

Formal Considerations on Argon-Argon Diagrams in $^{40}\text{Ar}/^{39}\text{Ar}$ Geochronometry

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A formal analysis of argon isochrons in terms of diffusion and Arrhenius' laws shows that the $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{39}\text{Ar}/^{40}\text{Ar}$ diagram exhibits a linear graph as a true isochron and allows regressions and extrapolations, only when activation energies of diffusion for radiogenic argon-40 and nucleogenic argon-39 are equal and that for atmospheric (*contaminating*) argon is lower. In cases of distorted kinetic parameters of argon isotopes, when activation energies of diffusion for radiogenic argon 40 and nucleogenic argon 39 are different, excess argon with lower activation energy, curvilinear arrays and spreading of points occur, which may produce misleading interpretations of ages and $^{36}\text{Ar}/^{40}\text{Ar}$ value of *contaminating* argon.

1 Introduction

The need of a diagram with the $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{39}\text{Ar}/^{40}\text{Ar}$ coordinates had arisen with the development of a step heating procedure of argon releasing in ^{40}Ar - ^{39}Ar technique. MERRIHUE & TURNER (1966) argued that, beside radiogenic ^{40}Ar and nucleogenic ^{39}Ar , a sample could contain *contaminating* argon, common for all fractions released during step heating. In the case of terrestrial samples, one had only to subtract from the total argon 40 released a part 295.5 times the measured atmospheric argon 36. In the case of meteoritic or planetary or incompletely degassed terrestrial samples, the problem was more complicated because the *contaminating* argon could differ by isotopic composition from the terrestrial atmospheric argon. The *contaminating* argon could be found by extrapolation to zero of an experimental point pattern due to the obviously linear relation

$$(^{40}\text{Ar}/^{36}\text{Ar})_{\text{tot}} = (^{40}\text{Ar}/^{36}\text{Ar})_c + (^{40}\text{Ar}/^{39}\text{Ar})_{\text{rad}} \times (^{39}\text{Ar}/^{36}\text{Ar}) \quad (1)$$

(subscripts are *tot* = total, *c* = contaminating, *rad* = radiogenic). For the perfection of the argon-argon diagram, it was suggested to use inverse coordinates: $x = ^{39}\text{Ar}/^{40}\text{Ar}$; $y = ^{36}\text{Ar}/^{40}\text{Ar}$. This was necessary to avoid infinities in samples extremely enriched with radiogenic argon when $^{36}\text{Ar} \rightarrow 0$ (TURNER, 1971; RODDICK & REX 1980).

The argon-argon diagram became a standard tool in routine $^{40}\text{Ar}/^{39}\text{Ar}$ age determinations. Linear regressions from scattered points were often used for determinations of *contaminating* argon (DICKIN 1995; MCDOUGALL & HARRISON 1999 and references therein). If temperature steps did not yield a plateau, a value at intercept of a regression line with abscissa was usually accepted as the most reliable approximation to the true age. There existed some verbal descriptions of the correlation diagram, but no strict mathematical deductions of its features combined with stepwise release of argon had been published yet.

In contrast to expression (1), the linearity of

$$(^{36}\text{Ar}_c / ^{40}\text{Ar}_{\text{tot}}) = f(^{39}\text{Ar} / ^{40}\text{Ar}_{\text{tot}}) \quad (2)$$

is not obvious. It may exist in several cases and in others not. Are linear regressions and extrapolations justified if (2) is not a straight line, but a curve? Could it not yield artefacts and mislead to false deductions? These questions have impelled us to perform the present theoretic investigation.

A formal analysis of Ar-Ar isochrons combined with stepwise heating is performed. It is based on standard solutions of differential equations in partial derivatives (TIKHONOV & SAMARSKY 1953; WEBSTER & SZEGÖ 1930; FRANK & MISES 1937; MADELUNG 1957), taught at mathematical, physical and even chemical faculties.

