Terra Antartica
1998, 5(3), 683-690

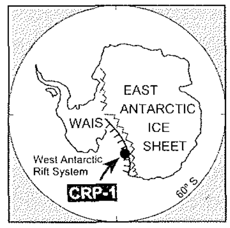
$^{40}Ar/^{39}Ar$ Geochronology of Volcanic Clasts and Pumice in CRP-1 Core, Cape Roberts, Antarctica

W.C. McIntosh

New Mexico Institute of Mining and Technology, Socorro, NM, 87801 - USA

Received 17 August 1998; *accepted in revised form* 15 October 1998

Abstract - $^{40}Ar/^{39}Ar$ dating of volcanic clasts and pumice provide constraints for depositional ages of the CRP-1 core. Laser-fusion analyses of anorthoclase phenocrysts from pumice clasts concentrated near 116 mbsf suggest a depositional age of 18.40 ± 1.12 Ma (± 2 σ). Results from anorthoclase phenocrysts and groundmass concentrates from five clasts of reworked lava provide maximum depositional ages of 19.73 ± 0.86 Ma to 17.15 ± 0.80 Ma for the 61 to 114 mbsf interval of CRP-1. The most precise age determination is 19.32 ± 0.14 Ma for a trachytic clast near 105 mbsf. Taken together, the $^{40}Ar/^{39}Ar$ results from the lower part of CRP-1 suggest that none of the sediment above 116 mbsf is older than 19.5 Ma, and the data are most compatible with rapid accumulation of the 61 to 116 mbsf segment of CRP-1 near 18 Ma. $^{40}Ar/^{39}Ar$ results from a basaltic clast from the carbonate-rich layer at 33 mbsf provide a maximum depositional age of 1.2 ± 0.1 Ma for this horizon.



INTRODUCTION

The CRP-1 core was drilled near Cape Roberts, Antarctica, as part of an international effort to sample marine sediments in McMurdo Sound, with the goal of reconstructing Cenozoic and possibly Cretaceous paleoenvironment and paleoclimate of Antarctica. Because of sea-ice break out, CRP-1 penetrated only to 147.9 mbsf, less than 20% of the planned depth.

Accurate interpretations of the paleoenvironmental record sampled by the CRP-1 drillcore require an accurate chronology for the core. Initial studies of diatom stratigraphy (Cape Roberts Science Team, 1998; Harwood et al., this volume) identified the upper interval of CRP-1 (19-43 mbsf) as Quaternary and the majority of the underlying interval (43-147.9 m) as lower Miocene. A number of other dating methods, including paleomagnetic analysis (Roberts et al., this volume) and Sr dating of shell material (Lavelle, this volume) have subsequently been applied to the question of CRP-1 chronology. This paper reports the results of $^{40}Ar/^{39}Ar$ dating of volcanic pumice and lava clasts recovered from the core at 33 mbsf and 62 to 117 mbsf. $^{40}Ar/^{39}Ar$ analysis is a well-established method of determining the eruption age of volcanic minerals and groundmass (McDougall &Harrison, 1988).

VOLCANIC CLASTS IN CRP-1

Most of the volcanic clasts observed in CRP-1 are fragments of basaltic to trachytic lava and scoria, commonly rounded, generally 1 to 10 mm in diameter, but in a few cases as large as 2-3 cm in diameter (Smellie, this volume). Because these lava clasts were transported and deposited by sedimentary (dominantly glacial) processes, they only provide maximum ages for the time of deposition. In addition to the clasts of reworked lava and scoria, a concentration of 0.5 to 3 mm clasts of rounded trachytic pumice are present in the 114 to 117 mbsf interval of CRP-1. Although these pumice have been reworked, their fragile nature and concentration in a restricted stratigraphic interval suggests that they were deposited soon after their eruption as pyroclastic ejecta, therefore their eruption age is inferred to closely approximate the time of their deposition (Armienti et al., this volume; Smellie, this volume). Their rounding may be due to wind transport and abrasion after their deposition on sea ice.

Volcanic clasts were separated from CRP-1 samples collected by John Smellie from the working half of the core after its arrival in Bremerhaven, Germany. Sandy to pebbly volcanic-clast-bearing sampling intervals were selected on the basis of observations from thin sections of samples collected from CRP-1 at McMurdo Station. A few larger volcanic clasts, visible on the outer surface or cut face of the half core, were hand picked directly from the core. Most clasts were hand picked from 100 to 200 g samples of sand-rich sediment, following ultrasonic disaggregation and washing. Small subsamples were chiseled from each clast and examined by electron microprobe to confirm their volcanic nature prior to dating. Compositionally, volcanic clasts ranged from basanite to peralkaline trachyte. Two clasts and the pumice were found to contain anorthoclase, a K-rich volcanic feldspar ideally suited for $^{40}Ar/^{39}Ar$ analysis. HF etching and hand picking were used to separate the anorthoclase from these samples. Clasts of lava lacking anorthoclase were prepared by washing in dilute HCl and, for a few phenocryst-rich clasts, by crushing and hand picking to remove K-poor phenocryst phases.

^{40}AR/^{39}AR ANALYTICAL METHODS AND RESULTS

Altogether, three anorthoclase separates, and 24 clasts were analyzed by ^{40}Ar/^{39}Ar methods in this study (Tab. 1, Appendices 1 & 2). The anorthoclase separates were analyzed by laser fusion of single crystals (two samples) or groups of four crystals (one sample). Whole-rock samples or groundmass concentrates of lava clasts were dated by the furnace incremental-heating age-spectrum method. Analytical methods are briefly summarized in table 1; further details of procedures used in the New Mexico Geochronology Research Laboratory are presented in McIntosh & Chamberlin (1994). Analyzed aliquots of lava clasts ranged in size from 1.9 to 102 mg. Only one clast, a basanitic fragment from 33 mbsf, was large enough to allow duplicate analyses of two different aliquots.

