SENIOR THESIS

$^{40}Ar/^{39}Ar$ AGE DETERMINATION OF THE SUZHOU GRANITE

by
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ABSTRACT

Two $^{40}Ar/^{39}Ar$ isotopic dating methods, the total fusion and the incremental heating techniques, were employed to determine the age of the Suzhou granitic intrusion in eastern China. Agreement between the results of the two experiments is excellent; both yield an age of about 121.7 Ma. A generally uncomplicated and concordant incremental heating spectrum is seen, with a plateau age of 122.2 Ma. This general concordance suggests a valid age, most likely reflecting the time that the biotite cooled to about 300°C. Since the intrusion is not very large and is shallowly emplaced, it probably cooled quickly; thus, the age most likely represents the actual intrusive event. This Cretaceous age is supported by previous ages determined by Chinese geologists.

Introduction

The purpose of this thesis is to determine the age of the Suzhou granitic intrusion in eastern China, using the $^{40}Ar/^{39}Ar$ variation of the K-Ar dating method.

The Suzhou Granitic Intrusion

The Suzhou granite is an elliptical stock about 10 km west of the city of Suzhou, in eastern China (see Fig. 1). The intrusion is in the Yangtze-Qiantang Paleozoic Depression Zone, west of the subduction zone of the Western Pacific Plate against the China Continental Plate, and borders on the southeastern flank of the huge Changjiang tension fault (Zhang Xinglong et al., 1987). The stock has an area of 40 km^2, only 11 km^2 of which is exposed due to Quaternary cover (Liu Yimao et al., 1987). The intrusion penetrated the axis of a north-northeastern-trending syncline composed of Silurian and Devonian sandstones and Permian shale. Faulting is prevalent in the area, which is common in zones of magmatic intrusion. Lamprophyres, quartz porphyry, rhyolitic porphyry, dacite porphyry, and quartz veining are also observed in the area (Liu Yimao et al., 1987).

Past studies have classified the Suzhou granitic intrusion into three stages, based on composition. The Ouyang Xinguei-Wang Xonggang study of 1985 is most representative of the general consensus. The three stages are:

1. Porphyritic-like hornblende biotite granite.
2. Porphyritic-like biotite and coarse-grained biotite granite.
3. Fine-grained biotite granite and albitized biotite granite.

