



# Ar-Ar Geo-/Thermochronology

(an introduction)

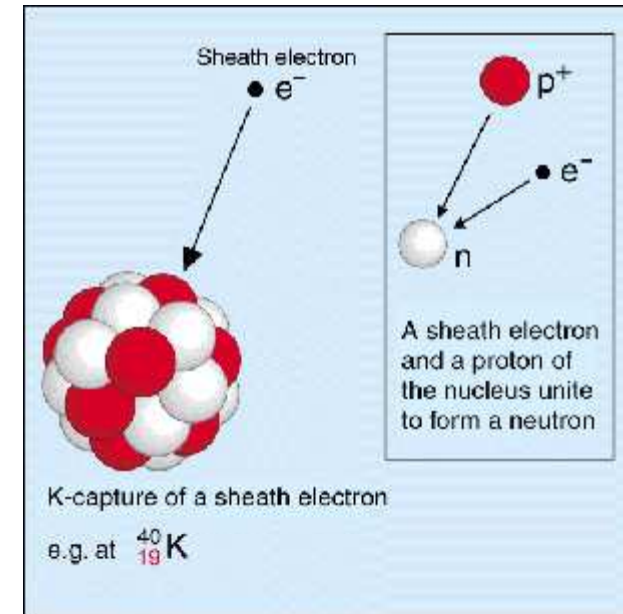
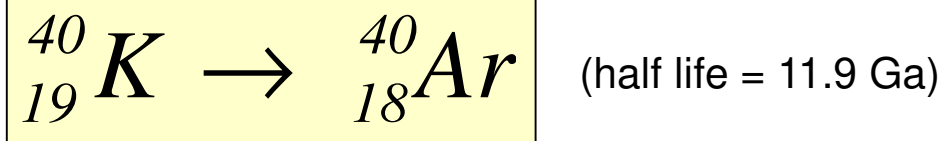
Jörg A. Pfänder

*TU Freiberg*

## Principle: K-Ar Method:

Electron capture decay

of  $^{40}\text{K}$  to  $^{40}\text{Ar}$ :



**Aldrich & Nier (1948):** *K-rich minerals* have elevated  $^{40}\text{Ar}/^{36}\text{Ar}$  ratios when compared to *atmospheric argon* – this suggests that  $^{40}\text{Ar}$  is a decay product of  $^{40}\text{K}$

(Aldrich L.T. & Nier, A.O., 1948: Argon 40 in Potassium Minerals, Phys. Rev. 74, 876–877 – Nachweis des Zerfalls von  $^{40}\text{K}$  zu  $^{40}\text{Ca}$  und  $^{40}\text{Ar}$ !)

### Argon 40 in Potassium Minerals

L. T. ALDRICH AND ALFRED O. NIER

Department of Physics, University of Minnesota, Minneapolis, Minnesota

(Received July 8, 1948)

An investigation has been made of the isotopic composition of the argon from four potassium minerals. In each case a high  $A^{40}/A^{36}$  ratio compared to that of atmospheric argon is observed showing directly that  $K^{40}$  decays to both  $Ca^{40}$  and  $A^{40}$ . From the absolute amounts of radiogenic  $A^{40}$  and  $K^{40}$  in the minerals a lower limit on the branching ratio  $\lambda_K/\lambda_\beta$  can be made. If the half-life,  $7 \times 10^8$  yrs., is assumed for  $K^{40} \rightarrow Ca^{40}$ ,  $\lambda_K/\lambda_\beta$  must be at least 0.02. The possibility of using this method for measuring geological age is suggested.

IT was suggested by von Weizsäcker<sup>1</sup> in 1937 that the abnormally high abundance of  $A^{40}$  in argon might be explained by assuming that  $K^{40}$  not only decays to  $Ca^{40}$  through  $\beta$ -emission but also to  $A^{40}$  through  $K$ -capture. Indirect evidence to support this view has been obtained by a number of investigators.<sup>2-6</sup> Bleuler and Gabriel<sup>7</sup> studied the  $X$ -radiation emitted during the decay process of potassium and concluded that there are 1.9 times as many  $K$ -captures occurring as there are  $\beta$ -rays emitted. Suess<sup>8</sup> was unsuccessful in his search for argon in sylvine and carnallite

minerals and concluded from this investigation together with general geological considerations that the branching ratio,  $\lambda_K/\lambda_\beta$ , was  $0.05 \pm 0.02$ .

An investigation of the gas evolved from four potassium minerals has been made with a high sensitivity mass spectrometer. In all cases small amounts of argon were discovered and in each case the  $A^{40}/A^{36}$  ratio was appreciably greater than that observed for atmospheric argon. Figure 1 shows a comparison between the spectra observed for atmospheric argon and for the argon found in one of the minerals.

The procedure employed in extracting the argon from the minerals was as follows: A weighed amount of the mineral was introduced into a high temperature vacuum furnace which was heated to a temperature above  $1000^\circ\text{C}$ . The condensable vapors were removed by a liquid oxygen trap. If the amount of gas remaining after this treatment was less than 20 std. cc, an investigation of the  $A^{40}/A^{36}$  ratio was made directly without further purification. The extremely high sensitivity of the spectrometer permitted one to do this. For samples larger than 20 std. cc the argon concentration was too low to permit accurate isotope analysis and the gas

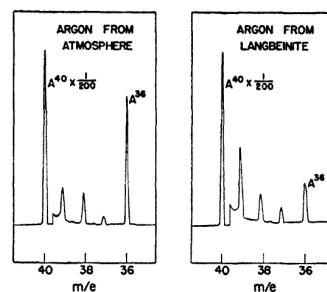
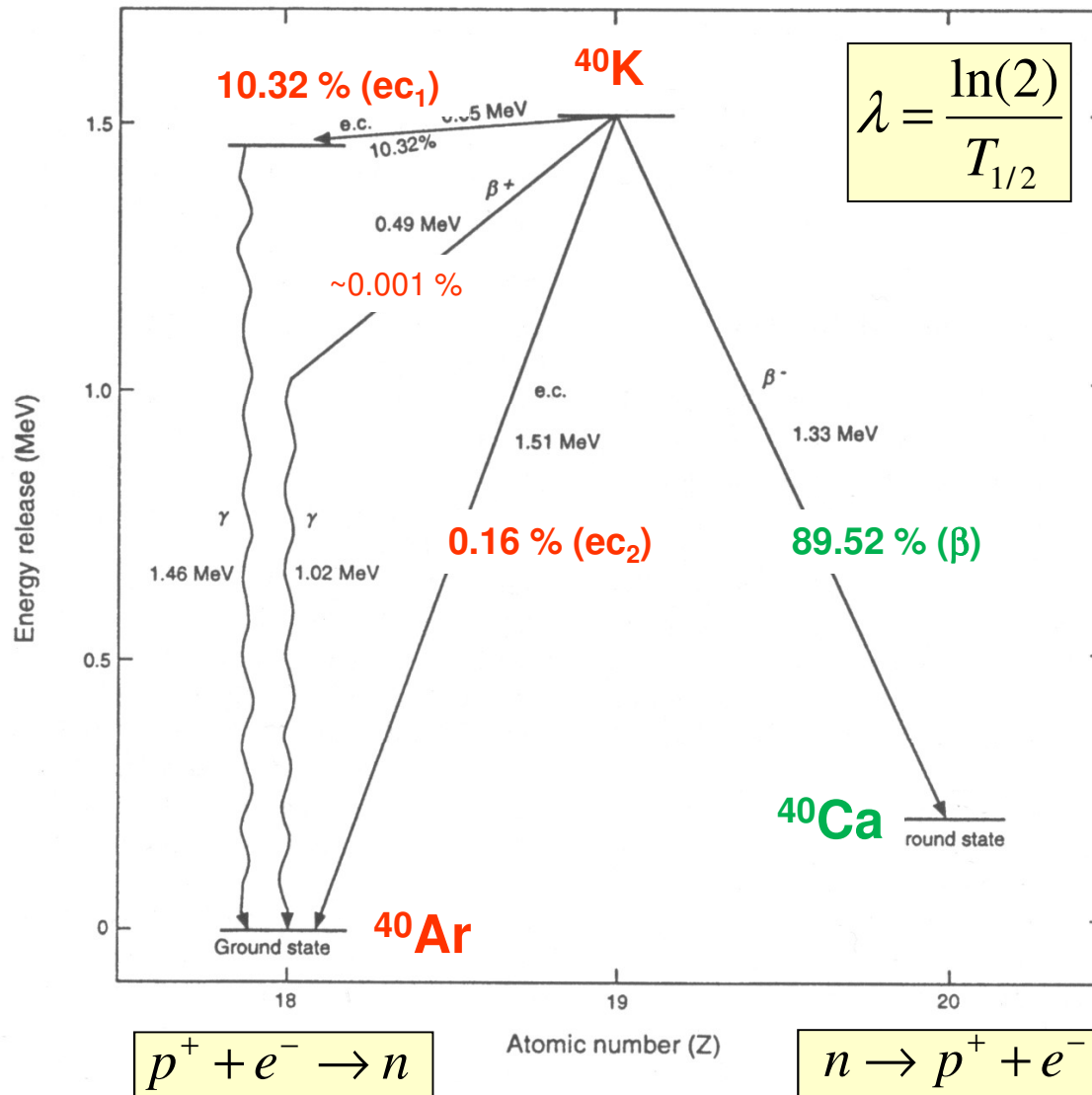


FIG. 1. Mass spectra for atmospheric argon and for argon from mineral Langbeinite. Note that this mineral has an  $A^{40}/A^{36}$  ratio greater than three times that for atmospheric argon. Peaks at 37 and 39 and part of 38 are due to residual

# Ar-Ar Geo-/Thermochronology

Introduction: K-Ar dating

Radioactive decay scheme of natural  $^{40}\text{K}$



Half life of the  $^{40}\text{K} - ^{40}\text{Ar}$  decay:

**11.93 Ga**

$$\lambda_{ec} = 0.581 \times 10^{-10} \text{ 1/a}$$

Half life of the  $^{40}\text{K} - ^{40}\text{Ca}$  decay:

**1.397 Ga**

$$\lambda_{\beta} = 4.962 \times 10^{-10} \text{ 1/a}$$

Half life ( $T_{1/2}$ ) of  $^{40}\text{K}$ :

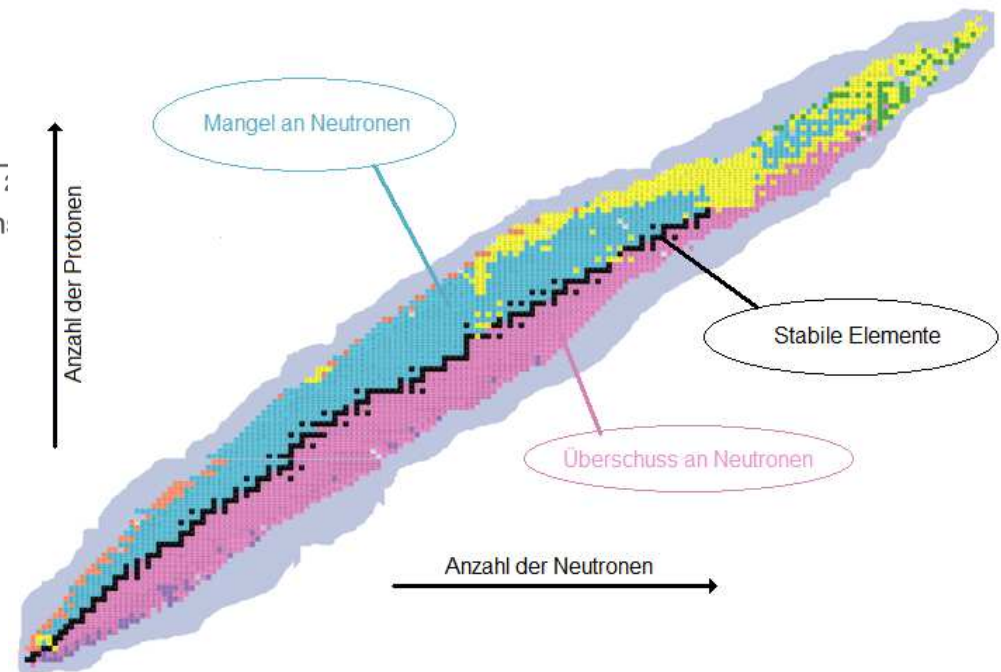
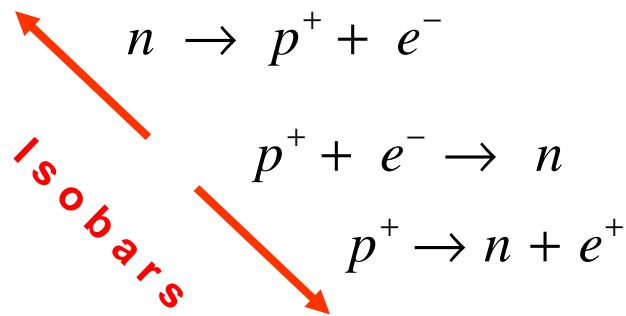
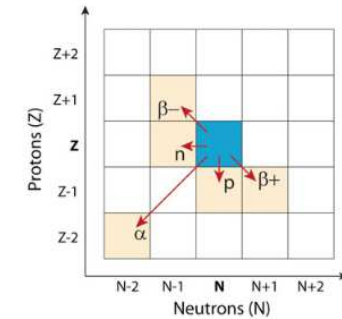
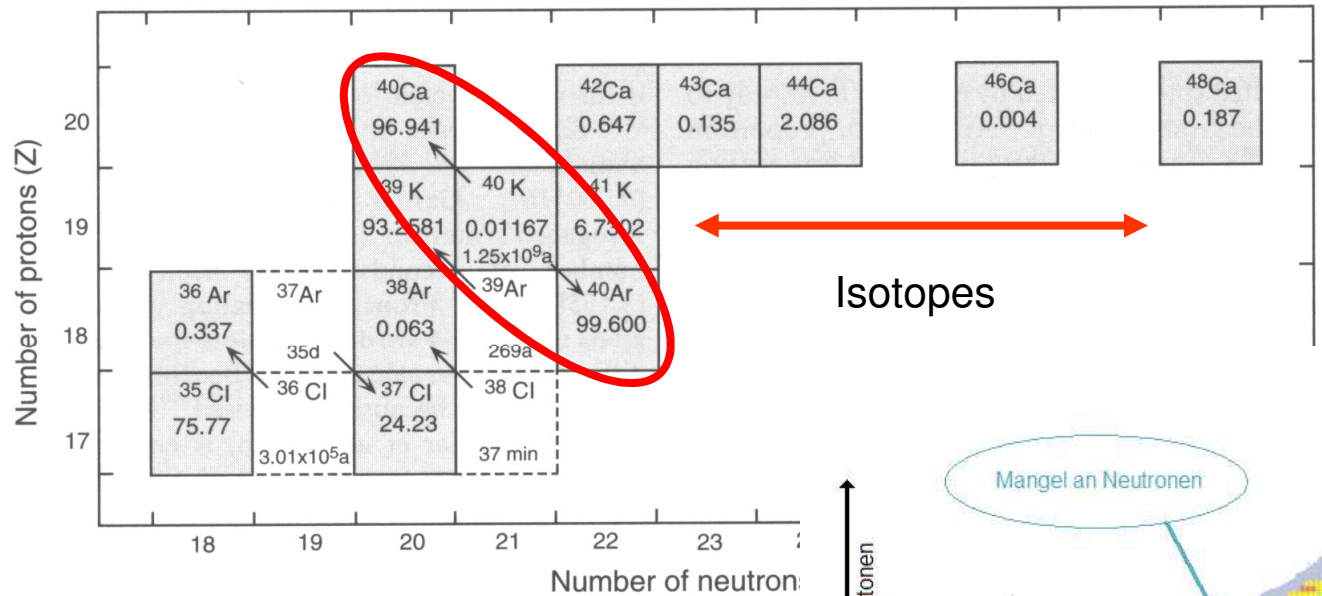
**1.25 Ga**

$$\lambda_{tot} = 5.543 \times 10^{-10} \text{ 1/a}$$

# Ar-Ar Geo-/Thermochronology

Introduction: K-Ar dating

$^{40}\text{K}$  branched decay to  $^{40}\text{Ca}$  (89.52%) and  $^{40}\text{Ar}$  (10.48%) in the chart of nuclides:



## Abundances of naturally occurring **Ar** isotopes:

$$^{36}\text{Ar} = 0.3364 \pm 0.0006 \%$$

$$^{38}\text{Ar} = 0.0632 \pm 0.0001 \%$$

$$^{40}\text{Ar} = 99.600 \%$$

Atmosphere: ~1% Argon  
with  $^{40}\text{Ar}/^{36}\text{Ar} = 298.56 \pm 0.31$   
(Lee et al., 2006)

## Abundances of naturally occurring **K** isotopes:

$$^{39}\text{K} = 93.2581 \%$$

$$^{40}\text{K} = 0.01167 \%$$

$$^{41}\text{K} = 6.7302 \%$$

Only ~0.012% of K consists of  $^{40}\text{K}$ ,  
and only ~10% of  $^{40}\text{K}$  decay to  $^{40}\text{Ar}$

**BUT**

**K is a major element in numerous  
rock forming minerals!**

*Formula to calculate the molar amount of  $^{40}\text{K}$  in a sample from  $\text{K}_2\text{O}$ :*

$$^{40}\text{K} [\text{mol}] = m_{\text{Min.}} [\text{g}] \times \{ \text{K}_2\text{O} [\text{wt}\%] / 100 \} \times 0.8301 \times 0.0001167 / 39.9637$$

## What can be dated by K-Ar and Ar-Ar?

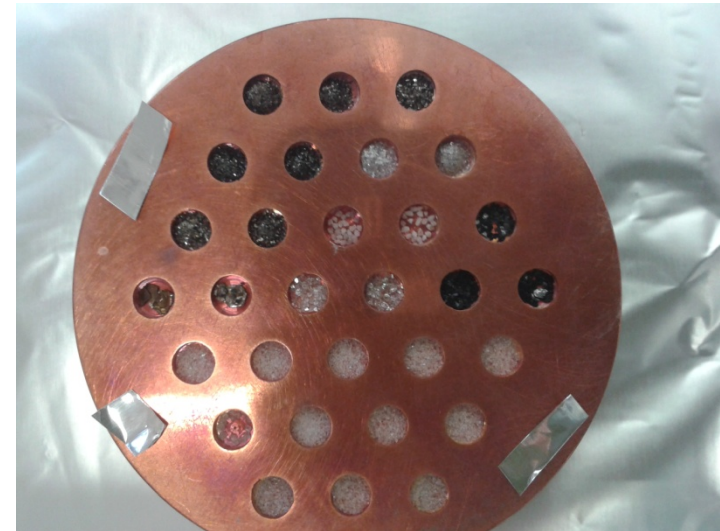
*Basically all K-bearing rocks and minerals !*

## Commonly used minerals:

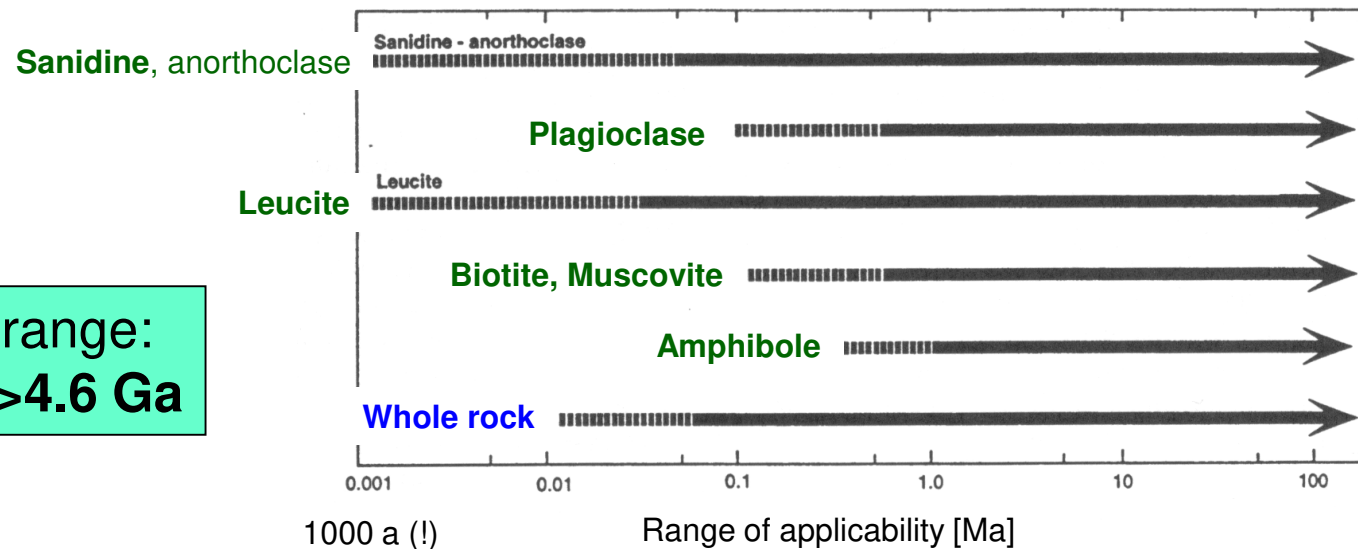
*K-feldspar, biotite, muscovite, hornblende, plagioclase*

## Whole rocks:

*Basalte, rhyolite, tuffs, meteorites, ....*

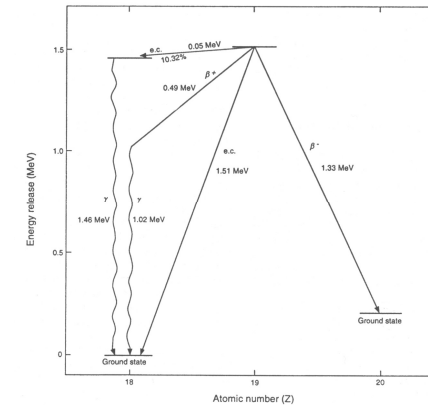


Dateable age range:  
~1000 a up to >4.6 Ga



Amount of **radiogenic**  $^{40}\text{Ar}$  in a sample/mineral is proportional to:

- **K-concentration** in sample/mineral
- **Age** of sample/mineral



Amount of radiogenic  $^{40}\text{Ar}$  ( $^{40}\text{Ar}^*$ ) in a sample or mineral as a function of **time** and **remaining** K-concentration:

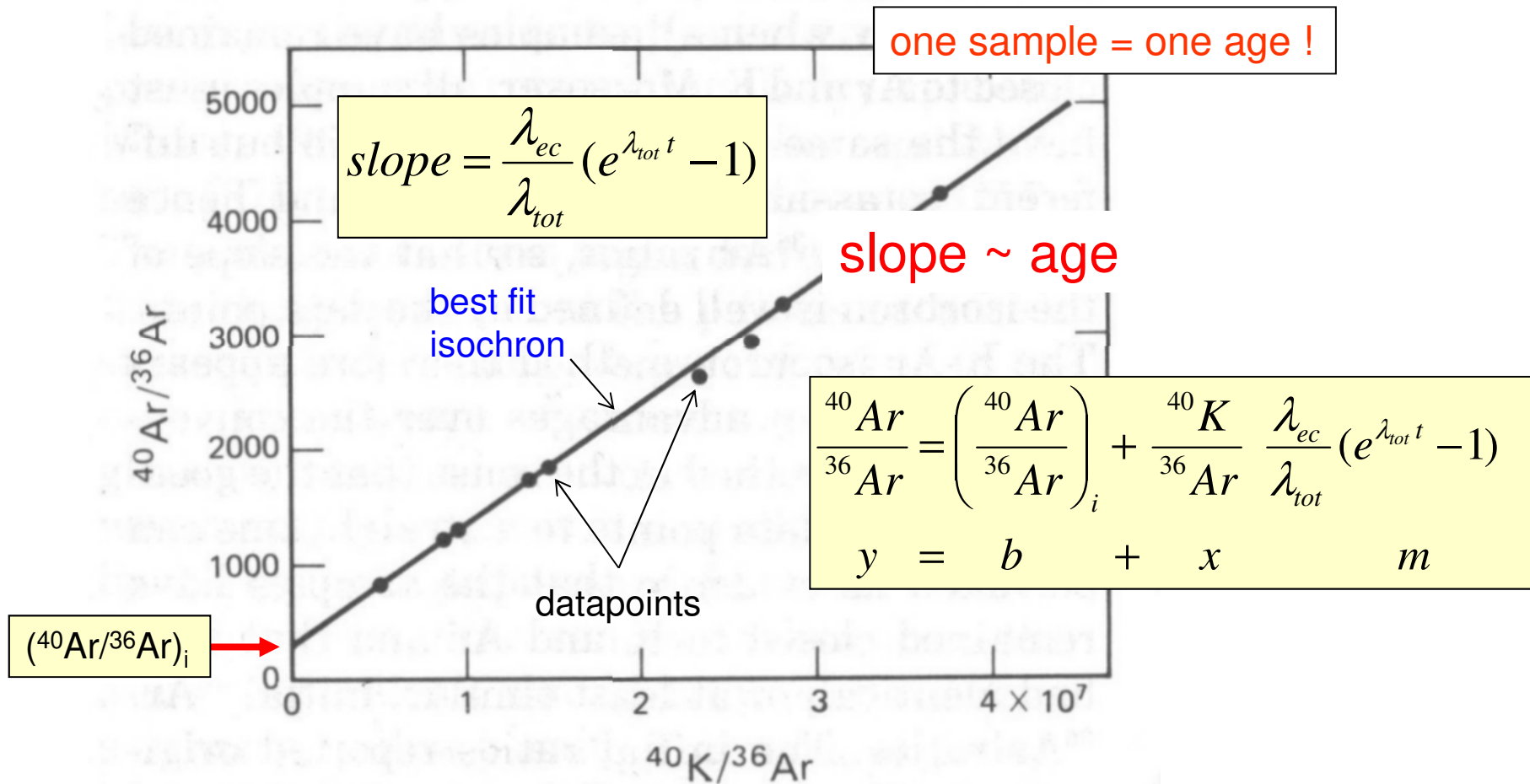
$$^{40}\text{Ar}^* = \frac{\lambda_{ec}}{\lambda_{tot}} {}^{40}\text{K} (e^{\lambda_{tot} t} - 1)$$

$$\lambda_{tot} = \lambda_{ec1} + \lambda_{ec2} + \lambda_{\beta} = 5.543 \times 10^{-10}$$

$$^{40}\text{Ar}_{tot} = {}^{40}\text{Ar}_i + {}^{40}\text{Ar}^*$$

$$\frac{\lambda_{ec}}{\lambda_{tot}} = \frac{0.581 \times 10^{-10}}{5.543 \times 10^{-10}} = 0.1048$$

Leading to the classical **K-Ar isochron** diagram:



*K-Ar whole-rock isochron of a **tuff sample** – each datapoint represents a split of the same sample*

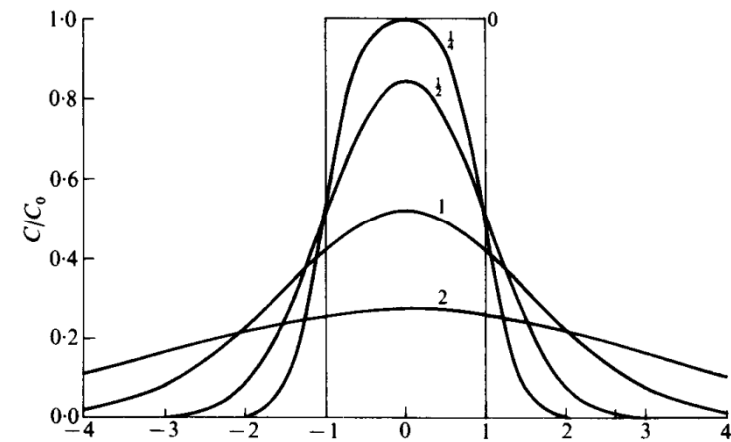
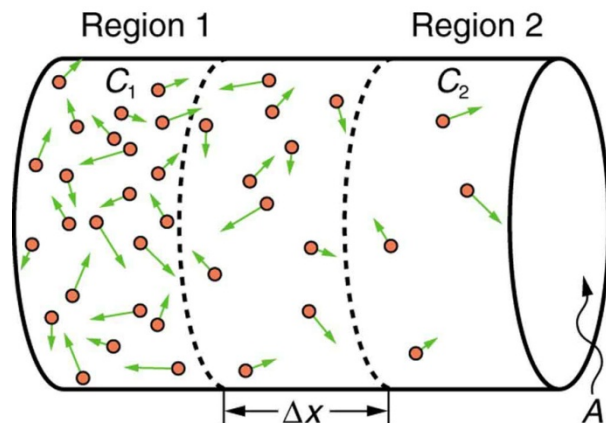
## Requirements to obtain a „correct“ K-Ar age:

- Decay constant's are *constant* over Earth's history...
- $^{40}\text{Ar}$  in a sample is *only* radiogenic ( $^{40}\text{Ar}^*$ ) ...
- ... *or non-radiogenic Ar* can be determined and *corrected* for
- Samples/minerals remain a *closed system* after crystallisation/cooling

What about ...