All three of the anorthoclase separates and six of the 24 analyzed clasts yielded relatively precise ^{40}Ar/^{39}Ar age data, which have some bearing on the depositional age of CRP-1 sediments. Data from the remaining 18 clasts are not detailed in this report. Eight of these 18 clasts were found to be much older than the depositional age of the surrounding sediment. These include two clasts with Paleozoic ages similar to McMurdo Sound basement rocks, four clasts with Jurassic ages characteristic of Kirkpatrick basalts, and two clasts from the uppermost Quaternary part of CRP-1, which gave early Quaternary to Pliocene ages. The remaining ten clasts yielded disturbed age spectra or imprecise age data, apparently due to various combinations of small sample size, low K_2O content, high glass content, and alteration. The rest of this paper focuses on the relatively precise and germane data obtained from three anorthoclase separates and the six clasts of lava that are inferred to yield reliable results.

Results from laser fusion analyses of the three anorthoclase separates are presented in figure 1 and summarized in table 1. Analytical data are provided in appendix 1. The precision and accuracy of the mean ages varies among the three samples. The nine anorthoclase phenocrysts from CRP-104-1, an approximately 1 cm fragment of peralkaline trachyte, yielded a weighted mean age 19.32 ± 0.14 Ma (all uncertainties reported as ± 2 σ, unless otherwise noted), which is the most precise and accurate age determination reported in this study (Fig. 1, Tab. 1). The high quality of this age determination results from the relatively large size of the crystals and consequent large amount of Ar isotopes available for analyses (Fig. 1). An identical but less precise and potentially less accurate weighted mean age of 19.32 ± 0.32 Ma was determined from CRP-90-3, a lithologically similar but much smaller clast of lava. Eight smaller fragments of anorthoclase phenocrysts from this sample were fused and analyzed as two groups of four crystals. The lower precision of this weighted mean age is in part due to the low number of data points (n = 2). The ages of both of these samples (as well as ages of all other lava clasts analyzed in this study) represent maximum ages for the time of deposition. A much less precise age of 18.40 ± 1.12 Ma was determined by single-crystal laser-fusion of three tiny anorthoclase phenocrysts separated from CRP-116/117, consisting of rounded, crystal-poor pumice fragments sparsely

Tab. 1 - Summary of ^{40}Ar/^{39}Ar results from CRP-1.

Sample	Depth (mbsf)	Rock type	Material	Age analysis	n	yield (%)	Age (Ma)	±2σ
CRP-33-1	33.23	basaltic clast	groundmass, 53 mg	plateau	4	24.4	1.16	0.15
CRP-33-1	33.23	basaltic clast	groundmass, 102 mg	plateau	5	19.8	1.18	0.12
CRP-61-7	61.82-63.00	trachyte clast	groundmass, 3.8 mg	plateau	7	48.1	18.12	0.84
CRP-61-8	61.82-63.00	trachyte clast	groundmass, 1.9 mg	plateau	6	86.8	17.30	0.95
CRP-90-6	90.50-90.75	trachyte clast	groundmass, 6.5 mg	plateau	8	91.5	17.92	0.41
CRP-90-11	90.50-90.75	basaltic clast	groundmass, 7.2 mg	plateau	7	84.4	17.15	0.80
CRP-90-3	90.50-90.75	trachyte clast	anorthoclase	laser fusion	2	99.3	19.32	0.32
CRP-104-1	104.88-104.89	trachyte clast	anorthoclase	laser fusion	9	96.9	19.32	0.14
CRP-114-22	114.94-115.70	trachyte clast	groundmass, 18 mg	near- plateau	12	13.3	19.73	0.86
CRP-116/117	116.52-117.45	pumice	anorthoclase	laser fusion	3	82.8	18.40	1.12

Notes: n denotes number of incremental heating steps on plateau, or number of single-crystal laser fusions used in mean age. Yield denotes unweighted arithmetic mean of percent radiogenic yield of plateau or fusion steps. All errors quoted at ± 2 sigma.

Sample preparation and irradiation: anorthoclase separates prepared by crushing, hand picking, and etching in 15% HF. Groundmass concentrates prepared by crushing and removal of phenocrysts. Samples were packaged in machined Al discs and irradiated for 7 hrs in the D-3 position, Nuclear Science Center, College Station, TX, along with neutron flux monitor Fish Canyon Tuff sanidine, (FC-1) with an assigned age of 27.84 Ma (Deino & Potts, 1990), relative to Mmhb-1 at 520.4 Ma (Samson & Alexander, 1987).

Instrumentation: Mass Analyzer Products 215-50 mass spectrometer on line with automated all-metal extraction system. Groundmass samples step-heated in Mo double-vacuum resistance furnace. Heating duration 7 to 8 minutes. Anorthoclase samples heated by single-crystal laser-fusion using 10 watt CO2 laser. Heating duration 45 seconds. Reactive gases removed by reaction with SAES GP-50 getters operated at ~450°C and 20°C

Analytical parameters: net electron multiplier sensitivity averaged 1×10^{-16} moles/pA for the furnace and 7×10^{-17} moles/pA for the laser extraction system. Total system blanks plus backgrounds for furnace-heated groundmass samples averaged: 1890, 10 , 2 , 5, and 8 x 10^{-18} moles at masses 40, 39, 38, 37, and 36, respectively for temperatures <1 300°C. Total system blanks plus backgrounds for laser-heated anorthoclase samples averaged: 175, 4, 0.1 , 3, 6 x 10^{-18} moles at masses 40, 39, 38, 37, and 36, respectively. J-factors were determined to a precision of ± 0.1% by CO_2 laser-fusion of 4 single crystals from each of 4 or 6 radial positions around the irradiation tray.

