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$^{40}Ar/^{39}Ar$ geochronology of biostratigraphically controlled Miocene tuffs from central Japan: Comparison with Italy and age of the Serravallian–Tortonian boundary

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Abstract

Two volcanic tuffs of the Tomioka area (central Japan) were selected from a fossiliferous marine succession of Late Serravallian age (Miocene) and dated using the incremental heating $^{40}Ar/^{39}Ar$ technique. The Kitamura Tuff, located 10 m below the regional first occurrence (FO) of the planktonic foraminifera *Globigerina nepenthes*, yielded a biotite age of 11.79 ± 0.08 Ma. The Baba Tuff, located 180 m above the previous one, yielded ages of 11.26 ± 0.09 Ma (biotite), 11.29 ± 0.12 Ma (sanidine) and ~ 11.3 Ma (plagioclase). Isochron ages are similar to the plateau ages.

The FO of *G. nepenthes* is estimated to have an age of 11.76 ± 0.10 Ma in Japan. Similarly precise results obtained from the Central Apennines (biotite: 11.48 ± 0.13 Ma) lead to an age estimate of 11.53 Ma for the same FO in an Italian section. The age difference between sections is of the same magnitude as the analytical errors. However, the potential diachronism of the biostratigraphic signal has been independently estimated at 0.25 Ma. The FO of *G. nepenthes* and FO of *Neogloboquadrina acostaensis* are commonly taken as index for the beginning of N14 and end of N15 foraminiferal biozones, respectively. In the Italian section, the time interval between the two signals — i.e. the duration of the two biozones — may be estimated at 0.3 Ma. The Serravallian–Tortonian boundary located near the dated layers can be estimated to have an age of $\sim 11.25 \pm 0.20$ Ma.

1. Introduction

The Miocene sedimentary sequence from Japan commonly has volcaniclastic horizons interbedded within it. This situation is of great interest in stratigraphy because a numerical time intercalibration is possible between biostratigraphy, sedimentology, and tectonics.

Recently, an international working group was established for gathering such intercalibration points connecting biostratigraphically and geochronologically characterized levels (Subcommission on Geochronology of IUGS, Working Group: Integrated Stratigraphy of the Miocene Sequence). Although a number of datable layers are now known from Miocene sedimentary successions, very few integrated studies exist. The inte-

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Abbreviations used

I.U.G.S.	= International Union of Geological Sciences
FO	= first occurrence
G.	= *Globigerina*
LO	= last occurrence
FAD	= first appearance datum
MSWD	= mean standard weigthed deviation

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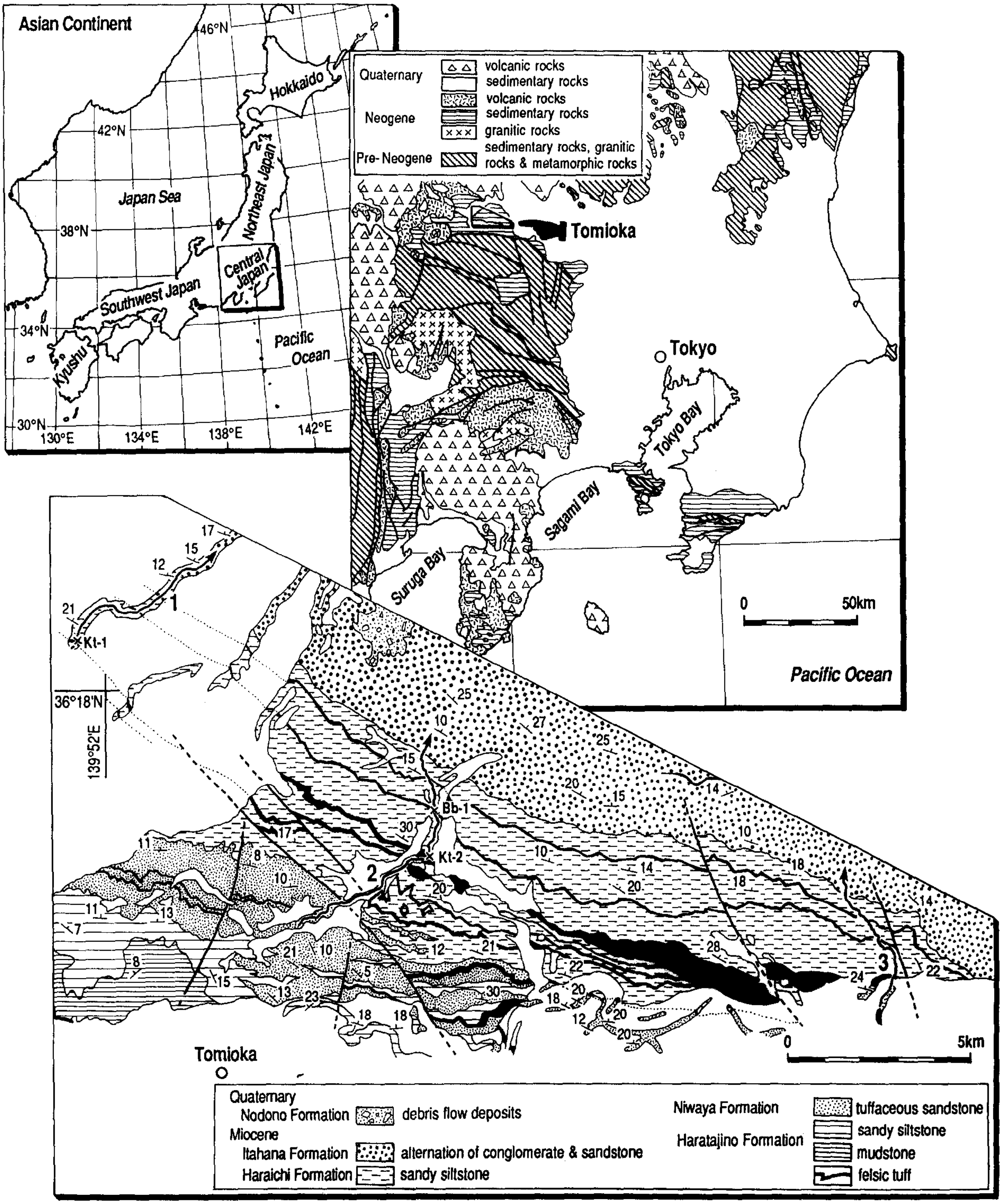

Fig. 1. Location of the study area and detailed geological map of the upper part of the Tomioka sequence. The sections (see Fig. 3) and localities of tuff samples used for radiometric dating are also shown. 1 = Usigawa section (studied sample Kt-1); 2 = Hoshigawa section (studied sample Kt-2 and Bb-1); 3 = Kamikoizawa section (from west to east, respectively).

grated stratigraphic approach has been focussed on the two potentially best areas: Italy and Japan where volcanic activity and oceanic sedimentary deposition are contemporaneous.

In this paper we present recent $^{40}Ar/^{39}Ar$ dating results on Late Serravallian levels from central Japan. In parallel with this work we also had the opportunity to study a time equivalent situation in Italy.

The resulting $^{40}Ar/^{39}Ar$ ages directly obtained from biostratigraphically documented sections permit comparisons and discussion of the reliability of the biostratigraphic information and the age of the Serravallian–Tortonian Stage (Middle–Late Miocene Series) chronostratigraphic boundary.