... „samples/minerals need to remain a **closed system** after crystallisation/cooling“ ?

Solid-state **Diffusion !**



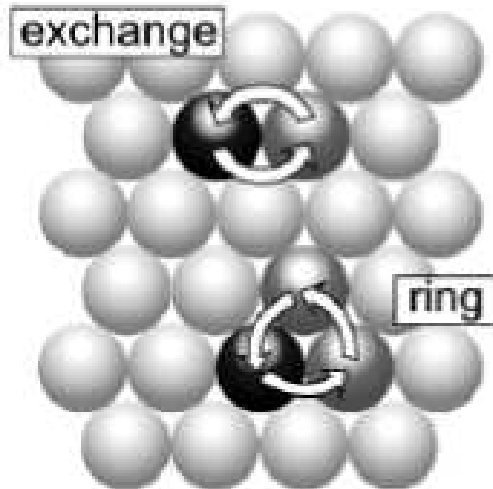
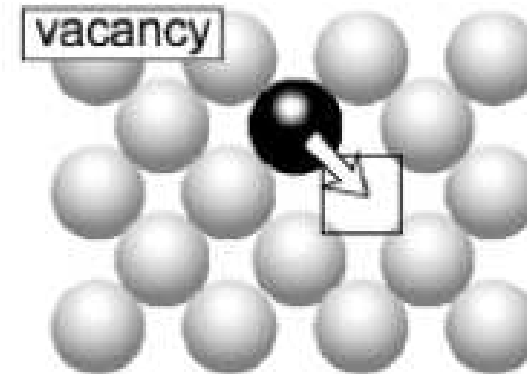
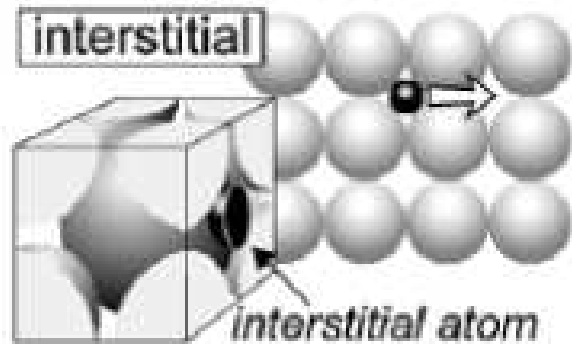
From: J. Crank – The Mathematics of Diffusion

$$J = -D \frac{\partial c}{\partial x} \quad \text{and} \quad D = f(T)$$

$J$  = mass flow per area (mol/cm<sup>2</sup>sec)  
 $c$  = concentration (mol/cm<sup>3</sup>)

$D$  = diffusivity (cm<sup>2</sup>/s)  
 $x$  = position (cm)

### Brief excursion: Diffusion pathways in minerals



$$J = -D \frac{\partial c}{\partial x} \quad \text{and} \quad D = f(T)$$

*one-dimensional & steady-state!*

*Fick's first law (Adolf E. Fick, 1855)*

*J = Mass flow per area and time (e.g.  $\text{mol}/\text{cm}^2\text{sec}$ ) in steady state*

*D = Diffusivity (diffusion coefficient in  $\text{cm}^2/\text{sec}$ )*

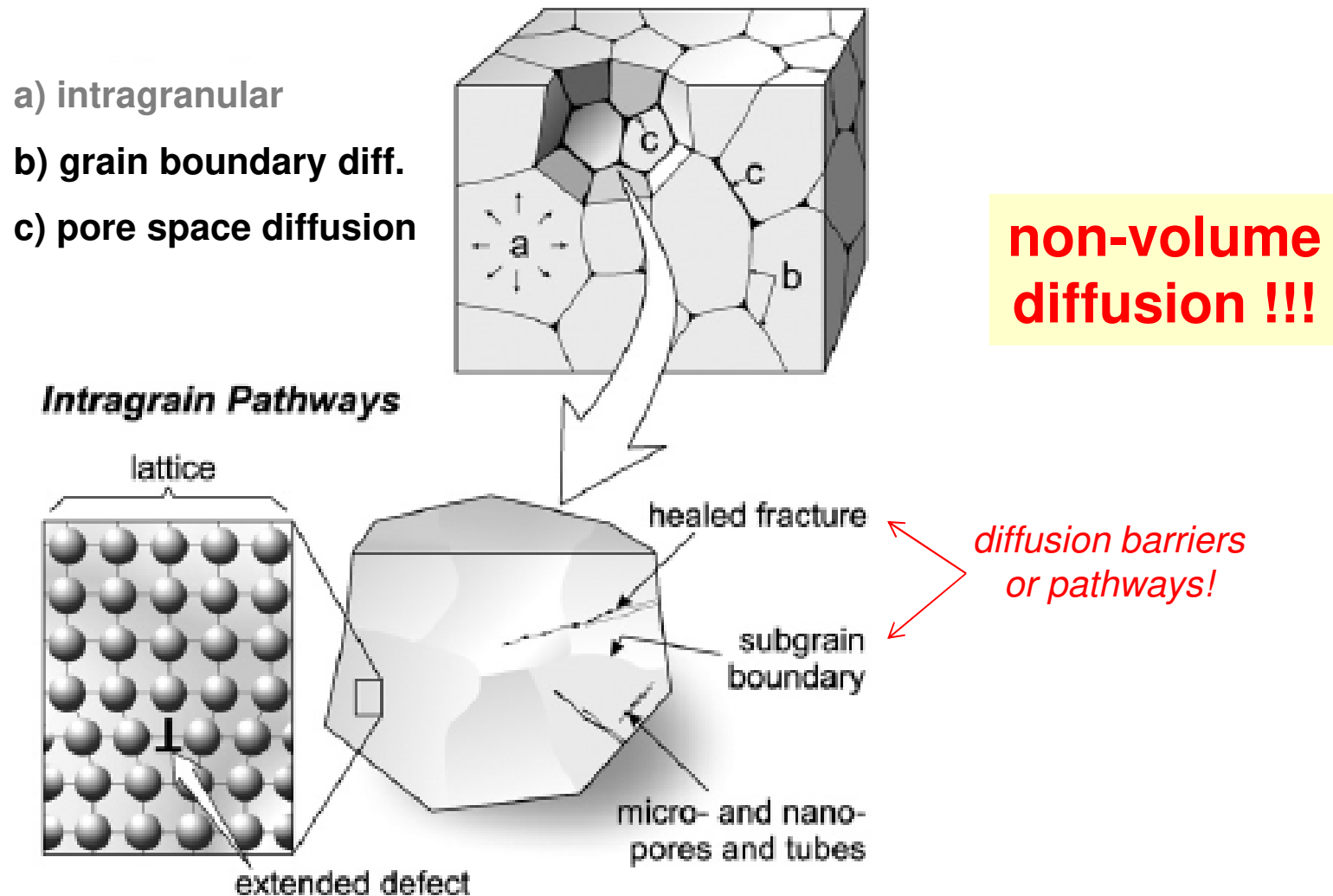
*dc/dx = Concentration gradient in x-direction*

*T = Temperature [K]*

All from: Watson & Baxter, 2006,  
*Earth Planet. Sci. Lett.*

**In three dimensions: Volume diffusion!**

## Brief excursion: 'Diffusion' pathways in rocks



## Non-steady-state requires to consider time:

Relation between Fick's first law and time (**Fick's second law**):

$$\frac{\partial c}{\partial t} = -\frac{\partial J}{\partial x} \quad \text{yields} \quad \frac{\partial c}{\partial t} = \frac{\partial}{\partial x} \left( D \frac{\partial c}{\partial x} \right)$$

$$J = -D \frac{\partial c}{\partial x}$$

$$\text{yields} \quad \frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2}$$

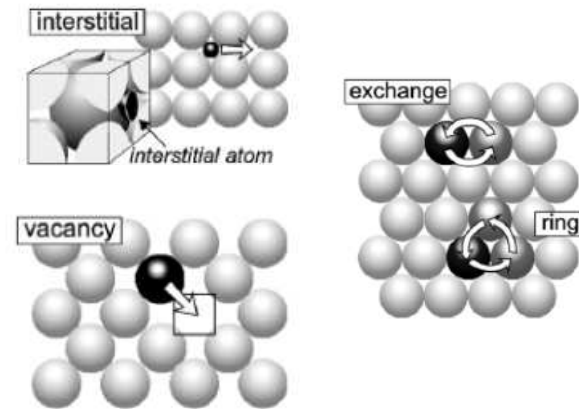
This **equation** describes the **concentration** of Argon in one dimension (along the x-direction) as a function of **space** and **time**

### Fick's second law in three-dimensions:

(and in cartesian coordinates):

$$\frac{\partial c}{\partial t} = D \left( \frac{\partial^2 c}{\partial x^2} + \frac{\partial^2 c}{\partial y^2} + \frac{\partial^2 c}{\partial z^2} \right)$$

$$\frac{\partial c}{\partial t} = D \nabla^2 c$$



This **equation** describes the **concentration** ( $c$ ) of a species (e.g. of argon in a **mineral**) at a defined **location** ( $x,y,z$ ) at a defined **time** ( $t$ ) (note that only volume diffusion is considered!)

## Solution of Fick's second law for a (infinite) **plane sheet**

(one dimensional analytical solution):

$$c = \frac{4c_0}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)} \times \exp(-D(2n+1)^2 \pi^2 \underline{t} / 4r^2) \times \cos \frac{(2n+1)\pi \underline{R}}{2r}$$

$D = f(T)$

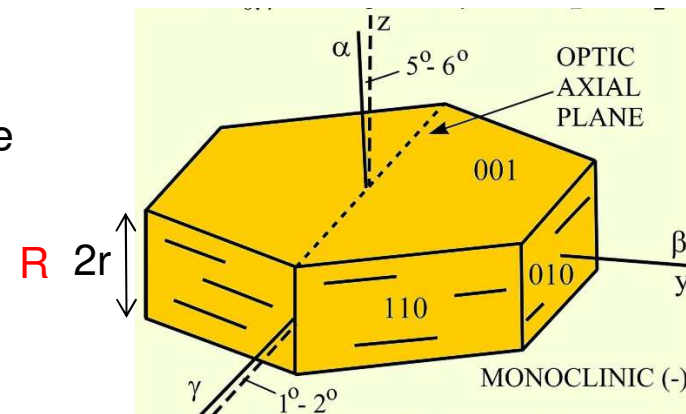
$c_0$  = Initial concentration at  $t = 0$  over the whole plate

$c$  = Concentration at **time t** at **position R**

$D$  = Diffusivity (diffusion coefficient)

$R$  = Position within the sheet

$2r$  = thickness of the sheet



This equation describes the **concentration** of Argon in a plane sheet as a function of **position R** and **time t** at a given temperature T

**Solution of Fick's second law for spherical geometry in spherical coordinates:**

$$c = \frac{c_0 2r}{\pi R} \sum_{n=1}^{\infty} \frac{(-1)^n}{n} \sin \frac{n\pi R}{r} \times \exp(-n^2 \pi^2 \underline{Dt} / r^2)$$

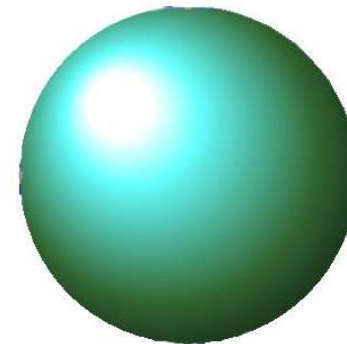
$c_0$  = Initial concentration at  $t = 0$  over the whole sphere

$c$  = Concentration at **time t** at **position R**

$D$  = Diffusivity (diffusion coefficient)

$R$  = Position within the sphere

$r$  = radius of the sphere



This equation describes the **concentration** of Argon in a sphere as a function of **space R** and **time t** at a given temperature T

## Numerical approaches to solve Fick's second law

### Finite differences method

Approximation of **differentials** by (small) **differences** within a defined (equidistant) 2D/3D grid, at defined starting and boundary conditions *(many textbooks are available on finite difference methods)*

### Lattice Boltzmann model

LB describes physics not in terms of **continuum mechanics**, but by the (timely) evolution of **particle distribution functions** upon translations and collisions (statistical mechanics)

*See for example: Huber et al. (2011), Geoch. Cosmoch. Acta, 75, 2170-2186, and refs. therein*

For practical reasons, a **simple approximation** can be used to calculate the diffusion length (i.e. the distance travelled by an atom or ion after time t):

$$x \approx \sqrt{Dt}$$

$$t \approx \frac{x^2}{D}$$

$x$  = diffusion length (cm)

$D$  = Diffusivity (diffusion coefficient;  $\text{cm}^2/\text{s}$ )

$t$  = time (s)

Example:

The diffusion coefficient of **Ar in hornblende** is  $\sim 10^{-12} \text{ cm}^2/\text{s}$  at **1250 K**. How long will it approximately take for a Hbl mineral with a diameter of 2 mm to lose most of its Argon? *Note: The maximum diffusion length is  $\frac{1}{2} \times 2 = 1 \text{ mm}$  assuming a spherical geometry*

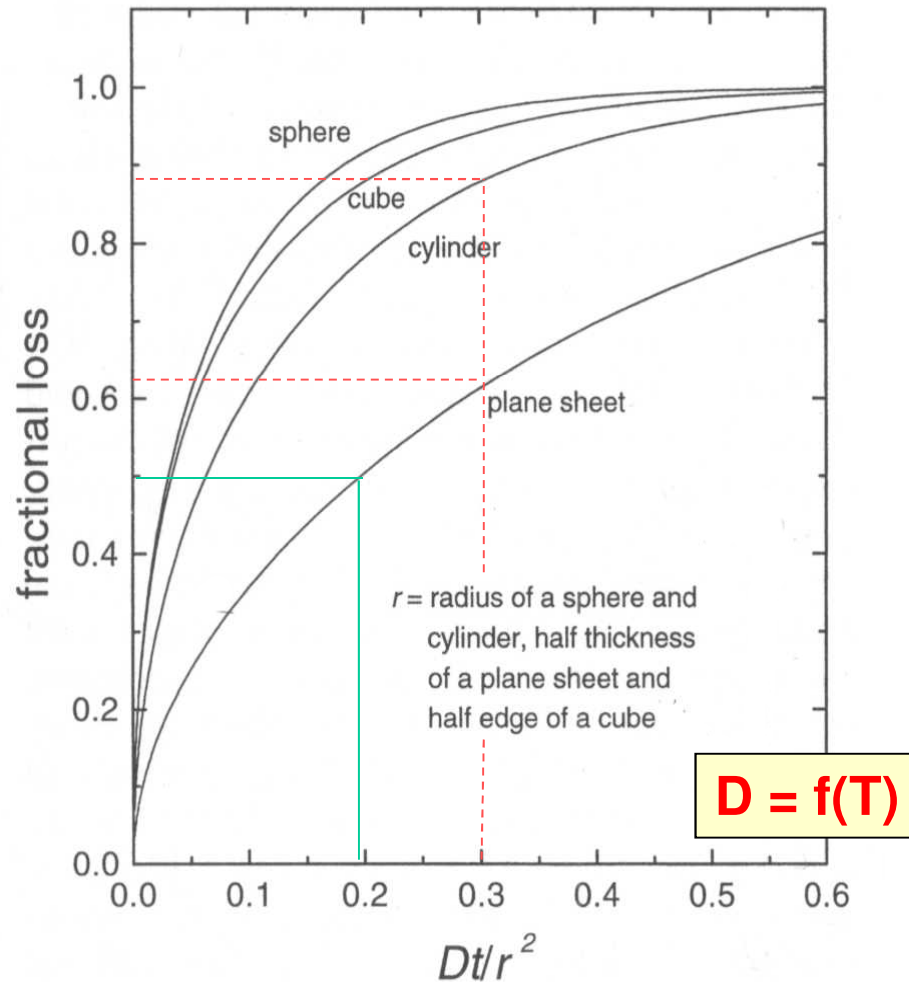
Solution:  $t = 0.1^2 / 10^{-12} = 1 \times 10^{10} \text{ s} = \underline{\underline{317 \text{ a}}} @ \underline{\underline{977^\circ\text{C}}}$  (1 a =  $31.56 \times 10^6 \text{ s}$ )

How long will it take at **1000 K** ( $D = \sim 10^{-16} \text{ cm}^2/\text{s}$ )?

Solution:  $t = 1 \times 10^{14} \text{ s} = \underline{\underline{3.1 \text{ Ma}}} @ \underline{\underline{727^\circ\text{C}}}$  (1 a =  $31.56 \times 10^6 \text{ s}$ )

**This illustrates: D and therefore the Ar loss of a mineral is strongly dependent on temperature !**

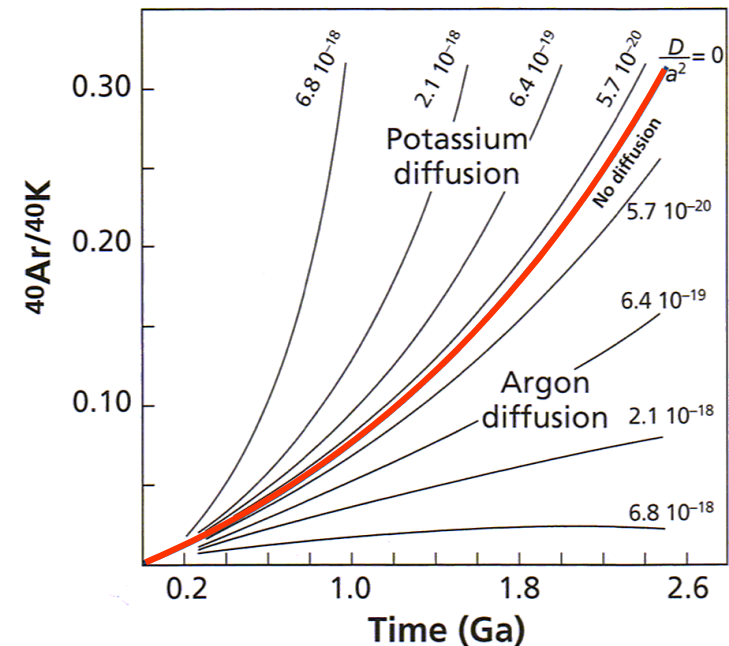
### Loss of Argon as a function of $Dt/r^2$



### Argon loss increases with:

- time ( $t$ )
- increasing diffusivity ( $D$ )
- temperature ( $T$ )
- decreasing particle size ( $r$ )

$^{40}\text{Ar}/^{40}\text{K}$  over time coupled to diffusive loss of Ar or K



From Allègre (2008) *Isotope Geology*, Cambridge U.P.

### Dependency of D from temperature T:

**Arrhenius relationship**

$$D = D_0 e^{-E/\mathfrak{R}T}$$

yields:

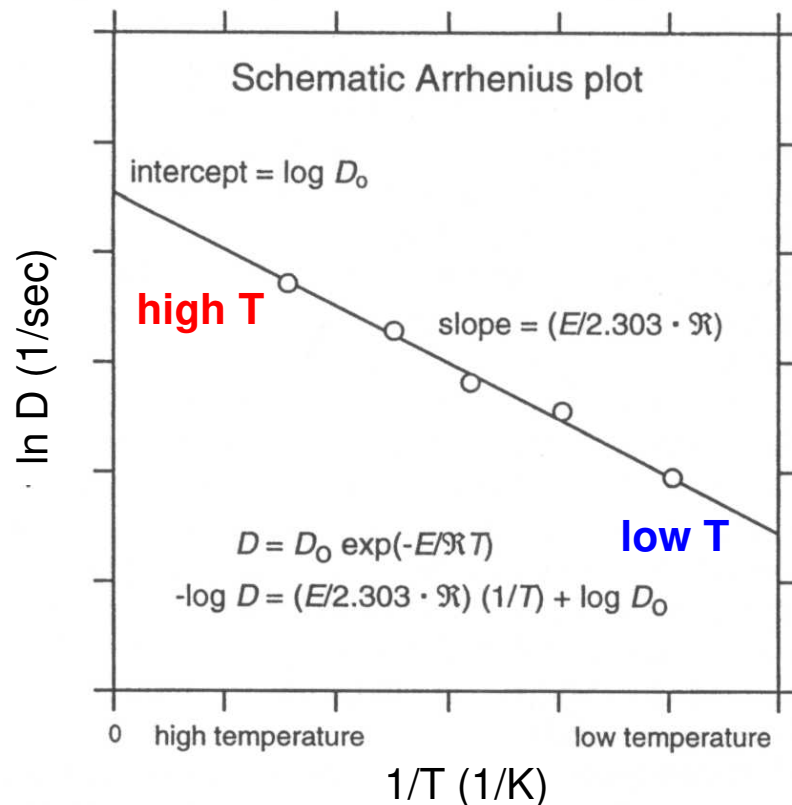
$$\ln D = \ln D_0 - \frac{E}{\mathfrak{R}} \frac{1}{T}$$

$$y = b - m x$$

Where **D** is determined from measured data and for a given temperature **T**:

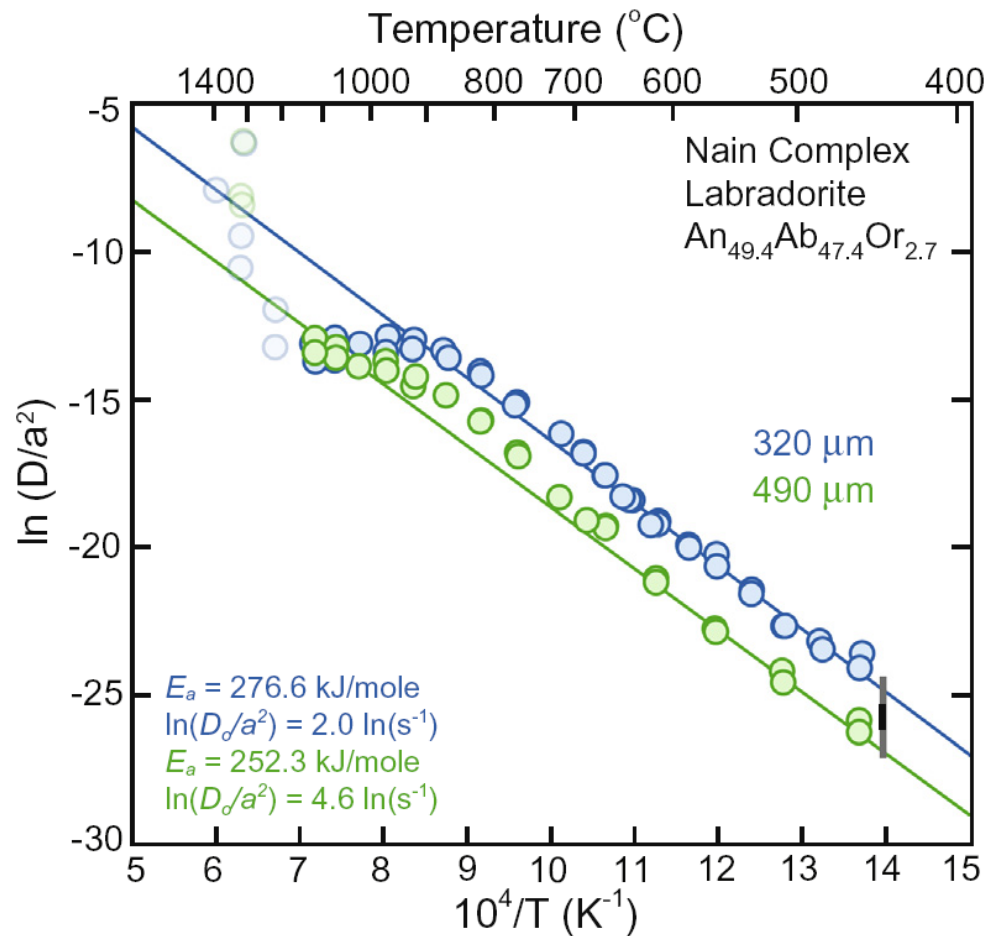
$$D = \frac{(qf)^2}{t}$$

q = geometry factor  
f = fraction of Ar released  
t = heating time

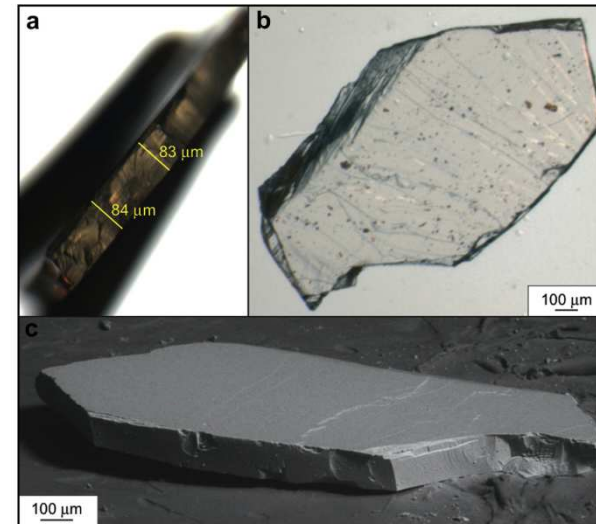


Strong dependency of **D** from **T** allows to define the **blocking temperature!**

### Example for a real sample:



From Cassata & Renne, 2013

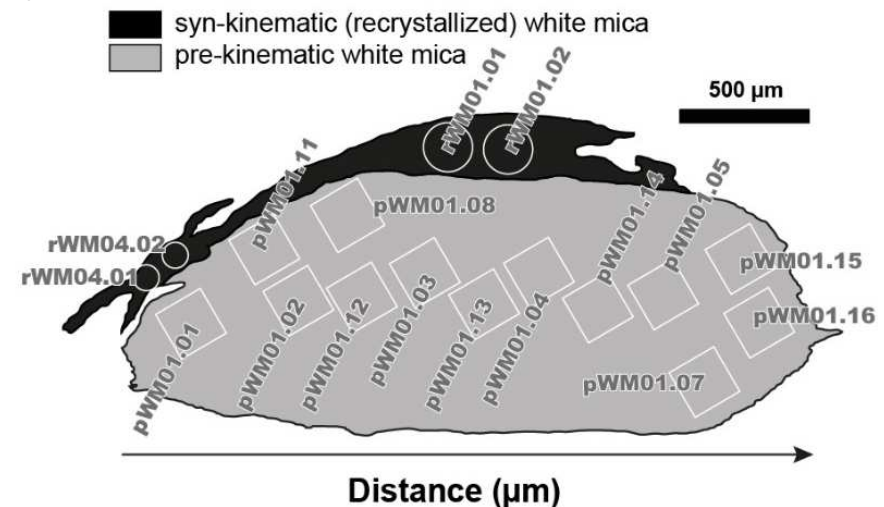
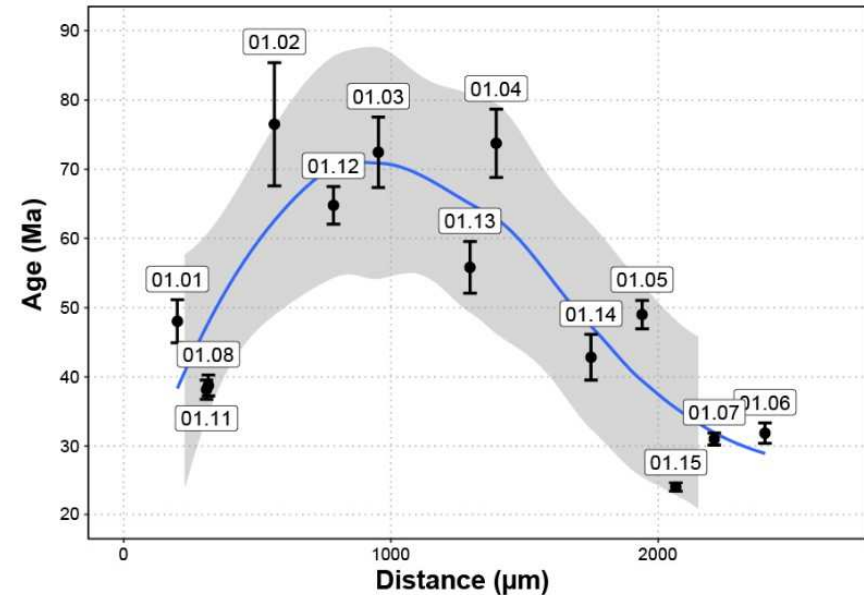
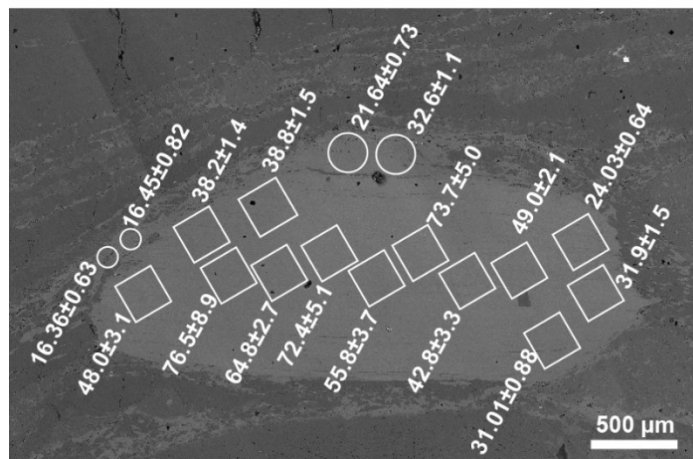


Arrhenius plot for two size fractions of labradorite (plagioclase). Note that the slopes (and thus  $E$ ) are similar, whereas the y-axis intercepts differ according to grain size

### Example from a 'real' sample

Inferred argon diffusion profile in white mica resolved by in-situ dating of a sample from a amphibolite-grade paragneiss out of a detachment zone of the Internal Dinarides

From Löwe et al., in prep.