2 Deduction of an Algebraic Expression for a $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{36}\text{Ar}/^{40}\text{Ar}$ Plot

Presuming contamination of a sample by atmospheric argon (Ar_{atm}), we aim to obtain an algebraic relation $y = f(x)$. Let be

$$y = \frac{^{36}Ar_{atm}}{^{40}Ar_{tot}}; \quad x = \frac{^{39}Ar}{^{40}Ar_{tot}}; \quad ^{40}Ar_{total} = ^{40}Ar_{atm} + ^{40}Ar_{rad} \quad (1abc)$$

Then we are compelled to assume, that

$$k = \frac{^{36}Ar_{atm}}{^{40}Ar_{atm}}; \quad l = \frac{^{39}Ar}{^{40}Ar_{rad}} \quad (2ab)$$

The expression (2a) means that the isotopic composition of *contaminating* argon should be constant for all steps of heating. According to the expression (2b), the nucleogenic argon 39 - radiogenic argon 40 ratio (or the ages of all thermal fractions) should be constant. Indeed, the latter rather severe condition presumes a straight line, parallel to the abscissa in the age-to-fraction-of-released-argon diagram. Thus, in the xy coordinates, this line means an isochron. The line becomes curvilinear, if conditions 2ab are not fulfilled.

From expressions (1ab) and (2ab) it follows that

$$y = \frac{k}{1 + \frac{^{40}Ar_{rad}}{^{40}Ar_{atm}}}; \quad x = \frac{l}{1 + \frac{^{40}Ar_{atm}}{^{40}Ar_{rad}}} \quad (3ab)$$

Express $^{40}Ar_{rad}$ from (3b) and substitute into (1a)

$$y = \frac{k}{1 + \frac{x}{l-x}} \text{ or finally } \frac{x}{l} + \frac{y}{k} = 1 \quad (4)$$

Equation (4) means a straight line with a negative slope $-k/l$ intercepting at the point k with the ordinate and at the point l with the abscissa. Both points are to be determined as a goal of the whole procedure.

3 Kinetic Relations Describing Step Heating

In any mineral, radiogenic argon obeys the laws of diffusion and that of Arrhenius. The former shows the space-time dependence of argon concentration (c). Its solution for the mean concentration in the one-dimensional case is

$$c(t) = \frac{8}{\pi^2} \sum_{\nu=0}^{\infty} \frac{1}{(2\nu+1)^2} e^{-(2\nu+1)^2 \pi^2 Fo} \quad (5)$$

Concentration is a function of a dimensionless parameter, the Fourier's number

$$Fo = \frac{Dt}{h^2}$$

(D = the coefficient of diffusion, t = time, h = the size of the grain, from which diffusion occurs) the latter controls the temperature dependence of argon release

$$\frac{D}{h^2} = \frac{D_0}{h^2} e^{-\frac{E}{RT}} \quad (6)$$

(D_0 = the frequency factor, meaning D at $T \rightarrow \infty$; E = the activation energy of diffusion; R = the gas constant; T = temperature in K).

For the case of step heating, we can apply a stepwise-constant-diffusion relation deduced by (AMIRKHANOV et al. 1960; BRANDT et al. 1996)

$$\Delta c_i = \frac{8}{\pi^2} \sum_{\nu=0}^{\infty} \frac{1}{(2\nu+1)^2} e^{-\sum_{i=1}^n \Delta F_{0i}} (1 - e^{-(2\nu+1)^2 \pi^2 \Delta F_{0n}}) \quad (7)$$

(Δc_i are fractions of ^{36}Ar , $^{40}Ar_{atm}$, $^{40}Ar_{rad}$ or ^{39}Ar released at the i -th step of a sample heating; Fo = a dimensionless Fourier's number,

$$Fo = \frac{Dt}{h^2} \quad (8).$$

This formula is easily programmed and tabulated. The whole procedure was described more explicit elsewhere (BRANDT et al. 2003).

4 Thermal Separation of Argon Components

We are dealing at least with three components of argon present in an irradiated sample: radiogenic, nucleogenic, and *contaminating* argon. Their behaviour during step heating is controlled by relations (5) and (6). Each of the argon components might have its own characteristic frequency factor D_0 and its activation energy of diffusion E . Differences in these parameters allow to separate them by thermal procedures.

