Age of Tephra Beds at the Ocean Point Dinosaur Locality, North Slope, Alaska, Based on K-Ar and ⁴⁰Ar/³⁹Ar Analyses

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Chapter C

Age of Tephra Beds at the Ocean Point Dinosaur Locality, North Slope, Alaska, Based on K-Ar and ⁴⁰Ar/³⁹Ar Analyses

By JAMES E. CONRAD, EDWIN H. McKEE, and BRENT D. TURRIN

A multidisciplinary approach to research studies of sedimentary rocks and their constituents and the evolution of sedimentary basins, both ancient and modern

U.S. GEOLOGICAL SURVEY BULLETIN 1990

EVOLUTION OF SEDIMENTARY BASINS----NORTH SLOPE BASIN

U.S. DEPARTMENT OF THE INTERIOR MANUEL LUJAN, JR., Secretary

March 3, 1849

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U.S. GEOLOGICAL SURVEY Dallas L. Peck, Director

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Cover. Looking upsection (north) along Colville River, Alaska, at 30-m-high bluffs of Cretaceous nonmarine siltstone and sandstone of Prince Creek Formation, unconformably overlain by Pliocene and Quaternary Gubik Formation. Low bench in foreground marks a dinosaur-bone-bearing bed. Drawing by J.F. Vigil, from photograph by E.M. Brouwers.

Age of Tephra Beds at the Ocean Point Dinosaur Locality, North Slope, Alaska, Based on K-Ar and ⁴⁰Ar/³⁹Ar Analyses

By James E. Conrad, Edwin H. McKee, and Brent D. Turrin

Abstract

Tephra layers exposed above and below dinosaur-bonebearing beds of the Prince Creek Formation, North Slope, Alaska, provide an opportunity to date these beds by radiometric methods. Although the tephra layers contain few or no potassium-bearing minerals, they are composed of apparently unaltered glass shards that appear to be suitable for K-Ar dating. ⁴⁰Ar/³⁹Ar incremental-heating analyses were conducted in order to evaluate the accuracy of the K-Ar ages, and these analyses indicate that most of the tephra layers have undergone only minor argon loss. Most of the K-Ar and * Ar/39Ar ages are analytically indistinguishable and indicate that the bone-bearing beds are between 68 and 71 Ma. The dinosaur-bone-bearing beds of the Prince Creek Formation therefore appear to be of latest Cretaceous age (the Cretaceous-Tertiary boundary is currently considered to be about 66 Ma).

INTRODUCTION

The age of dinosaur-bone-bearing beds of the Prince Creek Formation exposed along the Colville River near Ocean Point, Alaska (fig. 1), has aroused much interest in the geologic community because of its bearing on the hypothesis that the Cretaceous-Tertiary extinction was caused by an asteroid impact (Alvarez and others, 1980). Paleontological studies based on marine faunas have suggested that at least part of the section exposed along the Colville River is Paleocene in age (Marincovich and others, 1985), and a fission-track age on zircon of 50.9 ± 7.7 Ma from a tephra bed near the dinosaur-bone-bearing beds (Carter and others, 1977) seemed to confirm this age designation. If the Paleocene age is valid it would extend the age range of dinosaurs in Alaska into the Cenozoic and would cast doubt on the impact hypothesis as an explanation for the extinction of the dinosaurs. Other studies suggest that at least the part of the Prince Creek Formation that contains the dinosaur bones is Late Cretaceous in age (McDougall, 1987; Brouwers and others, 1987).

Abundant tephra beds exposed along the Colville River bluffs about 7 km west of Ocean Point (fig. 1) contain material suitable for dating by K-Ar techniques and provide an excellent means for establishing the age of these sedimentary rocks. This paper presents the results of conventional K-Ar and ⁴⁰Ar/³⁹Ar analyses of glass shards from the tephra beds and evaluates the suitability of these relatively old rhyolitic glasses for ⁴⁰Ar/³⁹Ar and K-Ar dating techniques.

GEOLOGIC SETTING

The Prince Creek Formation consists of a series of poorly consolidated sandstones, siltstones, and shales that dip gently to the northeast (Gryc and others, 1951; Detterman and others, 1975). The Colville River has cut into this formation, creating a bluff that stands some 20–30 m high and extends many kilometers upriver from Ocean Point.

At least 10 tephra beds are exposed in the section northwest of Ocean Point, and volcanic glass is a major component in the sedimentary rocks in much of the remainder of the section (fig. 2). Almost all the tephra beds are associated with dark beds that are rich in organic matter and probably represent flood-plain deposits (Phillips, 1988). Most of the dinosaur bones are found in these organic-rich horizons, although scattered bones occur in some sandstone beds.

The tephra beds stand out as resistant, buff-colored strata ranging from about 1 cm to 2 m in thickness. They interfinger with the organic-rich beds, pinching out over distances of hundreds of meters. The tephra is composed almost entirely of rhyolitic glass shards, although clays, quartz, and feldspar are minor constituents, and altered biotite grains occur in some of the tephra beds. The glass

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shards appear well preserved with sharp and ragged edges; whole bubbles remain (fig. 3). Microscopic examination revealed no traces of devitrification or alteration of the glass shards, suggesting that the glass is suitable for radiometric dating. The completely unabraided character of the fragile shards indicates they are airfall deposits that have not been transported by water for any distance. Clay minerals are generally a minor component of most of the tephra beds, although some beds are completely altered to clay. The differences in alteration are probably attributable to differences in initial composition of the glass and differences in local ground-water effects.

Because there are few or no potassium-bearing mineral phases in the tephra beds, glass shards were used for K-Ar and ⁴⁰Ar/³⁹Ar dating of the beds. Although relatively young tephra deposits are routinely dated by the K-Ar method, the reliability of glass older than about 15 million years for dating by this technique is questionable because glass tends to readily devitrify. This devitrification results in the redistribution or loss of radiogenic argon, which in turn leads to anomalous ages (Fleck and others, 1977). Glass shards from the Colville River tephra beds appear fresh and well preserved. In order to evaluate the accuracy of the ages, multiple K-Ar, ⁴⁰Ar/³⁹Ar total-fusion, and in-cremental, or age-spectrum, ⁴⁰Ar/³⁹Ar analyses were performed and the resulting ages compared. The age-spectrum analyses in particular provide a means to evaluate the possibility of alteration or diffusion that would lead to anomalous ages.