Correction factors for interfering nuclear reactions were determined using K-glass and CaF_2 and are as follows: $(^{40}Ar/^{39}Ar)_K = 0.0002 \pm 0.0003$; $(^{36}Ar/^{37}Ar)_{Ca} = 0.000279 \pm 0.00001$; and $(^{39}Ar/^{37}Ar)_{Ca} = 0.00077 \pm 0.00001$.

Age calculations: plateau definition: 3 or more analytically indistinguishable contiguous steps comprising at least 50% of the total ^{39}Ar (Fleck et al., 1977). "Near-plateau" age calculated for sample CRP-114-22, which approached but did not meet plateau criteria. Plateau and near-plateau ages calculated by weighting each step by the inverse of the variance. Plateau and near plateau age errors calculated using the method of Samson & Alexander (1987). Decay constants and isotopic abundances after Steiger & Jäger (1977). All errors reported at ±2σ.

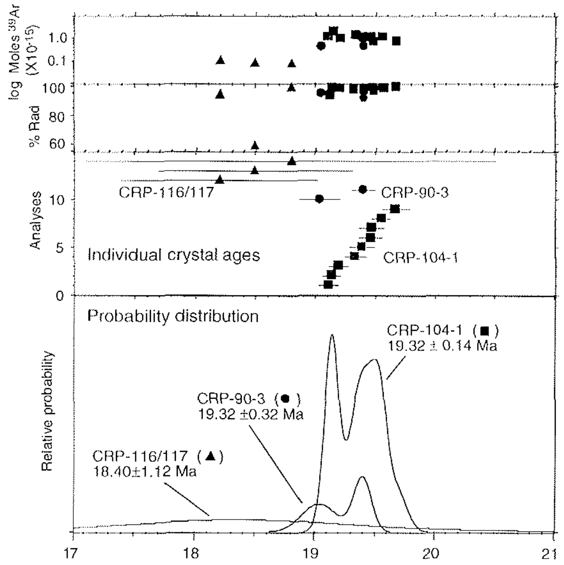


Fig. 1 - Age probability distribution diagram of laser-fusion dating results from anorthoclase phenocrysts. Additional panels show size of ^{39}Ar signal, radiogenic yield, and age with ± 1 σ errors for individual analyses. The apparent bimodality of CRP-104-1 results is not considered significant. The poor precision of results from CRP-116/117 pumice clasts results from the low number and small size of analyzed phenocrysts. See Deino & Potts (1992) for an explanation of the age probability distribution diagram.

distributed throughout the 116 to 117 mbsf interval. The low precision of this analysis reflects the low number of analyzed phenocrysts (n=3) and the small quantities of argon isotopes released from these tiny crystals, about an order of magnitude smaller than argon isotopes measured from CRP-104-1 and CRP-90-3. Although this age determination lacks precision, it is likely to be representative of the actual depositional age of the enclosing sediment, as discussed above.

Results from step-heated lava clasts are presented in figure 2 and summarized in table 1. Analytical data are given in appendix 2. Four clasts from 61 to 114 mbsf yielded early Miocene plateau ages ranging from 17.15 ± 0.82 Ma to 18.12 ± 0.84 Ma, and a fifth clast sample from 114 mbsf yielded a "near-plateau" age of 19.73±0.86 Ma (Fig. 2, Tab. 1, plateau criteria defined in Tab. 1 footnotes). As with the laser fusion results discussed above, precision and accuracy of these five age determinations varies among the five samples. The most precise age of 17.92 ± 0.41 Ma is from CRP-90-6, a holocrystalline trachytic clast which yielded a nearly flat age spectrum, high radiogenic yields, and highly precise individual steps (Fig. 2e; radiogenic yield is the fraction of ^{40}Ar not ascribable to atmospheric argon). Slightly lower quality plateau ages were obtained from CRP-90-11 (17.15 ± 0.89 Ma) and CRP-61-8 (17.30 ± 0.95 Ma), which have high radiogenic yields but slightly climbing age spectra. The remaining two early Miocene age determinations are considered to be of even lower quality. The age spectrum of CRP-61-7 meets plateau criteria (18.12 ±0.84 Ma) but individual steps have relatively low precision and highly variable radiogenic yields. Sample CRP-114-22 has a somewhat disturbed spectrum with low radiogenic yields which approach but does not meet plateau criteria (Fig. 2g). The "near-plateau age", calculated from all of the heating steps, is 19.73±0.86 Ma. Step-heating data from lower Miocene CRP-1 clasts were also examined using isochron analyses, an approach that avoids assumptions regarding the isotopic ratio of argon trapped during initial crystallization of the lava. In most cases, the isochron ages agreed with the plateau ages, and gave $^{40}Ar/^{36}Ar$ intercepts within ± 2 sigma of atmospheric argon ($^{40}Ar/^{36}Ar = 295.5$). Data from sample CRP-61-8 did not yield a well-defined isochron, in part due to the restricted range of radiogenic yields.