For instance, a problem on the separation of radiogenic and contaminating argon of the atmospheric composition (further referred to as atmospheric argon) have been considered quantitatively by RASSKAZOV et al. (2000). To extract

only a k -th atmospheric fraction and retain a l -th radiogenic fraction of argon, the sample is to be preheated at a temperature

$$T = \frac{E - e}{R} \cdot \frac{1}{\ln \left(\frac{fo}{Fo} \cdot \frac{D_0 / H^2}{d_0 / h^2} \right)} \text{ during a}$$

$$\text{time } t = \frac{(D_0 / H^2 Fo)^{\frac{e}{E-e}}}{(d_0 / h^2 fo)^{\frac{E}{E-e}}} \quad (9ab)$$

(capital letters D, H, Fo refer to radiogenic argon and small letters d, e, fo - to atmospheric argon;

$$Fo = \frac{\pi k^2}{16}; \quad fo = -\frac{1}{\pi^2} \ln \frac{\pi^2 l}{8}.$$

We see that a *conditio sine qua non* for separating these two components is a difference between E and e . If $E-e=0$, T becomes zero and t becomes indefinite. Therefore, if two or more argon components of a sample have identical kinetic parameters, they are not separable by any thermal procedure and become indistinguishable being released congruently.

In a case of equal kinetic parameters of argon components, points of i thermal release steps will be represented in the xy coordinates ($x = {}^{39}\text{Ar}/{}^{40}\text{Ar}_{\text{tot}}$; $y = {}^{36}\text{Ar}/{}^{40}\text{Ar}_{\text{tot}}$) by a single point of a

multiplicity of an order i . As fractions of argon components are inseparable, the point has no chronological meaning.

5 A Case of a True Isochron

During step heating, fractional release of an argon component with a lower activation energy occurs at lower temperature steps as compared to an argon component with an elevated activation energy (Fig. 1).

Taking into account expressions (9ab) suggest now that atmospheric argon is bound to the host structure looser (i.e. has a lower activation energy) than both radiogenic and nucleogenic. This is really plausible, as radiogenic and nucleogenic argon are produced within the crystalline structures themselves, whereas atmospheric argon is most likely impregnated into the structure under a huge pressure gradient.

For the sake of definiteness, we may consider a sample with a potassium content 2%, radiogenic ${}^{40}\text{Ar} = 1.0 \times 10^{-3} \text{ nmm}^3/\text{g}$ and an age

$$t = 1.885 \cdot 10^9 \ln \left(J \frac{{}^{40}\text{Ar}_{\text{rad}}}{K} + 1 \right) = 12.5 \text{ Ma}$$

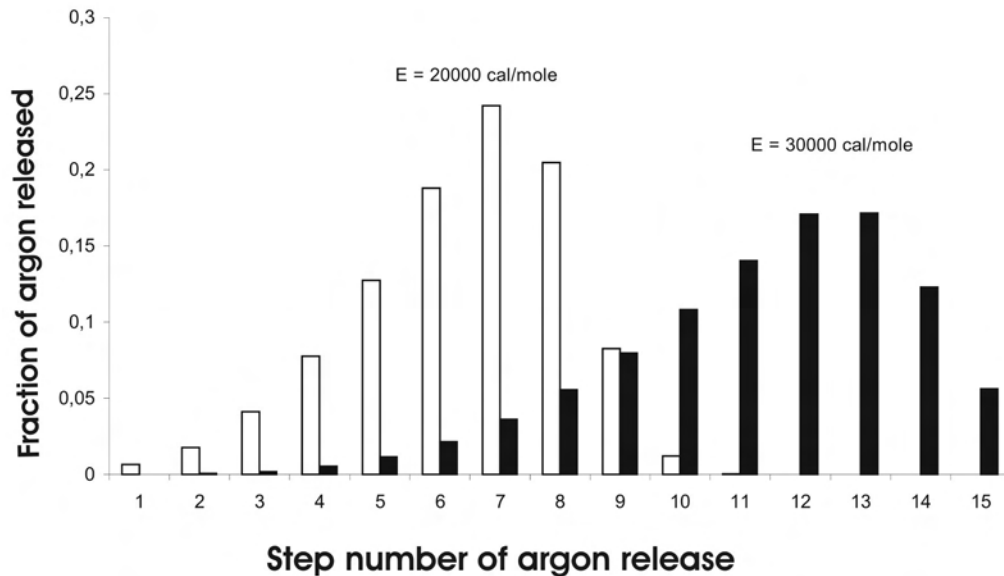


Figure 1: Comparison of argon release spectra at $E = 20000 \text{ cal/mole}$ and $E = 30000 \text{ cal/mole}$ as calculated by means of formula (7).