METHODS

Prior to analysis, the tephra samples were disaggregated and sieved to 100 to 140 mesh (149 to 105 micrometers). After ultrasonic cleaning to remove dust and clays adhering to the shards, a combination of magnetic and heavy-liquid separation techniques was used to produce a pure glass separate. In order to minimize contamination and insure homogeneity, a variable-density heavy-liquid column was used to separate the glass shards into a relatively narrow specific-gravity range. This technique also helps to eliminate grains with a greaterthan-average area of altered surfaces. For example, it was found that bubble-wall shards, with a higher surface-tovolume ratio than the bubble-corner shards, tended to be slightly less dense, presumably owing to a greater percentage of surface alteration.

The glass separates were etched in 5-percent hydrofluoric acid for approximately 45 seconds to remove surface alteration and reduce atmospheric-argon contamination. Conventional K-Ar analyses on shards not treated with acid gave ages as much as 10 percent younger than ages from the acid-treated samples. Furthermore, the nontreated samples had four times the atmospheric-argon contamination displayed by the treated samples; this contamination is significant in determination of the precision (\pm factor) of the age.

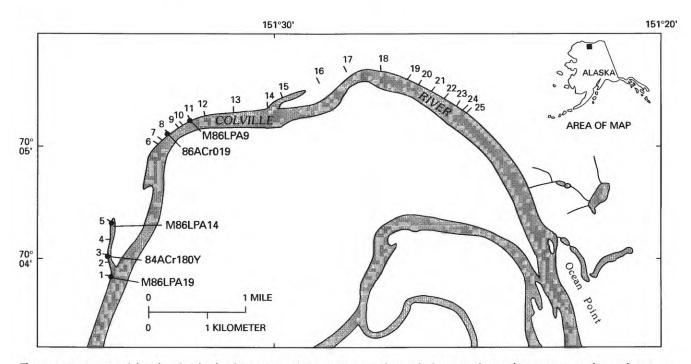
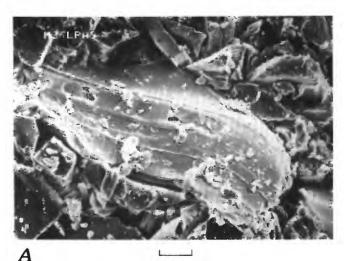


Figure 1. Locations of dated tephra beds of Prince Creek Formation, northern Alaska. Sample numbers correspond to tephra samples dated by K-Ar and (or) ⁴⁰Ar/³⁹Ar techniques, discussed in text. Numbers 1–25 mark locations of measured sections (Phillips, 1988).

Conventional K-Ar analyses were performed using standard isotope-dilution techniques similar to those described by Dalrymple and Lanphere (1969). A 60°-sector, 15.2-cm-radius, Nier-type mass spectrometer was used for argon analysis. Potassium analyses were performed by a lithium metaborate flux fusion-flame photometry technique, using lithium as an internal standard (Ingamells, 1970). Samples used in the ⁴⁰Ar/³⁹Ar experiments were heated by two different techniques—induction coil (Dalrymple and Lanphere, 1969) and continuous laser (York and others, 1981). Samples heated by induction coil—the more conventional technique—were irradiated in the U.S. Geological Survey TRIGA¹ nuclear reactor (Denver, Colo.) for a period of 20 hours at a power of 1 megawatt. Irradiation

¹TRIGA (Training Research Isotope General Atomic) is a registered trademark of General Atomics.





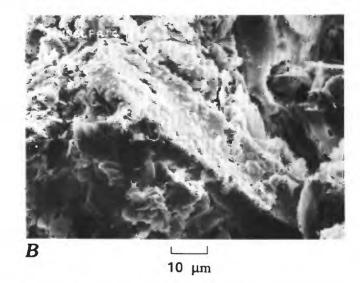
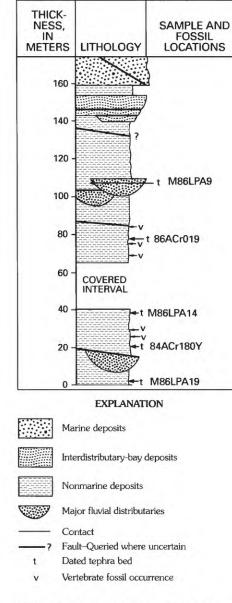


Figure 3. Scanning electron micrographs of untreated glass shards from Colville River tephra beds. *A*, Sample M86LPA9 shows little surface alteration and produced an undisturbed ⁴⁰Ar/³⁹Ar age spectrum. *B*, Sample M86LPA14 has significantly more surface alteration and showed evidence of recoil in ⁴⁰Ar/³⁹Ar analysis. See figure 1 for sample locations.



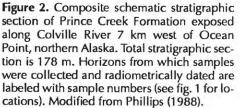


Table 1. Results of conventional K-Ar analyses of Colville River tephra beds [Samples listed in probable stratigraphic order, with sample from lowermost tephra bed at bottom of table. rad, radiogenic; σ , standard deviation. Constants: $\lambda_{\epsilon} + \lambda'_{\epsilon} = 0.581 \times 10^{-10} \text{ yr}^{-1}$, $\lambda_{\beta} = 4.962 \times 10^{-10} \text{ yr}^{-1}$, $\frac{40}{40} \text{ K/K}_{\text{total}} = 1.167 \times 10^{-4} \text{ mol/mol}$]

Sample number	Experiment number	Material	K ₂ O (weight percent)	⁴⁰ Ar rad (mole/g x 10 ⁻¹⁰)	⁴⁰ Ar rad (percent)	Age±1σ (Ma)
M86LPA9	871589	Glass	5.74	5.811	88.8	69.5±2.1
86ACr019	871535	Glass	3.87	3.732	77.0	65.8±2.0
M86LPA14	871590	Glass	3.83	3.817	70.7	68.5±2.0
M86LPA19	871452	Glass	5.46	5.647	76.7	70.4±2.1

flux was monitored by use of the SB-3 biotite standard, which has an age of 162.9 Ma. Heating steps were onehalf-hour intervals with higher temperatures (greater than about 750 °C) monitored by optical pyrometer. Temperatures of lower temperature steps are approximate, based on extrapolation from higher temperature steps along known curves of RF (radio-frequency) generator power plotted against temperature. Isotopic analyses were made at the U.S. Geological Survey in Menlo Park, Calif., using a multiple-collector mass spectrometer (Stacey and others, 1981). Sample handling techniques and corrections for calcium- and potassium-derived isotopes used in the Menlo Park laboratory are as described by Dalrymple and Lanphere (1971, 1974).