FIGURE 1a: LOCATION OF SUZHOU GRANITIC INTRUSION IN EASTERN CHINA

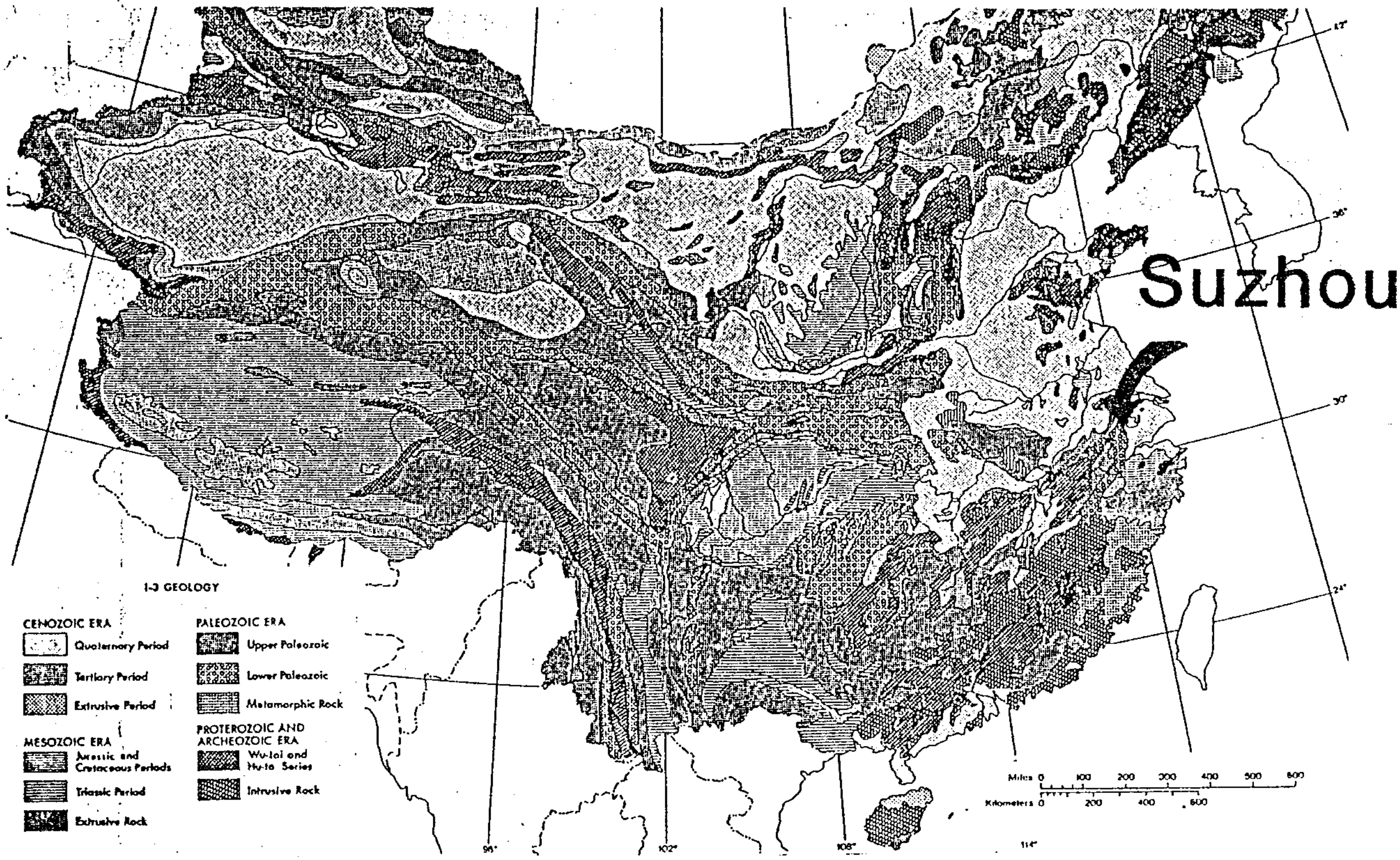
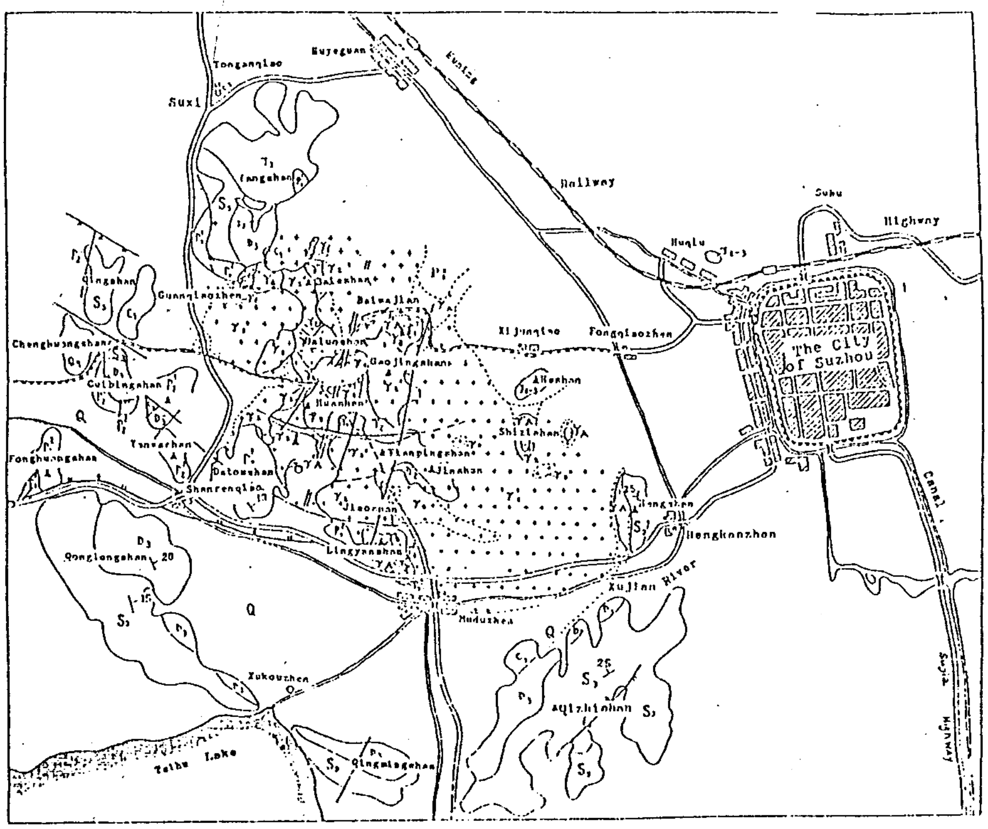