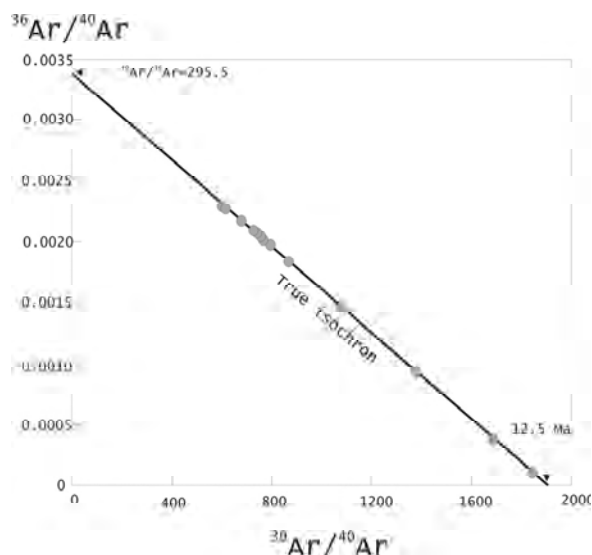


Figure 2: Argon-argon isotope plot for the case, when activation energies of diffusion for radiogenic and nucleogenic argon are equal and that for atmospheric is somewhat lower. An isochron in the form of an ideal straight line is obtained. Intercepts with the coordinate axes provide both the age of the sample and the isotopic composition of contaminating argon of atmospheric origin.

(for these units, the J -factor = 13.3 because argon is expressed in nm^3/g and potassium in %).

For both the radiogenic and nucleogenic argon, assume a frequency factor $D_0/H^2 = 0.5 \text{ s}^{-1}$. $E_{\text{rad}} = E_{39} = 25000 \text{ cal/mole}$. Finally, put the quantity of atmospheric argon remained in the sample at the instant of measurement as equal to the radiogenic argon. We ascribe to the atmospheric argon (composed of ^{40}Ar and a 1/295.5 part of ^{36}Ar) an activation energy (e_{atm}) of 23000 cal/mole . Results of calculations are plotted on Fig. 2.

The age of every fraction is 12.5 Ma. In the age-to-fraction-of-released-argon diagram, the age line is parallel to the abscissa (not shown). A shift of atmospheric argon towards lower activation energies stipulates an isochron with points corresponding to every thermal step ideally fitting the straight line in argon-argon diagram. Intercepts of the isochron with the coordinate axes determine compositions of the atmospheric argon and a single age of 12.5 Ma. And vice versa: if points fit a straight line intercepting with

the coordinate axes at points mentioned, then it may be stated that the activation energies for radiogenic and nucleogenic argon are equal and that of atmospheric argon is somewhat lower.

In practice, most frequently, however, distorted age spectra are met. They are saddle shaped, stepwisely increasing or decreasing. Conditions (2b) for them are not fulfilled and argon-argon diagrams of the distorted patterns exhibit curvilinear arrays. Now, let us consider three examples of 'false isochrons' resulted from varying kinetic parameters of argon components.

6 A Case of a 'False Isochron' due to Unequal Activation Energies for Atmospheric, Nucleogenic, and Radiogenic Argon: $E_{\text{atm}} < E_{39} < E_{\text{rad}}$

It is worth considering this case, as cross sections for nuclear impact for all the lattice elements differ from zero, have final values. Neutron bombardment may loosen the lattice, induce radiation damages and cause a shift of argon release spectra.

