by BEUKENS *et al.* (1986). This produces two machine-ready targets for accelerator mass spectrometry (AMS) analysis.

The procedure is repeated at melt temperatures, whereby the molybdenum crucible is heated by induction until it is white hot, melting its contents. The molybdenum also acts as a sulphur getter, effectively removing any SO_2 that may have evolved. The melt is maintained for 7 hours to ensure complete extraction of the carbon, which diffuses

Table 3. Background/contamination measurements.

		cc STP	dpm ($\times 10^{-3}$)
Carrier gas	^{14}C-free CO_2	(3.0)	0.05 ± 0.02
Crucible blank	CO_2: 500–900°C	0.13	0.27 ± 0.05
	CO: 500–900°C	0.04	0.74 ± 0.08
	CO_2: 900°C-melt	0.07	0.56 ± 0.06
	CO: 900°C-melt	0.09	0.35 ± 0.07

to the molten surface, reacts with oxygen from the silicate breakdown, and is swept away by the helium. This extended melt, however, introduces a background of 1.5×10^6 atoms ^{14}C to the CO component (mainly through leakage at the peristaltic pump, where Tygon®-pyrex seals are used), which currently limits this procedure to measuring samples with ages less than 35000 years, based on comparison to yields from Bruderheim (Table 3).

A 4 hour repeat of the melt procedure is then performed to ensure that all the carbon has been extracted. If more than 0.01 m*l* is retrieved, this is diluted and analysed for ^{14}C.

The $^{14}C/^{12}C$, measured using AMS, is converted to number of ^{14}C atoms, backgrounds are subtracted (Table 3), and results are converted to dpm/kg. The melt fractions are compared to equivalent fractions from the Bruderheim meteorite: this ratio is then converted to a terrestrial age assuming Bruderheim to be saturated with ^{14}C. The low temperature fractions are converted back to $^{14}C/^{12}C$ and can be compared to ambiemt levels in the atmosphere. These levels peaked shortly after the intensive bomb tests of the late 1950s and early 1960s to a level of 1.94×10^{-12} (NYDAL and LÖVSETH, 1983), falling to the present day level of 1.35×10^{-12}.

3. Results

To check the system for contamination, a crucible blank was run under identical conditions as for the samples. The results are listed in Table 3, together with the measurement on the CO_2 used as the dilutant. These backgrounds currently limit terrestrial age determinations to <35000 years, making accurate assessment of *in situ* levels difficult. Recently, blanks have been cut by a factor of three, making such studies feasible. All results for the Antarctic meteorites have had backgrounds subtracted as indicated in Table 3.

Results for the low and high temperature, and CO and CO_2 fractions are given in Tables 4 and 5. The high temperature fractions have been converted to terrestrial ages in Table 6. The results are shown in Fig. 2. Based on studies of the Bruderheim meteorite (CRESSWELL in prep.) the low temperature fractions are assumed to be derived from weathering, while the high temperature fractions are from spallation.

The low temperature component has previously been used to give a so-called 'weathering age' (*e.g.*, BROWN *et al.*, 1984; JULL *et al.*, 1984). The weathering component, however, is the sum of all ^{14}C accumulated by the meteorite, in the form of secondary

Table 4. Radiation in low temperature (500°C–900°C) extractions from Bruderheim and 9 Antarctic meteorites.