FIGURE 1b: LOCATION OF SUZHOU GRANITIC INTRUSION IN RELATION TO THE CITY OF SUZHOU



The $^{40}Ar/^{39}Ar$ Dating Method - Principles and Procedures

The K-Ar dating method is described in detail by Dalrymple and Lanphere (1969) and also by many standard treatises including Faure (1986).

The following are basic assumptions and must hold true for any K-Ar date to be considered valid:

1. The system was closed.

   A. No ^{40}Ar has escaped since formation.
   B. No ^{40}Ar has entered since formation.
   C. The system was closed to K gain or loss since formation.

2. No ^{40}Ar was initially present in the sample.

3. A proper correction is made to account for atmospheric ^{40}Ar.

4. Isotopic K composition is normal and has not changed except by decay of ^{40}K.

5. The decay constants are accurate.

The $^{40}Ar/^{39}Ar$ method of dating can alleviate some of the problems inherent in the conventional K-Ar method. The problems of different subsamples and difficulties of determining absolute K and Ar concentrations are avoided, since K and Ar are measured on the same subsample, and since only the isotopic ratio of Ar is needed. An added advantage to $^{40}Ar/^{39}Ar$ dating is that only a small amount of sample is required. This is attractive for use with rocks containing a small concentration of suitable dating mineral, or with small amounts of a very valuble sample, such as lunar rocks.

The $^{40}Ar/^{39}Ar$ dating method depends upon the production of ^{39}Ar by the irradiation of K-rich samples with fast neutrons in a nuclear reactor. The sample is sealed in a glass tube with flux monitors on either side (see below), placed in the reactor and irradiated for several days to allow for the formation of ^{39}Ar. The nuclear reaction is:

$$^{39}_{19}K\ (n,p)\ ^{39}_{18}Ar \qquad \text{(Eq.1)}$$

^{39}Ar decays to ^{39}K by beta emission, with a half-life of 269 years. Due to this slow decay rate, ^{39}Ar can be considered stable during the analysis period. The ratio of $^{40}Ar/^{39}Ar$ can then be measured using a mass spectrometer, and the age of the rock can be calculated.

When a K-rich sample is irradiated, several reactions produce isotopes of argon; only the n,p reaction (Eq.1) is used here. The starting point is Mitchell's (1968) formula which gives the number of ^{39}Ar atoms formed during neutron irradiation:

$$^{39}Ar = {}^{39}K \, \Delta T \int \phi(\epsilon) \, \sigma(\epsilon) \, d\epsilon \qquad \text{(Eq.2)}$$

where: ^{39}K = number of atoms in irradiated sample.

ΔT = period of irradiation.

$\phi(\epsilon)$ = neutron flux density at energy ϵ

$\sigma(\epsilon)$ = capture cross-section of ^{39}K for neutrons with energy ϵ.