2. Geology

A thick marine Miocene sequence is well developed in the Tomioka area of central Japan (Fig. 1). Sepa-

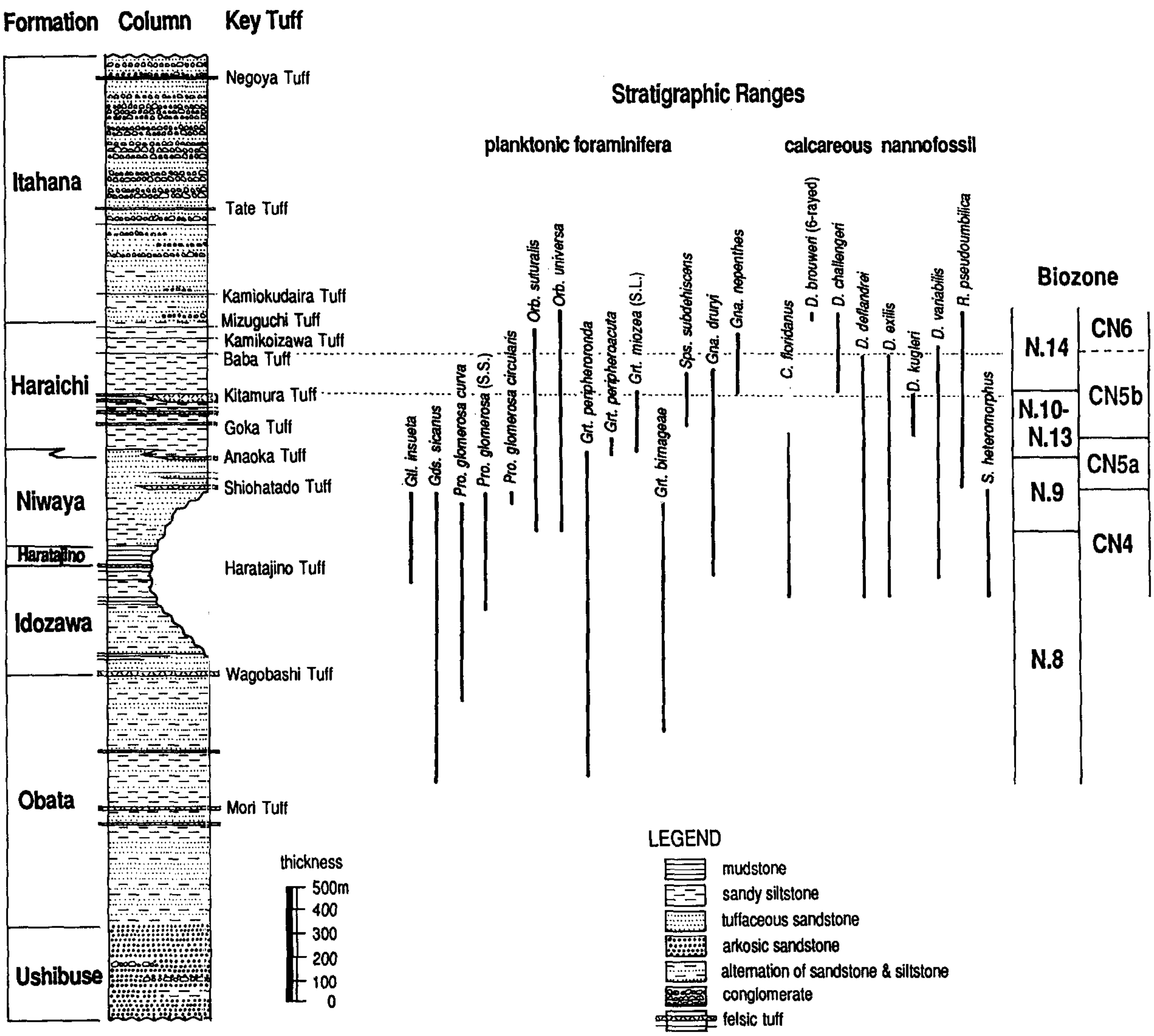

Fig. 2. Generalized stratigraphic column of the Miocene sedimentary sequence in the Tomioka area, also showing the relationships between stratigraphic ranges of selected microfossils and the levels of key tuff layers.

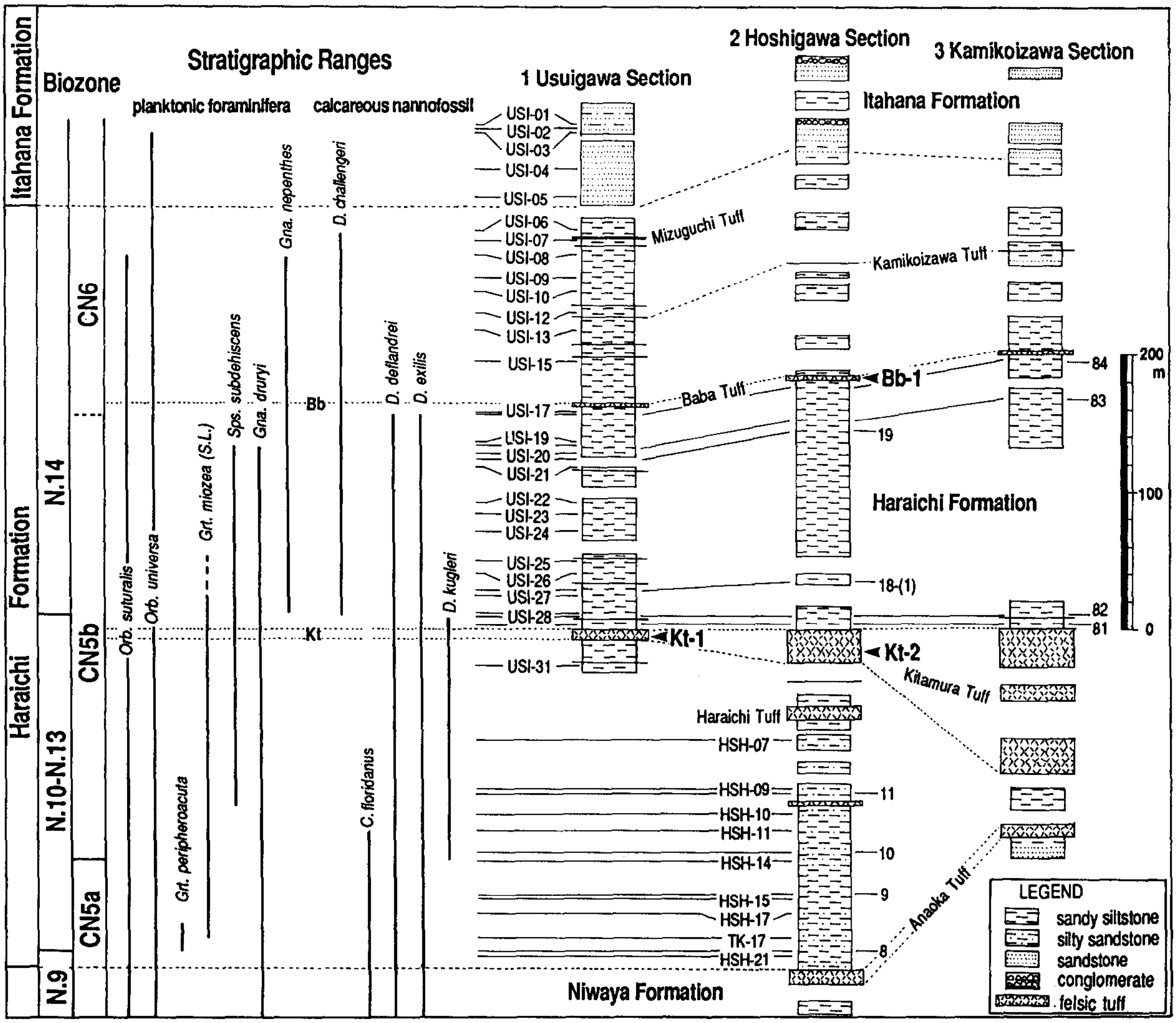

Fig. 3. The three sections. Collection sites for biostratigraphic study are plotted; samples shown to the left of the column (USI-01, ..., USI-31; HSH-07, ..., HSH-21) are after Takayanagi et al. (1976); samples to the right are after Chiji and Konda (1978). The local first occurrence of *G. nepenthes* is recognized from samples USI-23 (section 1) and 82 (section 3); the regional "first appearance datum" is adopted from the oldest level (sample 82) ~ 10 m above the Kitamura Tuff. Kt-1, Kt-2 and Bb-1 indicate the points of collected tuff samples.

rated from the pre-Miocene basement rocks by unconformities or faults, the Miocene strata are developed in a roughly homoclinal structure with an east-southeast–west-northwest strike and a north-northeast dip. This sequence is divided into the following seven units by Oishi and Takahashi (1990), in ascending order: Ushibuse (>400 m in thickness), Obata (900 m), Idozawa (450 m), Haratajino (40 m), Niwaya (<400 m), Haraichi (550 m) and the Itahana Formation (1200 m). The relationships between units are conformable except for the Niwaya Formation, which is unconformable on the underlying units (Fig. 2).