# Ar-Ar Geo-/Thermochronology

*Diffusion & closure temperature*

$D_0$  and  $E$  values for Phlogopite, Biotite, Muscovite and Hornblende

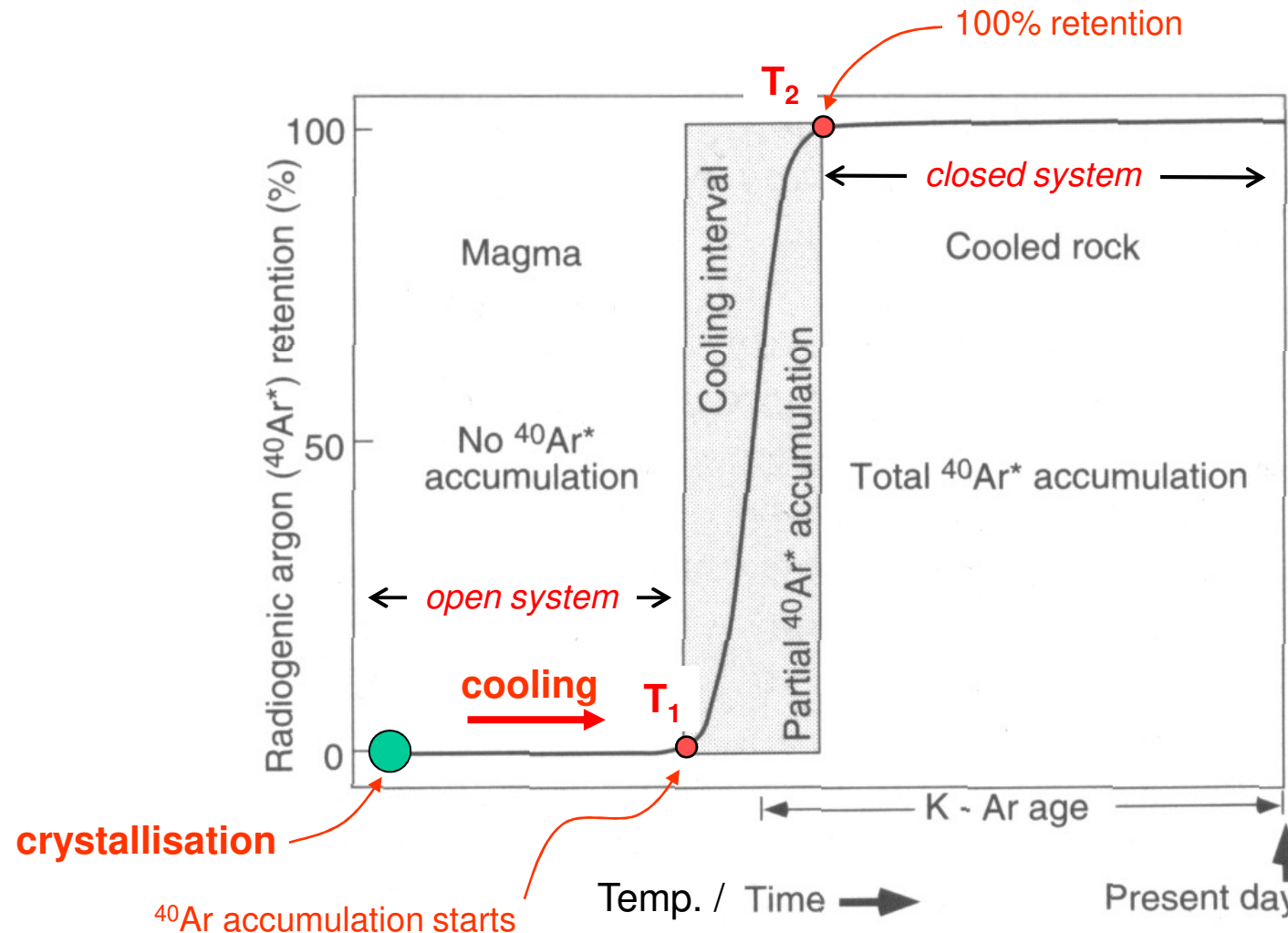
Mineral	$D_0$ (cm <sup>2</sup> s <sup>-1</sup> )	$E_a$ (kJ mol <sup>-1</sup> )	Reference
Phlogopite (Ann <sub>4</sub> )	$0.75^{+1.7}_{-0.52}$	$242 \pm 11$	Giletti (1974)
Biotite (Ann <sub>56</sub> )	$0.077^{+0.21}_{-0.06}$	$196 \pm 9$	Harrison et al. (1985)
Biotite (Ann <sub>56</sub> )	$0.075^{+0.049}_{-0.021}$	$197 \pm 6$	Combined data of Harrison et al. (1985) and Grove and Harrison (1996)
Biotite (Ann <sub>56</sub> )	$0.40^{+0.96}_{-0.28}$	$211 \pm 9$	Grove and Harrison (1996)
Muscovite	$0.033^{+0.213}_{-0.029}$	$183 \pm 38$	Hames and Bowring (1994)
Hornblende	$0.06^{+0.4}_{-0.01}$	$276 \pm 17$	Harrison (1981)

*From Braun et al., Quantitative Thermochronology*

# Ar-Ar Geo-/Thermochronology

Diffusion & closure temperature

Accumulation of **radiogenic  $^{40}\text{Ar}$**  in K-bearing minerals, closure temperature concept (Dodson, 1973)



Combining the **Arrhenius relation** to **diffusion models** leads to the closure (blocking) temperature concept (Dodson, 1973):

$$T_c = \frac{E}{\mathfrak{R} \ln(A \tau D_0 / r^2)}$$

from slope of Arrhenius plot

$$\tau = \frac{\mathfrak{R} T_c^2}{E dT / dt}$$

Intercept from Arrhenius plot  
(frequency factor)

see also Harrison et al., 2005,  
Rev. Min. Geochem.

Example (for Rb in biotite; Hofmann & Gilotti, 1970):

E = 21 kcal/mol

A = 27 (assuming a cylindrical model)

$D_0/r^2 = 10^{-12} \text{ 1/s} = 30 \text{ 1/Ma}$

$T_c$  = closure temperature

E = activation energy

$\mathfrak{R}$  = gas constant

A = geometry factor

$D_0/r^2$  = diffusivity

---

### Geometry factors:

A = 55 for a sphere

A = 27 for a cylinder

A = 8.7 for a plane sheet

*assuming volume diffusion !!*

Combining both terms:

$$\frac{E}{\mathfrak{R} T_c} = \ln \left( \frac{A \mathfrak{R} T_c^2 D_0 / r^2}{E dT / dt} \right)$$

*Harrison et al. (2005)*

This equation can be solved iteratively for a specific **type of mineral** (i.e. a given set of diffusion parameters **E** and **D<sub>0</sub>**) by assuming an appropriate **cooling rate** (dT/dt) and a specific **grain size** (r)!

T<sub>c</sub> = closure temperature

E = activation energy

℞ = gas constant

A = geometry factor

D<sub>0</sub>/r<sup>2</sup> = diffusivity

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### Geometry factors:

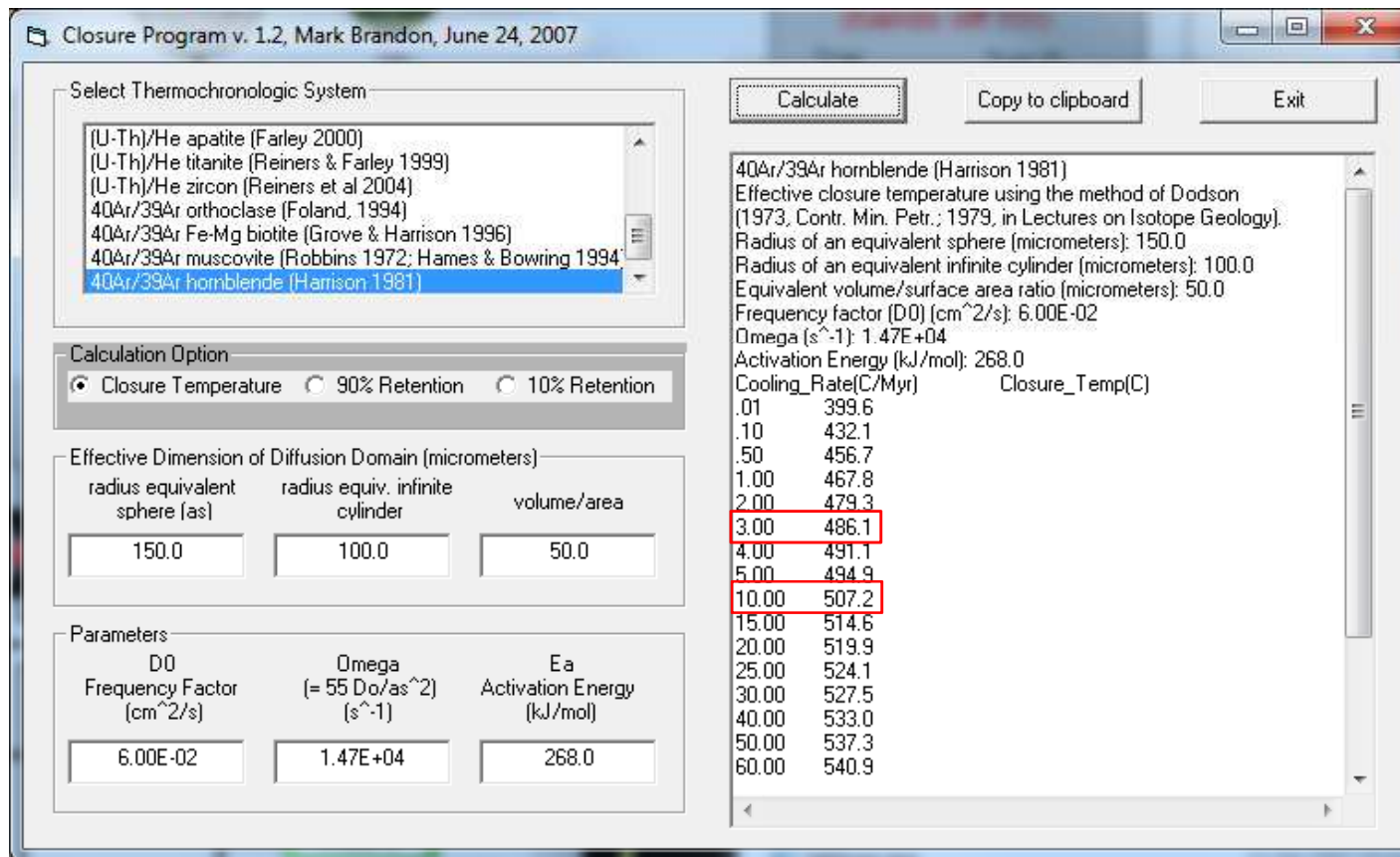
A = 55 for a sphere

A = 27 for a cylinder

A = 8.7 for a plane sheet

*assuming volume diffusion !!*

### Closure temperature calculation by using Mark Brandon's CLOSURE Program



**Example:** Hornblende with  $r = 100 \mu\text{m}$ :  $T_c \sim 486^\circ\text{C}$  at a cooling rate of  $3^\circ\text{C/Ma}$   
 $T_c \sim 507^\circ\text{C}$  at a cooling rate of  $10^\circ\text{C/Ma}$

### Closure temperatures of different minerals

Method	Mineral	Closure Temperature (°C)	Reference
K-Ar	Hornblende	$500 \pm 50$	Harrison (1981)
K-Ar	Muscovite	$350 \pm 50$	Hames and Bowring (1994)
K-Ar	Biotite	$300 \pm 50$	Harrison <i>et al.</i> (1985)
K-Ar	K-feldspar	150 – 350	Lovera <i>et al.</i> (1989)
(U-Th)/He	Zircon	200 – 230	Reiners <i>et al.</i> (2002)
(U-Th)/He	Titanite	150 – 200	Reiners and Farley (1999)
(U-Th)/He	Apatite	$75 \pm 5$	Wolf <i>et al.</i> (1998)
Fission track	Zircon	$240 \pm 20$	Brandon <i>et al.</i> (1998)
Fission track	Titanite	265 – 310	Coyle and Wagner (1998)
Fission track	Apatite	$110 \pm 10$	Gleadow and Duddy (1981)

### More closure temperatures...

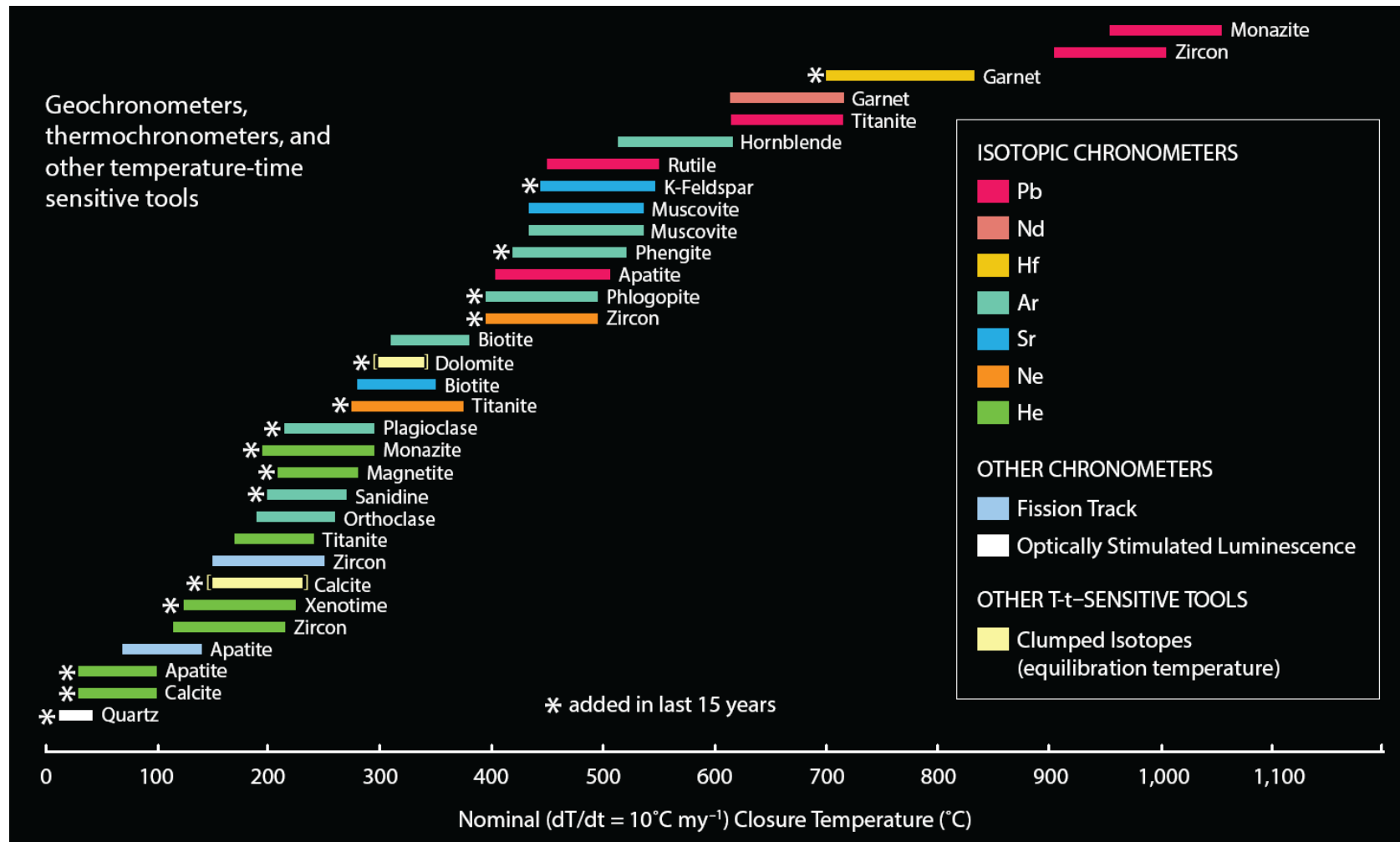
From Reiners et al., 2005, *Rev. Mineral.*, 58, 1-18

**Table 1.** Summary of commonly used thermochronometers and features.

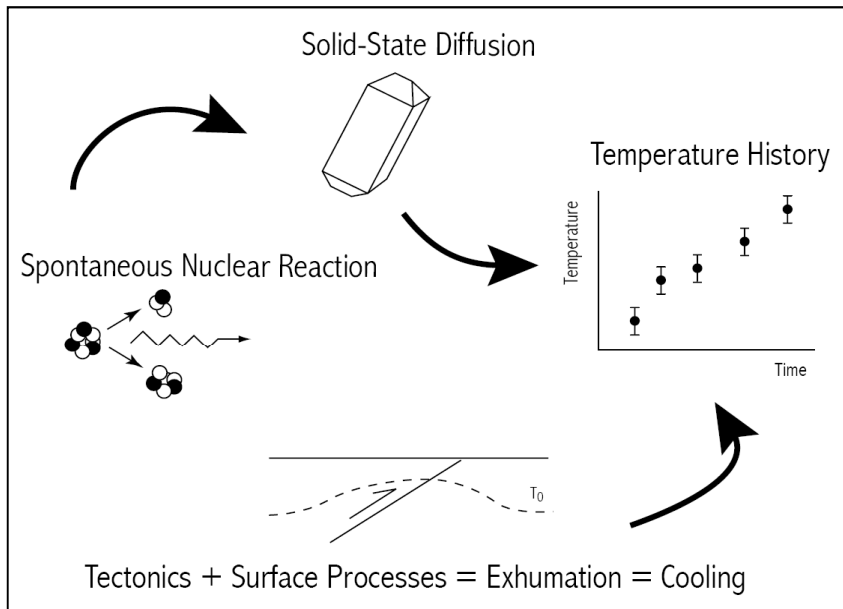
<i>Decay System</i>	<i>Mineral</i>	<i>Approximate precision (%<math>\sigma</math>)</i>	<i>Closure Temperature (<math>^{\circ}</math>C)</i>	<i>Activation Energy (kJ/mol)</i>	<i>References</i>
(U-Th)/Pb	zircon	1–2	>900	550	Cherniak and Watson (2001); Cherniak (2001)
	titanite	1–2	550–650	330	Cherniak (1993)
	monazite	1–2	~700	590	Cherniak et al. (2004)
	apatite	1–2	425–500	230	Chamberlain and Bowring (2001); Cherniak et al. (1991)
$^{40}\text{Ar}/^{39}\text{Ar}$	hornblende	1	400–600	270	Harrison (1981); Dahl (1996)
	biotite	1	350–400	210	Grove and Harrison (1996); Harrison et al. (1985)
	muscovite	1	300–350	180	Robbins (1972); Hames and Bowring (1994)
	K-feldspar	1	150–350	170–210	Foland (1994); Lovera et al. (1991; 1997)
Fission-track	titanite	6	(a) 240–300 (b) 380–420	440–480	(a) Coyle and Wagner (1998); (b) Watt and Durrani (1985); Naeser and Faul (1969)
	zircon	6	(a) 330–350 (b) 230	(a) 300–350 (b) 210	(a) Tagami et al. (1998); Rahn et al. (2004) (b) Brandon and Vance (1992); Brandon et al. (1998)
	apatite	8	90–120	190	Laslett et al. (1987); Ketcham et al. (1999)
(U-Th)/He	titanite	3–4	160–220	190	Reiners and Farley (1999)
	zircon	3–4	160–200	170	Reiners et al. (2004)
	apatite	3–4	55–80	140	Farley (2000)

**Note:** Approximate precisions are estimated values for age determinations; for TIMS U/Pb measurements precisions can be considerably better than cited here. Closure temperatures calculated using Dodson (1973) [or, for fission-track, Dodson (1979) using the 50% annealing isopleth (fanning models); also see Brandon et al. (1998)] using typical ranges of grain sizes and cooling rates (1–100  $^{\circ}$ C/m.y.) (small grains/low cooling rate and large grains fast/cooling rate). Also see Hodges (2003) for a similar and more complete compilation.

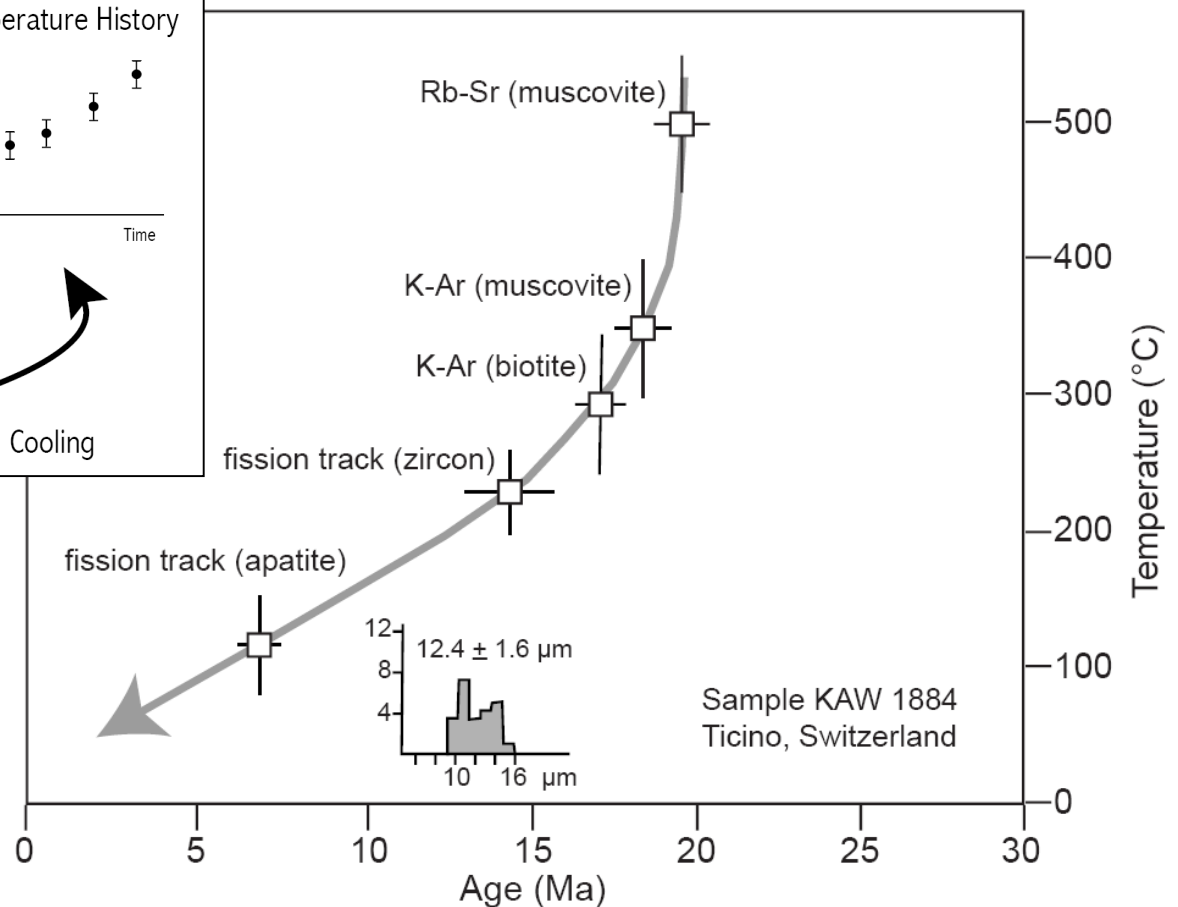
## Closure temperatures of different minerals - graphically



Cooling histories from different coexisting minerals having different closure temperatures: „**Bulk-closure approach**“:

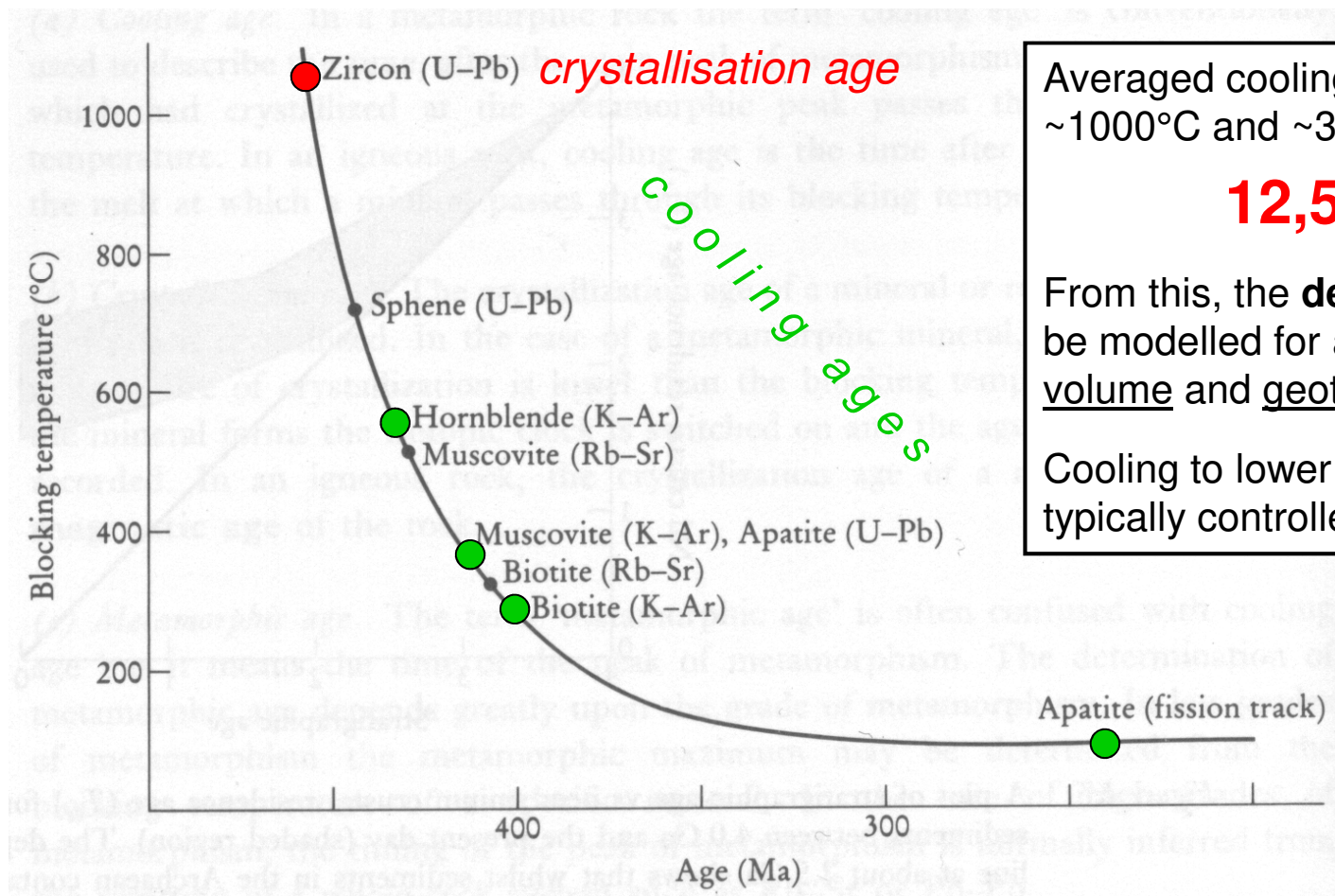


From Braun et al., *Quantitative Thermochronology*



Be aware that all minerals were recovered from **one piece** of rock, i.e. the cooling path is valid for one piece of rock!