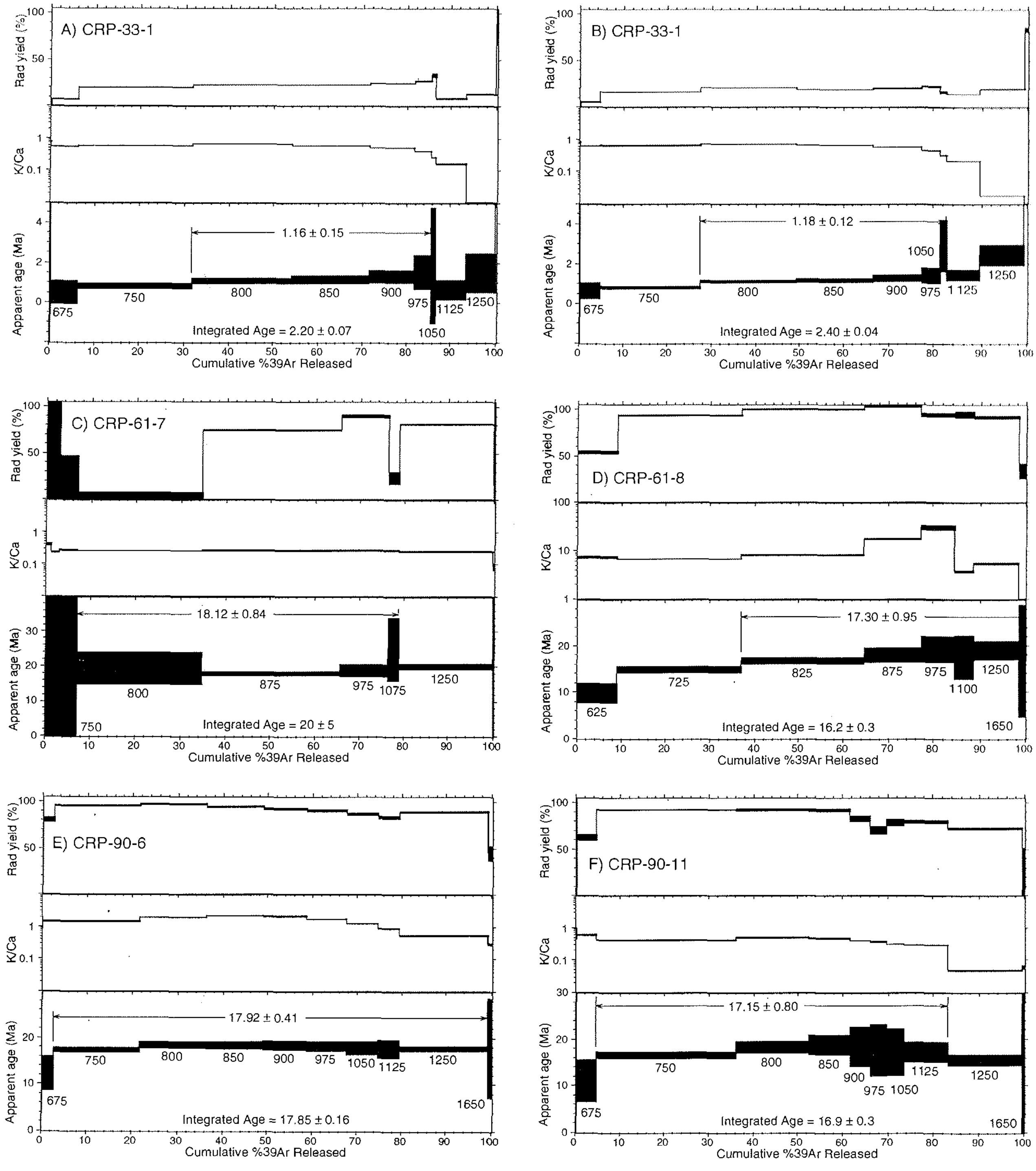
In addition to the dating study of the lower Miocene part of CRP-1, relatively precise plateau ages were obtained from two separately analyzed aliquots of CRP-33-1, a 1 cm basanitic clast from the calcareous-fossil-rich horizon near 33 mbsf (Taviani & Claps, this volume). These two aliquots yielded nearly identical but slightly climbing age spectra with plateau ages of 1.16 ± 0.15 Ma and 1.18 ±0.12 Ma (Fig. 2a & b). Although the radiogenic yields from analyses of this young clast are uniformly low, the analytical precision of individual steps is very high, mainly due to the large aliquot size (53 and 102 mg). This precision and high degree of reproducibility of the age spectra suggest that the plateau ages are accurate measurements of the eruption age of the clast. Neither of these analyses yield well defined isochrons, chiefly due to the restricted range of radiogenic yields.

DISCUSSION

AGE OF LOWER CRP-1

The early Miocene $^{40}Ar/^{39}Ar$ age determinations from pumice and lava clasts from 61 to 117 mbsf support the early Miocene age indicated by initial studies of diatom stratigraphy (Cape Roberts Science Team, 1998; Harwood et al., this volume). A probability distribution diagram of results from the clasts (Fig. 3) shows two relatively well defined peaks near 17.9 and 19.3 Ma. The only $^{40}Ar/^{39}Ar$ age which is actually interpreted as syndepositional is the relatively imprecise age of 18.4 ±1.1 Ma measured from anorthoclase from rounded pumice clasts concentrated near 114 to 117 mbsf. All other $^{40}Ar/^{39}Ar$ ages were measured from reworked clasts and therefore represent maximum ages for the enclosing sediments. Although the $^{40}Ar/^{39}Ar$ dates of the reworked clasts do not provide direct evidence for the depositional age of this part of CRP-1, the fact that the 24 analyzed clasts have a narrow range of ages constitutes indirect evidence that erosion of the clasts and deposition at the CRP-1 site occurred soon after their eruption. Taken together, the $^{40}Ar/^{39}Ar$ results from the lower part of CRP-1 suggest that none of the sediment above 116 mbsf is older than 19.5 Ma, and the data are most compatible with rapid accumulation of the 61 to 116 mbsf segment of CRP-1 near 18 Ma.