The amount of radiogenic ^{40}Ar ($^{40}Ar^*$) atoms from decay of ^{40}K is:

$$^{40}Ar^* = \frac{\lambda e}{\lambda} \, {}^{40}K \, (e^{\lambda t} - 1) \qquad \text{(Eq.3)}$$

where: λe = decay constant of ^{40}K for electron capture.

λ = total decay constant of ^{40}K.

The ratio of $^{40}Ar/^{39}Ar$ after initial irradiation is then:

$$\frac{^{40}Ar^*}{^{39}Ar} = \frac{(e^{\lambda t} - 1)}{J} \qquad \text{(Eq.4)}$$

where: $$J = \frac{\lambda}{\lambda e} \frac{^{39}K}{^{40}K} \Delta T \int \phi(\epsilon) \, \sigma(\epsilon) \, d\epsilon \qquad \text{(Eq.5)}$$

This is where use of flux monitors becomes crucial. Since the cross-sections of ^{39}K for capturing neutrons of various energies and the energy spectrum of incident neutrons are not well known, the neutron flux density and capture cross-sections are difficult to ascertain. The use of flux monitors solves this dilemma. Samples of known age (flux monitors) are irradiated next to the unknown sample in the sealed glass tube, and by plotting their J-values versus their position in the sample vial, J for the unknown sample can be interpolated. The age of the unknown sample can then be found by:

$$t = \frac{1}{\lambda} \ln \left(\frac{^{40}Ar^*}{^{39}Ar} J + 1 \right) \qquad \text{(Eq.6)}$$

Both the K-Ar and $^{40}Ar/^{39}Ar$ require a correction for atmospheric ^{40}Ar and also other Ar isotopes produced during irradiation.

The incremental heating technique is a variation of the $^{40}Ar/^{39}Ar$ dating method. A series of ages can be calculated for one sample by releasing argon from it by increasing the temperature in successive steps. If the sample has neither lost nor gained potassium and argon since it cooled below the temperature at which argon is quantitatively retained (known as the blocking temperature), the date found for each heating increment will be constant. If radiogenic argon was lost after cooling, however, a range of dates will emerge from the varying $^{40}Ar/^{39}Ar$ ratios. Since the loss of radiogenic argon is often attributed to reheating by a metamorphic event, this method can provide much insight in to the regional history of the sample area.

ANALYTICAL METHODS

Sample Preparation:

The samples studied were collected by Professors K. A. Foland and Chen Jiang-Feng in China in December, 1987. A total of six Suzhou granite samples were available. These samples were cut into slabs and thin sections were made. From subsequent hand sample and thin section examinations (see Table 1), the most promising sample (Sample #5) for K-Ar dating was chosen. This decision was based upon high biotite content (>50% for Sample #5) and upon freshness.

Part of the sample was then crushed into fragments using a jaw-crusher, and ground further using a disc grinder. A system of graded sieves was then employed to segregate the sample by grain size.

Each size fraction was examined under magnification. The goal was to separate the biotite from the quartz and feldspar in the crushed granite sample, since biotite is a K-rich mineral and thus very useful for K-Ar analysis. In the +40 mesh fraction, the grains of biotite were still mostly joined with the feldspar and quartz. In the -40/+60 mesh fraction, most of the biotite flakes had separated, yet were still intact. In the -60/+80 mesh fraction, the biotite flakes were so minute that separation would have been very difficult. Thus, fraction 2, the -40/+60 size fraction, was determined to be the best grade for separation of biotite for analysis.

The biotite was separated out using the paper-shake method. Using this procedure, the sample was placed at the top of a large piece of stiff card paper, which was then tilted to about a 45° angle and gently shaken. This method relies upon the fact that the biotite grains are relatively large and

TABLE 1:

THIN SECTION ANALYSES FOR BIOTITE CONTENT AND GRAINSIZE OF SUZHOU GRANITE SAMPLES.