Table 1
Results of the ^{40}Ar–^{39}Ar increment heating measurements on T217 biotite; T215 biotite, sanidine and plagioclase

HT (°C)	*T* (Ma)	±2σ	%^{39}Ar	% rad	K/Ca
T217 biotite (sample weight 12.62 mg):					
700	11.4	1.0	1.7	46	67
850	11.78	0.20	7.8	88	180
950	11.76	0.15	21.8	96	240
1,050	11.85	0.16	7.1	94	140
1,095	11.77	0.15	7.5	96	180
1,135	11.78	0.15	15.1	97	255
1,200	11.76	0.14	36.9	99	505
1,350	11.93	0.23	2.1	100	290
T215 biotite (sample weight: 13.83 mg):					
700	11.3	0.3	5.6	46	51
850	11.45	0.24	7.5	74	88
950	11.45	0.22	11.3	83	95
1,020	11.40	0.15	14.0	92	101
1,075	11.22	0.14	22.6	96	116
1,120	11.19	0.14	21.2	97	80
1,190	11.24	0.14	16.5	99	31
1,350	11.1	0.7	1.3	100	15
T215 sanidine (sample weight: 11.4 mg):					
900	10.6	0.6	1.7	62	39
1,050	10.89	0.21	4.5	86	56
1,150	11.18	0.15	8.2	98	69
1,185	11.35	0.15	5.9	99	82
1,230	11.31	0.16	9.4	98	84
1,270	11.29	0.14	16.4	98	88
1,320	11.25	0.14	34.2	99	87
1,370	11.35	0.14	15.8	99	83
(lost step)					
1,570	11.54	0.22	3.9	95	104
T215 plagioclase (sample weight 74.56 mg):					
730	9.5	1.0	3.5	43	0.10
875	10.8	0.4	5.6	57	0.08
1,025	10.97	0.22	20.2	86	0.07
1,175	11.25	0.16	23.8	94	0.08
1,325	11.39	0.18	16.1	93	0.08
1,475	11.59	0.26	24.2	54	0.07
1,600	12.0	0.4	6.5	53	0.06

HT = heating temperature; *T* = apparent age; error on $J = \pm 0.5\%$; monitor: biotite HD-B1 = 24.21 Ma; all samples irradiated for 10 MW-hr in the central thimble position, U.S.G.S. TRIGA reactor, Denver, Colorado, U.S.A.; measurements by G.S.O. in Lausanne, Switzerland; correcting factors used from our measurements of CaF_2 and K_2SO_4: $(^{40}Ar/^{39}Ar)_K = 0.0273 \pm 0.0022$; $(^{36}Ar/^{37}Ar)_{Ca} = 0.000253 \pm 0.000037$; $(^{39}Ar/^{37}Ar)_{Ca} = 0.000569 \pm 0.000013$.

High-confidence correlation is possible through the whole area owing to the presence of the many felsic tuffs. They can be identified in the field using their lithologic properties from diverse sections exposed by rivers dissecting the sequence (sections 1–3 in Fig. 1). Most of these tuffs have an individual name (key tuff layers) given according to the locality from where they were first described.

3. Biostratigraphy

The Miocene strata of the Obata to Haraichi Formations contain abundant planktonic and benthic foraminifera, calcareous nannofossils and radiolarians. They have been extensively studied (e.g., T. Saito, 1963; Kurihara, 1974; Takayanagi et al., 1976, 1978; Chiji and Konda, 1978; Konda, 1980).

The relations between the stratigraphic ranges of some important species of planktonic foraminifera and key tuff layers are shown in Fig. 2. The following eight horizons of first occurrences (FO) of planktonic foraminiferal species have been found in ascending order:
Praeorbulina glomerosa curva FO
P. glomerosa glomerosa FO
Orbulina suturalis FO and *O. universa* FO
P. glomerosa circularis FO
Globorotalia peripheroacuta FO
Sphaeroidinellopsis subdehiscens FO
Globigerina nepenthes FO

The collecting sites of samples studied and the location of each columnar section are shown in Fig. 1. The columnar stratigraphic sections of the Haraichi Formation with sample positions of microfossils and geochronology are also shown in Fig. 3; in this figure, samples represented on the left-and right-hand sides of each columnar section are after Takayanagi et al. (1976) and Chiji and Konda (1978), respectively.

The FO of *G. nepenthes*, which defines the local N13–N14 boundary (Blow, 1969), has been recognized in section 1 (sample USI-23) by Takayanagi et al. (1978) and in section 3 (sample 82) by Chiji and Konda (1978). While the horizon with the FO of the *G. nepenthes* in section 1 is intermediate between two tuff beds (Baba Tuff and Kitamura Tuff), Chiji and Konda (1978) have shown that the position of that FO in section 3 is older being only 10 m above the Kitamura

Tuff. The thickness between the Kitamura and the Baba Tuff in each section is almost the same (~180 m), which suggests that the difference of the stratigraphic positions of each FO is not due to the existence of a hiatus just above the Kitamura Tuff in section 3. We have adopted the lower horizon as the regional first appearance datum (regional FAD) of *G. nepenthes*. Therefore, the regional N13–N14 boundary is 150–190 m below the Baba Tuff and ~10 m above the Kitamura Tuff. *G. druryi*, which is considered to be the ancestor of *G. nepenthes*, disappears at a level located between the two tuffs.

Other important potential time markers of the calcareous nannofossil zonation are also recognized within the Haraichi Formation. The last occurrence (LO) of *Cyclicargolithus floridanus* was recognized in the lower part of the Haraichi Formation (sample HSH-11; Honda, 1981). The FO of *Discoaster kugleri* is also located in the lower horizon (sample HSH-14; Takayanagi et al., 1976). Consequently, the regional CN5a–CN5b boundary of calcareous nannofossil zones can be located 140–160 m below the Kitamura Tuff.

Discoaster deflandrei extends to the Baba Tuff horizon, while *D. challengeri* appears near the Kitamura Tuff (Fig. 3). The CN5b–CN6 boundary is probably located near the Baba Tuff, because the stratigraphic overlap of these two species is restricted to the last part of zone CN5b (Perch-Nielsen, 1989).

We collected tuff samples from two horizons; Baba Tuff and Kitamura Tuff. Both tuff samples have been geochronologically dated by several workers (Shibata et al., 1979, Kasuya, 1987; Takahashi et al., 1992) as the regional N13–N14 boundary of planktonic foraminiferal zones, which was recognized between the two layers as mentioned above.

4. Geochronology

4.1. Geochronometers

From the Kitamura Tuff (Haraichi Fm.), several samples were investigated and a single one selected from the Fujiki River section (Kt-2 purified as T217). The separated biotite flakes are typically idiomorphic and brilliant with abundant inclusions (up to 5 of them in a single 0.5-mm-wide crystal). Following magnetic treatment, hornblende and inclusions were eliminated by gentle crushing of the mica flakes with acetone in an agate mortar. Feldspars are present in that sample but most crystals are coated with white to red cement and easily separated.

From the Baba Tuff, one sample was collected from the Fujiki River section (Bb-1 purified as T215). Idiomorphic biotite flakes (0.1–0.6 mm in size) had no inclusions. The non-magnetic pyroclastic minerals including quartz and feldspars are abundant. Following

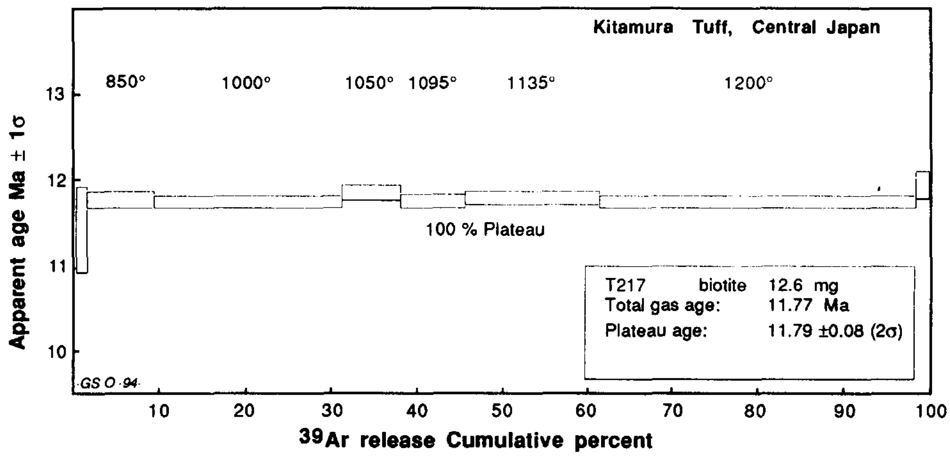

Fig. 4. Age spectrum of biotite T217 (Kitamura Tuff) dated in this paper. The vertical thickness of the *bars* for each step corresponds to 1σ analytical precision.