Samples used for the laser-heating analyses were irradiated in the TRIGA reactor at the University of California, Berkeley. Irradiation flux was monitored by use of standards MMhb-1 hornblende, with an age of 520.4 Ma, GHC 305 biotite, with an age of 103.76 Ma, and P-207 muscovite, with an age of 82.1 Ma. The following potassium and calcium corrections for the UC Berkeley TRIGA reactor were determined using optical grade CaF_2 and a laboratory potassium glass: ${}^{40}Ar_{K}/{}^{39}Ar_{K}=0.0086$; ${}^{39}Ar_{Ca}/{}^{37}Ar_{Ca}=0.0003$; and ${}^{36}Ar_{Ca}/{}^{37}Ar_{Ca}=0.0003$.

The laser-heated ⁴⁰Ar/³⁹Ar extractions and isotopic analyses were conducted at the Geochronology Center at the Institute of Human Origins (Berkeley, Calif.) using a laser-fusion microextraction system and an online, ultrasensitive mass spectrometer. Argon backgrounds for this system are approximately the following: ${}^{40}\text{Ar}=4.0\times10^{-12}$ cm³ (STP); ${}^{39}\text{Ar}=1.0\times10^{-13}$ cm³ (STP); ${}^{37}\text{Ar}=7.6\times10^{-14}$ cm³ (STP); and ${}^{36}\text{Ar}=3.9\times10^{-14}$ cm³ (STP). Detection limit for this mass spectrometer is on the order of 1.0×10⁻¹⁴ cm³ (STP). Use of laser-light energy for fusion greatly reduces the background argon because there is less nonfocused heating and subsequent outgassing of the ultrahigh vacuum system. This system is designed to analyze samples as small as single grains (less than 0.1 mg), but its chief advantage for this study is that as many as four individual fusions and analyses per hour are possible. For the age-spectrum experiments, less than about 0.25 mg (approximately 30 to 40 shards) was used. The temperature

not determined; progressive heating of the sample was achieved by gradually increasing laser output at each heating step to produce roughly equivalent gas fractions as determined by trial and error. Laser output started at 0.5 watts for the initial heating steps and was increased at 0.25- to 0.5-watt increments until fusion occurred. The duration of each heating step was approximately 20 seconds. The decay constants used in the age calculations are

of the heating steps for the laser-heating experiments was

those recommended by Steiger and Jäger (1977). Reported errors are an estimate of the standard deviation of analytical precision and were calculated using standard propagation-of-error methods (Taylor, 1982).

RESULTS

Conventional K-Ar Analyses

Conventional K-Ar ages on the Colville River tephra beds range from 65.8 ± 2.0 to 70.4 ± 2.1 Ma (table 1). The four samples listed in table 1 (see also fig. 1) are in probable stratigraphic order. (Sample 84ACr180Y is absent from table 1 because it was not dated by the K-Ar method.) Faults and covered intervals in the section prohibit a direct correlation between some of the dated tephra units, but in general correlation is good, and the section becomes younger to the northeast judging from the dip of the beds. Within the limits of analytical uncertainty, the ages are indistinguishable from one another, so these data are consistent with a general northeasterly younging of the section across faults and covered intervals. The age of sample 86ACr019 is concordant with the other ages, but ⁴⁰Ar/³⁹Ar data (described later) suggest that this sample may have undergone some argon loss. The K-Ar data indicate that the age of these beds ranges from about 70-71 Ma to about 66-68 Ma.

40Ar/39Ar Analyses

⁴⁰Ar/³⁹Ar analyses were performed to explore the possibility that some or all of the conventional K-Ar ages

may be too young. Anomalously young ages could result if the glass has lost argon via diffusion or alteration. The ⁴⁰Ar/³⁹Ar incremental, or age-spectrum, technique is used to evaluate the possibility of argon loss or redistribution caused by reheating or alteration of minerals. Although it is rarely used on terrestrial volcanic glasses, the fundamental concepts of the 40 Ar/ 39 Ar spectrum technique (Lanphere and Dalrymple, 1978) are the same as when applied to crystalline materials. A sample is heated to progressively higher temperatures, and a series of ages, termed apparent ages, is calculated from the argon expelled at each temperature step. Typically, a history of alteration or reheating of a mineral sample will result in younger ages for the low-temperature steps. Higher temperature steps will produce older ages that may form a plateau around a value thought to reflect the true cooling age of the rock. In general, this pattern is attributed to a higher argon retentiveness of the high-temperature sites in the crystal lattice. Although glass is not directly comparable to minerals in that it lacks a well-developed crystal structure, ⁴⁰Ar/³⁹Ar incremental heatings can nonetheless give insight into the argon retentiveness of glass. The assumption used for interpreting incremental heatings of glass is that the low-temperature steps (about 300-500 °C) release loosely held argon from the outside edges of the glass shards and from alteration products. Higher temperature steps (about 550 °C and higher) release argon (by volume diffusion) from inner portions of the shards that have been less affected by alteration and diffusion over geologic time. Alteration and diffusion of argon will be most pronounced along the edges of the shards; low-temperature steps will reveal these effects of argon loss by yielding anomalously young apparent ages.