SAMPLE NO.	ROCK TYPE	ESTIMATED BIOTITE CONTENT	RELATIVE GRAIN SIZE
87 SZ-1	AMPHIBOLE BIOTITE GRANITE	10%	VERY SMALL GRAINS 0.1-0.5 MM
87 SZ-2	BIOTITE GRANITE	15%	MEDIUM-SIZED GRAINS 0.5-1.5 MM
87 SZ-3	BIOTITE GRANITE	10%	VERY SMALL GRAINS 0.1-0.5 MM
87 SZ-4	BIOTIZATION GRANITE	5%	MEDIUM-SIZED GRAINS 0.5-1.5 MM
87 SZ-5	BIOTITITE	50%	LARGE GRAINS 0.5-3.0 MM
87 SZ-6	BIOTIZATION GRANITE	15%	MEDIUM SIZED GRAINS 0.5-1.5 MM CONCENTRATED IN A VEIN

much flatter than the more spherical and blocky quartz and feldspar grains. As the paper is shaken, the quartz and feldspar grains separate out and roll down the paper, leaving only biotite flakes (with their lower centers of gravity) remaining on the paper. This leftover biotite is shaken off into a container, and the process is repeated many times. In this manner, enough biotite can eventually be accumulated for analysis; due to the relatively high biotite content of the sample, about 2 g of biotite was collected in the space of a few hours.

The sample was then thoroughly washed in acetone, oven-dried, divided into two subsamples, and wrapped separately in Sn-foil in preparation for neutron irradiation.

$^{40}Ar/^{39}Ar$ Analyses

The two subsamples were irradiated as part of OSU Package No. 36 at the Ford Nuclear Reactor at the University of Michigan in April, 1988. The samples were irradiated for 100 hours, allowed to "cool" in the reactor pool for two weeks, and were returned back at OSU in May, 1988. The smaller sample was for regular $^{40}Ar/^{39}Ar$ gas dating by total fusion, the larger one for use with the incremental heating method. The smaller sample was fused using RF induction heating, and the larger sample was incrementally heated using a double vacuum system. The gases released during heating were treated in a number of stages to purify the inert Ar. The gas was cleaned using liquid nitrogen cold-finger traps to remove water vapor, SAES getters to remove active gases, and turbo pumps to remove hydrogen. Once purified, each Ar gas sample was run in three subdivisions, using a Nuclide Model 4.5-60-RSS mass spectrometer automated with an HP9825A computer. The flux monitors used for analysis were OSU intra-lab monitor Mon-4, a biotite with an age 121.66 Ma. More details on the OSU procedures may be found in Foland (1983).

RESULTS AND DISCUSSION

The agreement between the two $^{40}Ar/^{39}Ar$ dating experiments, total fusion and incremental heating, is excellent (see Table 2). The total fusion sample yielded an age of 121.8 Ma, compared to the weighted sum of all gas fractions from the incremental heating experiment, 121.9 Ma. This age is equivalent to a conventional K-Ar age.

Figure 2 is a plot of the incremental heating data, and shows a generally concordant spectrum; the various gas fractions yield nearly the same apparent ages. Minor variations existing are only slightly larger than their analytical uncertainties. The plateau age (see Faure, 1986 for discussion) of 122.2 Ma is found for 83.5% of the released ^{39}Ar. This internal agreement of the incremental heating experiment reflects a uniform K and ^{40}Ar distribution in the biotite and implies that the date is valid.

The age of the biotite is a cooling age, as is the case with most minerals, and represents the point at which the mineral cooled such that ^{40}Ar loss by diffusion was negligible. This closure temperature is about 300°C for biotite. The age of 122.2 Ma thus represents the time of cooling of the Suzhou granite below 300°C. Since the intrusion is not extremely extensive, and intruded to a shallow depth in the crust, it probably cooled relatively rapidly. This means that the age found in this study should agree closely with the actual time of the magmatic intrusion.

This age of 122.2 Ma for the Suzhou granite coincides with a Cretaceous age determined by Chinese geologists for the area (Liu Yimao et al., 1987). Many small, shallow intrusions occurred at about this time throughout eastern China (Chen, J-F, personal communication).