Although the $^{40}Ar/^{39}Ar$ data are in broad agreement with other age criteria which indicate an early Miocene age for the interval of CRP-1 below 43 mbsf, there is some



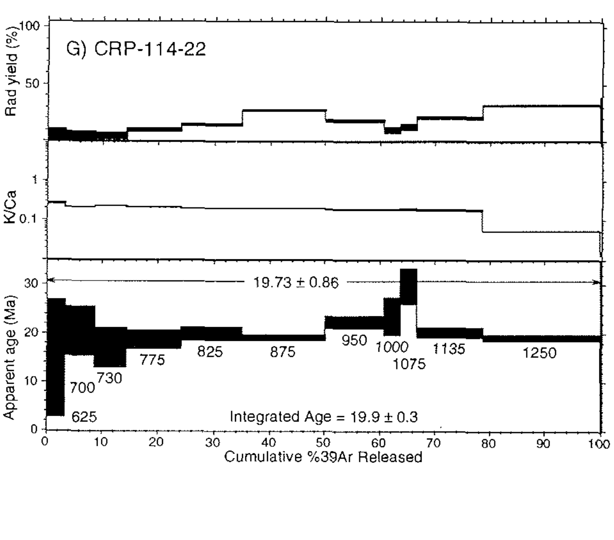


Fig. 2 - $^{40}Ar/^{39}Ar$ age spectra of whole-rock samples and groundmass concentrates of lava clasts from CRP-1. Upper two panels of each diagram show yield of radiogenic argon and K/Ca ratio, calculated from measured $^{39}Ar/^{37}Ar$ ratios. Plateau ages are determined using criteria defined in table 1 footnotes. Integrated age is the mean age of all steps, weighted by percent ^{39}Ar in each step.

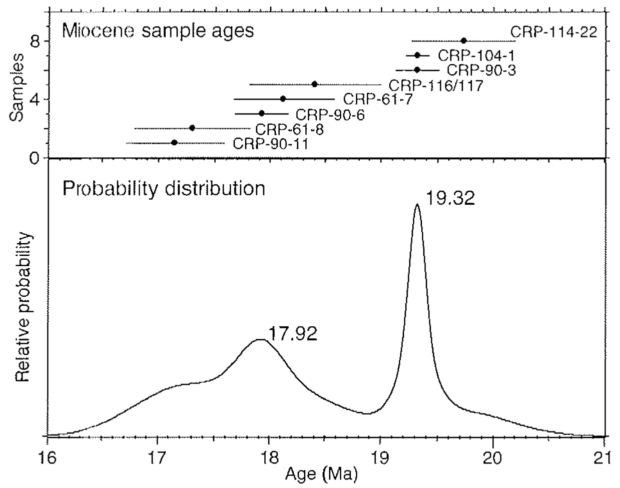


Fig. 3 - Probability age distribution diagram showing early Miocene age dates from CRP-1 lava and pumice clasts. Upper panel shows individual sample ages with ± 1 σ errors.

conflict with the initial interpretations of paleomagnetic data (Cape Roberts Science Team, 1998; Roberts et al., this volume) which suggest depositional ages of 21.0 to 19.5 Ma for the 117 to 90 mbsf interval of CRP-1 (Fig. 4). This interpretation of the paleomagnetic record conflicts with the 18.40 ± 1.12 Ma depositional age of the CRP-116/117 pumice, and with the relatively precise maximum depositional ages from clasts CRP-104-1 (19.32 ± 0.14 Ma) and CRP-90-6 (17.92 ± 0.41 Ma). The $^{40}Ar/^{39}Ar$ age of CRP-104-1 is considered to be particularly robust, because of the good analytical precision and because it was obtained from laser-fusion of individual anorthoclase phenocrysts.

There is also some lack of agreement between a Sr date of 18.5 ± 0.2 Ma from shell material at 61 mbsf and the maximum depositional age from CRP-61-8 of 17.30 ± 0.95 Ma. However, because these two age determinations nearly overlap at ±2 σ, and because (as presented above) the age spectrum for this analysis is not entirely flat, this lack of agreement is not considered significant.