All samples were analyzed by either induction-heating or laser-heating 40 Ar/39 Ar techniques, and one sample (M86LPA9) by both methods (table 2). Three of the samples (M86LPA19, 84ACr180Y, M86LPA9) produced well-defined plateaus in the argon-release sequence (fig. 4A) and show only minor radiogenic argon loss at low-temperature steps. The amount of argon loss can be estimated by comparison of plateau ages and recombined total-fusion ages. In a total-fusion analysis, a sample is completely fused after irradiation and all of the Ar released is analyzed in a single experiment. To simulate this technique, the data can be combined from the incremental Ar releases to calculate a total-fusion age, known as a recombined total-fusion, or total-gas, age for each sample. The recombined total-fusion ages for all samples are about 1 m.y. younger than the plateau ages (table 3), indicating loss of radiogenic argon of less than 2.5 percent. Two of the samples (M86LPA14, 86ACr019) give less welldefined plateaus, and their calculated ages appear anomalous on the basis of the inferred stratigraphic sequence. ⁴⁰Ar/³⁶Ar versus ³⁹Ar/³⁶Ar isochron diagrams and ${}^{36}\text{Ar}/{}^{40}\text{Ar}$ versus ${}^{39}\text{Ar}/{}^{40}\text{Ar}$ correlation diagrams for these samples also show evidence of disturbance (figs. 4*B*, 4*C*).

Sample M86LPA19

This tephra bed is inferred to be the oldest unit dated in this study on the basis of its stratigraphic position. Glass from the tephra was dated twice by the age-spectrum method and once by the total-fusion method using laser heating (table 2). The plateau ages from the two age-spectrum experiments are 70.0±0.9 and 71.4±0.6 Ma (table 3). Although temperatures of individual steps were not determined, the general plateau shape is similar to that of samples for which temperatures are known. Both age spectra for this tephra unit show evidence of minor loss of ⁴⁰Ar in the low-temperature steps. This loss is expressed by young ages in the first step of experiment L115-2, which involved about 7 percent of the total release of ³⁹Ar from the sample, and in the first two steps of experiment L115-3, which involved about 32 percent of the total ³⁹Ar release (fig. 4A). The difference in release patterns between the two experiments may be due to inhomogeneous heating by the laser heat source, leading to incomplete degassing at the lower temperature steps. In spite of this inconsistency, total radiogenic ⁴⁰Ar loss, estimated by comparison of the recombined total-fusion ages with the plateau ages, is less than about 2 percent in each analysis.

Ages calculated from the ⁴⁰Ar/³⁶Ar versus ³⁹Ar/³⁶Ar isochron diagrams (fig. 4B) and 36 Ar/ 40 Ar versus 39 Ar/ 40 Ar correlation diagrams (fig. 4C) for these experiments agree with the plateau ages according to the criteria of Lanphere and Dalrymple (1978). The error estimates (\pm) derived from the isochron and correlation diagrams are slightly higher than those calculated from the plateaus because of the extremely low percentage of atmospheric ³⁶Ar contamination in the laser-fusion line. ³⁶Ar for these samples was typically only slightly above background values, so analytic error is relatively high. In addition, measured ⁴⁰Ar/³⁶Ar ratios, especially for the higher temperature plateau steps, are greater than about 3,000 (fig.4B), so that the isochron must be projected relatively far back to the axis from which the initial ⁴⁰Ar/³⁶Ar ratio is derived. The combined effect of these factors is that the ages calculated from the isochron and correlation diagrams have errors approximately 2 to 3 times those indicated by the plateau calculations, but in all cases agree with the plateau ages within the error limits. Initial ⁴⁰Ar/³⁶Ar ratios also have large errors-of as much as 100 percent or more-due to the low ³⁶Ar content, but in both experiments, initial ⁴⁰Ar/³⁶Ar ratios were consistent with the expected ratio of 295.5, the ratio of ⁴⁰Ar to ³⁶Ar in air. The goodness-of-fit index for the regression of the lines in the isochron and correlation diagrams, given by SUMS/(N-2), ranges from 0.01 to 0.18. Values greater than about 2.5 indicate the presence of geologic error (Lanphere and Dalrymple, 1978); values less than 1.0 indicate that the scatter of points on the line is less than can be expected due to analytical error. In these experiments, values significantly less than 1.0 for the goodnessof-fit index suggest that the experimental errors were overestimated. The goodness-of-fit index nonetheless provides strong evidence that the tephra gives an undisturbed and geologically meaningful plateau age.

Table 2. Results of ⁴⁰Ar/³⁹Ar analyses of Colville River tephra beds

[Samples listed in probable stratigraphic order, with sample from lowermost tephra bed at end of table. Bold italic entries indicate data used in calculation of plateau and isochron ages. In third column, temperatures (°C) are reported for samples heated by induction coil, lettered steps for samples heated by continuous laser; TFU means total fusion. J, parameter used in age calculations; rad, radiogenic. Decay constants: $\lambda_{\epsilon} + \lambda'_{\epsilon} = 0.581 \times 10^{-10} \text{ yr}^{-1}$, $\lambda_{\beta} = 4.962 \times 10^{-10} \text{ yr}^{-1}$, $\frac{40}{K/K_{total}} = 1.167 \times 10^{-4} \text{ mol/mol}]$