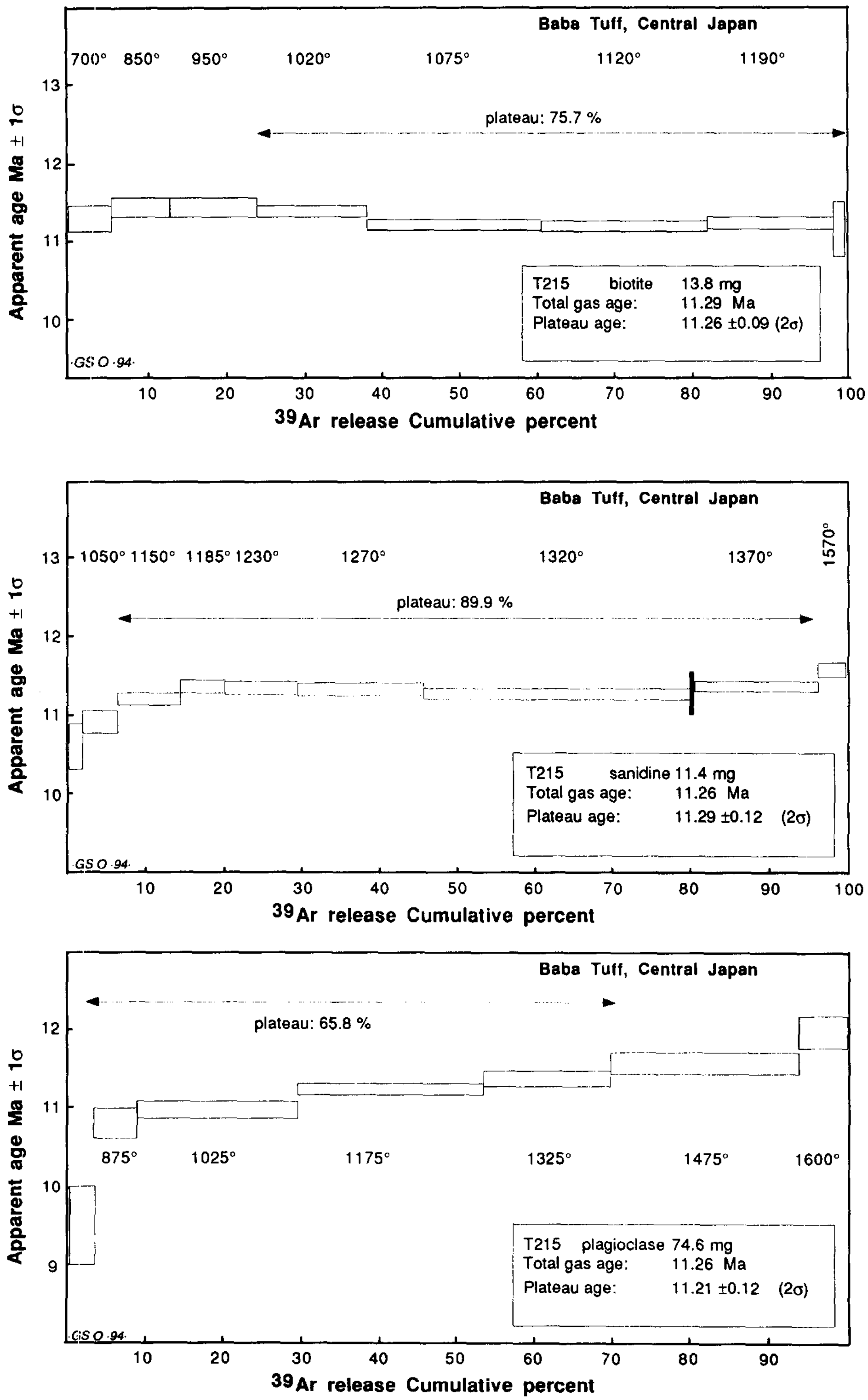

Fig. 5. Age spectra of the three mineral separates from sample T215 (Baba Tuff) dated in this paper.

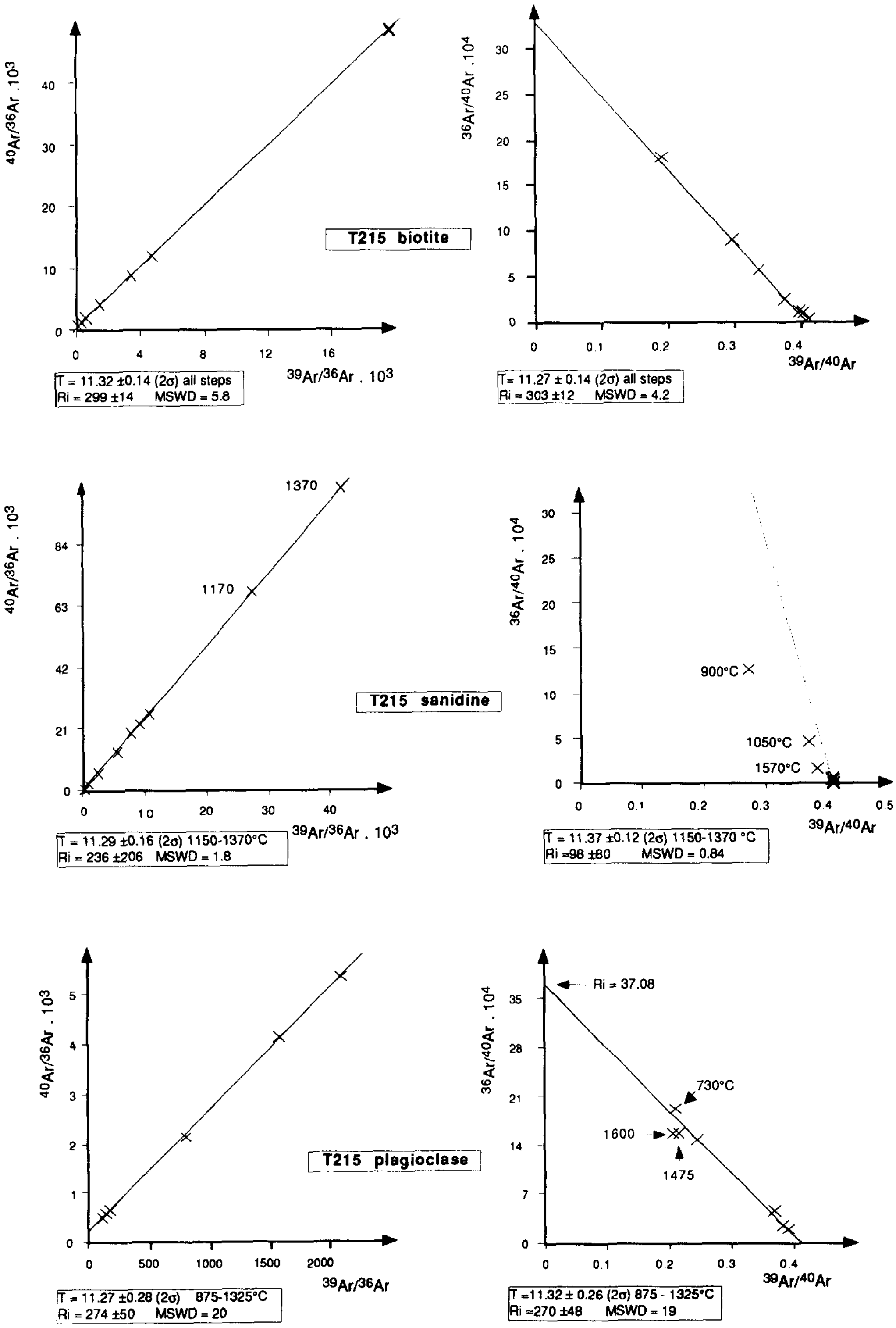

Fig. 6. Isochron plots for minerals extracted from the Baba Tuff.

ultrasonic cleaning for 20 min with a dilute acetic–nitric acid mixture, plagioclase and sanidine were purified in bromoform–acetone mixtures, and then hand-picked.