Meteorite	Class	weight (g)	CO_2		CO		CO / $(CO+CO_2)$	^{14}CO / $(^{14}CO+^{14}CO_2)$	Combined $CO+CO_2$ $^{14}C/^{12}C$ $(\times 10^{-12})$
			ppm C	$^{14}C/^{12}C$ $(\times 10^{-12})$	ppm C	$^{14}C/^{12}C$ $(\times 10^{12})$			
Bruderheim	L6	0.430	115	2.09 (0.20)	60	2.73 (0.40)	0.34	0.41	2.31 (0.13)
Y-8011	L6	0.327	149	1.49 (0.15)	147	1.51 (0.19)	0.50	0.60	1.86 (0.18)
Y-81132	H5	0.451	280	0.64 (0.03)	93	0.20 (0.02)	0.25	0.51	0.52 (0.05)
Y-82095	L3	0.454	72	1.37 (0.19)	83	4.50 (0.56)	0.54	0.79	3.04 (0.24)
ALH-77232	H4	0.420	119	3.52 (0.34)	93	—[1]	0.44	—[1]	1.97 (0.19)
ALH-78112	L6	0.383	162	0.53 (0.04)	85	—[1]	0.33	—[1]	0.33 (0.18)
ALH-78130	L6	0.458	58	1.63 (0.27)	58	—[1]	0.50	—[1]	0.82 (0.32)
BTN-78002	L6	0.423	82	1.19 (0.16)	94	2.38 (0.28)	0.53	0.69	1.82 (0.23)
MET-78028	L6	0.372	190	2.28 (0.16)	229	1.08 (0.06)	0.55	0.36	1.63 (0.10)
RPK-78002	H4	0.413	156	4.78 (0.38)	130	0.73 (0.06)	0.45	0.11	2.94 (0.14)
						mean =	0.45 (0.10)	0.51 (0.22)	

[1] ^{14}C measured at background levels.

Table 5. Radiocarbon in the high temperature (900°C-melt) fraction of Bruderheim and 9 Antarctic meteorites.

Meteorite	Class	Weight (g)	CO_2		CO		CO	^{14}CO
			ppm C	dpm/kg	ppm C	dpm/kg	$(CO+CO_2)$	$(^{14}CO+^{14}CO_2)$
Bruderheim	L6	0.430	214	8.85 (0.51)	682	45.72 (1.67)	0.76	0.84
Y-8011	L6	0.327	72	—[1]	334	5.80 (0.28)	0.82	—[1]
Y-81132	H5	0.451	101	1.78 (0.22)	95	4.30 (0.54)	0.48	0.71
Y-82095	L3	0.454	66	0.98 (0.17)	125	1.96 (0.18)	0.65	0.66
ALH-77232	H4	0.420	25	0.75 (0.38)	221	1.33 (0.27)	0.90	0.64
ALH-78112	L6	0.383	123	1.49 (0.30)	105	0.37 (0.30)	0.46	0.20
ALH-78130	L6	0.458	57	0.35 (0.25)	180	0.65 (0.25)	0.76	0.65
BTN-78002	L6	0.423	57	1.33 (0.30)	319	7.38 (0.59)	0.85	0.85
MET-78028	L6	0.372	62	0.67 (0.16)	287	1.75 (0.18)	0.82	0.72
RKP-78002	H4	0.413	39	0.53 (0.18)	169	2.41 (0.37)	0.81	0.82
						mean =	0.80 (0.15)[2]	0.72 (0.19)[2]

[1] ^{14}C measured at background levels.
[2] Excluding ALH-78112 and Y-81132 (see text).

Table 6. Terrestrial ages of 9 Antarctic meteorites.

Meteorite	Class	Weight (g)	CO_2 age (ka)	CO age (ka)	Combined age (ka)
Y-8011	L6	0.327	—	17.1 ± 0.5	18.5 ± 2.2
Y-81132	H5	0.451	13.2 ± 1.1	19.5 ± 1.1	18.1 ± 1.2
Y-82095	L3	0.454	18.2 ± 1.5	26.0 ± 1.1	24.1 ± 1.4
ALH-77232	H4	0.420	20.4 ± 4.2	29.3 ± 1.7	27.0 ± 2.2
ALH-78112	L6	0.382	14.7 ± 1.7	>26.4[1]	27.9 ± 4.0
ALH-78130	L6	0.458	>15.0[1]	35.2 ± 3.1	33.1 ± 3.8
BTN-78002	L6	0.423	15.7 ± 1.9	15.1 ± 0.7	15.2 ± 0.8
MET-78028	L6	0.372	21.3 ± 2.0	27.0 ± 0.8	25.8 ± 1.0
RKP-78002	H4	0.413	23.3 ± 2.8	24.3 ± 1.2	24.1 ± 1.5

[1] 2σ limits only.