**Example:** Cooling rate of a **syenite pluton** determined by dating of different minerals having different closure temperatures for different chronometers



Averaged cooling rate between  
~1000°C and ~300°C:

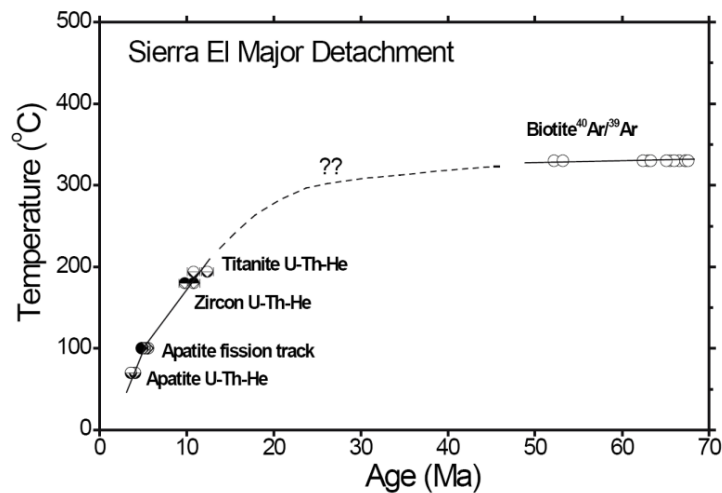
**12,5 °C / Ma**

From this, the **depth of intrusion** can be modelled for a given initial melt volume and geothermal gradient!

Cooling to lower temperatures is typically controlled by **exhumation**!

### Example: Thermal history of a **detachment fault** (NW Baja California, Mexico)

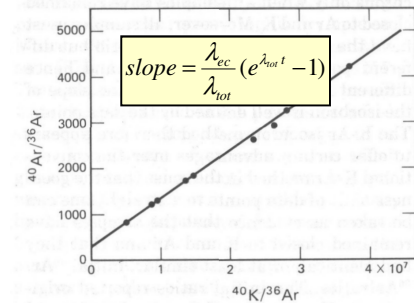
The picture as deduced from Ar-Ar, (U-Th)/He and fission track dating leaves a **significant gap** in the thermal history in the age range from ~50 to ~15 Ma



Applying 'continuous' temperature-time (T-t) monitors, such as K-feldspar multi-diffusion domain thermal modelling (MDD) and fission-track length thermal modelling fills this gap and reveals an unsteady thermal history

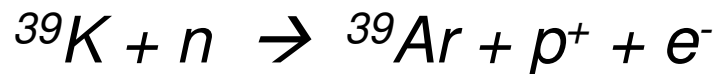
### Problems of the classical K-Ar method:

- Concentrations of  $^{40}\text{Ar}$  and  $\text{K}$  have to be precisely determined in representative sample aliquots – **difficult!**
- No (internal) control on potential argon-loss or -gain during thermal overprints (metamorphism): **one sample = one age**



### Solution to this problem:

- Irradiation of samples in a nuclear reactor to produce  $^{39}\text{Ar}$  by a (n,p) reaction:

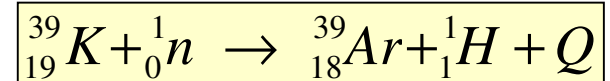


Natural abundance of  $^{39}\text{K} = 93.258\%$

- Amount of  $^{39}\text{Ar}$  produced from  $^{39}\text{K}$ :

$$^{39}\text{Ar}_K = ^{39}\text{K} T \int \phi(E) \sigma(E) dE$$

irradiation time



Picture taken from Research Centre Rez: cvrez.cz

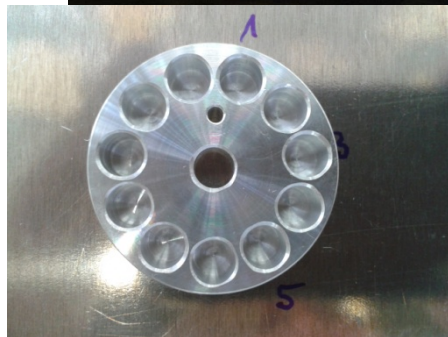
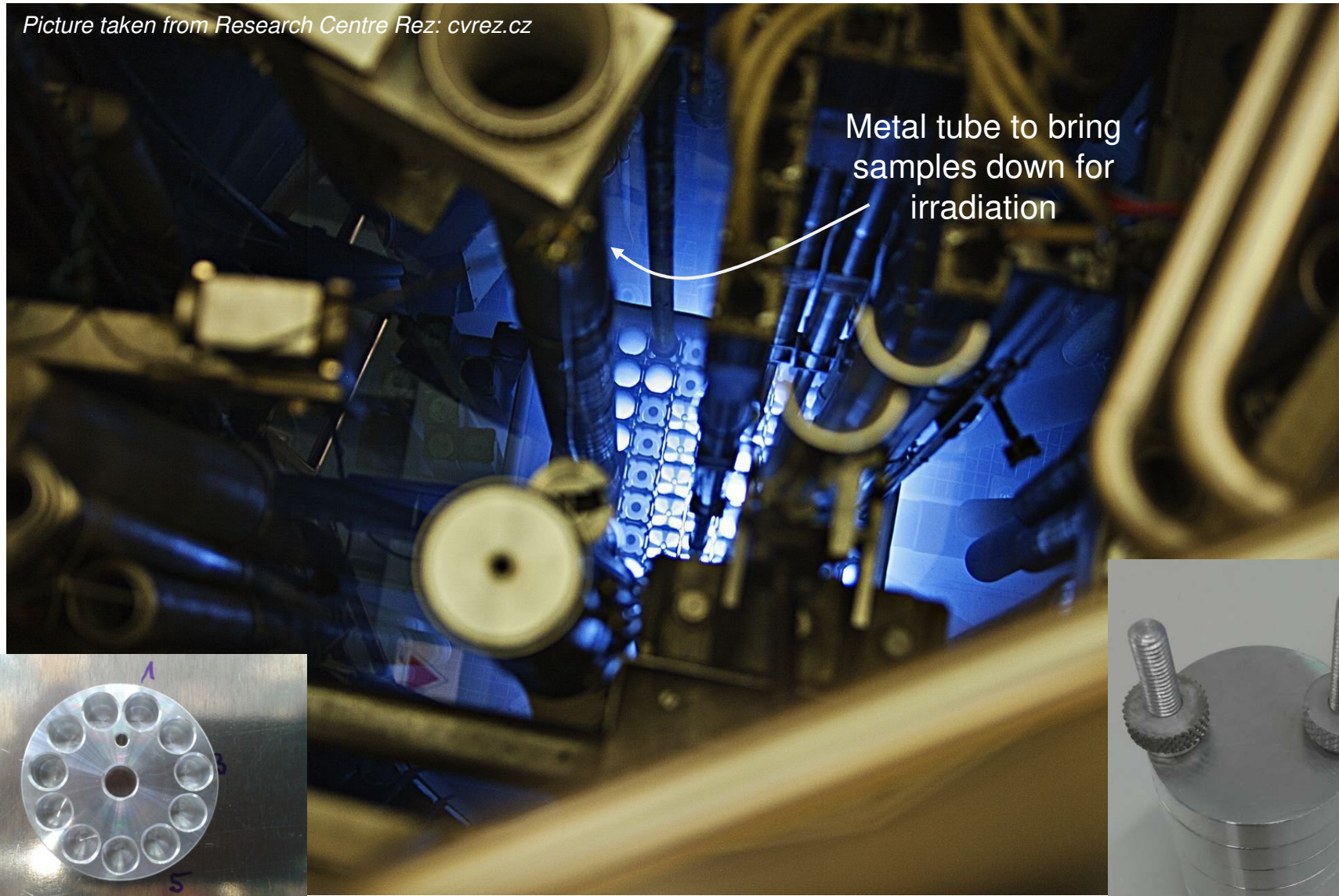
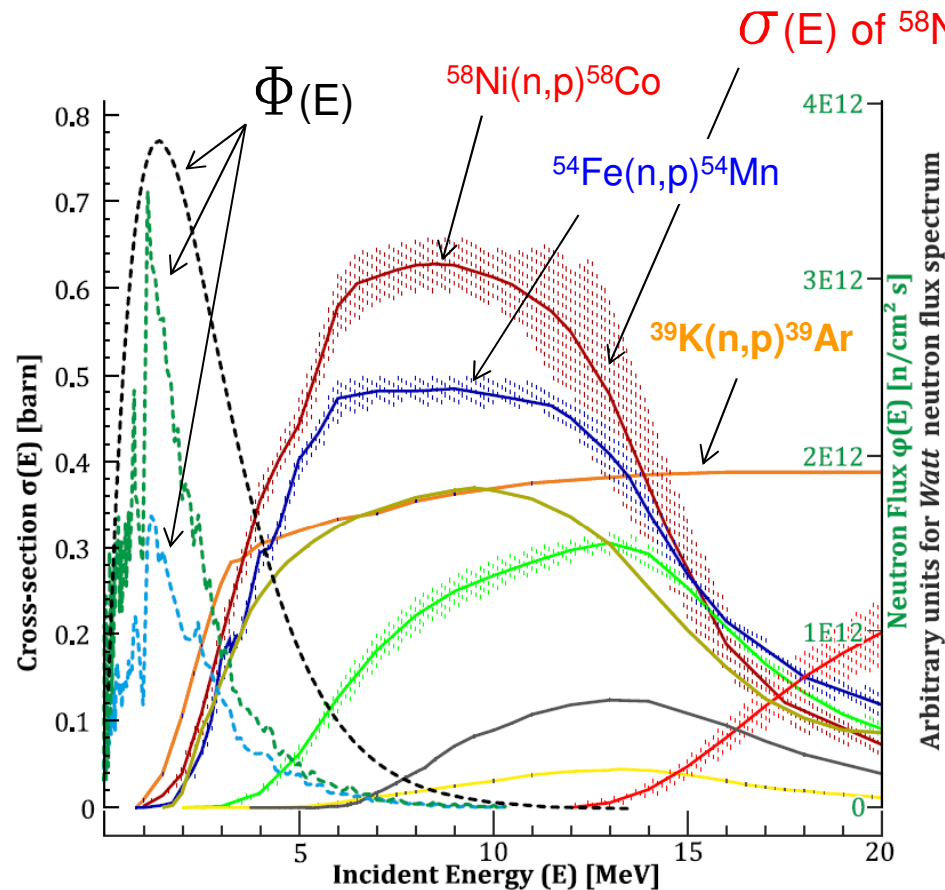


Plate for samples



Batch with samples



$\phi(E)$  calculated neutron-flux spectra in LVR-15:  
 --- FGA012  
 --- FGA014  
 ---  $\phi(E)$   $^{235}\text{U}$  fission neutron flux spectrum [Watt, 1952]

$\sigma(E)$  cross-sections:  
 ---  $^{39}\text{K}(n,p)^{39}\text{Ar}$   
 ---  $^{58}\text{Ni}(n,p)^{58}\text{Co}$   
 ---  $^{27}\text{Al}(n,a)^{24}\text{Na}$   
 ---  $^{32}\text{S}(n,p)^{32}\text{P}$   
 ---  $^{46}\text{Ti}(n,p)^{46}\text{Sc}$   
 ---  $^{47}\text{Ti}(n,p)^{46}\text{Sc}$   
 ---  $^{54}\text{Fe}(n,p)^{54}\text{Mn}$   
 ---  $^{63}\text{Cu}(n,a)^{60}\text{Co}$

$$^{39}\text{Ar}_K = ^{39}\text{K} T \int \phi(E) \sigma(E) dE$$

Reaction **cross sections** [ $\sigma(E)$ ] for different nuclear reactions along with **neutron energy spectra** [ $\Phi(E)$ ] from  **$^{235}\text{U}$  fission**

(calculated using different models and databases)

Some **nuclear reactions** (amongst others!) that **produce Argon isotopes** and typical production ratios:



For the 10 MW reactor (LVR-15) in  
Řež (Czech Republic):

$$(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} \sim 0.000227$$

$$(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} \sim 0.000602$$

$$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} \sim 0.00183$$

*(only slightly variable for different  
reactor types and irradiation positions  
within the reactor)*

## Types of „Argon“ in a sample (terminology):

- Atmospheric argon ( $\mathbf{Ar}_{\text{atm}}$ ):  $^{40}\text{Ar}/^{36}\text{Ar} = 298.6$  (today!)
- Radiogenic argon ( $^{40}\text{Ar}^*$ ): From natural  $^{40}\text{K}$ -decay
- Trapped (inherited) argon: has an *atmospheric* or *excess* composition or is a *mixture* of both
- Cosmogenic argon: Ar isotopes produced in *extraterrestrial rocks* by cosmic ray from other elements (e.g. Ca, Ti, Fe)
- Irradiation induced argon: Argon produce by neutronen-irradiation in a nuclear reactor (from K, Ca, Cl)

## Argon extraction, cleaning & measurement



*View of the Argonlab Freiberg (photo taken by Eva Enkelmann)*

Principle steps to obtain **Ar isotope data** from an **irradiated sample**:

- Thermal release of argon (and other gases) from mineral/rock
- Gas cleaning, i.e. sorption/freezing of all but the noble gases
- Measurement of argon isotope abundances in a gas mass spec

## Argon (gas) extraction: Thermal CO<sub>2</sub> laser system

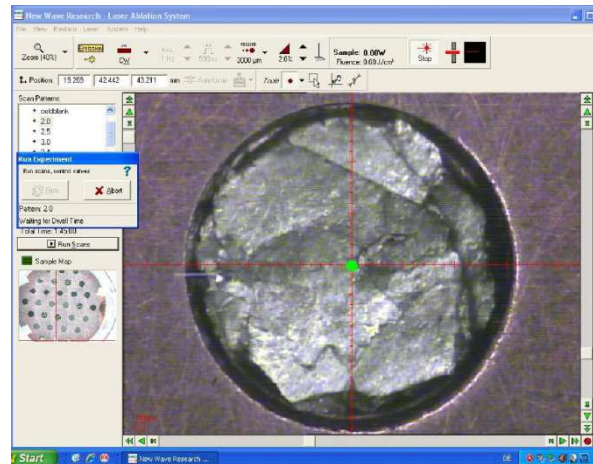
### CO<sub>2</sub> laser specifications:

*Wavelength: 10.6  $\mu\text{m}$*

*Energie: 30 Watts*

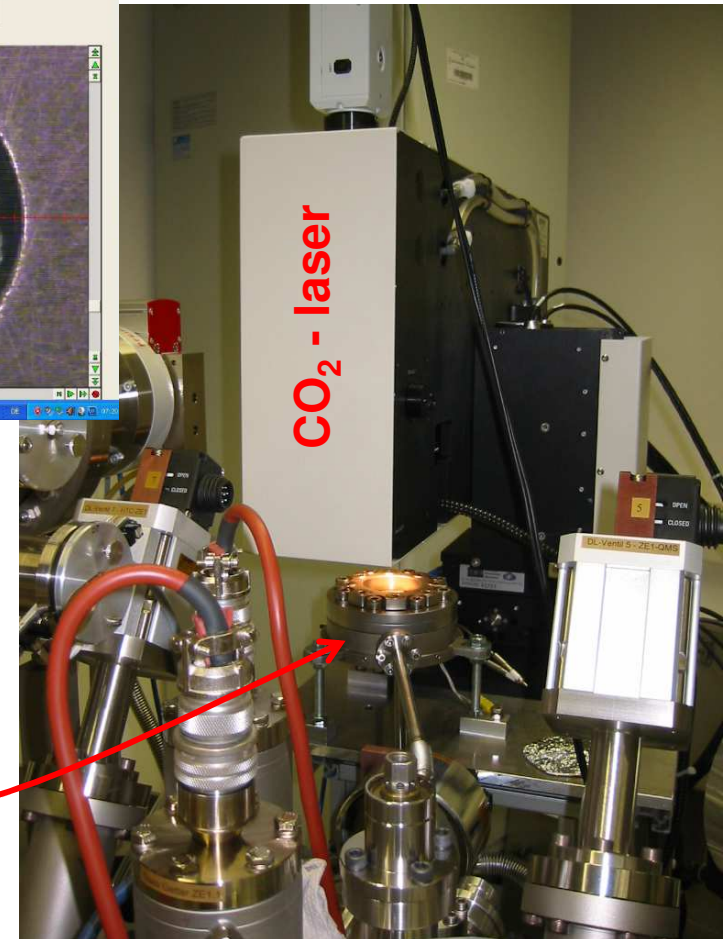
*Spot size: 200 – 3000  $\mu\text{m}$*

*Slightly glowing large mica flakes in a 3 mm hole seen through the video system of the laser* →



**Step wise degassing  
(heating) by a step wise  
increase of the laser power**

*Laser window &  
sample chamber*

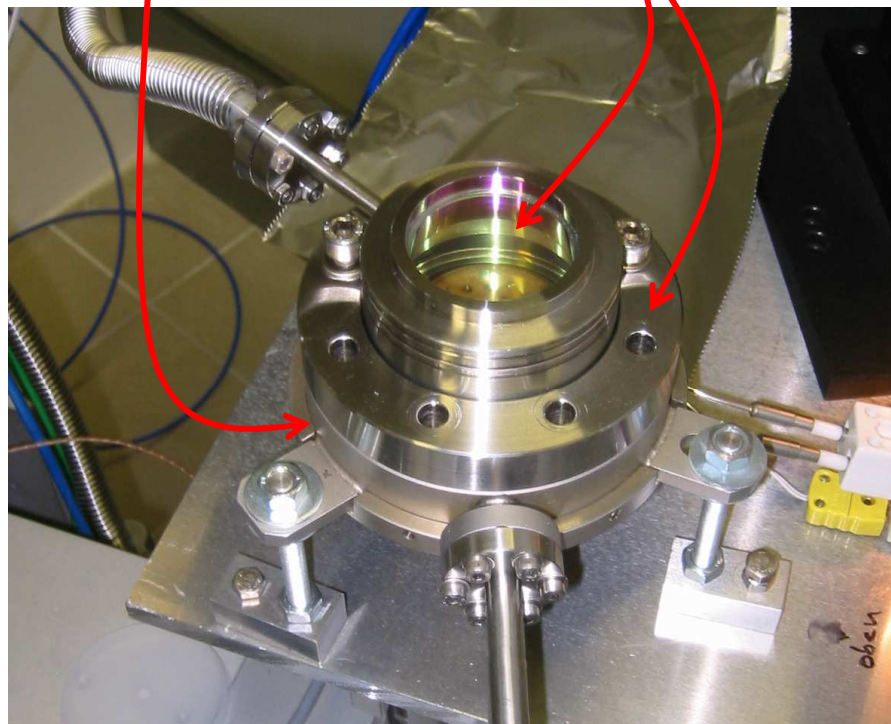


## Argon (gas) extraction

### Laser sample chamber

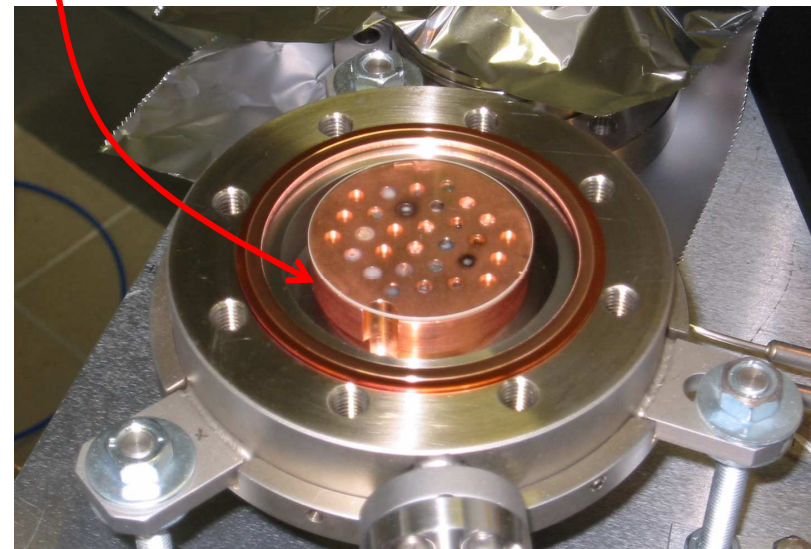
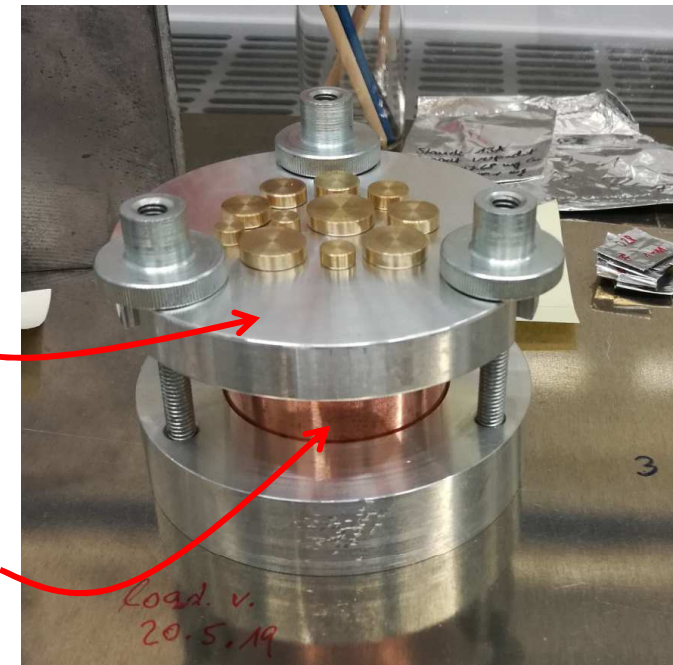
Sample chamber

10.6  $\mu\text{m}$  transparent ZnS window

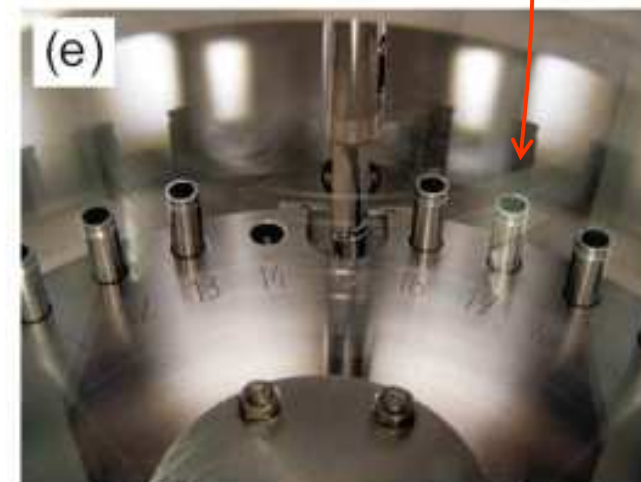
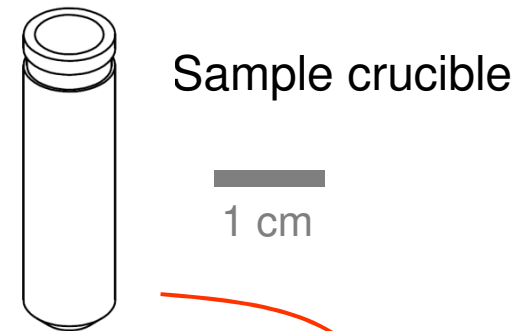
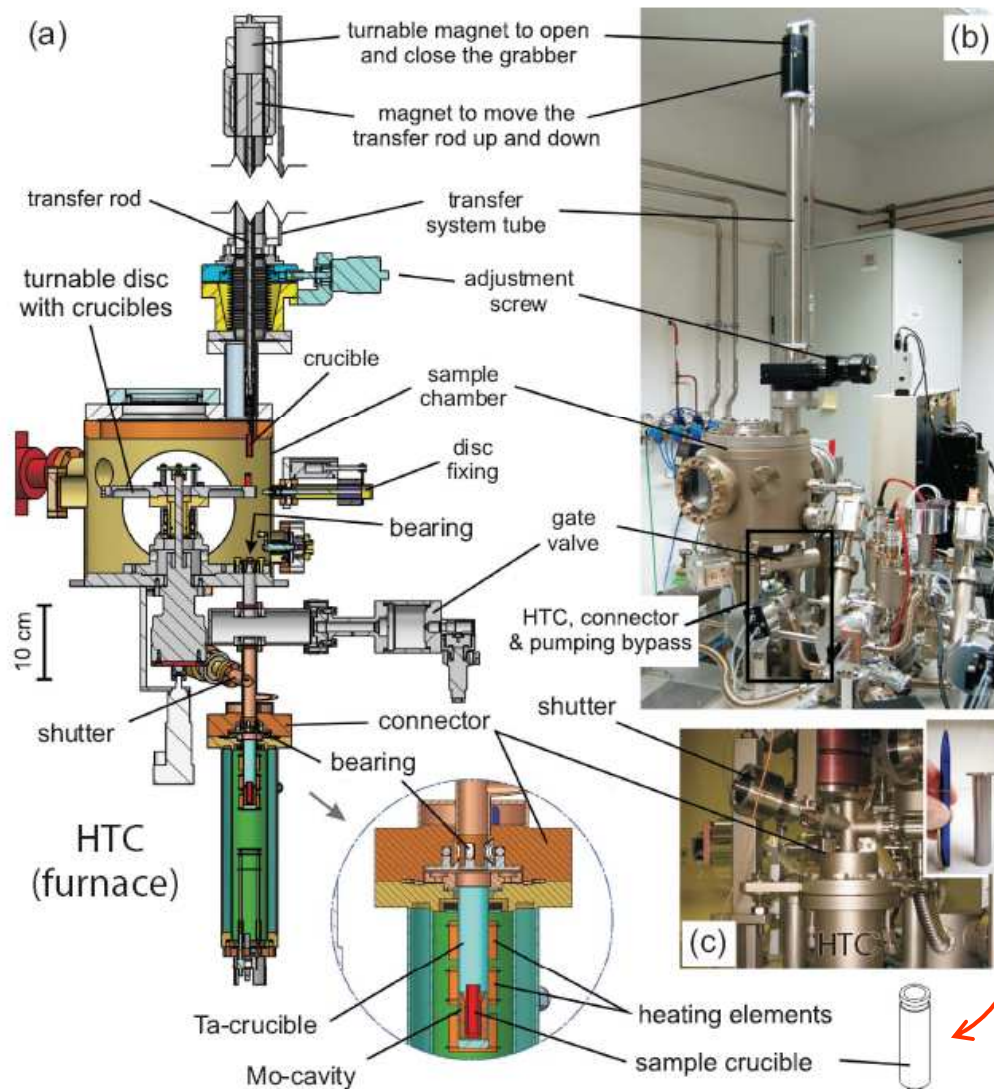