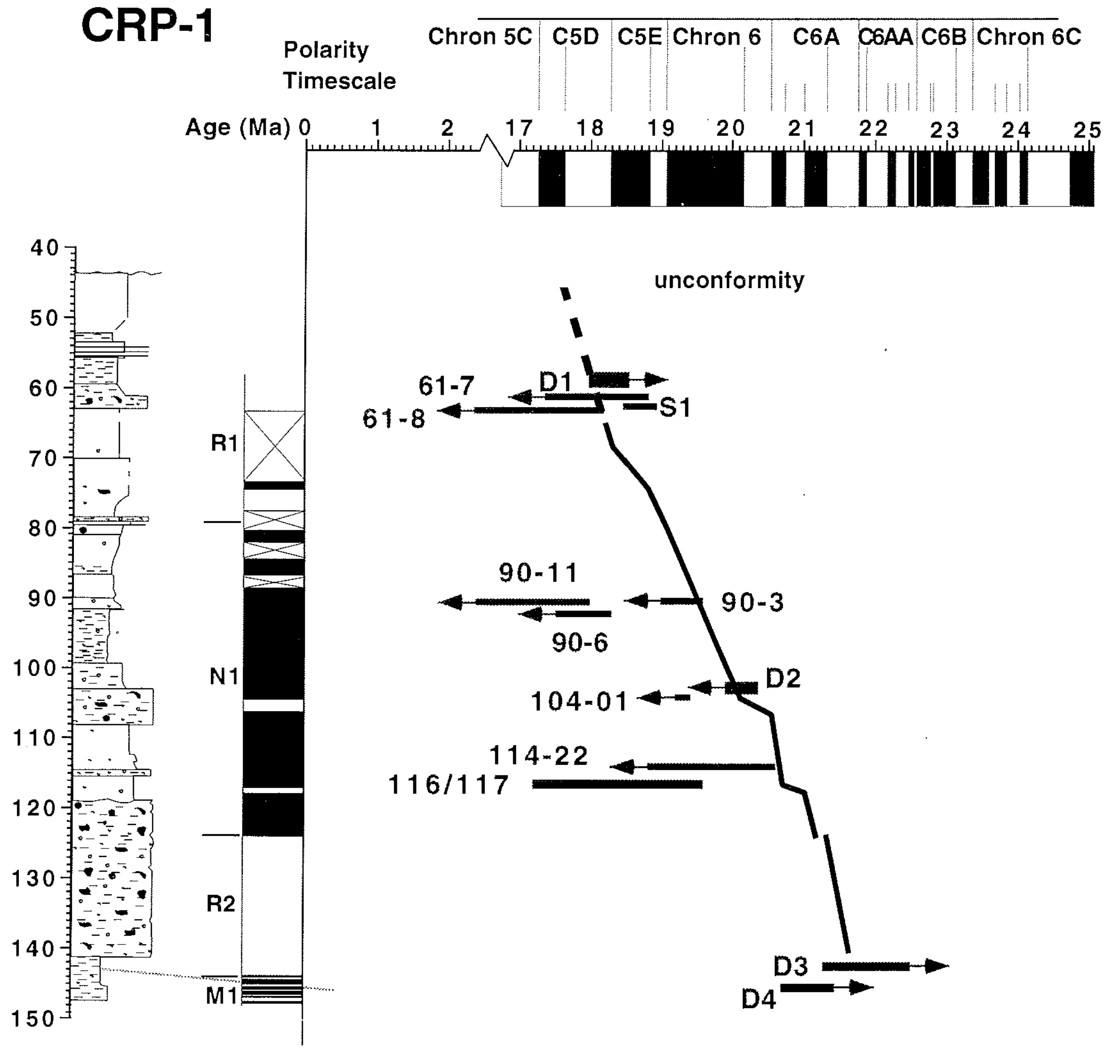


Fig. 4 - A comparison of $^{40}Ar/^{39}Ar$ results with the preferred interpretations of CRP-1 paleomagnetic record (Cape Roberts Science Team,1998; Roberts et al., this volume), diatom stratigraphy (Cape Roberts Science Team,1998; Harwood et al., this volume) and an Sr date from shell material (Lavelle, this volume). Bars labeled with sample numbers show $^{40}Ar/^{39}Ar$ ages with ± 2 σ uncertainties; arrows indicate the ages of clasts are maximum ages of deposition. Arrowed bars labeled "D1" through "D4" are stratigraphic constraints based on diatom stratigraphy, and the bar labeled "S1" is the Sr age with ± 2 σ uncertainty.

AGE OF UPPER CRP-1

Duplicate analyses of a basanitic clast from the carbonate fossil-rich layer at 33 mbsf indicate a maximum age of 1.2±0.1 Ma for this stratigraphic horizon. Although the $^{40}Ar/^{39}Ar$ data provide no evidence for depositional or minimum ages for the Quaternary part of CRP-1, the 1.2 ± 0.1 Ma maximum age is consistent with age determinations based on Sr dating (Lavelle, this volume) and diatom biostratigraphy (Harwood et al., this volume).

ACKNOWLEDGEMENTS

This work was supported by NSF grant OPP95277329. The New Mexico Geochronology Research Laboratory is supported in part by the New Mexico Bureau of Mines and Mineral Research and by NSF Grants EAR-92064438 and EAR-9706062. John Smellie's efforts in collecting dating samples from CRP-1 were essential to this study and are much appreciated.

REFERENCES

Cape Roberts Science Team, 1998. Summary of Results from CRP-1, Cape Roberts Project, Antarctica. *Terra Antartica*, **5**(1), 125-137.

Deino A. & Potts R., 1990. Age probability spectra for examination of single-crystal $^{40}Ar/^{39}Ar$ dating results: examples from Olorgesailie Formation, Southern Kenya Rift. *Quaternary International*, **13/14**, 47-53.

Deino A. & Potts R., 1992. Single-Crystal $^{40}Ar/^{39}Ar$ dating of the Olorgesailie Formation, Southern Kenya Rift. *J. Geophys. Res.*, **95**, 8453-8470.

Fleck R.J., Sutter J.F. & Elliot D.H., 1977. Interpretation of discordant $^{40}Ar/^{39}Ar$ age-spectra of Mesozoic tholieiites from Antarctica. *Geochim. Cosmochim. Acta*, **41**, 15-32.

McDougall I. & Harrison T.M., 1988. Geochronology and thermochronology by the $^{40}Ar/^{39}Ar$ method. *Oxford Monographs on Geology and Geophysics*, **9**, 212 p.

McIntosh W.C. & Chamberlin R.M., 1994. 40Ar/39Ar geochronology of Middle to Late Cenozoic ignimbrites, mafic lavas, and volcaniclastic rocks in the Quemado Region, New Mexico. *New Mexico Geological Society Guidebook*, **45**, 165-185.

Samson S.D. & Alexander E.C. Jr., 1987. Calibration of the interlaboratory $^{40}Ar/^{39}Ar$ dating standard, Mmhb-1. *Chem. Geol.*, **66**, 27-34.

Steiger R.H. & Jäger E., 1977. Subcommission on geochronology: Convention on the use of decay constants in geo- and cosmochronology. *Earth and Planet. Sci. Lett.*, **36**, 359-362.

Appendix 1 - Single-crystal laser-fusion $^{40}Ar/^{39}Ar$ results from CRP-1 K-feldspar.

ID	$^{40}Ar/^{39}Ar$	$^{37}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$ (x 10^{-3})	$^{39}Ar_K$ (x 10^{-15} mol)	K/Ca	%^{40}Ar*	Age (Ma)	±2σ (Ma)
CRP-90-3, J=0.000813612±0.09%, D=1.0024±.001, Lab#=9074								
02	13.70	0.0009	2.2380	0.417	558.2	95.2	19.04	0.29
01	13.48	0.0018	0.6442	0.821	278.7	98.6	19.40	0.15
weighted mean			n=2		418.4 ±395.3		19.32	0.32
CRP-104-1, J=0.000813399±0.09%, D=1.0024±.001, Lab#=9069								
07	13.45	0.0079	1.2170	1.29	64.2	97.3	19.11	0.12
06	13.20	0.0104	0.3177	1.76	49.2	99.3	19.135	0.092
04	13.18	0.0111	0.1195	1.20	46.0	99.7	19.19	0.12
05	13.40	0.0101	0.5367	1.13	50.4	98.8	19.33	0.13
09	13.55	0.0076	0.8966	0.838	67.5	98.0	19.39	0.16
03	13.34	0.0132	0.0398	0.895	38.5	99.9	19.46	0.15
02	13.26	0.0113	-0.2909	0.885	45.2	100.7	19.47	0.16
08	13.53	0.0104	0.4496	1.27	49.0	99.0	19.55	0.11
01	13.34	0.0086	-0.4717	0.852	59.2	101.0	19.67	0.18
weighted mean			n=9		52.1 ±19.0		19.32	0.14
CRP-116-21, 117-21, 117-22								
06	13.28	0.0049	2.409	0.098	104.3	94.6	18.2	1.6
18	13.58	0.0142	2.001	0.077	35.9	95.7	18.8	3.4
01	22.01	0.4182	31.37	0.082	1.2	58.0	18.5	1.6
weighted mean			n=2		52.7 ±145.7		18.36	1.18