deposits, since its arrival on earth. Hence, we here refer only to a low temperature activity, rather than a weathering age. As a first approximation for meteorites from Antarctica, we can assume a simple two stage accumulation of contemporary carbon. ^{14}C is first acquired when the meteorite falls on the ice cap, and is buried in the ice together with some air. Weathering of the meteorite may occur, with absorption of CO_2 in any effloresences. Accumulation of contemporary CO_2 is re-activated when the meteorite re-emerges at the stranding site. If all weathering occurred in the ice, the weathering age should be close to the terrestrial age derived from the cosmogenic component, and would plot along the exponential curve in Fig. 2. If all weathering occurred at the stranding site, and has continued to the present day, exchange with atmospheric carbon dioxide would give a modern signature, indicated by the horizontal lines. While four of the meteorites have clearly undergone recent weathering, three still

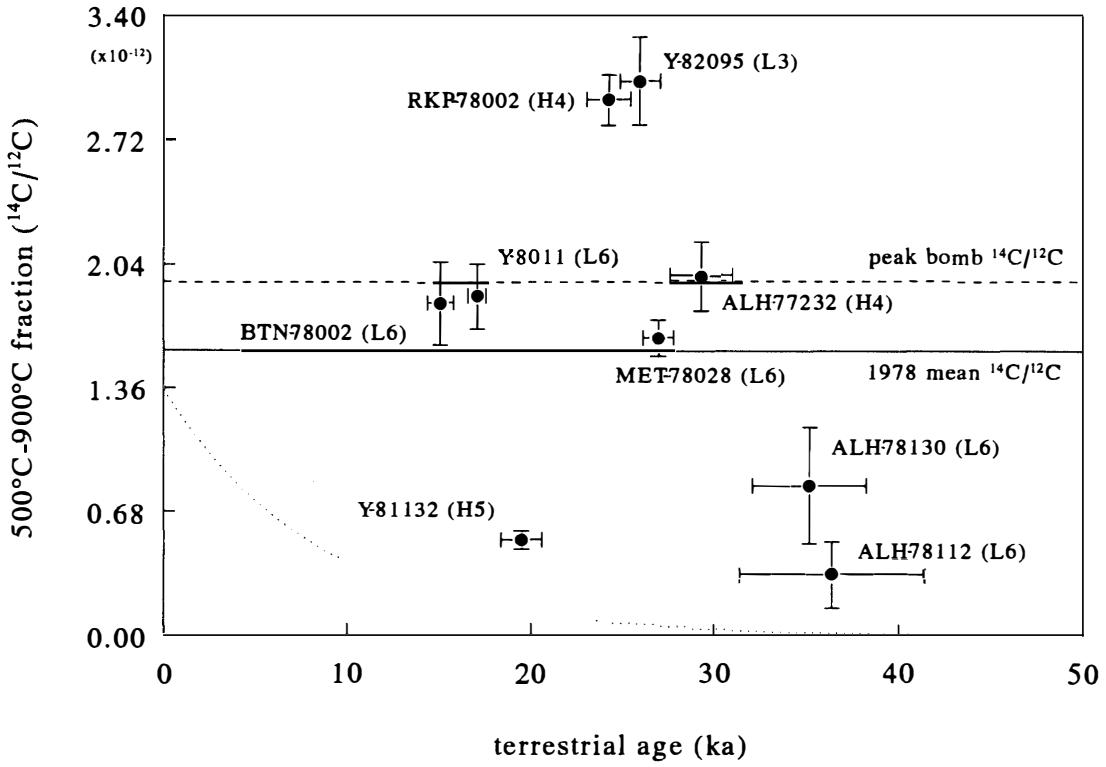

Fig. 2. *^{14}C data for nine Antarctic meteorites, described in the text. Terrestrial ages are calculated from the CO component of the 900°C to melt fraction, and compared to the activity of Bruderheim. Ages are calculated using $T_{1/2}=5730$ years. The abscissa plots the combined ($CO+CO_2$) activity from the low temperature (500°C–900°C) fraction. The dotted curve represents the locus of terrestrial age = low temperature activity, based on a present day ratio of $\sim 1.35 \times 10^{-12}$; the solid line represents the $^{14}C/^{12}C$ ratio for the southern hemisphere in 1978; the dashed line is the peak of atmospheric ^{14}C activity following nuclear bomb tests in the late 1950's and early 1960's for the southern hemisphere (NYDAL and LÖVSETH, 1983).*

retain a significant original component. Two meteorites plot above the modern signature line, suggesting that a cosmogenic component may have been present in the lower temperature fraction.