Microscopic examination and X-ray diffraction (XRD) analyses indicate that the material investigated corresponds to pure volcaniclastic tuffs. These tuffs comprise well-preserved volcanogenic minerals. The selected separates are pure with three different minerals separated for the most favourable sample (T215) and one for the other (T217).

4.2. Isotopic dating

4.2.1. Analytical techniques

$^{40}Ar/^{39}Ar$ analyses were performed at the University of Lausanne, Switzerland (on a Mass Analyser Products® 215-50 mass spectrometer) following the usual procedure in this laboratory for irradiation (U.S. Geological Survey's TRIGA reactor in Denver, Colorado), sample treatment, heating procedure (resistance-heated furnace), correction for mass discrimination, interfering derived isotopes of Ar, and neutron flux gradient, and age calculation using I.U.G.S. Subcommission on Geochronology standard decay constants (Cosca et al., 1992). During this particular set of analyses, we used a conservative error for the *J* curve of ±0.5% (deduced from inter-irradiation comparisons). The blank values for mass 40 increased from $\sim 4 \cdot 10^{-15}$ mol at temperatures up to 1350°C to $\sim 10 \cdot 10^{-15}$ mol at 1600°C; for masses 39 and 36 the blanks remain at or less than $0.02 \cdot 10^{-15}$ mol. Analytical flux calibration was made using the monitor biotite HD-B1 (Fuhrmann et al., 1987) for which a recently revised age at 24.21 Ma has been accepted (Hess and Lippolt, 1994). This interlaboratory comparison (based on conventional — isotope dilution — technique results) leads to an observed interlaboratory standard deviation of ±0.32 Ma which means a (2σ) error of ±2.5% for the age. This error is not due to sample inhomogeneity as the results show an acceptable intralaboratory reproducibility for both K and ^{40}Ar in most laboratories (standard deviation of ~ ±0.5% for each component).

In order to remove most of the adsorbed gas, the samples were heated in the crucible under vacuum for some time just before measurements: 40 min at 550°C (T215 plagioclase and T215 biotite — one step), or 600°C for 25 min (T215 sanidine; T215 biotite-spectrum; and T217 biotite). Our experience has shown that the portion of gas released at these temperatures is extremely small and does not influence the total gas age significantly for the well-preserved material dated.

All errors are given at the (2σ) 95% confidence level in this text.

4.2.2. Results

4.2.2.1. Data and age spectra

Summarized analytical data are gathered in Table 1. Detail on the analytical measurements may be found in Appendix A.

Argon in the biotite separated from the Kitamura Tuff was released in eight steps (T217, Table 1). The ages calculated for each step are internally concordant; the age spectrum is flat with a total gas age at 11.77±0.09 Ma (Fig. 4).

Argon in the biotite separated from the overlying Baba Tuff also was released in eight steps (T215, Table 1). The corresponding age spectrum (Fig. 5) is not perfectly flat but the whole spectrum is acceptable within error limits and the total gas age is 11.29±0.09 Ma. A second subsample of the same biotite (8.2 mg) was analysed two months later as part of a set of intra-laboratory comparisons. A three-step spectrum with a total gas age of 11.48±0.14 Ma agrees within error of the original measurements.

Nine steps were measured from the sanidine (the tenth was lost during purification procedure), and significant and precise ages obtained for most of them (Table 1). The age spectrum (Fig. 5) shows a large portion of the spectrum which is flat. Compared to the total gas age at 11.26 Ma, younger apparent ages exist for the low-temperature steps (probably due to the altered surface of the crystals) and there is a slightly higher apparent age for the highest-temperature step.

The plagioclase has a stair-shaped spectrum (Fig. 5) with apparent age of 11.0 Ma at low temperature to apparent age of 11.6 Ma. The integrated fusion age is 11.26 Ma (Table 2) overlapping the age of the biotite and the sanidine of the same sample.

4.2.2.2. Plateau ages

We may calculate "plateau ages" by combination of more than two successive steps showing internally consistent ages or $^{40}Ar^{\star}/^{39}Ar$ ratios within 2σ error margins and for which the sum of the gas released is

>50% of the total. Table 2 gives the results of these calculations. Three amongst the four dated phases (T217 biotite, T215 biotite and sanidine) give excellent plateaus comprising >75% of the total gas released and more than four successive steps. In all three cases, the resulting plateau ages are not significantly different from the total gas ages. This is usually considered a good criterion for reliability (geological significance) of the age calculated. The apparent ages are consistent with the stratigraphic order and the ages for the biotite (11.26 Ma) and sanidine (11.29 Ma) from the Baba Tuff are in excellent agreement.

Table 2
Plateau ages calculated from the incremental heating experiments

Sample	T (total gas) (Ma)	Gas released (%)	Heating steps (°C)	Plateau age (Ma)
T217 bio	11.77±0.09	98.3	850–1,350	11.79±0.08
T215 bio	11.29±0.09	75.7	1,020–1,350C	11.26±0.09
T215 san	11.26±0.12	89.9	1,150–1,370	11.29±0.12
T215 plag	11.26±0.12	65.8	875–1,325	11.21±0.12

bio = biotite; san = sanidine; plag = plagioclase.

Table 3
Results of isochron age calculations for minerals from the Baba Tuff

Sample	Heating steps (°C)	Isochron	T (Ma)	R_i	MSWD
T215 bio	1,020–1,190	^{36}Ar	11.26±0.17	319±94	4.9
		^{40}Ar	11.20±0.15	358±64	2.3
	all steps	^{36}Ar	11.32±0.14	299±14	5.8
		^{40}Ar	11.27±0.14	303±12	4.2
T215 san	1,150–1,370	^{36}Ar	11.29±0.16	236±206	1.8
		^{40}Ar	11.37±0.12	98±80	0.84
	all steps	^{36}Ar	11.20±0.14	268±44	5.6
		^{40}Ar	11.31±0.13	252±36	4.5
T215 plag	875–1,325	^{36}Ar	11.27±0.28	274±50	20
		^{40}Ar	11.32±0.26	270±48	19
	all steps	^{36}Ar	11.17±0.26	301±24	25
		^{40}Ar	11.22±0.24	302±22	22

T (Ma) = apparent age (±2σ); R_i = calculated initial ^{40}Ar/^{36}Ar ratio (±2σ). "36Ar" means ^{40}Ar/^{36}Ar vs. ^{39}Ar/^{36}Ar plot; "40Ar" means ^{36}Ar/^{40}Ar vs. ^{39}Ar/^{40}Ar plot; bio = biotite; san = sanidine; plag = plagioclase.

Data from T215 plagioclase forms a plateau at 11.21±0.12 Ma for the steps from 875° to 1325°C according to the conventions given above. Another similarly valid plateau age (~11.4±0.1 Ma for 64.1% of the gas released) may also be calculated from the steps from 1175° to 1475°C. Independently of any other information, we may propose that, given the small range of variation in individual step ages and the regular shape of the spectrum, the plagioclase appears to be only slightly disturbed since crystallization. Excluding the deviating age for the low-temperature step, it seems reasonable to suggest that the actual age of that plagioclase lies between (and including) extreme values at 11.0 and 11.6 Ma.

4.2.2.3. Isochron plots

Calculations were undertaken for sample T215 both using the "normal" (^{40}Ar/^{36}Ar vs. ^{39}Ar/^{36}Ar) and "inverse" (^{36}Ar/^{40}Ar vs. ^{39}Ar/^{40}Ar) isochrons. Results of calculations are given in Table 3; isochron plots are shown in Fig. 6. The isochron results agree with the plateau ages.

5. Discussion

5.1. Geochronology

The best phase dated in this study is the carefully purified T217 biotite. The plateau generated with 100% of the gas released indicates perfectly preserved material. Both biotite and sanidine from sample T215 have a well-defined plateau; the precisely similar ages obtained from the two phases of this sample point to a good quality of the data and of the dated material.

The plagioclase of the same tuff gives a stair-shaped age spectrum. However, the individual step ages increase only slightly from 11.0 to 11.6 Ma with increasing temperature. "Pseudo-plateau" ages at 11.2 Ma (low temperatures) or 11.4 Ma (higher temperatures) can be calculated. The total gas age of the plagioclase is 11.26 Ma which is similar to the "real" age of the rock given by the other two phases: very near 11.3 Ma. The most appropriate age for the plagioclase seems to be obtained by combining all but the most deviate low-temperature step. In the light of the present case study, the best way to derive a significant age from a plagioclase characterized by an increasing stair-shaped age spectrum, and an analytically small difference in age between steps, is not to calculate a plateau age for a portion of the spectrum but to combine all steps. Depending on the spectrum obtained, it may be useful to eliminate the low-temperature (< 800°C) and the high-temperature (> 1500°C) steps if clearly discordant.