TABLE 2: AGE DETERMINATION DATA FOR TOTAL FUSION AND INCREMENTAL HEATING METHODS

a: TOTAL FUSION METHOD

OUTPUT DATA

TEMP (C)	40/39 Meas.	36/39 Meas.	37/39 Corr.	36/39 Meas.	F VALUE	% 39Ar TOTAL	% 40Ar TOTAL	% 36Ar (Ca,Cl)	K/Ca	K/Cl	AGE (Ma.)	UNCERTAINTY (Ma.) J CONST.	J+/- 1.5 %
FUSE	1.0028E+01	2.4104E-03	9.3750E-03	1.2424E-01	9.3420	100.00	92.61	0.26	5.57E+01	4.20E+01	121.840	0.259	1.786
SUM	1.0088E+01	2.4104E-03	9.3750E-03	1.2424E-01	9.3420	100.0	92.67	1.12	5.57E+01	4.20E+01	121.840		

AGE 121.8 MA

b: INCREMENTAL HEATING METHOD

OUTPUT DATA

TEMP (C)	40/39 Meas.	36/39 Meas.	37/39 Corr.	38/39 Meas.	F VALUE	% 39Ar TOTAL	% 40Ar TOTAL	% 36Ar (Ca,Cl)	K/Ca	K/Cl	AGE (Ma.)	UNCERTAINTY (Ma.) J CONST.	J+/- 1.5 %
500	5.0070E+01	1.4087E-01	6.0035E-02	1.1370E-01	8.4267	1.02	16.83	0.01	8.70E+00	4.59E+01	111.147	24.769	24.821
450	1.0198E+01	2.4641E-03	1.0830E-02	1.2092E-01	9.4368	14.02	92.53	0.28	4.83E+01	4.32E+01	124.021	0.318	1.826
750	9.5213E+00	6.6251E-04	4.5886E-03	1.2233E-01	9.2919	20.80	97.59	0.88	1.14E+02	4.27E+01	122.180	0.263	1.791
825	9.6475E+00	1.1352E-03	8.1170E-03	1.2112E-01	9.2786	13.21	96.18	0.57	6.44E+01	4.31E+01	122.011	0.277	1.791
875	9.5178E+00	9.2058E-04	8.9738E-03	1.2353E-01	9.2125	23.24	96.79	0.73	5.82E+01	4.23E+01	121.169	0.249	1.775
925	9.4179E+00	4.5994E-04	8.7228E-03	1.2474E-01	9.2487	21.19	98.20	1.47	5.99E+01	4.18E+01	121.630	0.245	1.781
975	9.4588E+00	4.6585E-04	1.1188E-02	1.2353E-01	9.2879	5.08	98.19	1.55	4.67E+01	4.23E+01	122.129	0.322	1.800
1000	9.5577E+00	5.4965E-04	3.5291E-02	1.1751E-01	9.3636	0.79	97.97	2.14	1.49E+01	4.44E+01	123.091	0.250	1.902
FUSE	9.5650E+00	1.2752E-03	2.2916E-02	1.0467E-01	9.1556	0.65	95.72	0.69	2.28E+01	4.99E+01	120.446	0.415	1.796
SUM	1.0022E+01	2.4185E-03	9.0878E-03	1.2258E-01	9.2735	100.0	92.54	0.28	5.75E+01	4.26E+01	121.946		