Take $E_{\text{rad}} = 25000$; $E_{39} = 24500$ and $e_{\text{atm}} = 23000 \text{ cal/mole}$. Calculations quite similar to the previous case exhibit results plotted on Fig. 3.

As compared to radiogenic argon, a lower activation energy of nucleogenic argon results in its elevated concentrations in fractions released at lower temperature steps. This is why an age-to-fraction-of-released-argon diagram shows a plateau somewhat lower than the reference line of the true age. Condition (2b) is violated in this case: the ^{39}Ar - ^{40}Ar ratio is not constant

In argon-argon coordinates, points are not determining a straight line anymore, but are belonging to a curved array. Intercepts of the curve with the coordinate axes have another meaning than in the case of the true isochron. Attempts to "stratify" the curve by means of extrapolations and least square procedures are likely leading to artefacts. For regression calculations, an experimenter would be tempted to use the seemingly straight array of points shown by filled circles. The fictive regression equation is

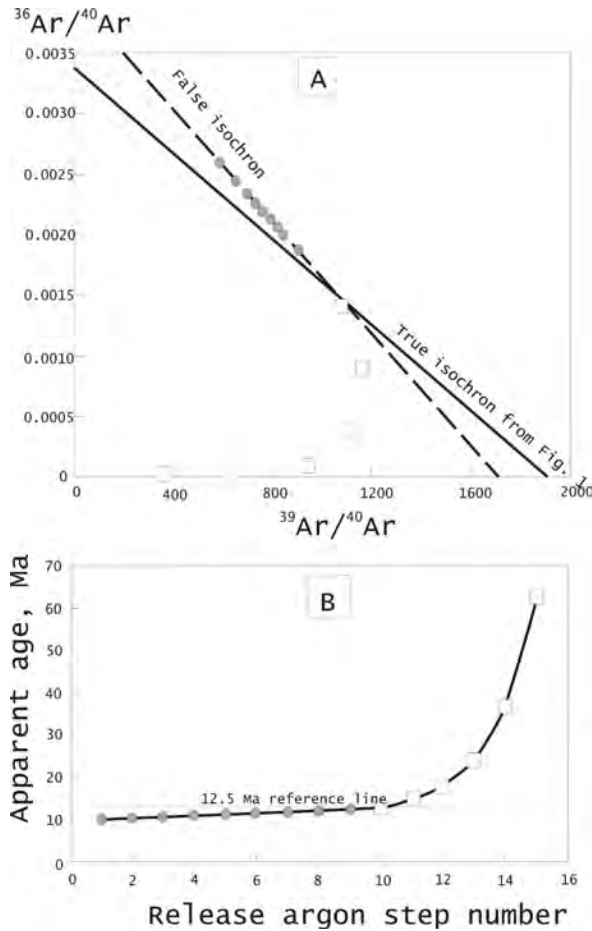


Figure 3: Argon-argon isotope plot (A) and age spectrum (B) for the case, when the activation energy of diffusion for nucleogenic argon ^{39}Ar lies between those of radiogenic and atmospheric. Intercepts with coordinate axes give distorted results. A locus of points is not a straight line. In the array, filled circles mark a portion exhibited a 'false' isochron. Open squares are omitted from the isochron calculations.

$$\frac{^{36}\text{Ar}}{^{40}\text{Ar}} = -3 \cdot 10^{-4} \frac{^{39}\text{Ar}}{^{40}\text{Ar}} + 0.0040$$

This yields an intercept with the $^{36}\text{Ar}/^{40}\text{Ar}$ -axis at 0.0042 instead of 0.003384 - the input value of our calculation and an intercept with the $^{39}\text{Ar}/^{40}\text{Ar}$ -axis at 1700 instead of 1900.

So, in the case of unequal activation energies for atmospheric, nucleogenic, and radiogenic argon ($e_{\text{atm}} < E_{^{39}\text{Ar}} < E_{\text{rad}}$), the experimenter obtains a plateau younger than the true age, a 'false isochron' with an older age and a high $^{36}\text{Ar}/^{40}\text{Ar}$ ratio of *contaminating* argon. And vice versa: this pattern of age spectrum and high $^{36}\text{Ar}/^{40}\text{Ar}$ ratio of *con-*

taminating argon on an argon-argon diagram is likely a symptom of a shift in the activation energies of radiogenic and nucleogenic argon.