Isochron age calculations commonly are used to identify the trapped $^{40}Ar/^{36}Ar$ ratio. In the case of volcaniclastic rocks erupted in the atmosphere, the presence of non-atmospheric argon is *a priori* unlikely. We have observed during the present study that all phases have a normal atmospheric-like argon contribution [$^{40}Ar/^{36}Ar = 295.5$, see initial ratios (R_i) in Table 3]. Sanidine T215 yields an imprecise $^{40}Ar/^{36}Ar$ value because the data points are highly clustered.

The analytical ages deduced from this study are precise. For T217 biotite the age at 11.79 ± 0.08 Ma is well qualified by the perfect plateau: any disturbance would have disturbed the biotite spectrum because that mineral is extremely sensitive vis-à-vis the irradiation procedure as soon as a very small proportion of the sheets are altered.

Concerning sample T215, the differences between various calculation procedures are statistically insignificant; all calculated ages are between 11.20 and 11.35 Ma from which a pooled age at 11.28 ± 0.08 Ma is proposed; sanidine ages are on the old side and biotite on the young one. This pooled age is well substantiated as it is derived from several phases with different geochemical properties.

The ages of the two samples are in correct sequence and they bracket a time interval of 0.5 ± 0.1 Ma which is consistent with the thickness of sediments between them and the relevant biostratigraphic control. Previous data (Shibata et al., 1979; Kasuya, 1987; K. Saito and Takahashi, 1992) of 13.1 ± 0.8 Ma for the Kitamura Tuff and 11.6 ± 0.8 Ma for the Baba Tuff suggested an age difference of ~ 1.5 ± 1.5 Ma between the two tuffs. The present study was undertaken partly in order to obtain a more precise comparison.

We have been particularly careful to prepare highly pure separates and to try to document their a priori reliability which had not been tried in previous studies. Especially for sample T217, the presence of very common inclusions within the biotite flakes (possibly also of hornblende or pyroxene) are sources of potential increase in apparent ages (trapped ^{40}Ar).

Our new results indicate: (1) an age (11.28 Ma) similar to the previously published ones (11.6 Ma) for the Baba Tuff within error limits and a younger age for the Kitamura Tuff (11.79 vs. 13.1 Ma); (2) a more precise estimate of the age difference (~0.5 Ma) between the two dated layers; and (3) a good substantiation of the pair of ages deduced from the recognized quality of the dated material and the consistency of the analytical data.

The intralaboratory precision of ±0.08 Ma for the two ages at 11.79 and 11.28 Ma is relative to the accepted value of 24.21 Ma for the monitor allowing calibration of the neutron flux. However, the actual age of HD-B1 is only known with an error bar of ±2.5% of the age (2σ) which should be propagated to the analytical age to derive the actual accuracy of the geological age. Therefore, for interlaboratory comparisons and location of the dated layers in the numerical time scale, the ages to be considered are 11.79 ± 0.37 Ma (Kitamura Tuff) and 11.28 ± 0.36 Ma (Baba Tuff).

5.2. *Chronostratigraphy*

5.2.1. *Age of the* FO *of G. nepenthes in central Japan*

In central Japan, the Kitamura Tuff and the Baba Tuff are located ~10 m below and ~170 m above, respectively, the first occurrence of the *G. nepenthes*. From the $^{40}Ar/^{39}Ar$ ages of 11.79 and 11.28 Ma, the 180 m of sediments represent ~0.5 Ma. From this, we may suggest a mean rate of sediment accumulation of ~360 m Ma^{-1}; thus the age of the FO of *G. nepenthes* can be extrapolated to be 11.79 Ma − (10 m/360 m Ma^{-1}) ≈ 11.76 Ma.

5.2.2. *Age of the* FO *of G. nepenthes in the Central Apennines*

In the Monte dei Corvi section (Central Apennines, Italy), a biotite-rich layer (Livello Ancona) lies 1.5 m above the FO of *G. nepenthes* (Coccioni et al., 1992; Appendix B). In that section, the mean rate of sedimentation may be estimated from the fact that ~ $\frac{1}{2}$–$\frac{2}{3}$ of the Serravallian Stage is represented by 60 m of sediments. Assuming a duration for the Serravallian Stage of 3.5–4 Ma (Odin, 1994) a mean sedimentary accumulation rate between 25 and 35 m Ma^{-1} may be accepted. That estimate is consistent with preliminary geochronological data (Deino and Montanari, 1992) which indicate that two tuffs, 48.7 m from each other vertically, represent ~1.5 Ma or a mean sedimentation rate of ~30 m Ma^{-1}. If we use our age of 11.48±0.13 Ma (Appendix B) then the age of the FO of *G. nepenthes* in the Italian section (Livello Ancona) is 11.48 Ma + (1.5 m/30 m Ma^{-1}) = 11.53 Ma.

5.2.3. Potential diachronism of the FO *of G. nepenthes*

Because the two Japanese and Italian ages were obtained using the same analytical facility during the same set of measurements (same monitor, same irradiation), the age difference between the FO in the two areas is well constrained at ~0.23 Ma (11.76 Ma − 11.53 Ma). But the error in the difference (derived by combining the analytical errors of ~0.1 Ma for each estimated age) is of the same magnitude as the difference itself.

That difference appears small. However, if the derived difference of 0.23 Ma is correct, this would correspond to a sedimentary thickness of ~7 m in the Monte de Corvi section. In the same section, the two foraminiferal biozones N14 (the base of which is defined by the FO of *G. nepenthes*) and N15 (the top of which is defined by the first occurrence of *Neogloboquadrina acostaensis*) are represented by only 9 m of sediments. In this situation, a possible age difference of 0.23 Ma for a biozonal boundary would be of considerable significance.

There is another way to document the order of magnitude of the possible diachronism of the *G. nepenthes* FO biostratigraphic signal. We may consider the information from sections 1 and 3 of the Tomioka area. In section 3 *G. nepenthes* appears 10 m above the Kitamura Tuff and its extrapolated age is 11.76 Ma; in section 1 the same species appears at a level located in the middle of the interval between the Kitamura and the Baba Tuff. At the latter place, its age may therefore be estimated as the mean between the ages of the two tuffs, i.e. (11.79 Ma + 11.28 Ma):2 = 11.53 Ma. The age difference between the location of the signal in the two sections, 20 km apart, is ~0.25 Ma.

Finally, a biostratigraphic uncertainty for the location of the FO of *G. nepenthes* can be derived from the sedimentary thickness where the two taxa (ancestor and derived forms) are identified together. In Japan, this thickness (23 m) is equivalent to ~0.35 Ma; in Italy, the corresponding thickness (1.5 m) is equivalent to only 50 ka.

The chronologic significance of the FO of the taxon has already been discussed; Spencer-Servato et al. (1994) estimate its "reproducibility" to be at ±2.3 Ma. Between Italy and Japan, our data indicate a better reproducibility. This may be connected to the latitude of study (~45°N in Italy and ~35°N in Japan) in agreement with Spencer-Servato et al. (1994) who observe a better reproducibility for sites located between 25° and 40°S.

5.2.4. The age of the Serravallian–Tortonian boundary

In open ocean, the Serravallian–Tortonian boundary is commonly approximated using the FO of *N. acostaensis*. Spencer-Servato et al. (1994) derive a potential diachronism of ±2 Ma for this FO. The taxon is present in Italy. In the historical stratotype of the Tortonian Stage (northern Italy), the FO of *N. acostaensis* occurs within the base of the section (Iaccarino, 1985) and is therefore of early Tortonian age. Let us assume that the FO of *N. acostaensis* is contemporaneous in the different portions of the Italian sedimentary basins and is only slightly younger than the chronostratigraphic limit of the Tortonian Stage.

In the Central Apennines, a short extrapolation using sediment thicknesses (30 m Ma^{-1}) can be done: the FO of *N. acostaensis* is located 7.5 m above the Ancona layer dated from the Apennines (Appendix B). Thus a rounded age estimate of ~11.25±0.20 Ma can be derived (11.48 Ma − 0.25 Ma).