Loading device

Sample holder



## Argon (gas) extraction: Resistance furnace (HTC)



## Gas cleaning

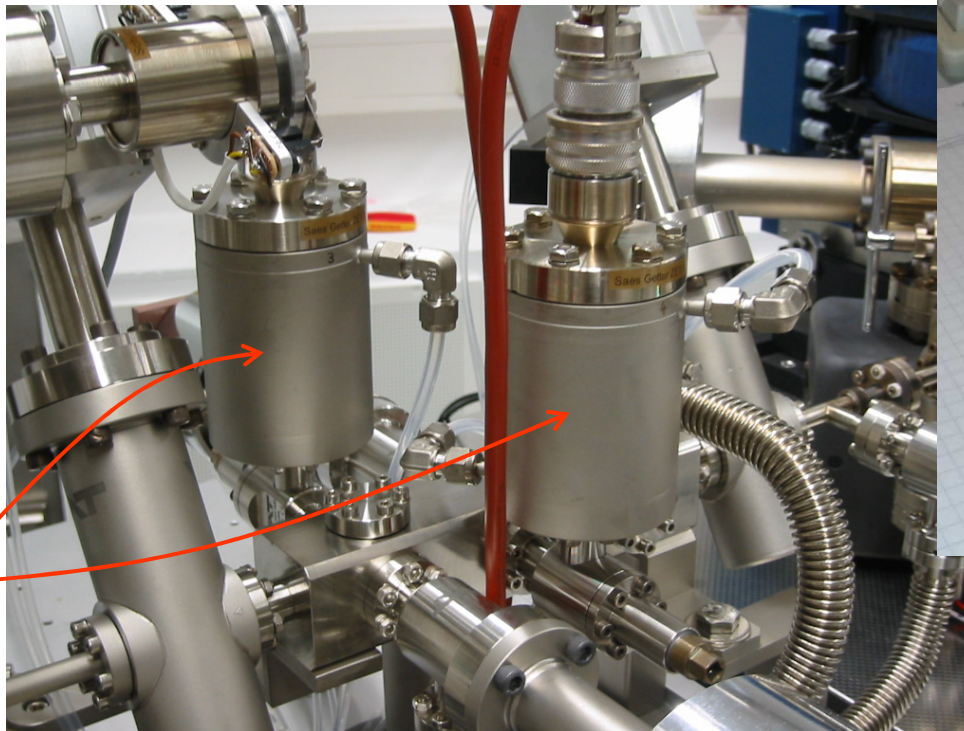
„Getters“ made from a Zirconium metal alloy were used for gas cleaning: Ab-/Adsorption of active gases

One operates at 400°C, one at room temperature

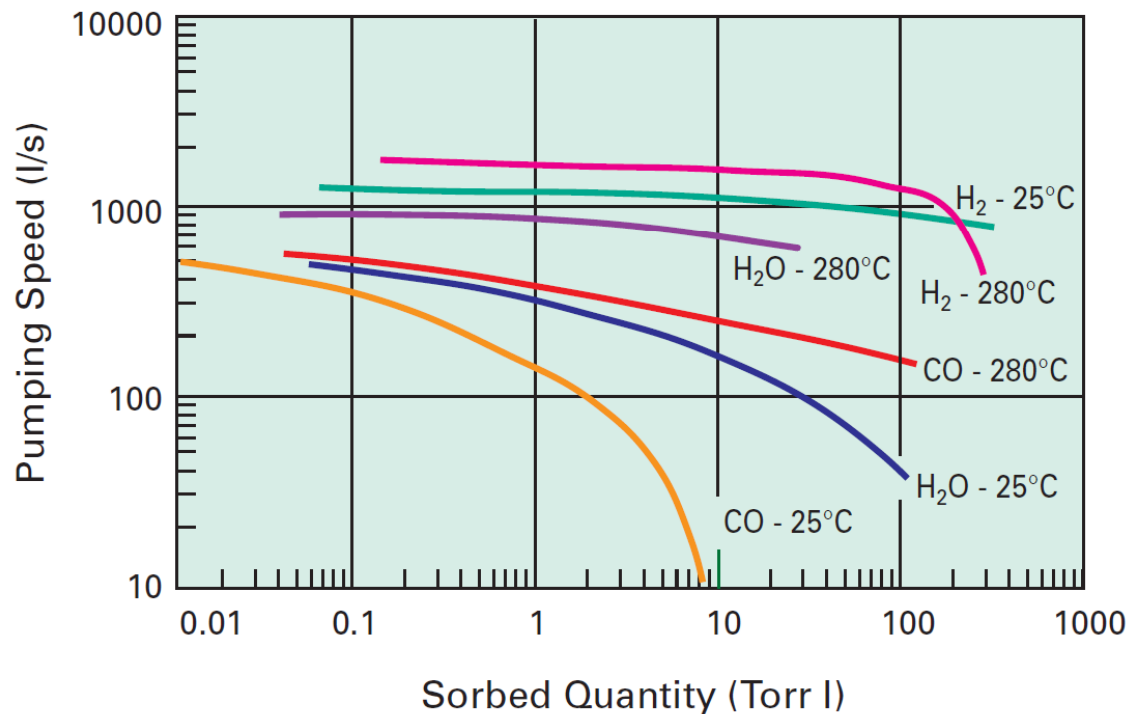
*Zirconium alloy  
getter*



*Water cooled  
getter housings*



## Gas cleaning: Pumping speed vs. sorbed quantity for St101 getter material (Zr-Al alloy)



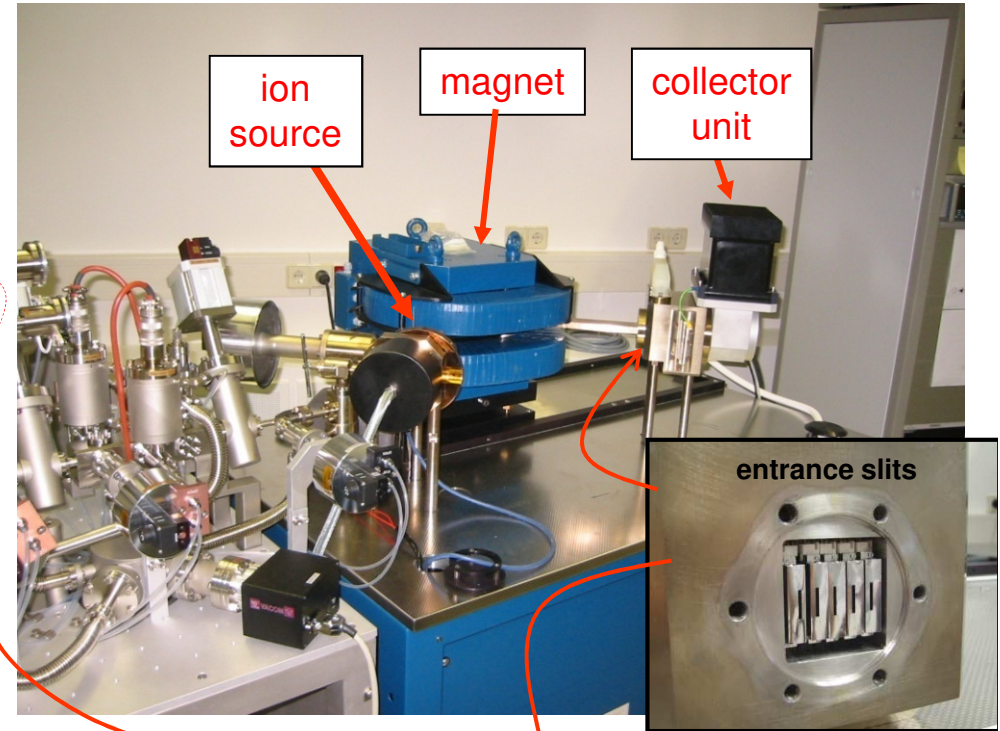
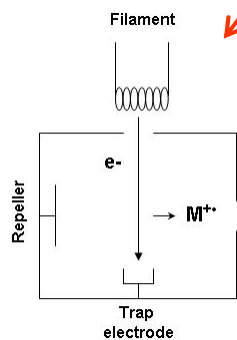
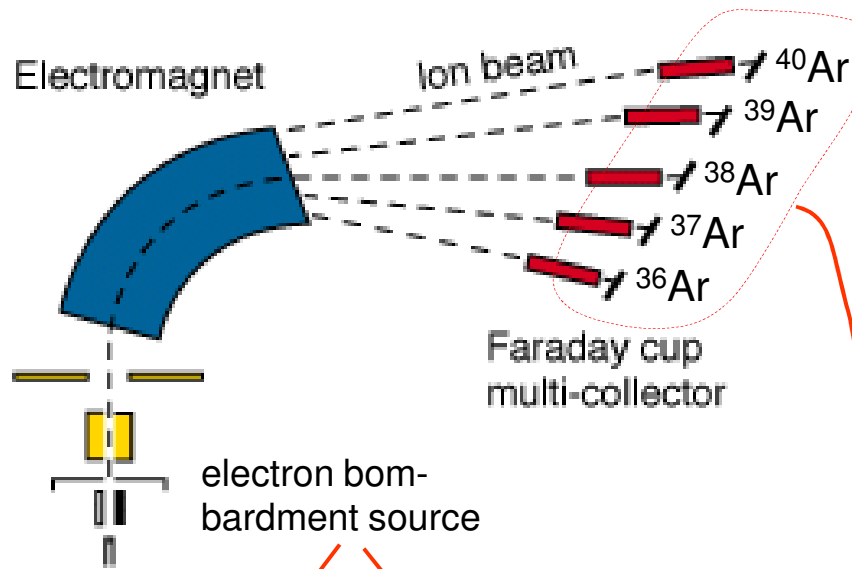
getter alloy having a large surface

*From SAES webpages*

*Most (active) gases (CO, CO<sub>2</sub>, NO<sub>x</sub>, N<sub>2</sub>, H<sub>2</sub>, ...) are sorbed, inert noble gases not!*

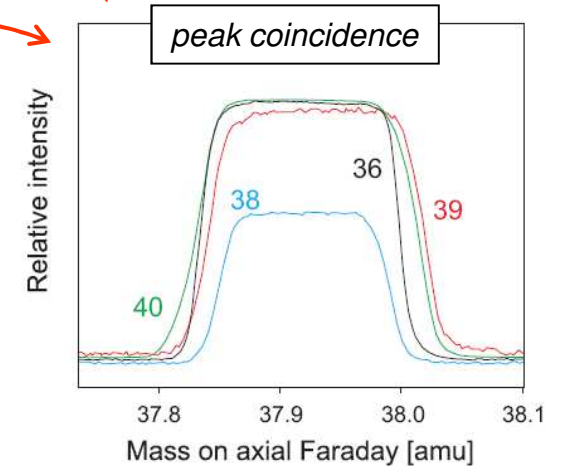
## Argon isotope measurement

All five Ar-isotopes (mass 36 – 40) were measured simultaneously



$$r = \frac{1439}{H} \sqrt{\frac{m}{e} U}$$

$r$  = radius of ion flight path [mm]  
 $H$  = magnetic field strength [Gs]  
 $m$  = mass [amu]  
 $e$  = charge units  
 $U$  = acceleration voltage [V]



How can we calculate an **age** from the **measured Ar isotope composition** of an **irradiated sample**?

Age equation from decay law:

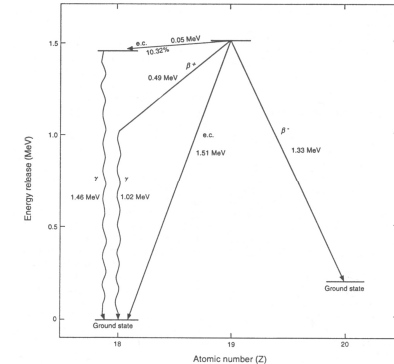
$$t = \frac{1}{\lambda} \ln \left( 1 + \frac{D}{N} \right) \quad (1)$$

$t$  = age of the sample

$\lambda$  = decay constant

$D$  = number of daughter isotopes (atoms) today

$N$  = number of parent isotopes (atoms) left (i.e. today)



Quantity of  $^{40}\text{K}$  that decays to  $^{40}\text{Ar}$  (~10.5%):

$$f = \frac{\lambda_{ecI} + \lambda_{ecII} + \lambda_{\beta}}{\lambda} \quad (2)$$

with

$$\lambda = \lambda_{ecI} + \lambda_{ecII} + \lambda_{\beta}$$

Yielding

$$t = \frac{1}{\lambda} \ln \left( 1 + \frac{{}^{40}\text{Ar}^*}{f \times {}^{40}\text{K}} \right) \quad (3)$$

which is:

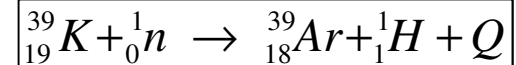
$$t = \frac{1}{\lambda} \ln \left( 1 + 9.54 \frac{{}^{40}\text{Ar}^*}{{}^{40}\text{K}} \right) \quad (4)$$

Age equation of the K-Ar method

How can we get  $^{40}\text{K}$  from measured  $^{39}\text{Ar}$ ?

Using the natural isotope abundances:

$$^{40}\text{K} = 0.000125 \times ^{39}\text{K} \quad (5) \quad (\text{natural } ^{40}\text{K}/^{39}\text{K} = 0.000125)$$



Quantity of  $^{39}\text{Ar}$  produced by neutron irradiation from  $^{39}\text{K}$  :

$$^{39}\text{Ar}_K = ^{39}\text{K} T \int \phi(E) \sigma(E) dE \quad (6)$$

$T$  = duration of irradiation

$\Phi$  = neutron flux (neutrons per  $\text{cm}^2 \times \text{sec}$ )

$\sigma$  = effective cross section of  $^{39}\text{K}(n,p)^{39}\text{Ar}$  reaction

$E$  = Energy of the neutrons

$J'$

„Conversion factor“

Combining yields:

$$^{40}\text{K} = 0.000125 \times \frac{^{39}\text{Ar}_K}{J'} \quad (7)$$

$$t = \frac{1}{\lambda} \ln \left( 1 + 9.54 \frac{^{40}\text{Ar}^*}{0.000125 \times (^{39}\text{Ar}_K / J')} \right) \quad (8)$$

Combining all constants yields the  **$^{40}\text{Ar}/^{39}\text{Ar}$  age equation**:

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

where the constant **J** contains the **abundances of the K isotopes**, the **decay constants (i.e. the branching ratio)** and the **neutron fluence, reaction cross section** and the **duration of irradiation** and is commonly termed **„irradiation parameter“** or **„J-value“**

**J can not be calculated precisely – therefore, it needs to be determined by using samples (minerals) with known ages („fluence monitors“)** that were co-irradiated with the samples

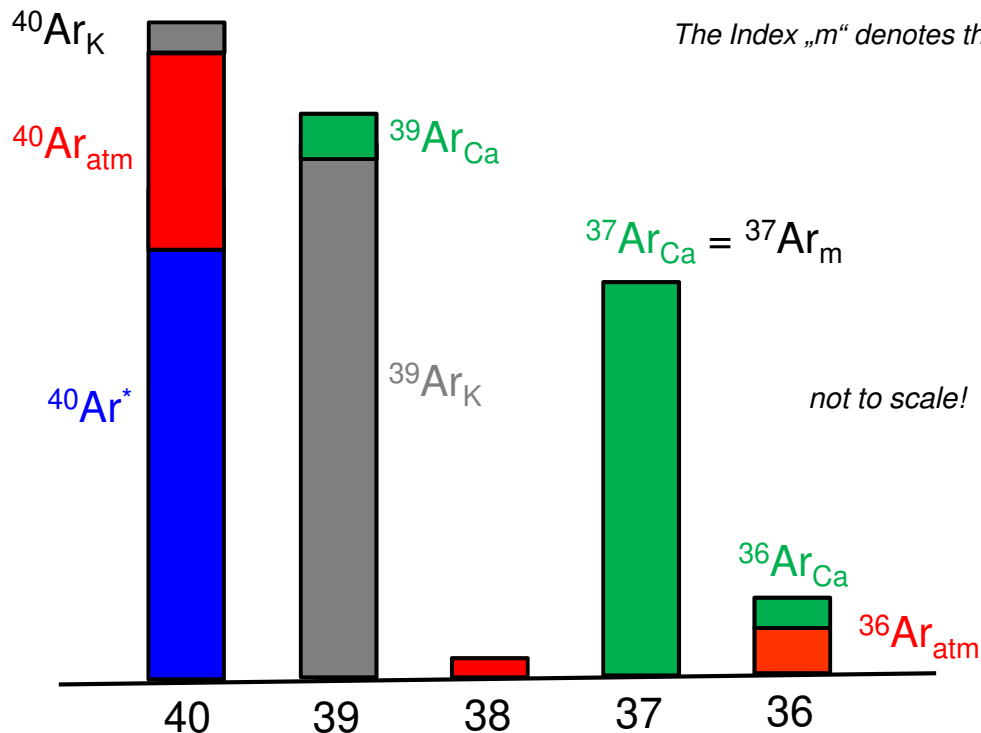
## How to determine $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ? Simplified version:

$$^{36}\text{Ar}_{\text{atm}} = ^{36}\text{Ar}_m - ^{37}\text{Ar}_m \times (^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$$

$$^{40}\text{Ar}^* = ^{40}\text{Ar}_m - 298.6 \times ^{36}\text{Ar}_{\text{atm}} - ^{39}\text{Ar}_K \times (^{40}\text{Ar}/^{39}\text{Ar})_K$$

$$^{39}\text{Ar}_K = ^{39}\text{Ar}_m - ^{37}\text{Ar}_m \times (^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$$

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

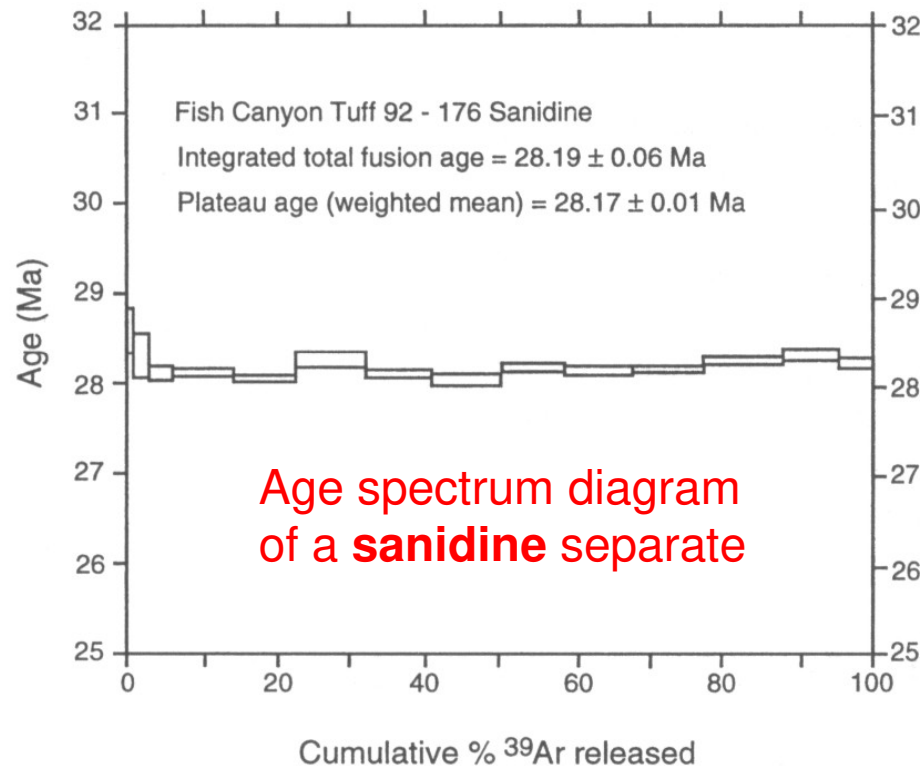


$$(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} \sim 0.000227$$

$$(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} \sim 0.000602$$

$$(^{40}\text{Ar}/^{39}\text{Ar})_K \sim 0.00183$$

### Data presentation in $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology (step heating)



1) For **each** temperature step, an **age** is calculated using:

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

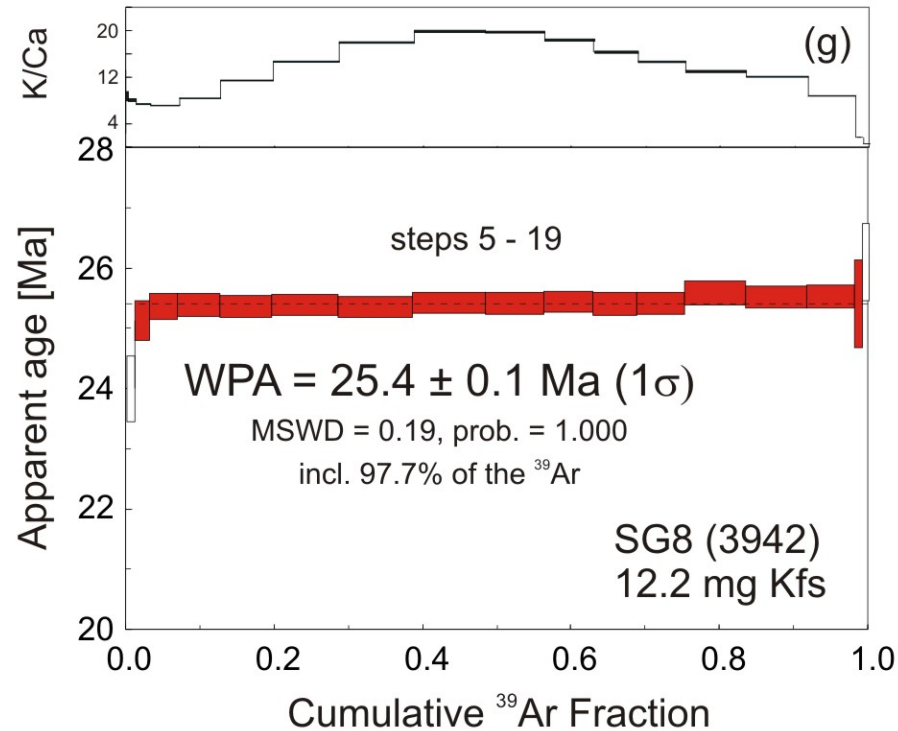
2) For the **plateau steps** (if any), a **Weighted Mean Age/Average** (WMA or WPA) is calculated using:

$$WMA = \frac{\sum_{i=1}^n \frac{1}{\sigma_i^2} t_i}{\sum_{i=1}^n \frac{1}{\sigma_i^2}}$$

3) The **goodness of the plateau age** is quantified by calculating the **Mean Square of Weighted Deviates**:

$$MSWD = \frac{1}{n-1} \sum_{i=1}^n \frac{\Delta t^2}{\sigma_i^2}$$

### Data presentation in $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology (step heating)



Age spectrum diagram yields:

Total gas age (TFA) = 25.4 Ma  
**Plateau age = 25.4 ± 0.1 Ma**

The advantages of the Ar-Ar method over the K-Ar method are therefore:

> **One sample = many ages!**

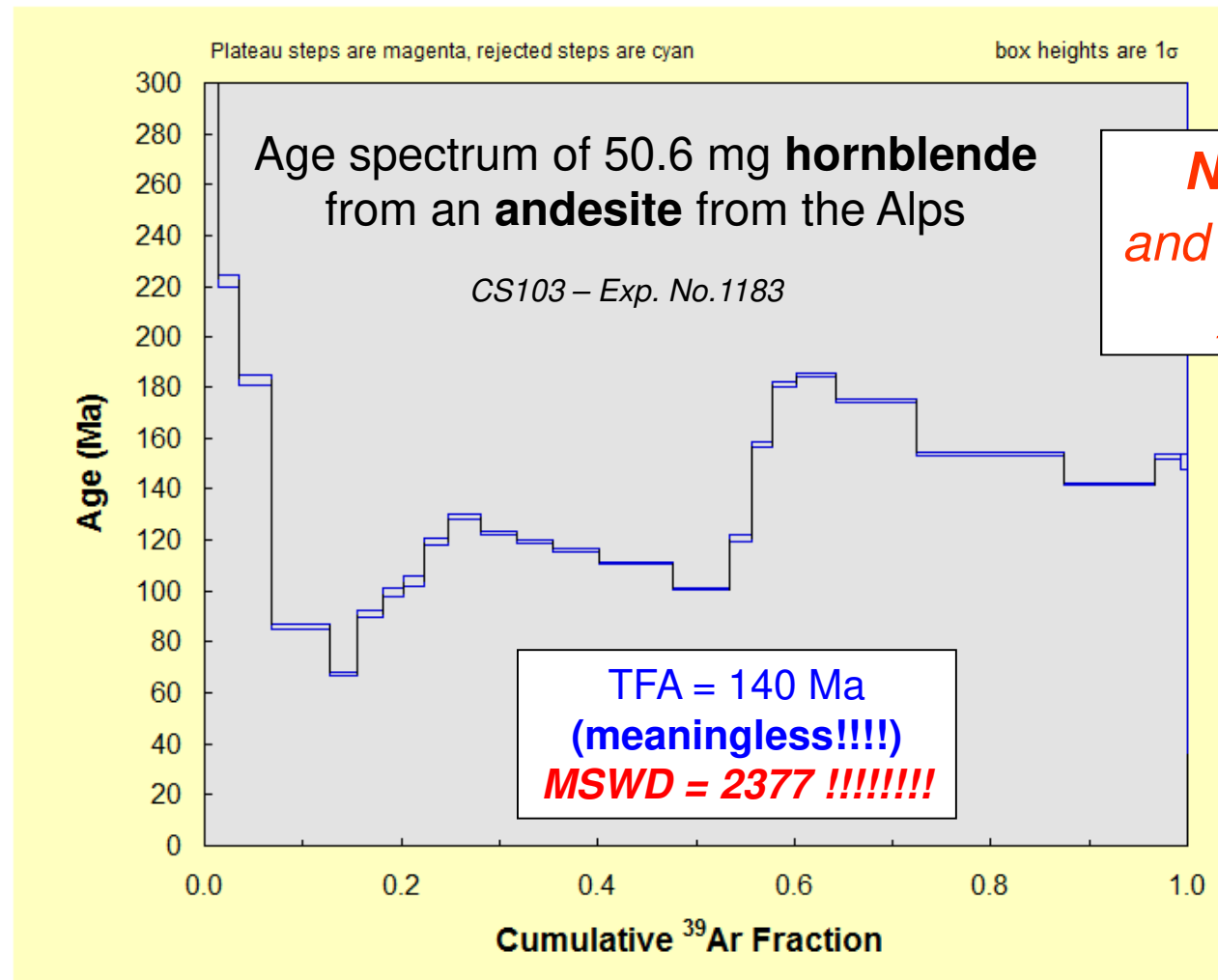
*Step-wise degassing of a sample provides information about potential „artificial“ argon not related to radioactive ingrowth, or about Ar-loss / K-gain, etc. ....!*

> **„Easy“ to measure ...**  
*(everything is relative!)*

Age spectrum of **12.2 mg** sanidine from a **latite** from the Siebengebirge Volcanic Province, Central Germany (*Przybyla et al., 2018*)

### Data presentation in Ar-Ar geochronology (step heating)

Complex release pattern of a hornblende (excess argon and thermal overprint, i.e. Ar loss)