Note: isotopic ratios corrected for blank, radioactive decay, and mass discrimination, not corrected for interferring reactions. Individual analyses show analytical error only; mean age errors also include error in J and irradiation parameters. D = mass dicrimination. Analyses in italics are excluded excluded from mean age calculations. Correction factors: $(^{39}Ar/^{37}Ar)_{Ca}$ = 0.00070±0.00005; $(^{36}Ar/^{37}Ar)_{Ca}$ = 0.00026±0.00002; $(^{38}Ar/^{39}Ar)_K$ = 0.0119; $(^{40}Ar/^{39}Ar)_K$ = 0.0002±0.0003.

Appendix 2 - Bulk-sample step-heating $^{40}Ar/^{39}Ar$ results from CRP-1 clasts.

ID	Temp (°C)	$^{40}Ar/^{39}Ar$	$^{37}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$ (x 10^{-3})	$^{39}Ar_K$ (x 10^{-15} mol)	K/Ca	$^{40}Ar^*$ (%)	^{39}Ar (%)	Age (Ma)	±2σ (Ma)
CRP-33-1, 53.33 mg, J=0.000789602±0.10%, D=1.0024±0.001, NM-87, Lab#=9091-01										
B	600	85.88	0.6554	300.4	0.108	0.78	-3.3	0.3	-4.00	14.6
C	675	5.406	0.8988	17.29	2.40	0.57	6.8	6.4	0.52	0.57
D	750	2.950	0.8406	8.189	9.92	0.61	20.2	31.5	0.85	0.13
E	800	3.374	0.7455	8.999	8.74	0.68	22.9	53.6	1.10	0.15
F	850	3.600	0.8582	9.635	6.89	0.59	22.7	71.1	1.17	0.20
G	900	3.753	0.9714	9.802	4.10	0.53	24.8	81.5	1.33	0.32
H	975	4.010	1.243	10.23	1.55	0.41	27.0	85.4	1.54	0.85
I	1050	3.928	1.934	9.362	0.509	0.26	33.4	86.7	1.90	2.90
J	1125	5.421	3.104	17.62	2.70	0.16	8.4	93.6	0.65	0.49
K	1250	7.925	47.16	35.65	2.44	0.011	12.8	99.7	1.49	0.55
L	1650	350.7	114.0	166.5	0.099	0.004	88.5	100.0	425.8	23.9
total gas age			n=11		39.5	0.53			2.1	0.40
plateau			n=4	steps E-H	21.3	0.60	24.4	53.9	1.16	0.15
CRP-33-1, 101.93 mg, J=0.000787124±0.10%, D=1.0024±0.001, NM-87, Lab#=9090-01										
B	600	87.80	0.6218	301.1	0.224	0.82	-1.3	0.3	-1.6	7.1
C	675	8.927	0.8253	28.83	3.55	0.62	5.3	4.7	0.67	0.39
D	750	3.533	0.7849	10.21	18.6	0.65	16.3	27.6	0.819	0.080
E	800	3.826	0.7053	10.44	17.6	0.72	20.7	49.2	1.127	0.083
F	850	4.317	0.7762	11.99	14.1	0.66	19.3	66.6	1.19	0.10
G	900	4.440	0.8732	12.17	8.91	0.58	20.5	77.6	1.30	0.15
H	975	4.511	1.093	12.10	3.36	0.47	22.6	81.7	1.45	0.37
I	1050	12.79	1.560	36.72	0.975	0.33	16.1	82.9	2.9	1.3
J	1125	7.260	2.321	21.68	5.29	0.22	14.2	89.5	1.47	0.25
K	1250	8.685	27.56	30.83	7.92	0.019	19.5	99.2	2.45	0.21
L	1650	125.5	48.78	83.41	0.615	0.010	83.3	100.0	147.6	2.6
total gas age			n=11		81.0	0.55			2.36	0.23
plateau			n=5	steps E-I	44.9	0.65	19.8	55.4	1.18	0.12
CRP-61-7, 3.79 mg, J=0.000808551±0.09%, D=1.0024±0.001, NM-88, Lab#=9127-01										
A	625	4828.8	1.269	16252.3	0.050	0.40	0.5	1.2	38.0	719.9
B	700	2369.9	2.115	7892.5	0.074	0.24	1.6	3.1	54.5	219.4
C	750	1486.7	1.900	4988.1	0.162	0.27	0.9	7.2	18.7	66.4
D	800	330.9	1.915	1074.8	1.12	0.27	4.1	35.2	19.5	4.5
E	875	16.53	1.907	14.59	1.23	0.27	74.8	65.9	17.97	0.60
F	975	14.34	1.873	5.238	0.408	0.27	90.2	76.1	18.8	1.6
G	1075	73.38	1.870	190.6	0.095	0.27	23.4	78.4	25.0	8.9
H	1250	16.92	2.005	11.17	0.855	0.25	81.4	99.8	20.01	0.87
I	1650	235.3	5.802	665.1	0.010	0.088	16.7	100.0	56.6	138.7
total gas age			n=9		4.01	0.27			20.1	18.1
plateau			n=4	steps D-G	2.86	0.27	48.1	71.3	18.12	0.84
CRP-61-8, 1.91 mg, J=0.000809219±0.09%, D=1.0024±0.001, NM-88, Lab#=9128-01										
A	625	12.53	0.0678	19.41	0.339	7.5	54.3	9.1	9.9	2.1
B	725	10.83	0.0741	2.143	1.06	6.9	94.2	37.4	14.84	0.69
C	825	11.42	0.0612	-0.4170	1.03	8.3	101.1	64.9	16.79	0.70
D	875	11.93	0.0291	-1.5324	0.469	17.5	103.8	77.4	18.0	1.5
E	975	14.07	0.0168	2.833	0.250	30.3	94.1	84.1	19.2	2.8
F	1100	12.72	0.1355	2.149	0.155	3.8	95.1	88.2	17.6	4.7
G	1250	14.23	0.0921	3.686	0.378	5.5	92.4	98.3	19.1	1.9
H	1650	33.80	0.5192	75.56	0.063	0.98	34.1	100.0	16.7	12.1
total gas age			n=8		3.74	9.9			16.2	1.5
plateau			n=6	steps C-H	2.34	11.6	86.8	62.6	17.3	1.0
CRP-90-6, 6.46 mg, J=0.00079615±0.10%, D=1.0024±0.001, NM-87, Lab#=9076-01										
B	600	92.89	0.3849	262.0	0.011	1.3	16.7	0.1	22.1	140.9
C	675	10.72	0.3475	7.083	0.350	1.5	80.7	2.6	12.4	3.6
D	750	12.71	0.3444	1.856	2.59	1.5	95.9	21.6	17.43	0.48
E	800	13.17	0.2660	0.9221	2.00	1.9	98.1	36.4	18.47	0.62
F	850	13.55	0.2299	2.287	1.72	2.2	95.1	49.0	18.42	0.72
G	900	13.99	0.2395	3.636	1.32	2.1	92.4	58.7	18.48	0.95
H	975	14.03	0.2978	4.415	1.23	1.7	90.9	67.7	18.2	1.0
I	1050	14.33	0.4130	6.492	0.955	1.2	86.8	74.8	17.8	1.3
J	1125	14.75	0.5679	8.299	0.665	0.90	83.7	79.6	17.7	1.8
K	1250	13.87	0.9548	5.324	2.66	0.53	89.2	99.2	17.69	0.48
L	1650	28.00	1.723	52.83	0.113	0.30	44.7	100.0	17.9	10.7
total gas age			n=11		13.6	1.5			17.8	1.0