A preliminary dating result on this volcaniclastic layer was taken as indicating that the Serravallian–Tortonian boundary was in the interval 11.0±0.3 Ma (Odin, 1994). Considering the geochronologic data presented here and that the Serravallian–Tortonian boundary with regard to that biostratigraphic information is lower dawn the section, the older side of the interval seems more probable.

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Appendix A

Table A-1

Incremental heating analytical measurements for samples dated in this paper (gas in 10^{-15} mol; analytical error bars ($\pm 1\sigma$); 40/39 = ^{40}Ar radiogenic/$^{39}Ar_K$)

(°C)	$^{40}Ar^{\star}$	^{39}Ar	^{38}Ar	^{37}Ar	^{36}Ar	40/39
T217 biotite (J=0.002584):						
700	67.4±0.9	12.34±0.13	1.16±0.01	0.090±0.006	0.125±0.002	2.457±0.11
850	167.6±0.3	58.33±0.3	5.38±0.03	0.16±0.01	0.065±0.0015	2.536±0.02
950	429.1±0.9	162.6±0.3	14.81±0.05	0.332±0.015	0.057±0.003	2.530±0.01
1,050	142.4±0.3	52.65±0.06	4.780±0.012	0.182±0.012	0.268±0.001	2.550±0.01
1,095	147.7±0.3	55.82±0.11	5.117±0.011	0.150±0.013	0.020±0.001	2.534±0.01
1,135	294.1±0.6	112.32±0.22	10.277±0.007	0.216±0.007	0.293±0.002	2.536±0.01
1,200	701.2±1.4	274.5±0.5	24.93±0.05	0.266±0.002	0.017±0.002	2.531±0.01
1,350	40.09±0.09	15.83±0.03	1.429±0.007	0.027±0.010	0.00±0.00	2.568±0.02
T215 biotite (J=0.002573):						
700	234.7±0.47	44.49±0.10	3.898±0.014	0.426±0.015	0.426±0.004	2.44±0.03
850	200.28±0.02	59.6±0.3	5.10±0.04	0.332±0.015	0.177±0.003	2.474±0.02
950	266.3±0.5	89.8±0.4	7.62±0.02	0.462±0.012	0.147±0.004	2.475±0.002
1,020	298.0±0.6	111.8±0.2	9.38±0.03	0.539±0.012	0.074±0.003	2.465±0.01
1,075	452.8±0.9	180.0±0.4	15.22±0.03	0.761±0.012	0.052±0.001	2.425±0.01
1,120	420.6±0.8	169.2±0.3	14.28±0.02	1.043±0.019	0.035±0.002	2.419±0.01
1,190	322.9±0.7	131.9±0.3	10.85±0.01	2.104±0.019	0.0067±0.0011	2.429±0.01
1,350	23.7±0.3	10.46±0.05	0.807±0.003	0.340±0.018	0.000±0.000	2.402±0.07
T215 sanidine (J=0.002574):						
900	53.2±0.1	14.41±0.05	0.209±0.03	0.181±0.006	0.068±0.003	2.285±0.06
1,050	101.4±0.2	37.15±0.19	0.501±0.003	0.326±0.010	0.0469±0.001	2.352±0.02
1,150	168.7±0.3	68.12±0.19	0.896±0.006	0.482±0.007	0.0127±0.001	2.416±0.01
1,185	121.6±0.2	49.29±0.11	0.656±0.002	0.295±0.012	0.0019±0.001	2.451±0.01
1,230	193.7±0.4	77.9±0.3	1.022±0.005	0.456±0.008	0.0098±0.001	2.443±0.01
1,270	337.0±0.7	136.0±0.3	1.795±0.010	0.761±0.015	0.0152±0.001	2.440±0.01
1,320	698.4±1.4	283.5±0.6	3.75±0.02	1.61±0.02	0.027±0.001	2.430±0.01
1,370	321.9±0.6	130.6±0.3	1.719±0.007	0.775±0.022	0.003±0.001	2.452±0.01
1,470	-	-	(lost)	-	-	-
1,570	84.0±0.3	31.97±0.10	0.431±0.007	0.151±0.024	0.0138±0.002	2.495±0.02
T215 plagioclase (J=0.002584):						
730	38.20±0.06	7.996±0.018	0.131±0.002	37.91±0.10	0.084±0.003	2.038±0.10
875	53.19±0.20	13.01±0.06	0.207±0.003	80.44±0.22	0.099±0.001	2.324±0.04
1,025	127.0±0.3	46.63±0.10	0.647±0.003	306.2±0.5	0.139±0.003	2.360±0.02
1,175	140.0±0.3	54.89±0.13	0.760±0.002	353.0±0.4	0.118±0.002	2.420±0.01
1,325	97.88±0.18	37.18±0.08	0.528±0.005	227.9±0.6	0.083±0.001	2.451±0.01
1,475	257.6±0.5	55.90±0.11	0.957±0.004	393.2±0.7	0.505±0.004	2.494±0.03
1,600	71.94±0.18	14.949±0.018	0.268±0.003	121.4±0.2	1.145±0.002	2.586±0.04

Appendix B — Geochronology of a Late Serravallian biotite-rich layer; dating of the FO of *Globogerina nepenthes* in the Central Apennines, Italy

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In the Central Apennines (east-central Italy), volcaniclastic layers are present within a Miocene pelagic sedimentary suite (Monte dei Corvi, south of Ancona) with good bio-magneto- and chemostratigraphic potential for global correlation. This Appendix reports the biostratigraphic framework and geochronological results obtained from a 15-cm-thick biotite-rich layer (Ancona layer) sampled at meter level 150.0. This layer has a biostratigraphic control similar to that of the above studied Japanese material. In parallel to this

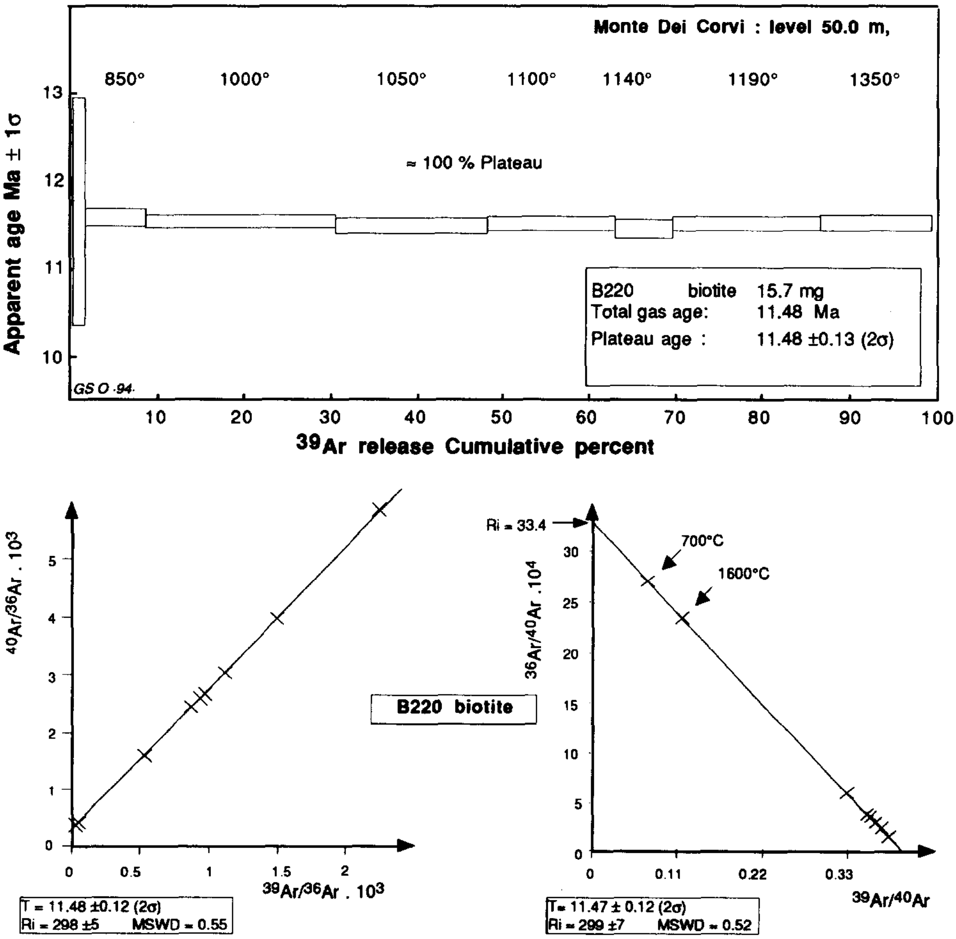

Fig. B-1. Age spectrum of biotite sample B220 from the Monte dei Corvi section and isochron representation of the results. Ages are similar using various calculation procedures; the $^{40}Ar/^{36}Ar$ initial ratio is consistent with an atmospheric composition; the MSWD's of isochrons are small: excellent colinearity of the points.

study, another study was undertaken in Berkeley, California, U.S.A., on the same (Ancona) layer and on an older one located at meter level 101.3 in the same section (Respighi layer; Deino and Montanari, 1992).