Sample	Experiment	Temp (°C	C)			³⁹ Ar released	⁴⁰ Ar rad	Apparent
number	number	or step	⁴⁰ Ar/ ³⁹ Ar	³⁷ Ar/ ³⁹ Ar	³⁶ Ar/ ³⁹ Ar	(percent)	(percent)	age (Ma)
					0.040.54			
M86LPA9	L121-2	Α	22.28	0.08604	0.06051	1.28	19.7	22.1±25.8
		B	16.41	.003099	.008048	3.90	85.5	69.8±6.5
		С	14.73	.01691	.003862	16.24	92.2	67.6±1.7
		D	14.26	.01690	.001943	27.32	95.9	68.1±0.8
		E	14.18	.01330	.001050	20.67	97.7	68.9±1.0
		F	14.07	.01413	.0004771	10.11	98.9	69.2±1.0
		G	14.14	.01139	.0004163	5.80	99.0	69.7±1.0
		H	14.08	.01649	.0008217	14.68	98.2	68.8±1.3
J=0.00281					10 00			
Reactor cor	rections: (³⁶	Ar/ ³⁷ Ar)	$a = 0.0003; (^{39})$	Ar/ ³⁷ Ar) _{Ca} =0.00	003; (⁴⁰ Ar/ ³⁹ A	r)K=0.0086		
M86LPA9	87A076	350	222.3	0.05515	0.7520	0.17	0.03	0.6±26.7
	0/110/0	400	27.77	.0111	.07389	1.97	21.4	50.0±1.8
		400	11.27	.01715	.01176	13.36	69.1	65.5 ± 0.4
		600	8.803	.01713 .01659	.01170 .002101	62.55	92.9	68.7±0.4
		750	9.160		.003466	19.41	88.8	68.3±0.3
		750 865	9.100 17.38	.01430 .003415	.003466 .03180	19.41 2.46	00.0 45.9	67.0±1.3
		FUSE	338.2	.1253	1.153	.08	43.9 7	-20.9±78.8
J=0.004745	:	LOSE	220.4	.1433	1.133	.00	/	-20.71/0.0
			∼ _a =0.000234: (39 Ar/ 37 Ar)Ca=0	.000654: (⁴⁰ A)	r/ ³⁹ Ar)ĸ=0.005	7	
								- <u></u>
86ACr019	88A003	450	16.67	0.1756	0.003098	9.35	45.0	63.0±1.8
		550	9.126	.1562	.004647	42.38	84.7	65.0±0.5
		625	8.788	.1621	.004158	29.18	85.8	63.4±0.7
		700	10.01	.1809	.008652	11.89	74.3	62.6±1.4
		800	13.49	.2357	.01977	5.67	56.6	64.2±2.8
		FUSE	41.95	1.256	.1153	1.52	18.9	66.7±2.8
J=0.004745					(a` a	•		
Reactor cor	rections: (³⁶	Ar/ ³⁷ Ar)	<u>Ca</u> =0.000278; ($^{39}Ar/^{37}Ar)Ca=0$.0007; (⁴⁰ Ar/ ³	9 Ar) _K =0.0332		
M86LPA14	88A004	450	17.30	0.1889	0.03943	4.59	32.5	47.4±2.9
		525	13.01	.1510	.01474	23.36	66.3	72.1±0.8
		600	10.07	.1439	.004354	32.74	87.0	73.2±0.6
		700	9.355	.1463	.002891	26.64	90.6	70.9±0.7
		775	9.517	.1630	.004225	9.73	86.7	69.0±1.4
		FUSE	64.77	.3460	.1842	2.94	16.0	86.0 ± 5.1
J=0.004725	1	1006	v .		.1074	<i>4</i>	10.0	00.01.1
		Ar/ ³⁷ Ar)	∑a=0.000278; (39 Ar/ 37 Ar)Ca=0	.0007; (⁴⁰ Ar/ ³	⁹ Ar) _K =0.0332		
				_			12.0	20.010.0
84ACr180Y	(87 A 075	350	35.37	0.05557	0.1041	0.58	13.0	39.0±8.9
		450	16.69	.05418	.03560	6.26	36.9	52.0±0.9
		525	10.31	.04298	.007529	34.06	78.4	68.0±0.3
		600	8.773	.03772	.001751	24.51	94.1	69.3±0.3
		700	8.497	.04671	.008265	25.86	97.1	69.3±0.3
		850	13.02	.2258	.01610	7.38	63.6	69.5±0.8
J=0.004745		FUSE	14.75	1.956	.02166	1.34	57.5	71.3±3.9
		A-137 A-N	0 000024- /	$^{39}Ar/^{37}Ar)_{Ca}=0.00$	000654. 40	39 1-1-0 005-	,	
Reactor cor	rections: (50	AI/~'AI)	<u>_a=0.000234;</u> (·	Ar/3' Ar)Ca=0	.000654; (^{¬v} Ar	/~~Ar)K=0.005/	·	

Table 2. Results of ⁴⁰Ar/³⁹Ar analyses of Colville River tephra beds—Continued

[Samples listed in probable stratigraphic order, with sample from lowermost tephra bed at end of table. Bold italic entries indicate data used in calculation of plateau and isochron ages. In third column, temperatures (°C) are reported for samples heated by induction coil, lettered steps for samples heated by continuous laser; TFU means total fusion. J, parameter used in age calculations; rad, radiogenic. Decay constants: $\lambda_{g} + \lambda'_{g} = 0.581 \times 10^{-10} \text{ yr}^{-1}$, $\lambda_{\beta} = 4.962 \times 10^{-10} \text{ yr}^{-1}$, $\frac{40}{K}/K_{total} = 1.167 \times 10^{-4} \text{ mol/mol}]$

Sample number	Experiment number	Temp (°C or step) ⁴⁰ Ar/ ³⁹ Ar	³⁷ Ar/ ³⁹ Ar	³⁶ Ar/ ³⁹ Ar	³⁹ Ar released (percent)	Radiogenic ⁴⁰ Ar	Apparent age (Ma)
M86LPA19	L115-1	TFU	15.29	0.02571	0.005813	100	88.7	67.5±1.5
	L115-2	Α	18.15	0.07131	.02405	6.29	60.8	55.1±7.4
		B	15.36	.03294	.004176	20.85	91.9	70.2±2.4
		С	14.88	.02771	.002610	17.40	94.8	70.1±2.1
		D	14.85	.02393	.003381	20.15	93.2	68.8±2.0
		E	14.87	.03198	.003107	11.37	93. 8	69.3±2.9
		F	14.45	.03979	.001380	1.83	<i>97.1</i>	69.8±3.0
		G	14.77	.02632	.001768	22.12	96.4	70.8±1.4
	L115-3	А	17.34	0.007356	0.01290	2.80	78.0	67.3±4.2
		В	15.01	.02244	.003973	28.68	92.1	68.8±0.6
		С	14.77	.02248	.001475	47.44	97. 0	71.2±0.8
		D	14.65	.01742	.0007919	15.67	98.4	71.6±0.7
		E	14.92	.01306	.001839	2.76	96.3	71.4±3.3
		F	15.03	.01364	.002773	2.64	94.5	70.6±3.4