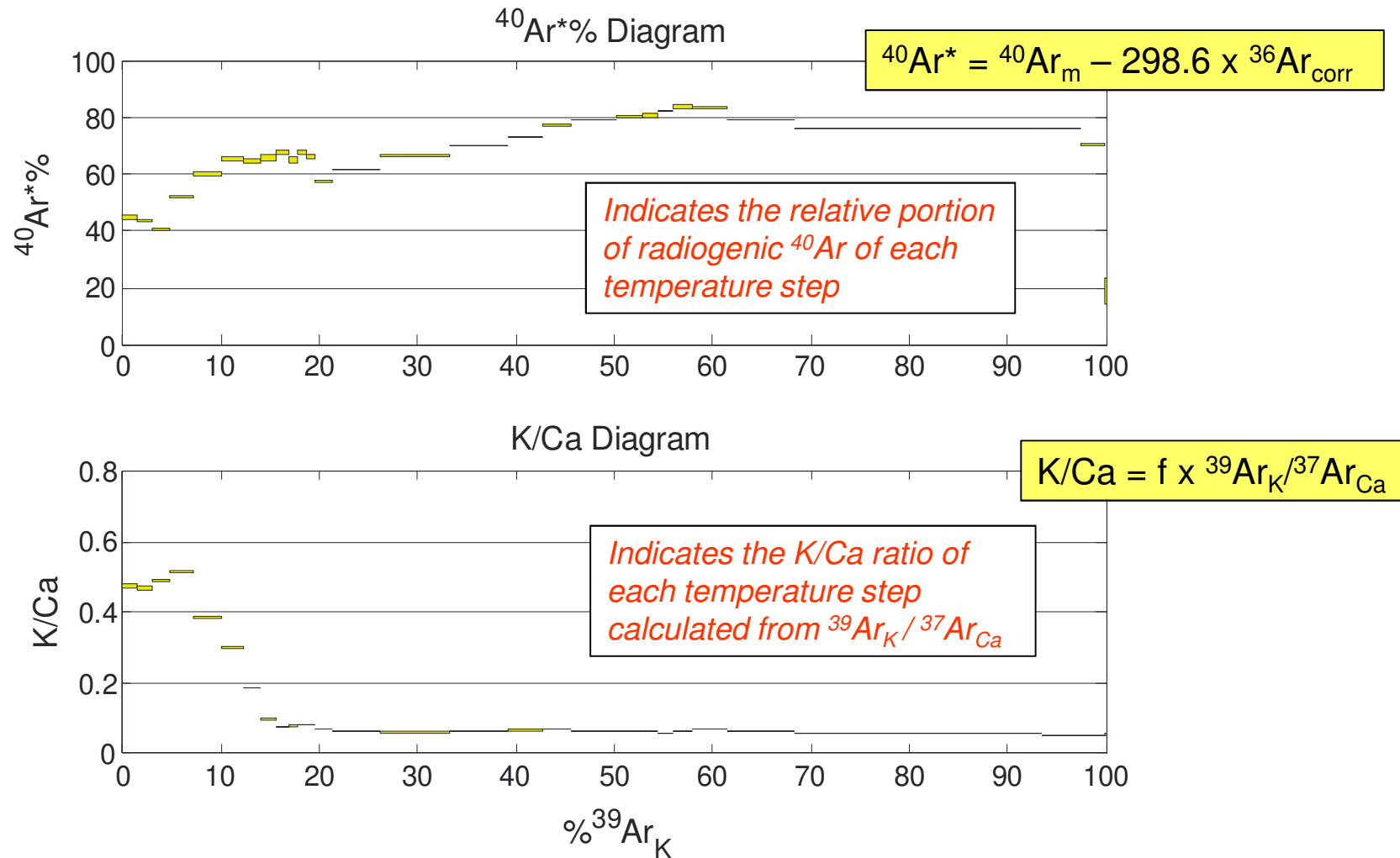


**NO PLATEAU,  
and thus in this case  
„NO AGE“ !**

**K-Ar dating would  
not resolve this  
circumstance but  
instead would  
provide a completely  
meaningless age!!!**

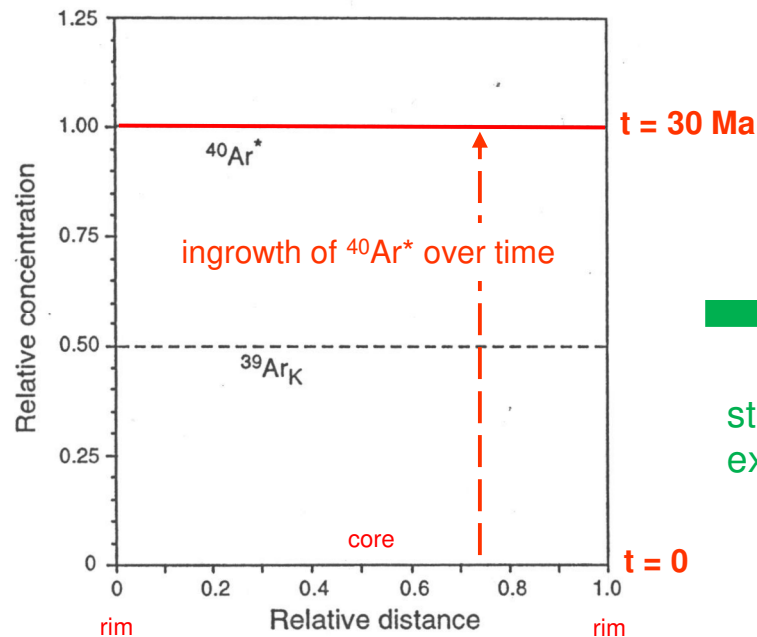
### Data presentation in Ar-Ar geochronology (step heating)

Amount of  $^{40}\text{Ar}^*$  and K/Ca ratio of individual temperature steps



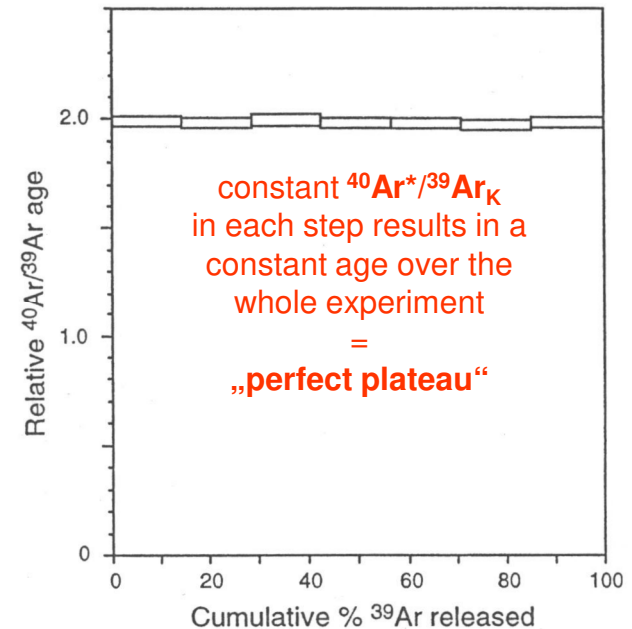
### Degassing behaviour of an undisturbed sample

Concentration profile within a mineral



step heating  
experiment

Resulting age spectrum



Evolution of radiogenic  $^{40}\text{Ar}$  over time

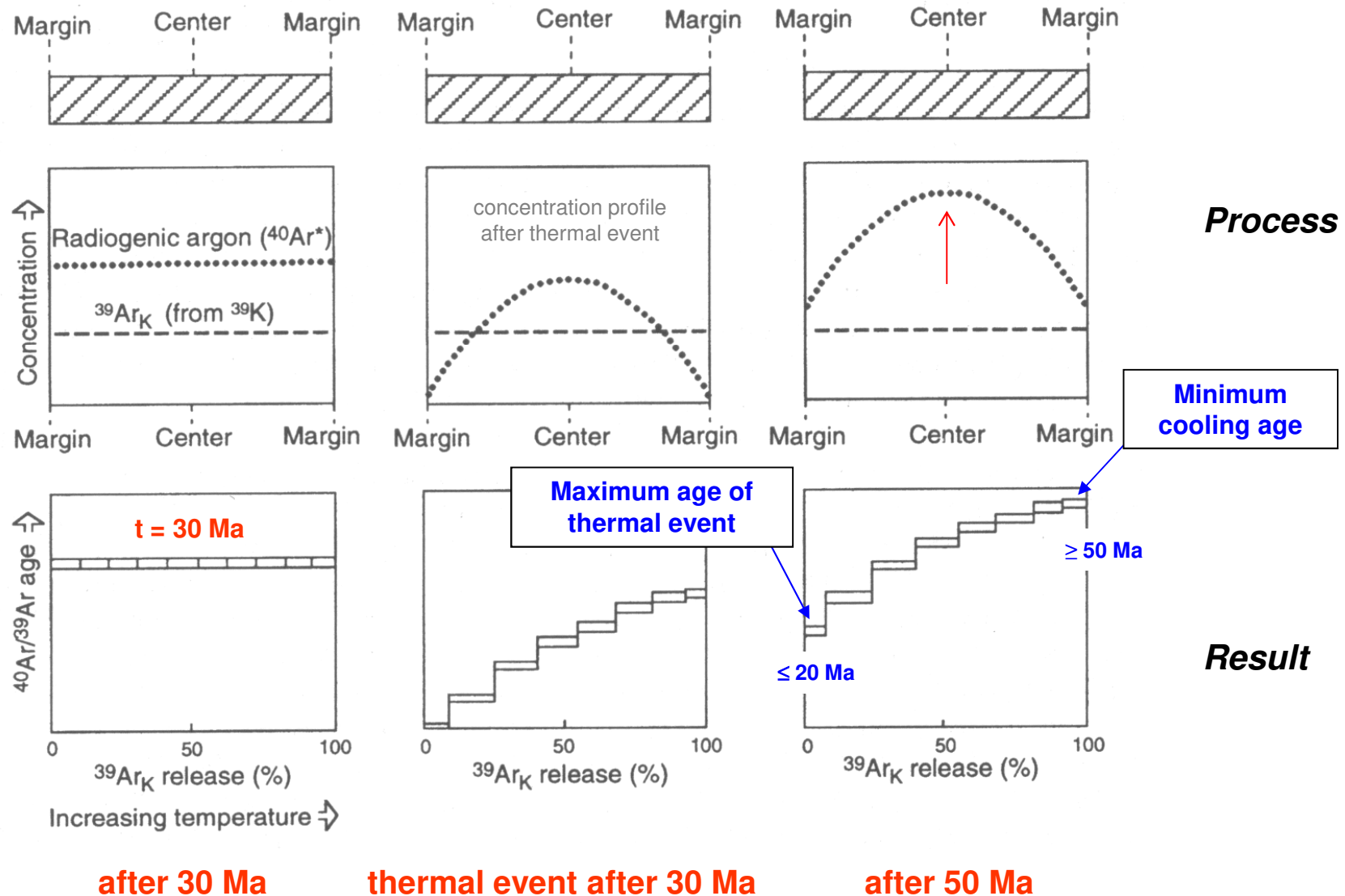
Undisturbed age spectrum

For (thermally) **undisturbed pure minerals**, the released gas fractions were expected to have **constant  $^{40}\text{Ar}^*/^{39}\text{Ar}_K$  ratios**, i.e. in an ideal case, no Ar-isotope fractionation does occur during a step-heating experiment

# Ar-Ar Geo-/Thermochronology

Data presentation & interpretation

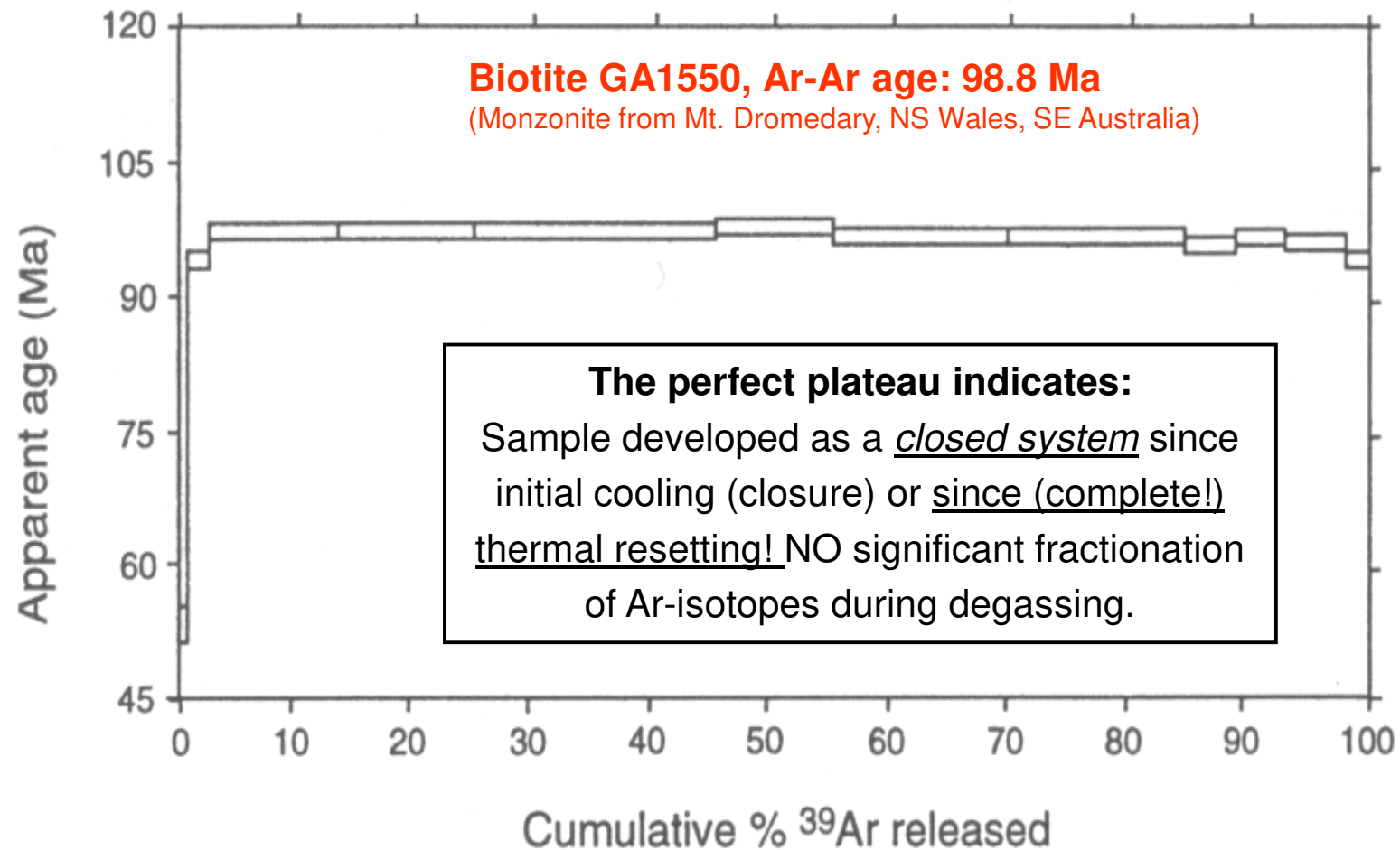
## Degassing behaviour of a thermally disturbed sample



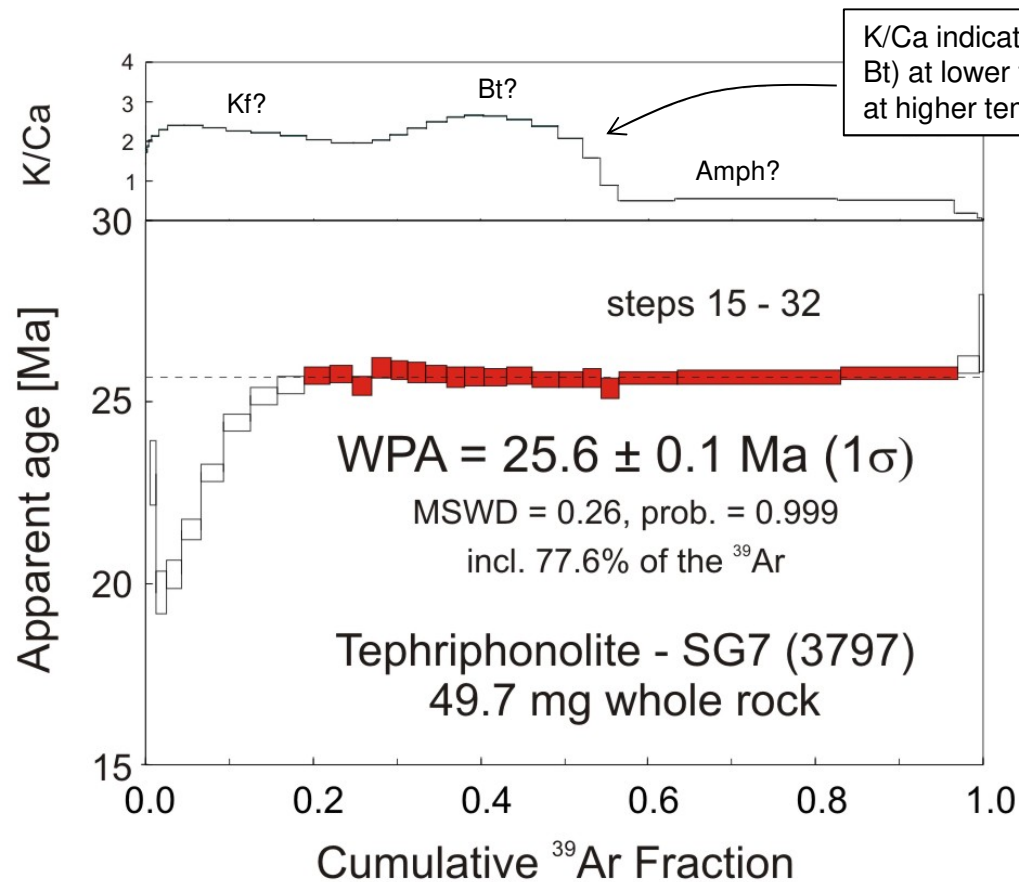
# Ar-Ar Geo-/Thermochronology

Data presentation & interpretation

**Example 1:** Undisturbed age spectrum (minimal loss indicated by first steps)



**Example 2:** Disturbed age spectrum (Argon loss indicated by first steps)



**Age spectrum**  
(34 temperature steps) of  
49.7 mg **whole-rock**  
**fragments** from a tephri-  
phonolite

Note the **partial Ar-loss**  
revealed by the **low**  
**temperature steps**

### Three (of much more!) age plateau definitions:

(or: what is disturbed, what is undisturbed?)

- **>50%  $^{39}\text{Ar}_K$  and no age difference at the 95% confidence level ( $2\sigma$ ) between any two steps** (Fleck et al., 1977)
- **$\geq 5$  contiguous steps** where all (or all except one) agree in age at the 95% confidence level (a single discordant value is acceptable if it represents a small gas fraction; Berger & York, 1981)
- Maximal number of steps where the **first** and **last** step coincide within the  **$2\sigma$  level**, and all steps inbetween coincide with the plateau age at the  **$1\sigma$  level** (Foland et al., 1986)

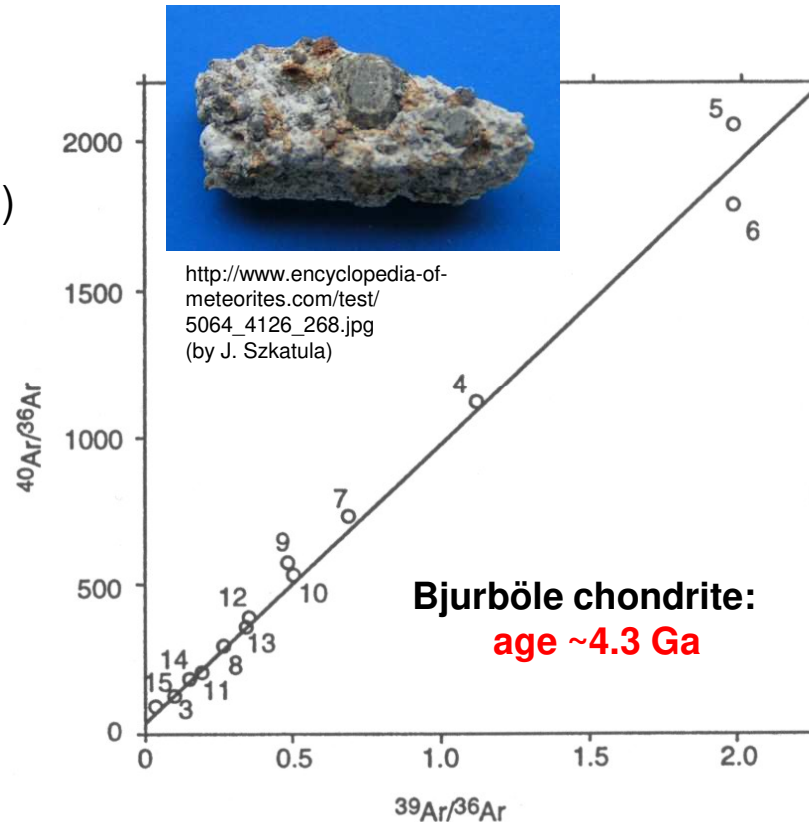
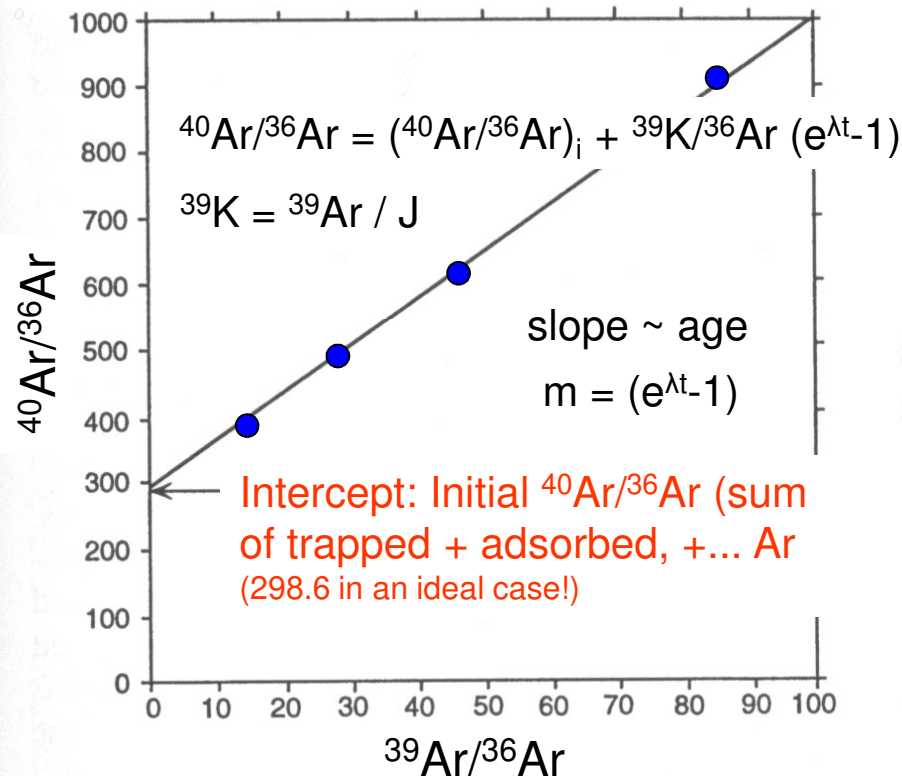
---

**Problems:** Measurement **precision** increased over the years, as did the number of measured steps! **Statistical tests** are model dependent!

**Recommendation:** A plateau can be defined if it represents **more than 50% of  $^{39}\text{Ar}_K$  released** and if the steps are **contiguous** and concordant with the inferred plateau age **within the  $2\sigma$  level** (exceptions will follow...)

### Isochron diagram

(each point represents a single temperature step)

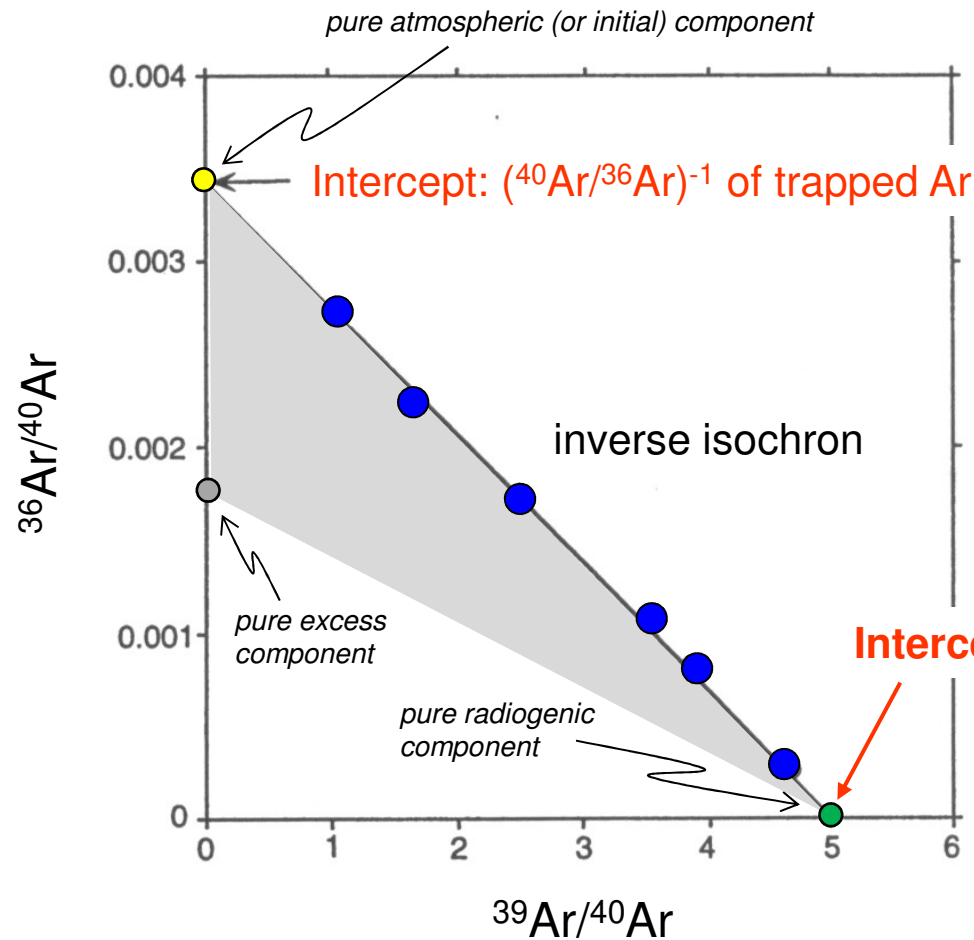


Intercept provides the composition of the „initial“ (trapped) Argon component!

*Problem:* Virtually  $^{36}\text{Ar}$ -free steps and/or samples with high amounts of  $^{40}\text{Ar}^*$  dominate the regression and yield **erroneous initial  $^{40}\text{Ar}/^{36}\text{Ar}$** . *Solution: inverse isochron diagram*

### Inverse isochron diagram

(each *blue* point represents a single temperature step)



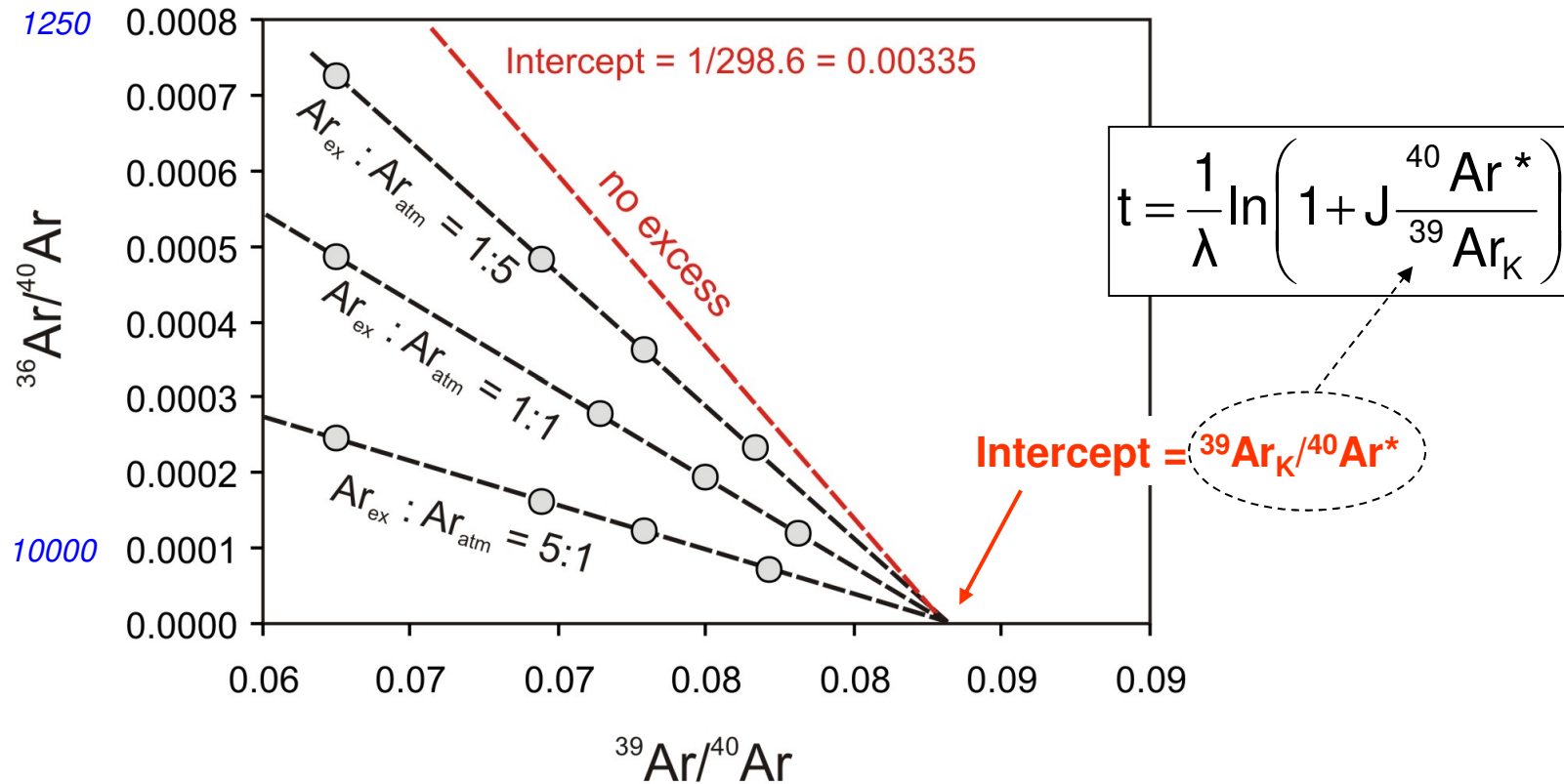
If **more than two** isotopically different **Ar components** are present (e.g. a radiogenic, an excess and an atmospheric component), and if these components are present in variable proportions in **each** step:

**NO LINEAR CORRELATION !!**

Instead, the scatter then indicates a **three- or multi-component** isotope mixture!