Preliminary planktonic foraminiferal biostratigraphy of the upper Serravallian–lower Tortonian interval has been presented by Coccioni et al. (1992) who locate the FO of *G. nepenthes* at level 148.5 m; the LO of *G. druryi* (suspected ancestor of *G. nepenthes*) at meter level 149.9; the FO of *Neogloboquadrina acostaensis* at meter level 157.6. The biostratigraphic control of the Monte dei Corvi section indicates that the Ancona layer is located slightly above the base of the N14 planktonic foraminiferal biozone and that the N14 + N15 biozones are represented by ~9 m of sediments.

The observed FO (and last occurrence: LO) of Cenozoic foraminiferal taxa are commonly used for correlation and chronologic interpretation. The FO and LO observed in the sections are commonly interpreted (and equated to) as evolutionary "first" appearance (or "last" appearance). The latter are theoretical markers which cannot be proved to be such anywhere. According to Bolli and Saunders (1985) *G. nepenthes* Todd 1957, is a "distinctive, easily recognizable form" and is used as a zonal marker, among others, by Blow (1969), Cita (1978), and Kennett and Srinisivan (1983); the first occurrence defines the base of biozone N.14 but it is erratic in its appearance. Bolli and Saunders also note the "record of transitional forms between the species and such forms as *G. druryi* Akers and *G. woodi* Jenkins at the beginning of its range" which is in contrast to the above quoted "distinctive form". According to Cita (1978), "it is worth mentioning how difficult it is to define the lower boundary of Zone N14 because of the inherent difficulties in pinpointing the first evolutionary occurrence of *G. nepenthes* in the slow and gradual evolution from *G. druryi*". Kennett and Srinisivan (1983) indicate that "*G. nepenthes* differs from its ancestor *G. druryi* primarily in its protruding thumb-shaped final chamber"... but also that the species "exhibits a wide range of variation in size and shape of the final chamber".

On the basis of those comments, the FO of *G. nepenthes* would seem to be a problematic key marker. But it involves a lineage and the successive presence of the ancestor and later form is a good criterion for chronologic significance. In the Monte dei Corvi section, transitional forms between *G. druryi* and *G. nepenthes* are present. In agreement with most authors, the FO of *G. nepenthes* has been placed at the level of appearance of forms that are completely similar to the holotype; it is assumed that this is a useful biostratigraphic signal (valid chronologic "event") in a lineage which can be traced in the series. The Japanese sections show a similar trend with *G. druryi* replaced by *G. nepenthes* with a short rock interval where the two forms are found together.

The Ancona layer is one of the many volcaniclastic layers present in the Priabonian (Late Eocene) to Messinian (Late Miocene) sedimentary succession from the Apennines (Montanari et al., 1991; Odin et al., 1991). This biotite-rich layer is not purely volcanic in origin being a mixture of oceanic (limestone with glaucony, abundant foraminiferal tests, fish teeth and scales), detrital (composite clay with 14-, 10- and 7-Å peaks on XRD patterns) and volcanic material (idiomorphic biotite, ~1‰ of the whole rock, and feldspar).

Table B-1
Summarized analytical data on biotite B220 from the Monte dei Corvi section

HT (°C)	T (Ma)	$\pm 2\sigma$	$\%^{39}Ar$	% rad	K/Ca
T220 biotite (sample weight 15.67 mg):					
700	11.6	2.5	1.5	18	27
870	11.55	0.18	7.0	88	71
970	11.51	0.14	22.0	95	145
1,050	11.46	0.15	17.9	92	110
1,100	11.48	0.15	15.0	90	30
1,140	11.41	0.21	6.9	88	15
1,190	11.48	0.15	17.0	88	17
1,350	11.48	0.16	1.25	81	26
1,600	10	11	0.1	25	4

Results of the $^{40}Ar/^{39}Ar$ increment heating measurements (irradiation ulrd7, same conditions as for Japanese samples). Data by G.S.O. in Lausanne. HT = heating temperature; T (apparent age) for total gas = 11.48 ± 0.13 Ma, for plateau (700–1350°C) = 11.48 ± 0.13 Ma (99.9%).

The biotite was magnetically separated, cleaned by gentle crushing, and then with 10% acetic acid for 5 min in an ultrasonic bath. XRD analysis did not show any trace of alteration of the mineral. The biotite from this level was investigated by Montanari (1988) for geochemical homogeneity. A MgO/FeO ratio at 1.03 ± 0.11 was found for 12 flakes and the material concluded to be compositionally homogeneous, unaltered, and suitable for dating purpose.

Table B-2
Incremental heating analytical measurements for biotite B220, Ancona Layer from the Monte dei Corvi section (Central Apennines)

HT (°C)	$^{40}Ar^{\star} \pm 2\sigma$	$^{39}Ar \pm 2\sigma$	$^{38}Ar \pm 2\sigma$	$^{37}Ar \pm 2\sigma$	$^{36}Ar \pm 2\sigma$	$40/39 \pm 2\sigma$
B220 biotite (J=0.002584):						
700	182.4±0.4	13.37±0.18	0.770±0.010	0.245±0.02	0.503±0.006	2.5±0.3
870	180.6±0.4	63.79±0.24	3.287±0.008	0.44±0.02	0.073±0.002	2.486±0.02
970	522.3±1.0	199.8±0.4	10.12±0.03	0.675±0.011	0.0892±0.0014	2.476±0.01
1,050	432.5±0.9	162.0±0.3	8.17±0.03	0.727±0.013	0.109±0.003	2.466±0.01
1,100	374.0±0.8	136.45±0.27	7.13±0.03	2.20±0.02	0.123±0.003	2.471±0.01
1,140	174.8±0.4	62.9±0.2	3.439±0.014	2.132±0.017	0.068±0.003	2.456±0.02
1,190	431.1±0.9	154.7±0.3	8.40±0.02	4.52±0.03	0.164±0.004	2.470±0.01
1,350	343.8±0.7	113.3±0.2	6.033±0.019	2.175±0.019	0.214±0.003	2.471±0.01
1,600	5.2±0.4	0.62±0.04	0.036±0.004	0.075±0.009	0.013±0.002	2.1±1.2

HT = heating temperature; gas in 10^{-15} mol; analytical errors are 1 standard deviation ($\pm 1\sigma$); 40/39 = ^{40}Ar radiogenic/^{39}Ar(K). Isochron calculation for steps from 700° to 1350°C: $^{40}Ar/^{36}Ar$ vs. $^{39}Ar/^{36}Ar$ plot = 11.48±0.12 Ma (R_i = 298.2±5.0; MSWD = 0.55), $^{36}Ar/^{40}Ar$ vs. $^{39}Ar/^{40}Ar$ plot = 11.47±0.12 Ma (R_i = 299.0±6.6; MSWD = 0.52).

The analytical data obtained together with other samples from Japan are summarized in Table B-1 and details are given in Table B-2. The age spectrum is flat with no step deviating within error. The corresponding plateau age at 11.48±0.13 Ma is similar to the isochron ages (Fig. B-1). The calculated age reflects the time of eruption of the biotite and comparison with Japanese samples can be made using the restricted error bar given.

The sedimentary thickness combined with the preliminary biostratigraphic and geochronological control (Deino and Montanari, 1992) suggest that, in the Monte dei Corvi section, deposition occurred at 30m Ma^{-1} on average. The FO of *G. nepenthes* is located ~1.5 m below the dated level and, therefore, the FO of *G. nepenthes* is only slightly older than 11.5 Ma in this section.

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