7 A Case of a 'False Isochron' due to Radiogenic Argon Losses in the Past

Underestimated ages due to radiogenic argon losses are met very often. According to kinetic theory, argon losses are negligible in young rocks and increase in the older ones. This is why experimenters are compelled to abandon the conventional potassium-argon method for rocks older than Late Cenozoic. When the distribution of argon inside an undisturbed mineral grain is homogeneous, the rate of argon release is higher, than in a case when the grain had lost some of its argon. Consider the equation (7), putting it $f(F_0)$.

$$f(F_0 + \Delta F_0) \neq f(\Delta F_0)$$

(F_0 = a Fourier's number characterizes argon losses, ΔF_0 is acquired during laboratory thermal treatment). This follows from an expansion into a Taylor series.

In a numeric example, suppose that the sample had recently lost one half of its argon. This loss corresponds to an initial Fourier's number $F_{0i} = 0.049$. The calculation is performed quite similar, but to every value is added the Fourier's number 0.049. The results are shown on Fig. 4.

Apparent ages are relatively young. Radiogenic argon depletion results in a combination of increasing and then decreasing steps. On an argon-argon plot, the array has a double curvature. This pattern may be mistakably accepted as a result of usual 'spreading of experimental points' or 'damages in crystalline nuclei'. Some points may be omitted and a part of the array accounted for. The points shown by filled circles yield a regression line

$$\frac{^{36}\text{Ar}}{^{40}\text{Ar}} = -2.3 \cdot 10^{-4} \frac{^{39}\text{Ar}}{^{40}\text{Ar}} + 0.0035$$

An intersection of the line with the $^{39}\text{Ar}/^{40}\text{Ar}$ -axis indicates a distorted value 1550 without physical meaning and the one with the $^{36}\text{Ar}/^{40}\text{Ar}$ -axis gives the value 0.0035 accidentally close to atmospheric.

Samples with radiogenic argon losses in the past may be recognized through a quite specific age spectrum with ascending and descending steps

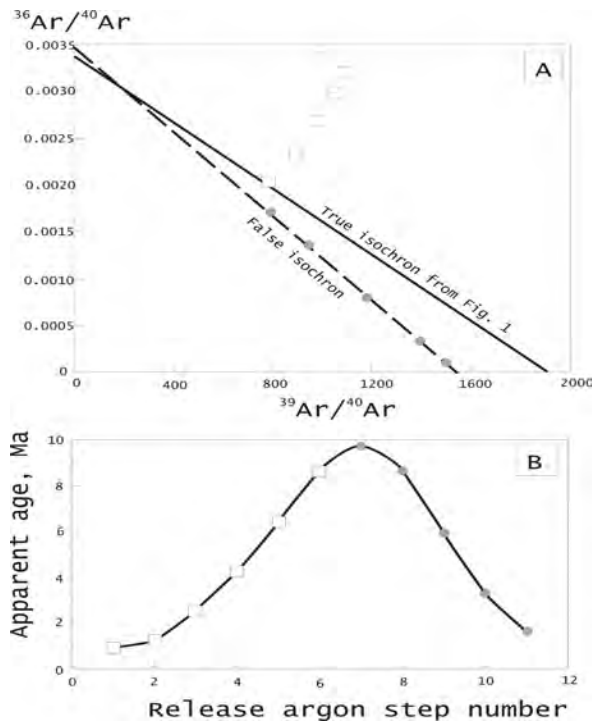


Figure 4: Argon-argon isotope plot (A) and age spectrum (B) for the case, when a sample had lost 50% of its radiogenic argon in the past. Symbols as in Fig. 3.

branches and large scattering points on the argon-argon plot.

8 A Case of a 'False Isochron' due to Excess Radiogenic Argon

Excess and inherited argon are often present in intrusive rocks due to incomplete degassing and/or incomplete substitution of magmatic argon by the atmospheric. It has been identified in submarine lavas, in pyroxenes from intrusive rocks etc. Qualitatively an inclusion of excess ^{40}Ar could be thought as a capture of argon-gas bubbles at crystallization, and inherited argon as a capture of individual argon particles by the main lattice during precipitation.