Appendix 2 - Continued.

ID	Temp (°C)	$^{40}Ar/^{39}Ar$	$^{37}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$ (x 10^{-3})	$^{39}Ar_K$ (x 10^{-15} mol)	K/Ca	$^{40}Ar^*$ (%)	^{39}Ar (%)	Age (Ma)	±2σ (Ma)
CRP-90-11, 7.18 mg, J=0.00079663±0.10%, D=1.0024±0.001, NM-87, Lab#=9081-01										
B	600	130.5	0.7480	460.4	0.011	0.68	-4.2	0.2	-7.9	159.6
C	675	12.28	0.8245	15.65	0.277	0.62	62.8	4.5	11.1	4.4
D	750	12.57	1.217	3.481	2.01	0.42	92.6	36.2	16.65	0.62
E	800	13.76	1.011	3.546	1.04	0.50	92.9	52.4	18.3	1.2
F	850	14.20	1.072	3.998	0.593	0.48	92.3	61.8	18.7	2.1
G	900	15.45	1.253	9.084	0.287	0.41	83.2	66.3	18.4	4.2
H	975	17.41	1.374	17.44	0.221	0.37	71.0	69.8	17.7	5.5
I	1050	15.32	1.622	11.48	0.244	0.31	78.7	73.6	17.3	4.9
J	1125	15.09	1.727	10.72	0.603	0.30	79.9	83.1	17.3	2.0
K	1250	14.90	10.34	16.60	1.04	0.049	72.4	99.5	15.6	1.2
L	1650	59.73	8.359	155.1	0.033	0.061	24.4	100.0	20.9	39.1
total gas age			n=11		6.36	0.37			16.9	2.2
plateau			n=7	steps D-J	5.00	0.42	84.4	78.6	17.15	0.80
CRP-114-22, 17.64 mg, J=0.000806498±0.09%, D=1.0024±0.001, NM-88, Lab#=9113-01										
A	625	528.8	1.921	1755.6	0.462	0.27	1.9	3.2	14.8	12.0
B	700	292.3	2.320	941.8	0.807	0.22	4.8	8.8	20.5	5.0
C	730	232.9	2.221	749.0	0.812	0.23	5.0	14.4	17.0	4.0
D	775	124.7	2.364	379.1	1.38	0.22	10.3	23.9	18.7	1.9
E	825	95.36	2.521	276.8	1.58	0.20	14.4	34.9	20.0	1.4
F	875	49.21	2.484	122.3	2.16	0.21	26.9	49.8	19.21	0.59
G	950	85.93	2.646	239.6	1.54	0.19	17.8	60.4	22.2	1.2
H	1000	162.0	2.677	494.0	0.460	0.19	10.0	63.6	23.5	3.8
I	1075	159.9	2.551	472.5	0.440	0.20	12.8	66.6	29.6	3.6
J	1135	66.91	2.730	179.7	1.74	0.19	21.0	78.6	20.32	0.94
K	1250	41.71	9.264	98.83	3.07	0.055	31.7	99.8	19.25	0.51
L	1650	217.4	33.59	721.3	0.030	0.015	3.1	100.0	10.2	51.4
total gas age			n=12		14.5	0.17			19.9	2.0
near plateau			n=12	steps A-L	14.5	0.17	13.3	100.0	19.73	0.86

Note: see note appendix 1.