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

### Three component isotope mixing:

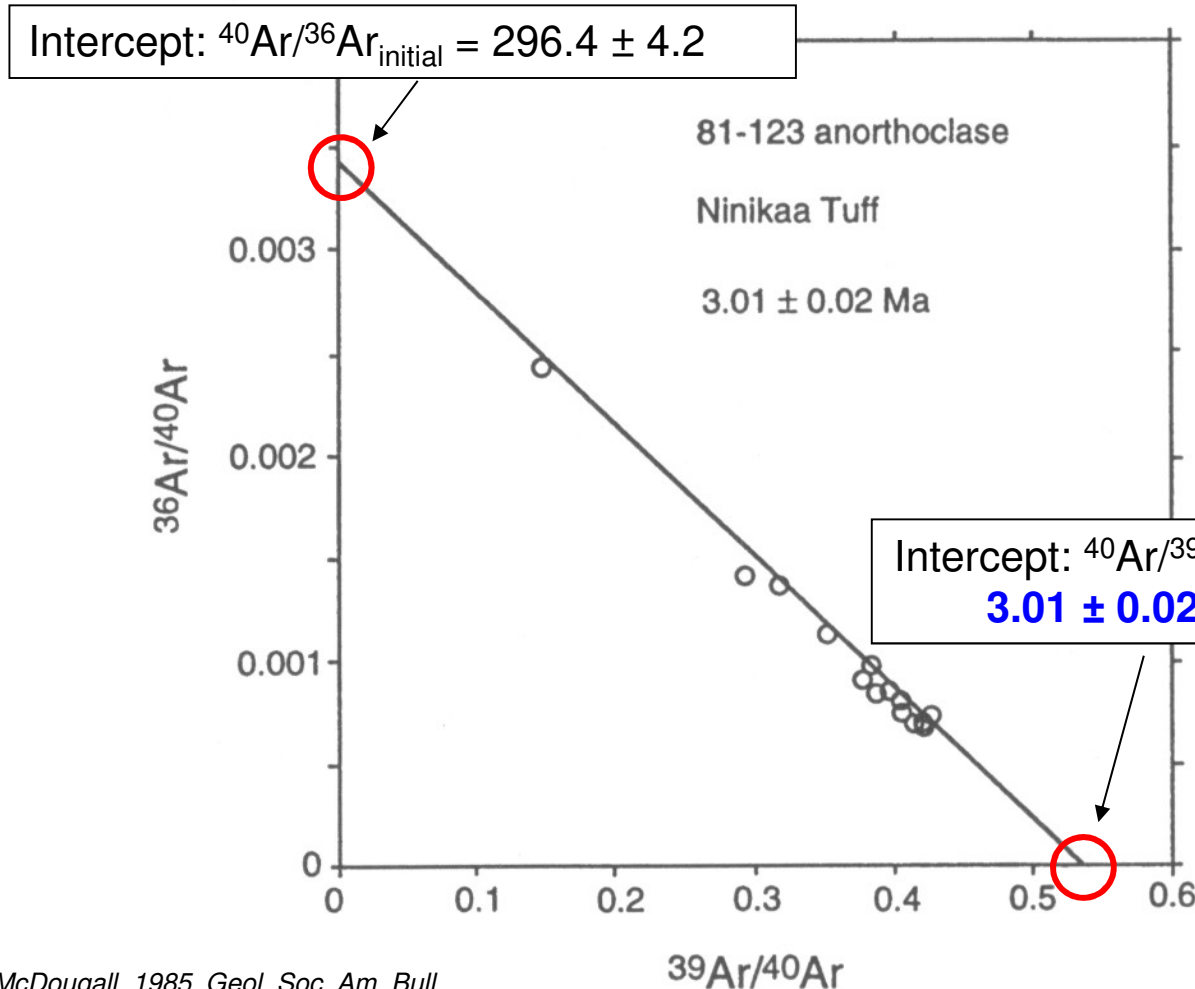


Whenever the **RATIO** between **EXCESS** and **ATMOSPHERIC** Ar changes from one to another step, there will be scatter in the linear correlation!

# Ar-Ar Geo-/Thermochronology

Data presentation & interpretation

**Example:** Anorthoclase from **a volcanic tuff** (Ninikaa tuff) within a **sedimentary strata** (Koobi Fora Fm.) east of Lake Turkana (Northern Kenya)



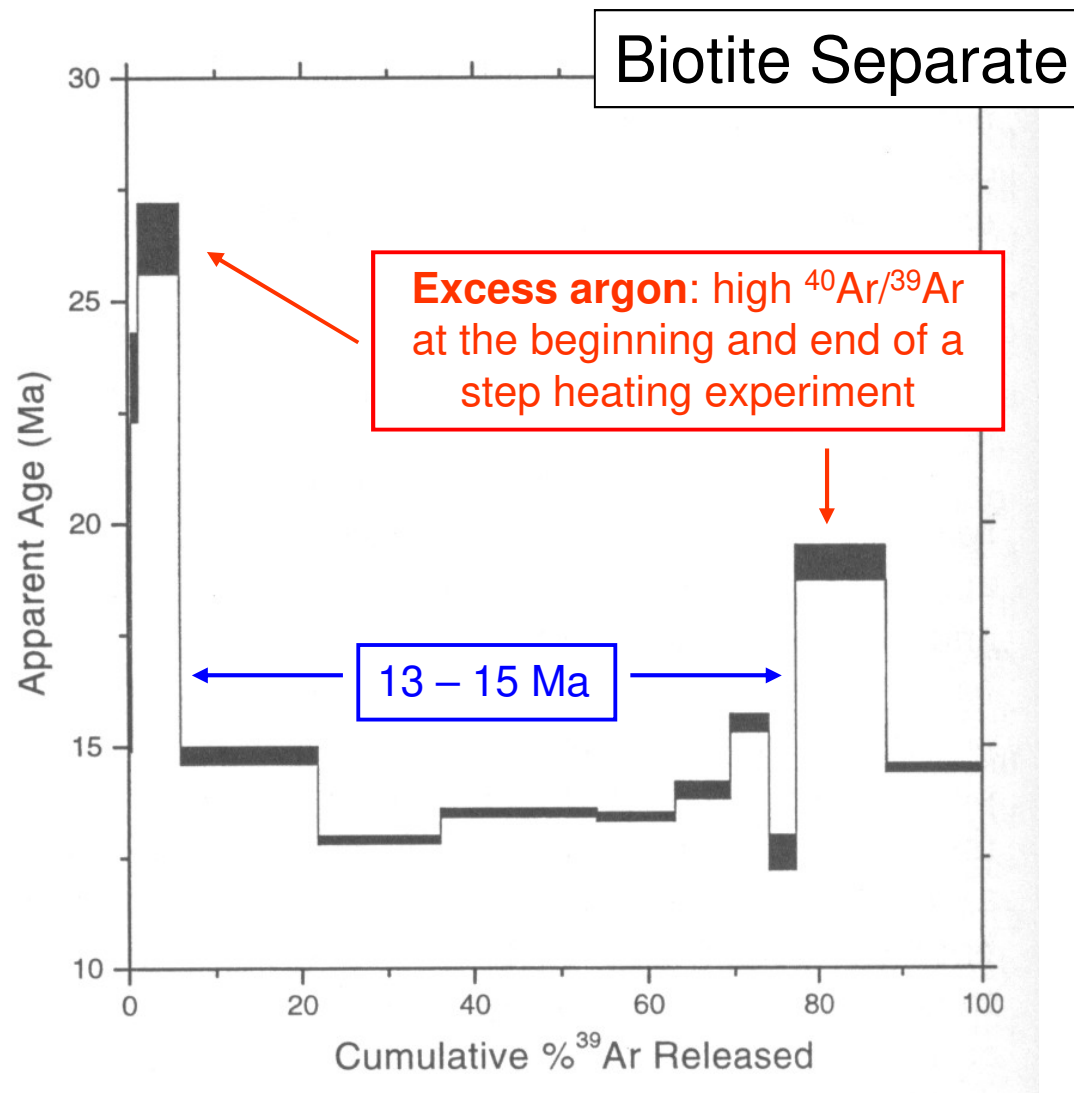
Intercept value indicates: Only two argon components are present: atmospheric and radiogenic Argon



# Ar-Ar Geo-/Thermochronology

Data presentation & interpretation

**Example:** Age spectrum diagram indicating excess argon

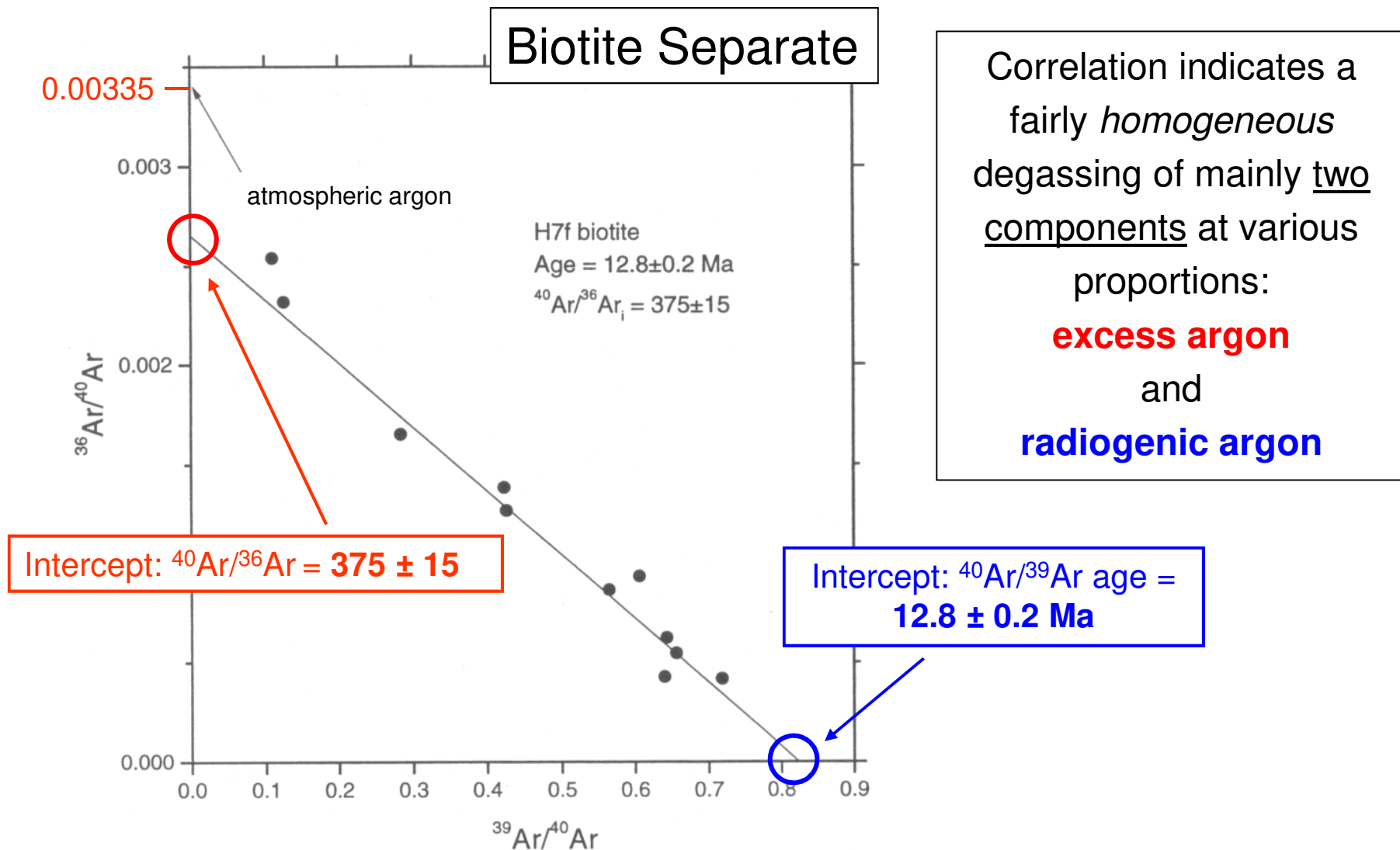


Underestimation of **initial** <sup>40</sup>Ar/<sup>36</sup>Ar results in the typical shape of the age spectrum (undercorrection of measured <sup>40</sup>Ar by using an atmospheric <sup>40</sup>Ar/<sup>36</sup>Ar ratio for low (LT) and high temperature (HT) steps)

# Ar-Ar Geo-/Thermochronology

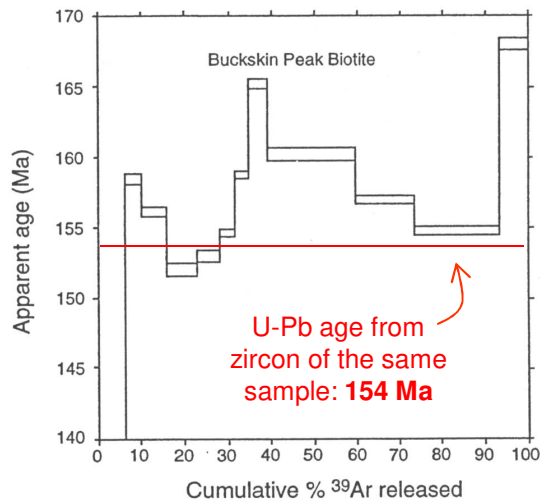
Data presentation & interpretation

The same **biotite** sample plotted in an **inverse isochron** diagram:

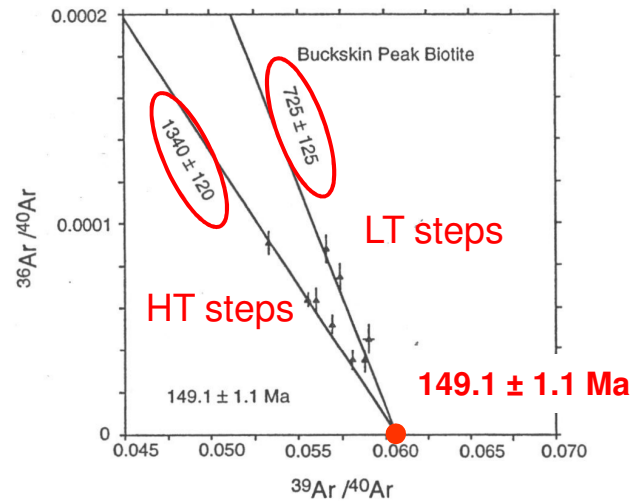


### Example: Biotite from a Qz-Diorite

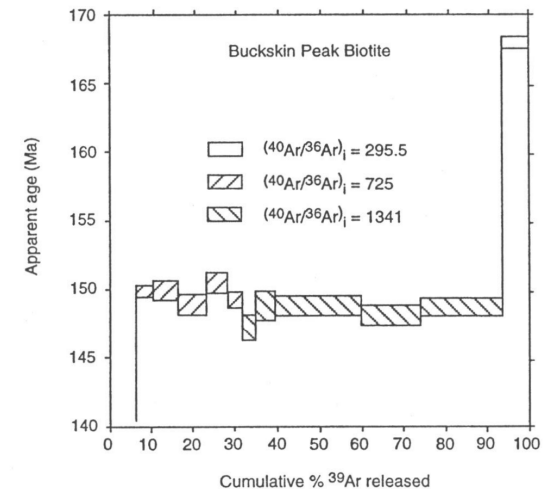
Age spectrum



Inverse isochron diagram



Recalculated age spectrum



Highly disturbed age spectrum – **no plateau!**

Maximum age exceeds the maximum possible **emplacement age** derived from U-Pb on zrn (**154 Ma**)!

Heating steps of the same sample in an **inverse isochron** diagram.

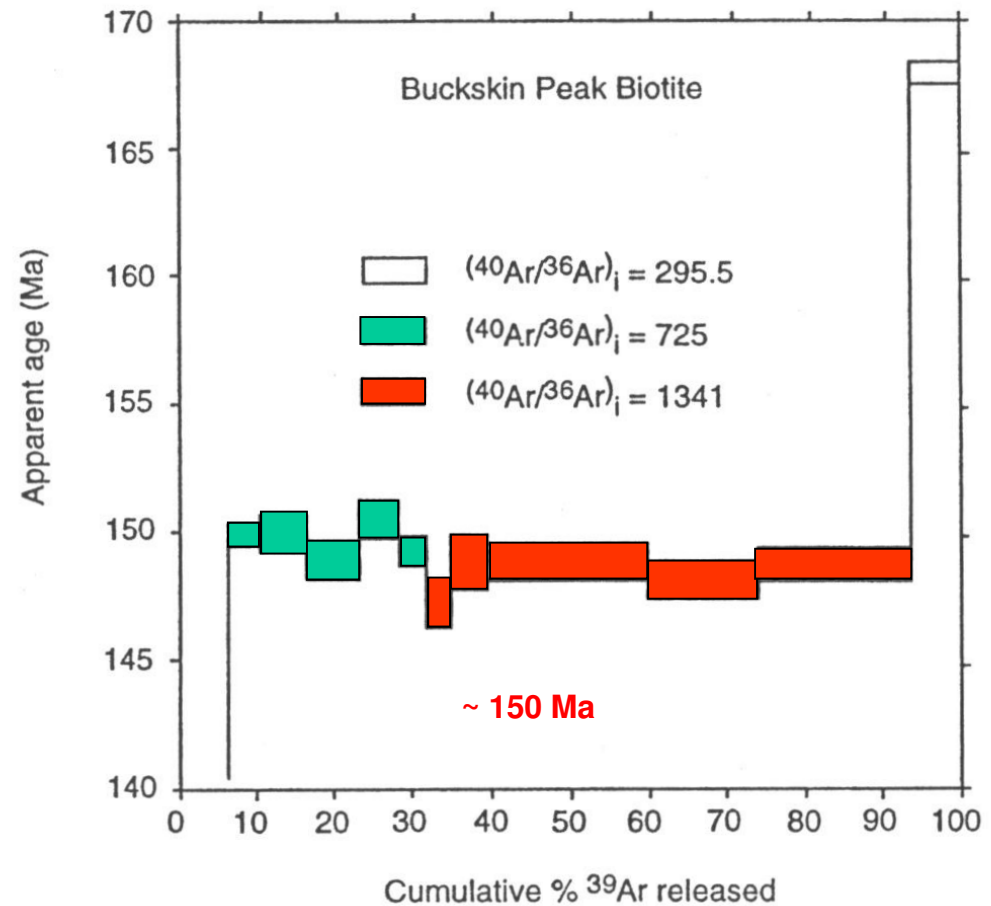
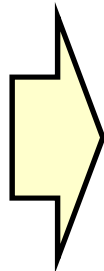
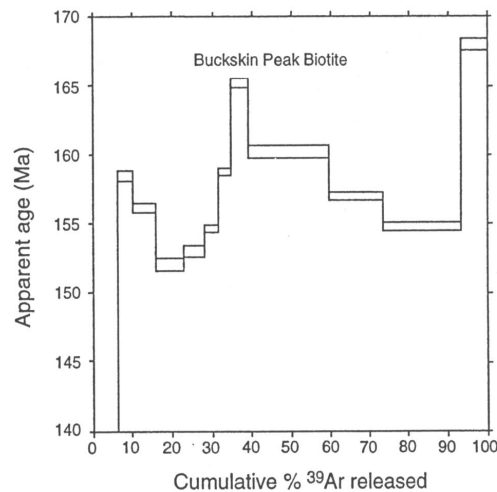
Two **non-atmospheric** argon components were detected, degassing from **different sites** within the crystals at different temperatures

Age spectrum **recalculated** using the two different initial argon isotope compositions as derived from the inverse isochron diagram

*see next overhead!*

### Example: Biotite from a Qz-Diorite

Age spectrum **recalculated** using the **initial (trapped, excess) argon** compositions as derived from the inverse isochron diagram:



## „Disturbed“ samples (phases) - reasons

Argon **loss**, **gain** or **redistribution** between minerals may result from:

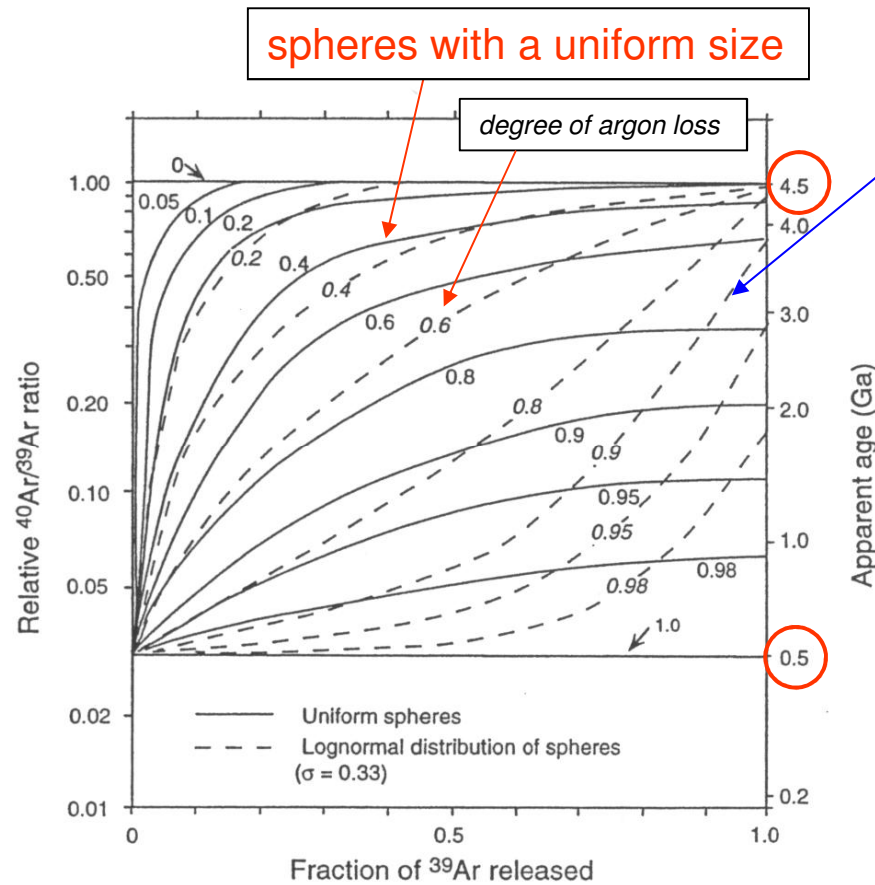
- Thermally induced (volume) diffusion („reheating“)
- Recrystallisation during **metamorphism**
- Recrystallisation during **alteration**

What are the consequences of such processes with respect to the **measured isotope composition** of a sample, i.e. how do such effects affect age spectra, inverse- and normal isochron diagrams?