Consider first equal diffusion parameters for radiogenic, nucleogenic, and excess argon and lower activation energy for atmospheric argon. Quite similar to the section 5, we may see that at any thermal procedure, the three types of argon are released congruently. A shift of argon isotopic composition due to excess argon results in a 'false isochron'. Its intercepts yield abnormally low value of the ^{36}Ar - ^{40}Ar ratio at the y-axis and an older age at the x-axis (not shown).

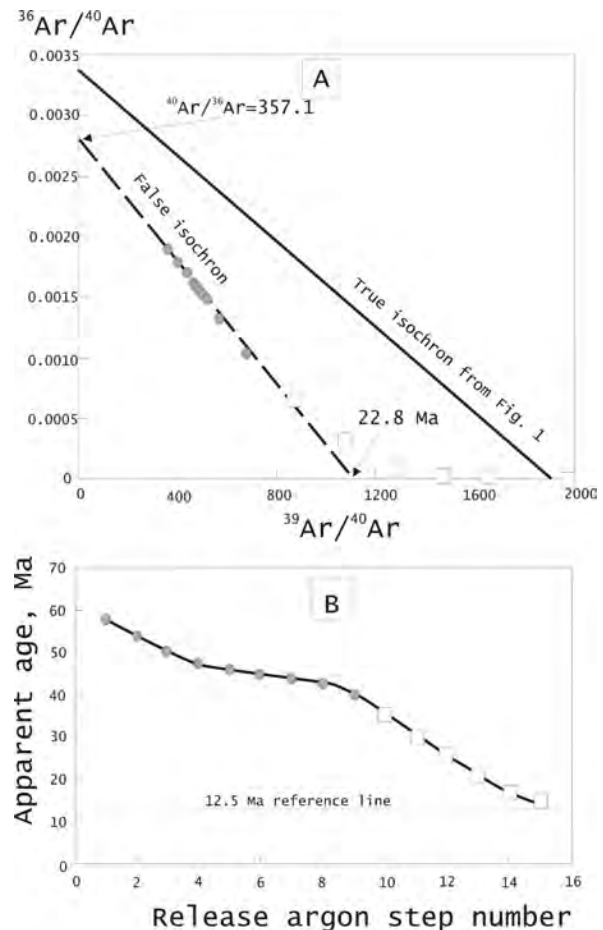


Figure 5: Argon-argon isotope plot (A) and age spectrum (B) for the case, when the activation energy of diffusion for excess argon lies between those of radiogenic (equal to nucleogenic) and atmospheric. Symbols as in Fig. 3.

Take the activation energy of excess argon (E_{exc}) slightly lower than those of radiogenic and nucleogenic: $E_{rad}=E_{39}=25000$; $E_{exc}=24500$, and $e_{atm}=23000$ cal/mole; $D_0=0.5$ s $^{-1}$. Take a sample with the age of 12.5 Ma having 2% potassium and 10^{-3} nmm 3 /g radiogenic argon. Further let be the contents $^{40}\text{Ar}_{rad} = ^{40}\text{Ar}_{exc} = ^{40}\text{Ar}_{atm}$. Calculations for this case are quite similar to those of section 5.

On Fig 5B, we can see a descending succession of steps above the 12.5 reference line. On Fig. 5A, the points show curvilinear array. The part of the latter is interpreted as a 'false isochron' plotted lower than a 'true' one with no excess argon. The 'true isochron' is usually unknown to the experimenter and he has to draw a straight regression line through some points at the 'false isochron'. So, the experimenter should obtain an

intercept with the y-axis at 0.0028, corresponding to an $^{40}\text{Ar}/^{36}\text{Ar}$ ratio 357.1, instead of 295.5 and an intercept with the x-axis, corresponding to an age of 23.88 Ma, instead of 12.5 Ma. Interesting is that both the values are fictitious showing presence of excess argon.