AGE 121.9 MA

c: PLATEAU AGE

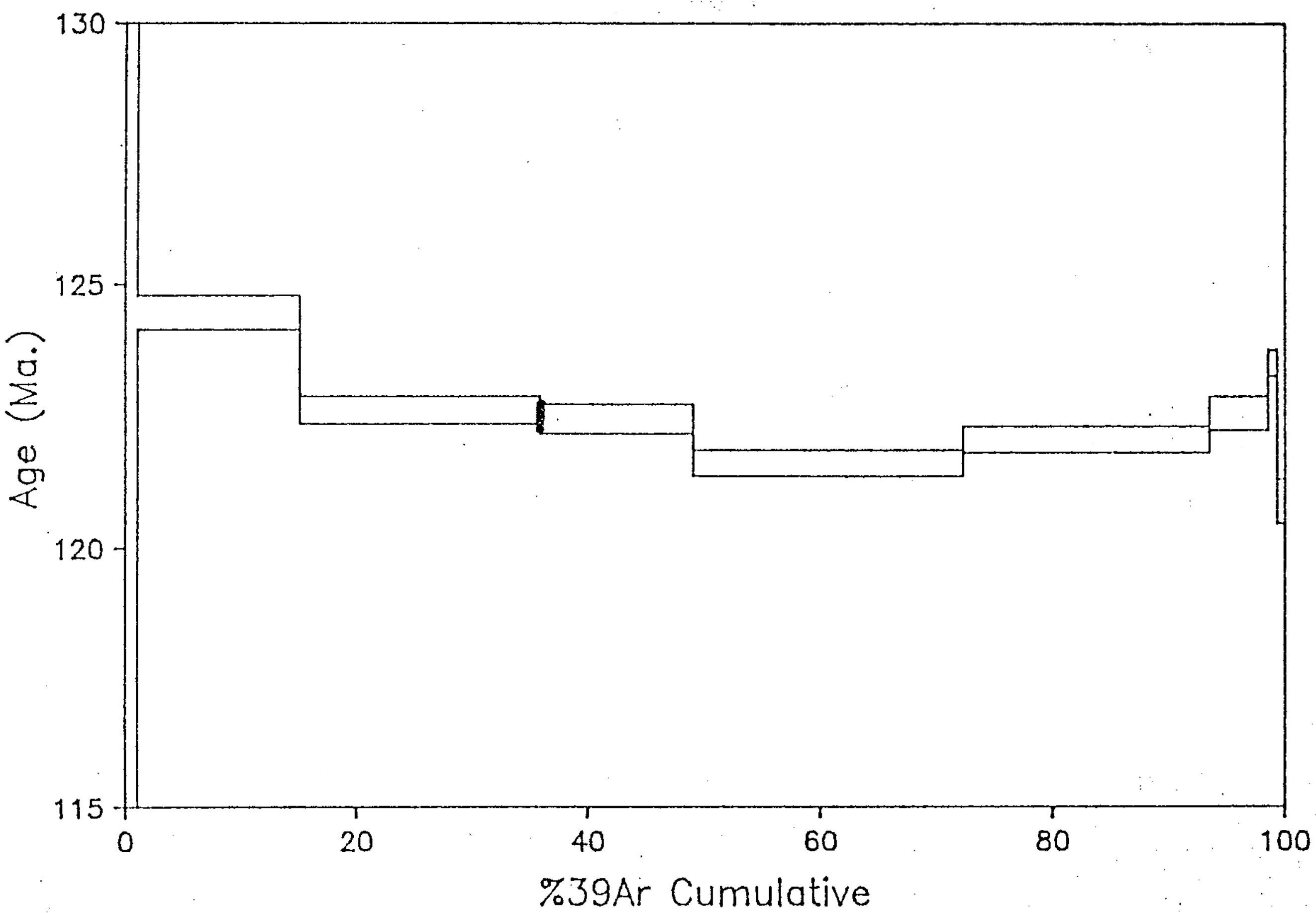
J= .007541

Fraction	F value	% 39Ar	Age (m.y.)	delta (m.y.)
3	9.291899	20.8	122.1798	-.4501953
4	9.2786	13.21	122.0107	-.28101135
5	9.212499	23.24	121.17	.5596008
6	9.2487	21.19	121.6304	9.927368E-02
7	9.2879	5.08	122.1289	-.3992157

Total amount of 39Ar = 83.52001 %

AGE 122.2 MA

FIGURE 2: $^{40}AR/^{39}AR$ INCREMENTAL RELEASE SPECTRUM FOR A BIOTITE FROM A GRANITE FROM SUZHOU, CHINA



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GRC
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