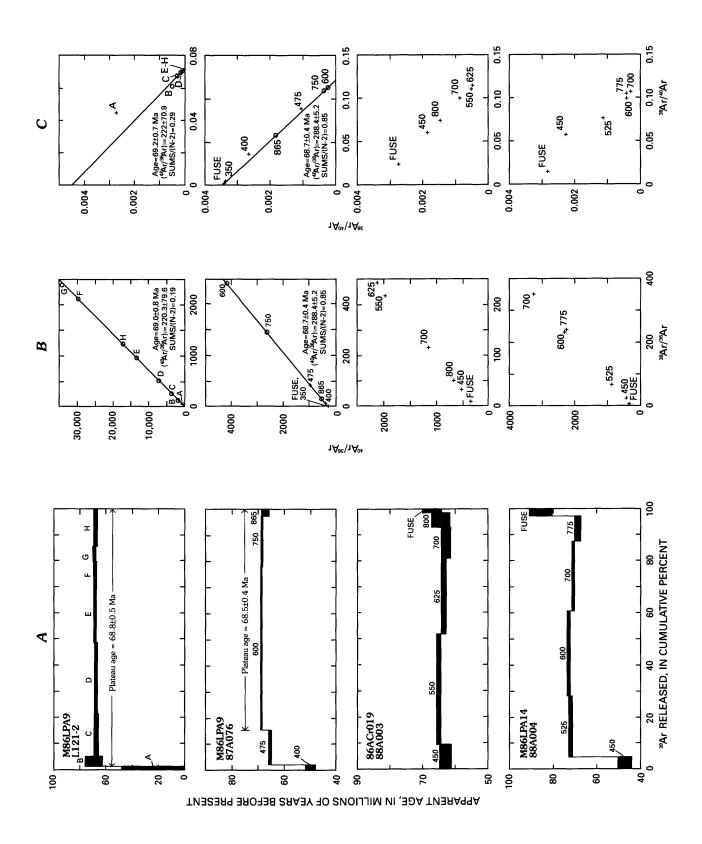
Sample 84ACr180Y

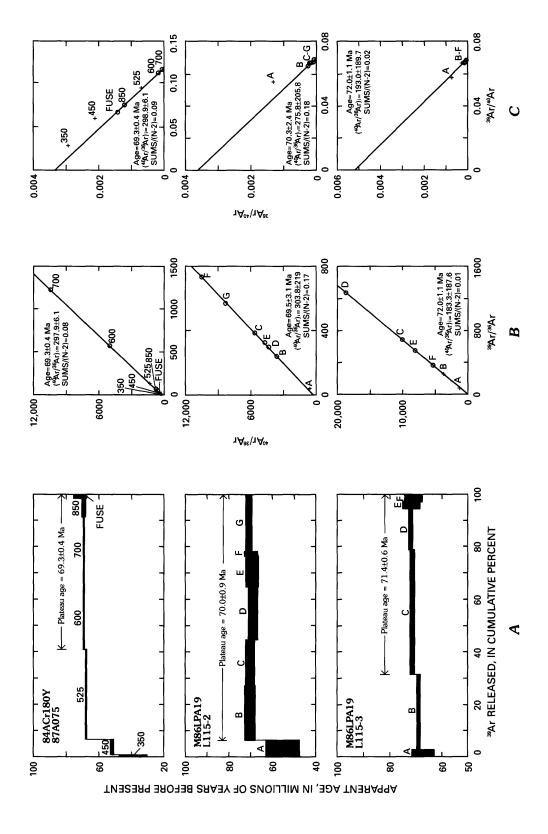
Sample 84ACr180Y was collected from a tephra bed inferred to be the second oldest in the area of study. A fault of unknown displacement separates it from the M86LPA19 tephra bed, the oldest tephra in the study area. Conventional induction-heated ⁴⁰Ar/⁸⁹Ar incremental-heating analysis on glass from this tephra sample gives a plateau age of 69.3±0.4 Ma (table 3). Minor radiogenic ⁴⁰Ar loss is apparent in the 350-, 450-, and 525-°C steps (fig. 4A, table 2), which total nearly 41 percent of the total ³⁹Ar release. Comparison with the recombined total-fusion age of 67.6±0.4 Ma (table 3) indicates that total radiogenic 40 Ar loss is about 2.5 percent. The 40 Ar/ 36 Ar versus 39 Ar/ 36 Ar isochron diagram (fig. 4*B*) and 36 Ar/ 40 Ar versus 39 Ar/ 40 Ar correlation diagram (fig. 4C) yield identical ages of 69.3 \pm 0.4 Ma and initial 40 Ar/ 36 Ar ratios of 297.9 \pm 6.1 and 298.0 ± 6.1 , respectively; these ratios are identical to the expected value of 295.5. The values of SUMS/(N-2)are about 0.08 and 0.09 for these diagrams, suggesting that the age is geologically accurate. Inclusion of the 525-°C step on the plateau does not significantly change the plateau age or the ages derived from the isotope and correlation diagrams, but it changes the SUMS/(N-2) value for the diagrams to about 5, suggesting geologic error, in this case most likely due to minor radiogenic ⁴⁰Ar loss.

Sample M86LPA14

Sample M86LPA14 was taken from a tephra bed about 18 m stratigraphically above the 84ACr180Y tephra

bed. ⁴⁰Ar/³⁹Ar age-spectrum analysis suggests some disturbance of argon in this sample. As in the two previous samples, the initial low-temperature (450 °C) step shows evidence of radiogenic ⁴⁰Ar loss; it is followed by an apparent plateau defined by the 525-, 600-, 700-, and 775-°C steps (fig 4A). This plateau, however, shows significantly more scatter than those of the previous samples, with apparent ages ranging from 69.0 to 73.2 Ma (table 2). The ${}^{40}\text{Ar}/{}^{36}\text{Ar}$ versus ${}^{39}\text{Ar}/{}^{36}\text{Ar}$ isochron diagram (fig. 4*B*) and the ³⁶Ar/⁴⁰Ar versus ³⁹Ar/⁴⁰Ar correlation diagram (fig. 4C) also suggest that there is some argon redistribution in this sample. No combination of steps from the apparent plateau yields ages in the isochron and correlation diagrams that are concordant with the corresponding plateau age and have a SUMS/(N-2) value of less than about 4; most combinations give higher values. Initial ⁴⁰Ar/³⁶Ar values are highly variable and range from about 120 to more than 500 (expected value is 295.5), depending on the heating increments used in the regression. One explanation for the anomalous data for this sample is that it contains extremely fine-grained alteration products, most likely clays, that have lost small amounts of ³⁹Ar by recoil during or shortly after irradiation, thus giving slightly old apparent ages. Samples of glass shards from this tephra unit viewed under the scanning electron microscope (fig. 3) show significantly more surface alteration than shards that give apparently undisturbed spectra. This alteration may have penetrated the glass from this tephra bed deeply enough that the hydrofluoric acid treatment did not remove