9 Discussion

A formal analysis of the $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{39}\text{Ar}/^{40}\text{Ar}$ diagram shows that the true isochron is represented by a straight line allowing proper extrapolations and regressions only when activation energies of diffusion for radiogenic argon-40 and nucleogenic argon-39 are equal and the one for atmospheric argon is lower. It is the lower activation energy of atmospheric argon that makes possible using the argon-argon diagram. The latter allows a proper test for the plateau age. The true results are obtained if the plateau age is equal to the isochron value and intercept of the isochron with ordinate yields the atmospheric value of the $^{36}\text{Ar}/^{40}\text{Ar}$ at 0.003384.

In cases, when activation energies of diffusion for radiogenic argon-40 and nucleogenic argon-39 differ for some reasons, curvilinear arrays and spreading of points occur, which may produce misleading interpretations. If age spectrum do not yield a plateau, argon-argon diagram can not be used directly for determinations of the age and $^{36}\text{Ar}/^{40}\text{Ar}$ ratio of *contaminating* component. There arises the idea to approximate experimental curves by means of a proper choice of parameters of equation (7). In this study, we discuss some the most general features of argon-argon diagram for the cases considered.

An array rectilinear at the beginning and curvilinear at the end may be caused by the lower activation energy of nucleogenic argon than radiogenic as well as by presence of excess argon (Fig. 3, 5). A pattern of curvilinear array at the beginning and rectilinear at the end may be resulted from the argon losses in the past (Fig. 4).

More reliable information on kinetic relation of argon components is obtained from comparisons of age spectra and argon-argon diagrams. An ascending step pattern of age spectrum along with high $^{36}\text{Ar}/^{40}\text{Ar}$ value of the *contaminating* argon exhibits a case of lower activation energy for nucleogenic argon than radiogenic. If the plateau yields the age lower than the intercept of a correlation line with x-axis in argon-argon coordinates, the true age lies between the ages of the plateau and the 'false isochron'. It is note-

worthy, that the lower activation energy of ^{39}Ar causes its losses (recoil) at a stage of irradiation. This effect makes confusing results during the step heating argon release.

A descending sequence of apparent ages along with lower $^{36}\text{Ar}/^{40}\text{Ar}$ indicates a 'false isochron' related to excess radiogenic argon with the lower energy of activation than radiogenic and nucleogenic. There exists no procedure to discriminate radiogenic and excess argon. The array on Fig. 5A has, however, a quite characteristic shape: rectilinear at the beginning and curved at the end. An age spectrum with ascending and descending steps and large scattering points on argon-argon plot is likely a result of argon losses in the past

10 Conclusions

A formal analysis of argon components in terms of diffusion and Arrhenius' laws shows that the $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{39}\text{Ar}/^{40}\text{Ar}$ diagram exhibits a linear graph as a true isochron and allows regressions and extrapolations only, when activation energies of diffusion for radiogenic argon 40 and nucleogenic argon 39 are equal and that for *contaminating* argon of atmospheric composition is lower.

The character of the point arrays on argon-argon diagram may provide indications on correlations of activation energies of its components: radiogenic, nucleogenic, and *contaminating*. Some portions of those curvilinear arrays (initial or final) may not correspond to the 'true isochrons' and their intersections with coordinate axes gain no physical sense. Strict rules for selection of these portions at present do not exist. Their formulation is an object for further investigations. There exists a possibility to determine kinetic parameters of the argon components in crystal structures through configurations of arrays of experimental points in argon-argon coordinates.

It is noteworthy that in terms of the present paper features of argon components can be characterized by higher or lower activation energies. It remains the question, whether these 'nuclei' or 'domains' or, in our terms, 'components of argon with a characteristic activation energy' are spatially separated or are distributed homogeneously over the whole host structure. In perthites, they are separated. On the other hand, it is plausible to suggest that particles of ^{39}Ar are surrounded by 'individual radiation damage zones' and distributed over the whole crystal structure. In our opinion, priority should be given to the advan-

tage of a quantitative estimation of array patterns. We have considered a few characteristic cases. Actually there exists an infinite lot of combinations of parameters. By a proper choice of the latter, every array could be approximated.

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