The mean $CO/(CO+CO_2)$ for the low temperature (500°C–900°C) fractions suggests equal formation of CO and CO_2 at the extraction conditions (900°C under a helium atmosphere). This is corroborated by the similar mean for $^{14}CO/(^{14}CO+^{14}CO_2)$ (Table 4).

Terrestrial ages (Table 6) are consistent with previous measurements on samples from the Yamato and the Trans-Antarctic Mountains (NISHIIZUMI, 1990), in so far that the Yamato samples are generally younger than those from the Trans-antarctic Mountains. The Allan Hills samples give terrestrial ages that are essentially at background levels. In particular, ALH-78130 yields a combined $CO+CO_2$ ^{14}C content of 1.00 ± 0.51 dpm/kg, which is comparable to the *in situ* level of 0.93 ± 0.02 dpm/kg, measured in a volcanic glass near the same site (BEUKENS *et al.*, 1988).

The mean $CO/(CO+CO_2)$ ratio for melt fractions of 0.80 ± 0.15, is close to that expected for samples held at the iron/wustite buffer at elevated temperatures

(COUDURIER *et al.*, 1985). The $^{14}CO/(^{14}CO + ^{14}CO_2)$ ratio of 0.72 ± 0.19 is in agreement with the results of ROWLAND and LIBBY (1953) on irradiated solids (CO_2 and $NaHCO_3$), and more recently with studies on *in situ* ^{14}C produced in ice (LAL *et al.*, 1990). ALH-78112 and Y-81132 have not been included in this analysis as the low $CO/(CO + CO_2)$ ratios suggest non-equilibrium conditions.

CO_2 terrestrial ages are generally younger than CO ages. This is due to the smaller amount of gas liberated as CO_2, commonly only 0.02 m*l* (equivalent to ≈ 30 ppm for a 350 mg sample) above background, and thus containing a greater proportion of contaminant CO_2 from the atmosphere relative to the CO component. This also results in errors generally twice those of the CO results (Table 6). CO suffers less from this source of contamination, and the CO ages, therefore, represent a better estimate of the true terrestrial age of the sample. This is also reflected in the combined age (calculated from the sum of ^{14}C atoms), which is generally closer to the age given by the CO than by CO_2.

The excess over modern seen in the low temperature fractions from meteorites Y-82095 (L3) and RKP-78002 (H4), and probably due to spallation ^{14}C, can be added to the ^{14}C in the melt to estimate the total spallation component. This yields a revised terrestrial age of 18.1 ± 2.1 ka for Y-82095 (L3), within error of the age determined from the melt CO_2 fraction. Applying the same rational for the excess ^{14}C from the low temperature fraction from RKP-78002 (H4), a revised terrestrial age of 14.5 ± 1.7 ka is obtained. ALH-77232 (H4) also yielded high ^{14}C in the low temperature fraction though when combined with the CO fraction a $^{14}C/^{12}C$ ratio in accord with ambient levels is obtained. It is not clear whether this is due to a spallation component, or from partitioning of the heavy isotope to the CO_2 phase. These two results also indicate that the extraction procedure may not be applicable for ^{14}C analysis of all meteorite classes.

4. Conclusions

By taking temperature and gas separations from the pyrolysis of meteorites, we feel that a better understanding of the nature of ^{14}C in meteorites has been achieved. The CO component of the high temperature fraction yields a better estimate of the meteorite's true terrestrial age than that derived from a combined CO and CO_2 age, due to a reduced contribution from atmospheric contamination.

Low temperature (500°C–900°C) fractions give some insights into the meteorite's terrestrial history. Recent weathering at the stranding site appears to contribute significantly to many samples' ^{14}C content, although this is not universal, nor isolated to one region.

Ideally, mass pyrograms should be constructed for each sample prior to ^{14}C analysis, to delimit the optimum temperatures for each gas separation. While the L6 meteorites that have been analysed give results that may be simply explained, the two H4 chondrites, ALH-77232 and RKP-78002, appear to yield a lower temperature spallation component, which evolves as CO_2. The L3 chondrite, Y-82095, yields anomalously high ^{14}C in the low temperature CO fraction. The origin of this elevated level has not yet been ascertained, though sample contamination cannot be ruled out.

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