show range of heating steps used to calculate plateau ages. *B*, Isochron diagrams. Straight lines are isochrons. Open circles indicate data used to fit straight lines; crosses indicate data not used in fit. (⁴⁰Ar/³⁶Ar)i is initial ⁴⁰Ar/³⁶Ar value; SUMS/(N-2) is goodness-of-fit index for regression of lines. C, Correlation diagrams; symbols as in figure 4*B*. For samples that did not yield reliable ages, no plateau age is reported in figure 4A and no lines are drawn in figures 4*B* and 4*C*. For sample M86LPA9, experiment 87A076, heating steps "350" and "FUSE" are too narrow to show in figure 4A.

Figure 4. ⁴⁰Ar/³⁹Ar analyses (see table 2). In upper left corner of first graph in each row are sample number (first) and experiment number (second) that apply to graphs in that row. Samples are in probable stratigraphic order, with sample from lowermost tephra bed at bottom of figure. A, Argon-release spectra from incremental heating. Solid bars indicate apparent ages for intervals shown; uncertainties in age (1-sigma) indicated by vertical width of bars. Numbers above bars indicate temperatures (°C) of heating steps; letters indicate heating steps whose temperatures were not monitored (see text). Horizontal arrows

 Table 3. Summary of K-Ar and ⁴⁰Ar/³⁹Ar ages of Colville River tephra beds

[Samples listed in probable stratigraphic order, with sample from lowermost tephra bed at bottom of table. ---, not determined]

Sample	Experiment	K-Ar	⁴⁰ Ar/ ³⁹ Ar	⁴⁰ Ar/ ³⁹ Ar	⁴⁰ Ar/ ³⁹ Ar total-
number	number	age (Ma)	plateau age (Ma)	isochron age (Ma)	fusion age (Ma)
M86LPA9	L121-2		68.8±0.5	69.0±0.8	68.0±0.7*
	87A076		68.5±0.4	68.7±0.4	67.6±0.4*
	871589	69.5±2.1			
86ACr019	88A003		No plateau		64.1±0.5*
	871535	65.8±2.0			
M86LPA14	88A004		No plateau		71.1±0.5*
	871590	68.5±2.0			
84ACr180Y	87A075		69.3±0.4	69.3±0.4	67.6±0.4*
M86LPA19	L115-1				67.5±1.5
	L115-2		70.0±0.9	69.5±3.1	69.0±1.0*
	L115-3		71.4±0.6	72.0±1.1	70.4±0.6*
	871452	70.4±2.1			

*Recombined age.

all the alteration products. The conventional K-Ar analysis of this sample gave an age of 68.5 ± 2.0 Ma, analytically indistinguishable from the age of the underlying tephra bed (see table 2). Thus, alteration of this tephra bed, which appears to have caused some argon redistribution, apparently did not result in appreciable loss of radiogenic 40 Ar.

Sample 86ACr019

Sample 86ACr019 was taken from a tephra bed several meters stratigraphically above two major dinosaur-bonebearing beds and about 3 m below a third bone bed. This tephra bed is inferred to be the second youngest in this study, although a covered interval separates this part of the section from the three tephra beds described in the previous paragraphs, and a fault of unknown displacement separates it from the younger beds to the east (figs. 1, 2). ⁴⁰Ar/³⁹Ar age-spectrum analysis of this sample reflects some degree of argon loss and (or) redistribution. Although a plateau appears to be present at about 64 Ma (table 2), the overall shape of the plateau is similar to that of sample M86LPA14, which is interpreted to indicate a disturbed age (fig. 4A). The initial 450-°C step gives a slightly young age, and it is followed by a series of steadily decreasing ages for the middle-temperature steps. The final two steps give slightly higher ages, similar to the final step for M86LPA14. Moreover, the apparent plateau age of about 64 Ma is slightly young on the basis of the ages of the other samples. The ⁴⁰Ar/³⁶Ar versus ³⁹Ar/³⁶Ar isochron diagram (fig. 4B) and ³⁶Ar/⁴⁰Ar versus ³⁹Ar/⁴⁰Ar correlation diagram (fig. 4C) bear out the conclusion that this sample is slightly disturbed. Ages calculated from the isochron and correlation diagrams are concordant with the age calculated from the apparent plateau, and initial ⁴⁰Ar/³⁶Ar ratios are close to 295.5, but the goodness-of-fit index, SUMS/(N-2), is about 3.5, indicative of geologic error. The pattern of steadily decreasing ages of the plateau steps is a characteristic of recoil of ³⁹Ar after irradiation. The young apparent ages, however, are not typical of recoil but instead suggest loss of radiogenic ⁴⁰Ar. Possibly, devitrification and recrystallization of very fine grained alteration products that were susceptible to ³⁹Ar recoil in the reactor also led to significant loss of radiogenic ⁴⁰Ar over geologic time. This age spectrum is in contrast to that of M86LPA14, which apparently was affected by recoil effects but does not show evidence of argon loss.

Sample M86LPA9

Sample M86LPA9 is from the youngest tephra bed exposed near Ocean Point. ⁴⁰Ar/³⁹Ar age-spectrum analysis was performed by both induction-heating techniques (experiment 87A076) and laser-heating techniques (experiment L121-2), giving plateau ages of 68.5±0.4 and 68.8±0.5 Ma, respectively (table 3). Both spectra show very well defined plateaus and similar release patterns (fig. 4A). Only minor loss of radiogenic argon is apparent in the low-temperature release steps. Comparison of the plateau ages with recombined total-fusion ages (table 3) indicates total radiogenic argon loss is less than 1.5 percent for both types of analysis. The 40 Ar/ 36 Ar versus 39 Ar/ 36 Ar isochron diagrams and 36 Ar/ 40 Ar versus ³⁹Ar/⁴⁰Ar correlation diagrams for both these analyses also indicate that the plateau ages represent undisturbed ages (figs. 4B, 4C). Ages derived from the isochron and correlation diagrams are concordant with the plateau ages, initial ⁴⁰Ar/³⁶Ar ratios are not significantly different from 295.5 at a 95-percent confidence level, and the goodness-of-fit indexes (SUMS/(N-2)) are less than 1.

DISCUSSION

The results indicate that rhyolitic glass at least as old as Late Cretaceous can give geologically reasonable K-Ar and ⁴⁰Ar/³⁹Ar radiometric ages. Analysis of age spectra and isochron and correlation diagrams using the criteria of Lanphere and Dalrymple (1978) readily distinguishes between samples that have undergone some degree of argon redistribution and those that are essentially undisturbed and therefore give reliable cooling ages. The 40Ar/39Ar age spectra for three of the glass samples (M86LPA19, 84ACr180Y, M86LPA9) have concordant, well-defined plateaus and show only minor evidence of argon loss caused by alteration or diffusion. Samples M86LPA14 and 86ACr019 gave disturbed age spectra and appear to have some degree of argon redistribution, but only one of these (86ACr019) shows evidence of significant argon loss. ⁴⁰Ar/³⁹Ar ages derived from isochron diagrams-termed isochron ages-are in general preferred ages because the method by which they are determined minimizes the effects of alteration, and thus argon loss, and takes best account of analytical errors. The plateau and isochron ages also have greater precision than conventional K-Ar ages, in part because they are an average of several individual age determinations.

Although all of the samples show evidence of minor argon loss, three of the five glass samples we dated yielded ages that represent true cooling ages on the basis of normal criteria for analysis of ⁴⁰Ar/³⁹Ar age spectra. The minor (<2.5 percent) ⁴⁰Ar loss observed in the low-temperature heating steps can be attributed to loss of ⁴⁰Ar due to surface alteration and (or) diffusion near shard boundaries. Nonetheless, the ⁴⁰Ar/³⁹Ar ages are indistinguishable from the conventional K-Ar ages within the limits of analytical uncertainty. Of the two samples giving apparently disturbed ⁴⁰Ar/³⁹Ar age spectra, one (M86LPA14) gave a K-Ar age (68.5±2.0 Ma) that is reasonable on the basis of the other radiometric dates, suggesting that some argon redistribution occurred without significant argon loss. The slightly old recombined totalfusion age of 71.1±0.5 Ma obtained on this sample by 40 Ar/ 39 Ar analysis may be an artifact of the irradiation process (recoil of ³⁹Ar). Sample 86ACr019 appears to have suffered significant ⁴⁰Ar loss due to alteration or diffusion on the basis of the incremental heating data. The K-Ar age of 65.8±2.0 Ma for this sample is therefore considered a minimum age.

The "best" or preferred age for each of the tephra beds is shown in figure 5. For samples M86LPA9 and M86LPA19, preferred ages are 68.8 ± 0.4 and 70.4 ± 0.8 Ma, respectively, and are calculated from weighted means of 40 Ar/ 39 Ar isochron ages, 40 Ar/ 39 Ar total-fusion ages, and K-Ar ages for each tephra bed. The preferred age for sample 84ACr180Y is the 40 Ar/ 39 Ar isochron age of 69.3±0.4 Ma.

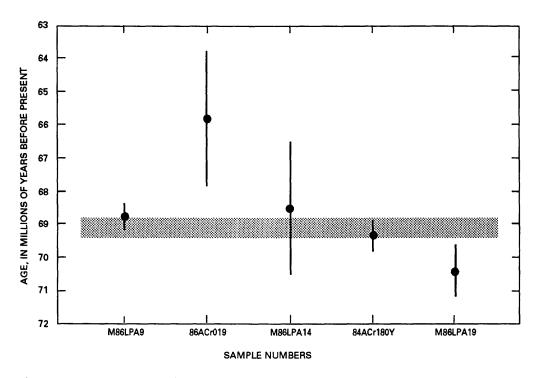


Figure 5. Best age estimates for Colville River tephra beds (dots) and associated 1-sigma errors (bars). Shaded area shows best estimate of age of bone-bearing beds on basis of all analyses. See text for discussion.

The best age estimate for sample M86LPA14 is the K-Ar age of 68.5±2.0 Ma, because of possible irradiationinduced disturbance in the ⁴⁰Ar/³⁹Ar analysis. The K-Ar age of 65.8±2.0 Ma for sample 86ACr019 is considered a minimum age because of significant argon redistribution and loss apparent in the ⁴⁰Ar/³⁹Ar analysis. Although there appears to be a slight decrease in age upward through the section, none of the tephra beds are demonstrably different in age at a 95-percent confidence level. Since the ages are concordant, a best age estimate of 69.1±0.3 Ma for the dinosaur-bonebearing beds was calculated using the weighted mean of all the analyses. Assuming that offsets across the faults and covered intervals are small, the location of the Cretaceous-Tertiary boundary, considered to be about 66 Ma (Palmer, 1983), appears to be at least 100 m above the youngest dated tephra bed (M86LPA9), provided depositional and environmental conditions remained constant. This new dating, by establishing a Late Cretaceous age for the dinosaur-bone-bearing beds, is compatible with the impact theory for the extinction of the dinosaurs. Unfortunately, no tephra beds have been identified near the probable location of the Cretaceous-Tertiary boundary to allow for K-Ar or ⁴⁰Ar/³⁹Ar dating. About 40–60 m above the youngest tephra bed, the section passes into a marine depositional environment (Phillips, 1988). Thus, any tephra beds deposited at this time would likely be reworked and altered by diagenetic interaction with sea water.

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