*Note, that commonly only  $^{40}\text{Ar}$  and  $^{36}\text{Ar}$  are affected by such processes, not  $^{39}\text{Ar}$  that is produced by neutron irradiation just prior to measurement!*



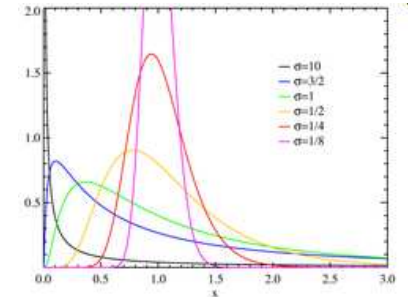
### Disturbed samples: *Single site diffusion model*



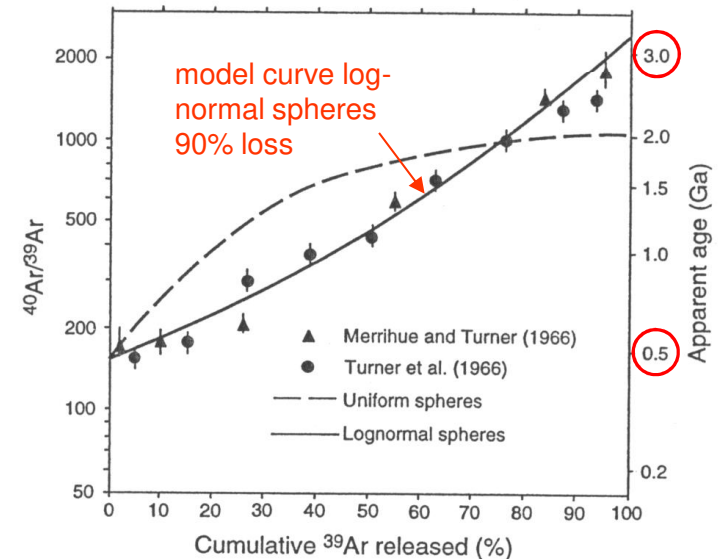
spheres with a lognormal size distribution

spheres with a uniform size

log-normal distributions



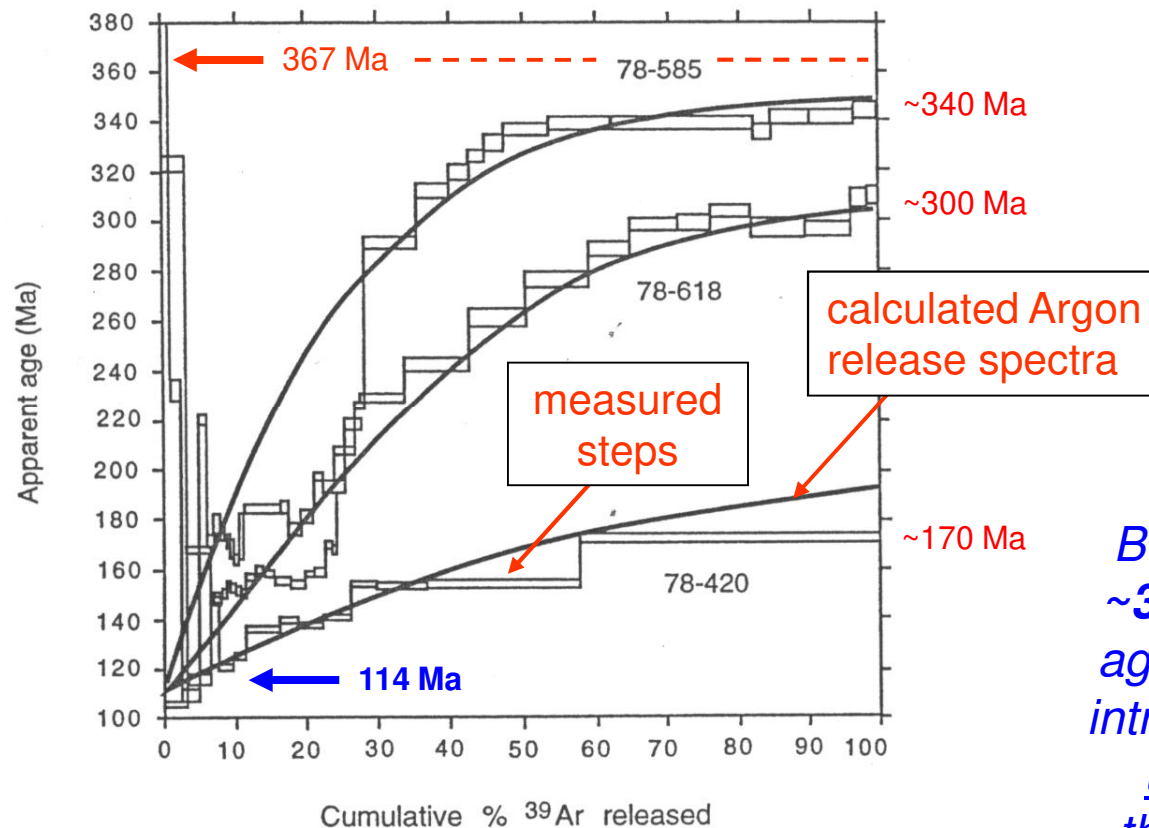
### Bruderheim meteorite, whole-rock



Calculated **Ar release** patterns of **uniform spheres** and **spheres with a lognormal size distribution** assuming ideal volume diffusion (**age = 4.5 Ga**, thermal event at 0.5 Ga)

**Real meteorite sample: Age >3 Ga**, Ar-loss at around **0.5 Ga**  
(from Turner et al., 1966)

## Disturbed samples: Example from host rocks adjacent to an intrusion

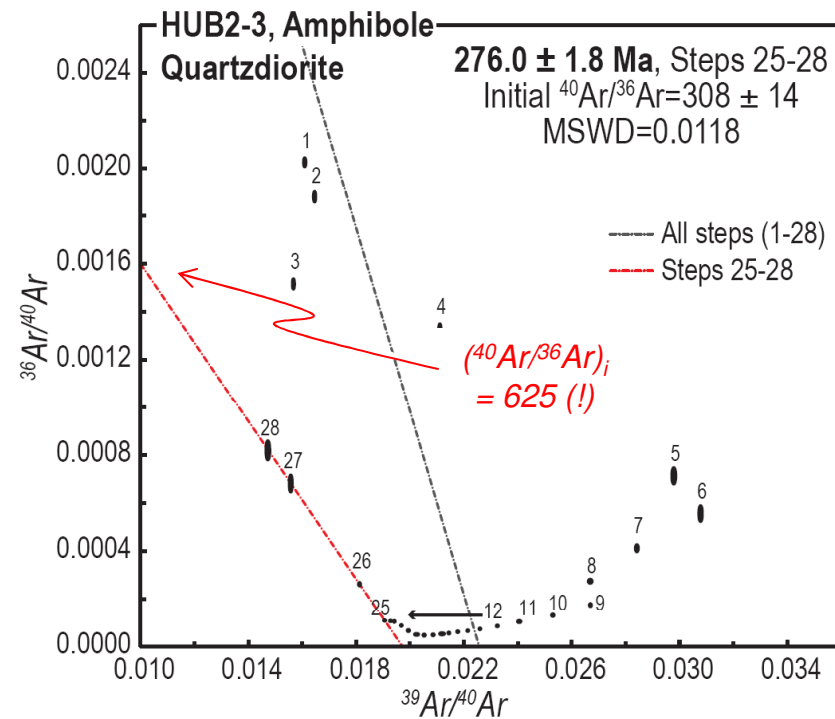
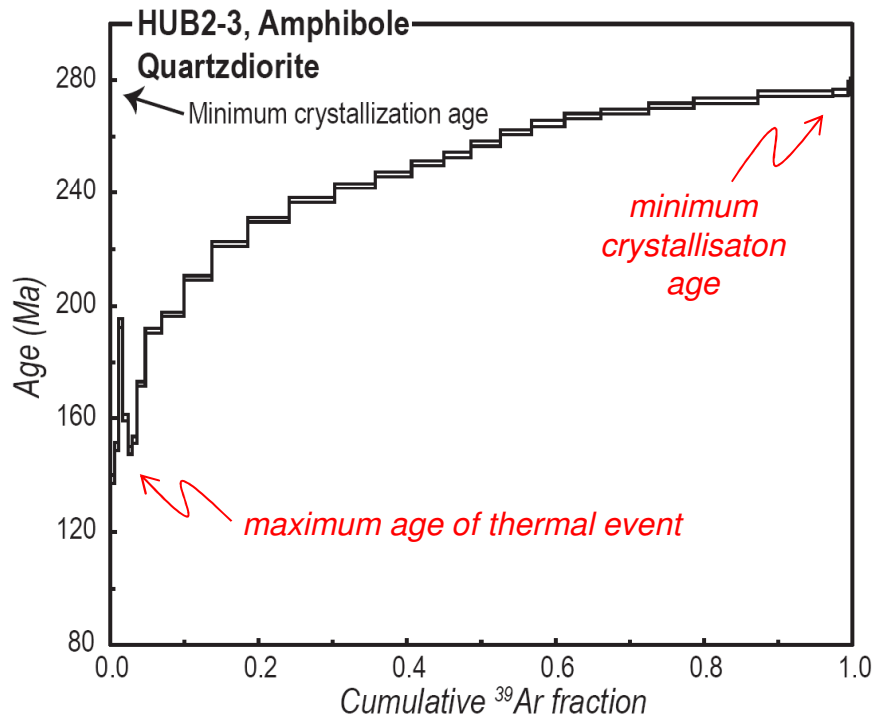


**No initial age preserved !!**

*But the lower estimate of ~340 Ma for the formation age is close to the 367 Ma intrusion U/Pb age, and the upper estimate for the thermal event is close to 114 Ma!*

Age spectra of three **367 Ma** old hornblende samples from a diorite taken at different distances from the contact to a younger, **114 Ma** old granodiorite intrusion

### Disturbed samples: Hbl from a Qz-Diorite from Alaska



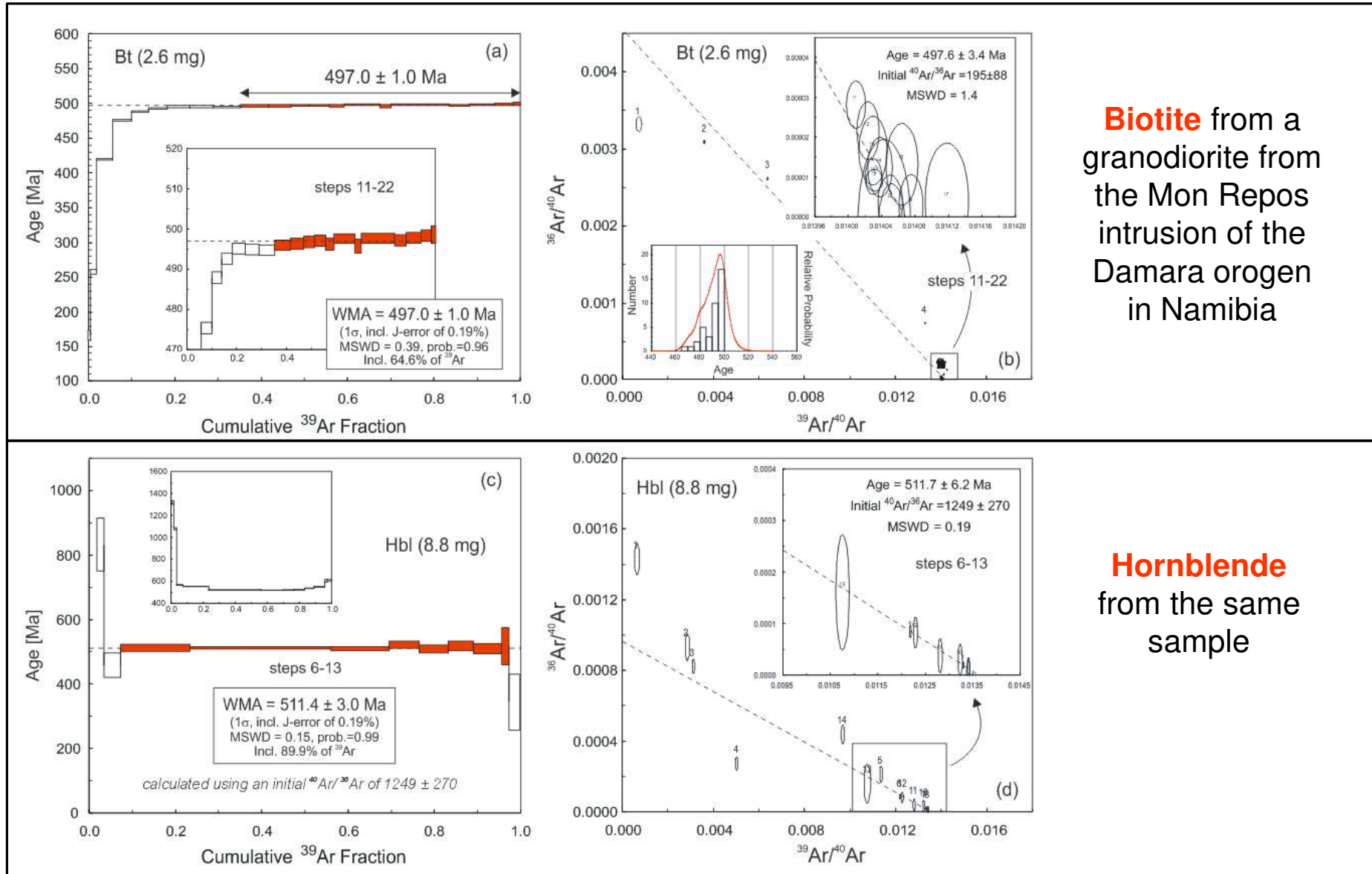
Total gas age: 248.3 ± 0.2 Ma

Minimum crystallization age: 276.0 ± 1.8 Ma

completely meaningless!!!!

**Age spectrum and inverse isochron diagram of a hornblende separate from a Qz-Diorite from Alaska with a typical **Ar-loss profile**. Minimum initial cooling age: **276.0 ± 1.8 Ma**, maximum age of metamorphism: **~140 Ma** (from Falkowski et al., 2016)**

### Disturbed samples: Bt and Hbl from a granodiorite from Namibia



**Biotite** from a granodiorite from the Mon Repos intrusion of the Damara orogen in Namibia

**Hornblende** from the same sample

## Mixed phases I: Whole rocks

Whole rock samples contain different minerals that may provide different (virtual) ages due to:

- Different **closure temperatures** (relevant for **slow** cooling!!)  
(at 10°C/Ma cooling, a **Hbl** will be ~20 Ma older than a **Bt** from the same sample!)
- Different degrees of **Ar loss** during reheating (according to  $T_c$ ,  $D_0$ )
- Various amounts of **excess Ar** (inherited or gained)
- **Recrystallisation** and/or **formation of new minerals** during metamorphism, synkinematic recrystallisation and alteration

**Therefore: Separate individual minerals out of a rock!**

**Where not possible: Apply high-resolution dating!**

## Mixed phases I: Whole rocks

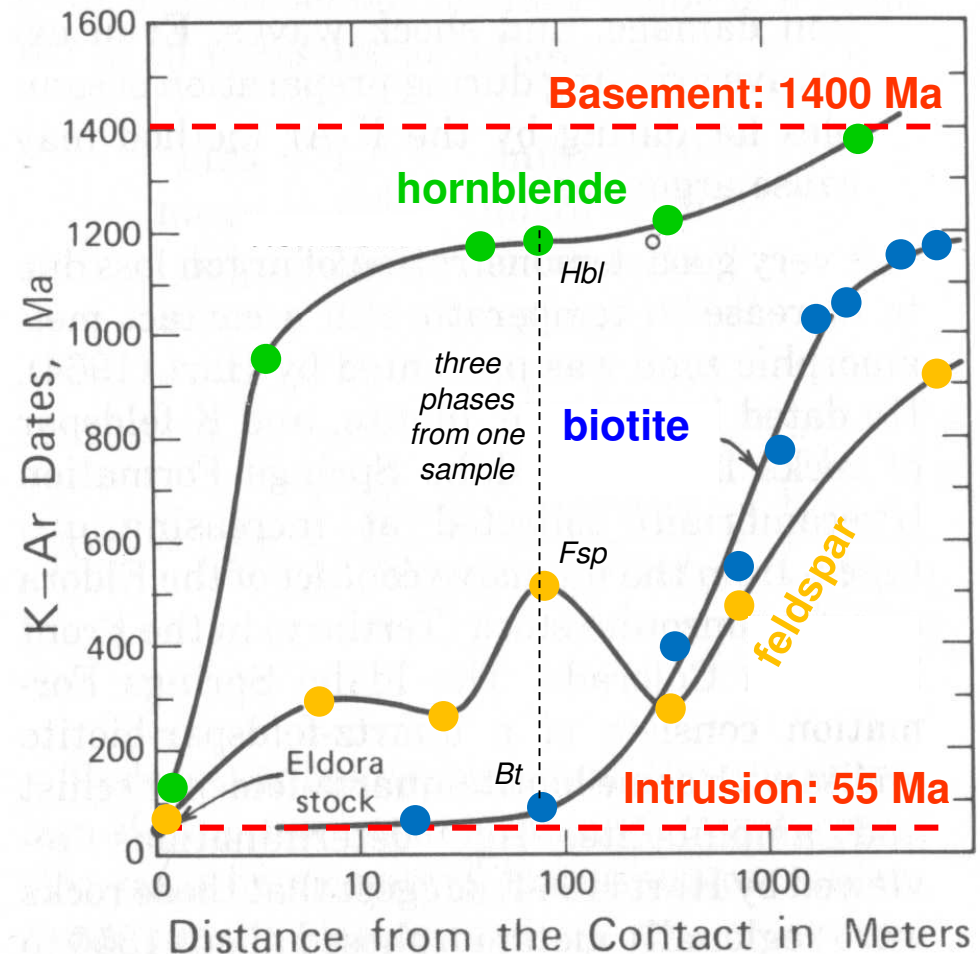
**Example:** K-Ar ages of **different minerals** isolated from samples taken at **different distances** from a young intrusion, hosted by precambrian basement

Age of host rock: **1.4 Ga**

Age of intrusion: **55 Ma**

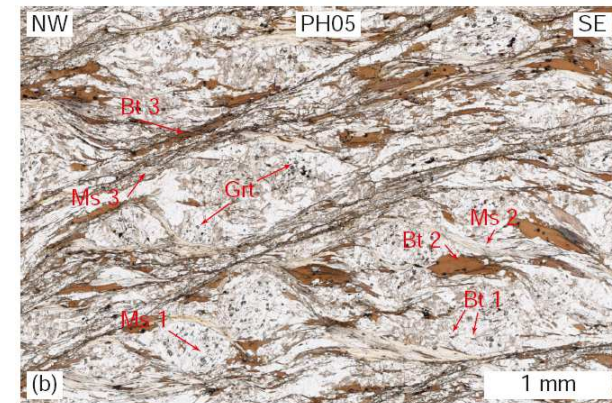
**Different temperature profiles due to different closure temperatures**

(i.e. different diffusion activation energies of different minerals)

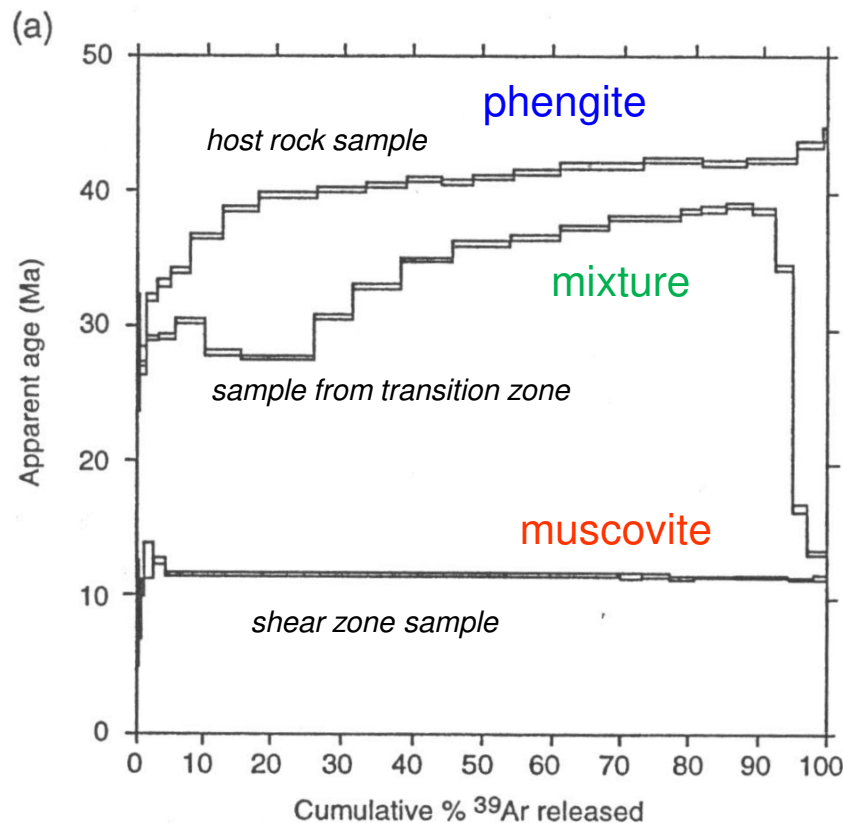


### Mixed phases II: Different mica generations in a shear-zone

**Example:** Shear zone samples with recrystallized muscovite



From: Hallas, P., 2020, PhD



Degassing patterns of pure, pre-existing ~40 Ma old phengite, of pure ~12 Ma old muscovite (recrystallized during shear zone metamorphism); and degassing pattern of a mixture of both.

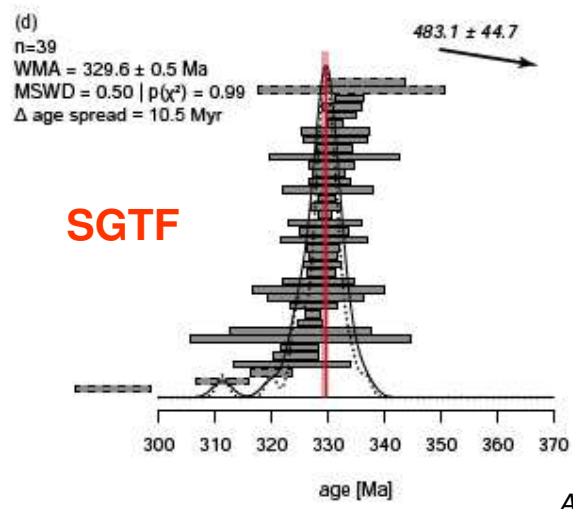
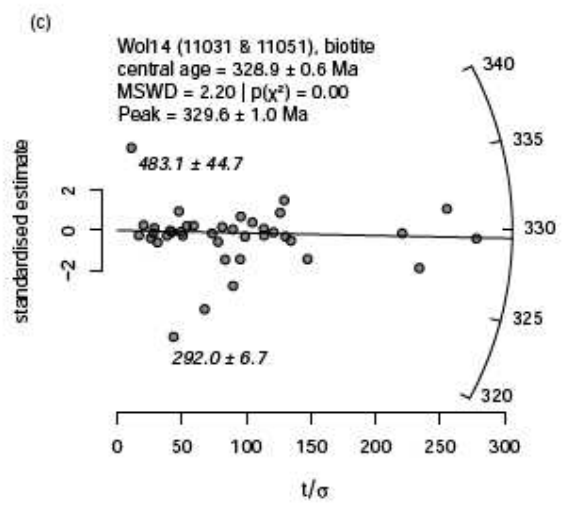
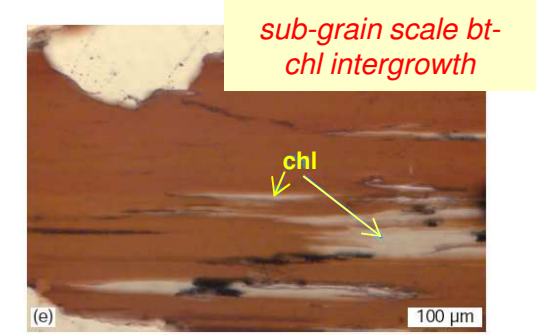
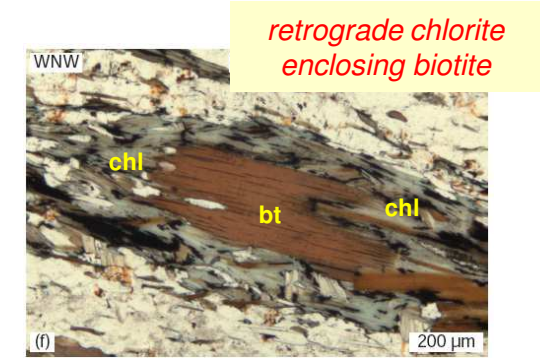
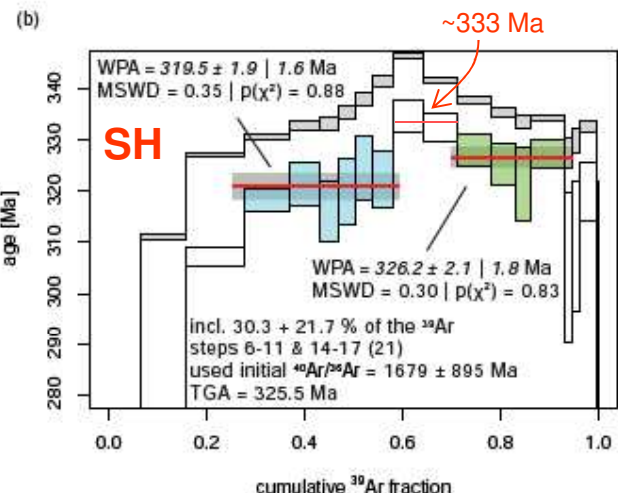
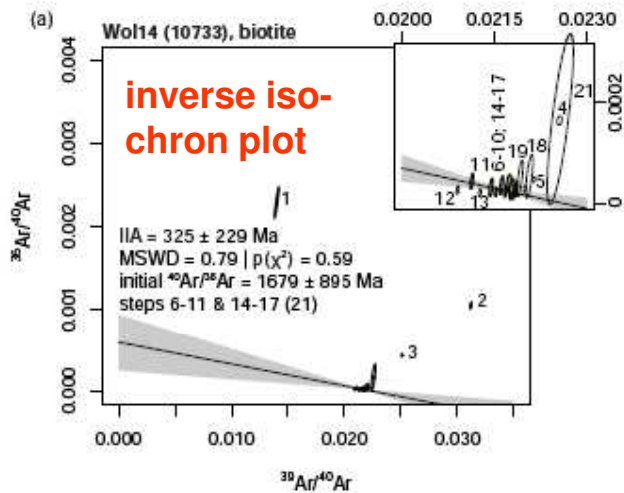
The three samples were taken at different distances from a shear zone (after Wijbrans & McDougall, 1986)

Therefore: **Separate** phases properly **before** dating!

Alternative:  
**Single grain total fusion dating (SGTF)**

### Mixed phases III: Chlorite intergrowth with biotite

Example: Mica schist sample with *chloritized* biotite

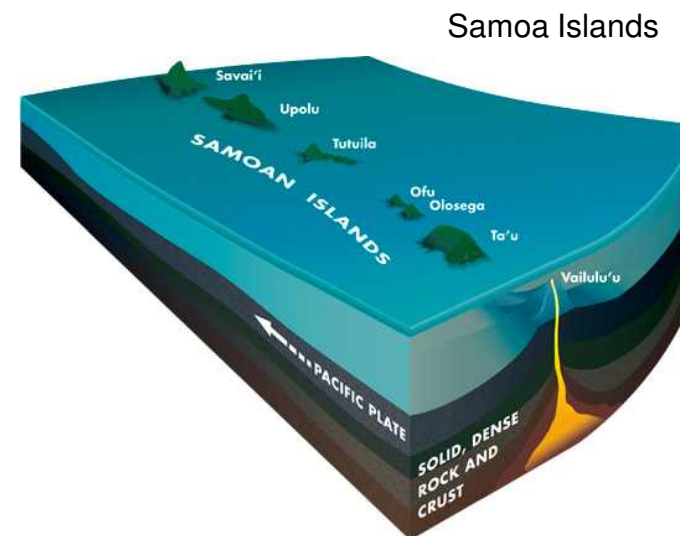
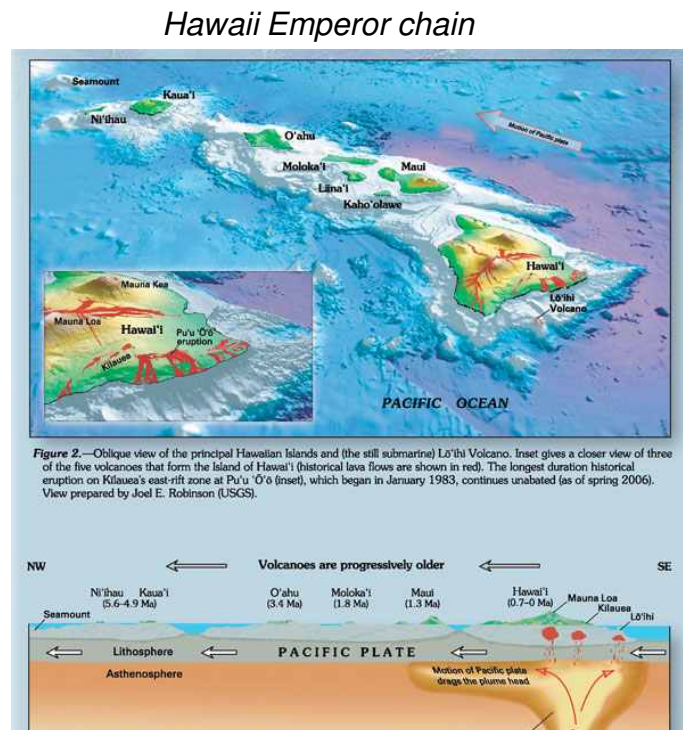


Chlorite (nearly K-free) hosts **recoil  $^{39}\text{Ar}$**  from biotite, resulting in **hump-shaped** age spectra

### Mixed phases IV: Volcanic rocks („fine grained“)

Dating of (fine-grained) **volcanic rocks** applying (high resolution)  $^{40}\text{Ar}/^{39}\text{Ar}$  dating

### Why?

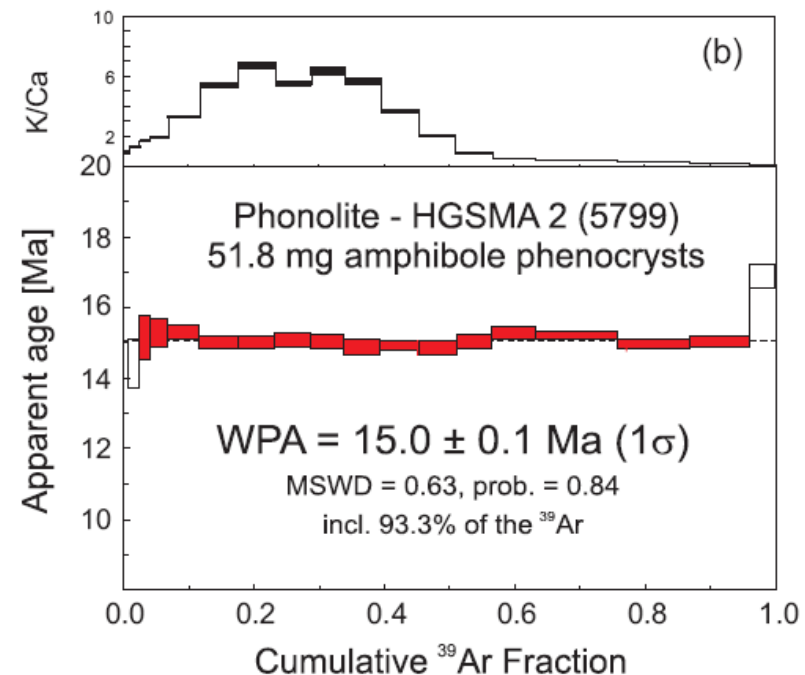
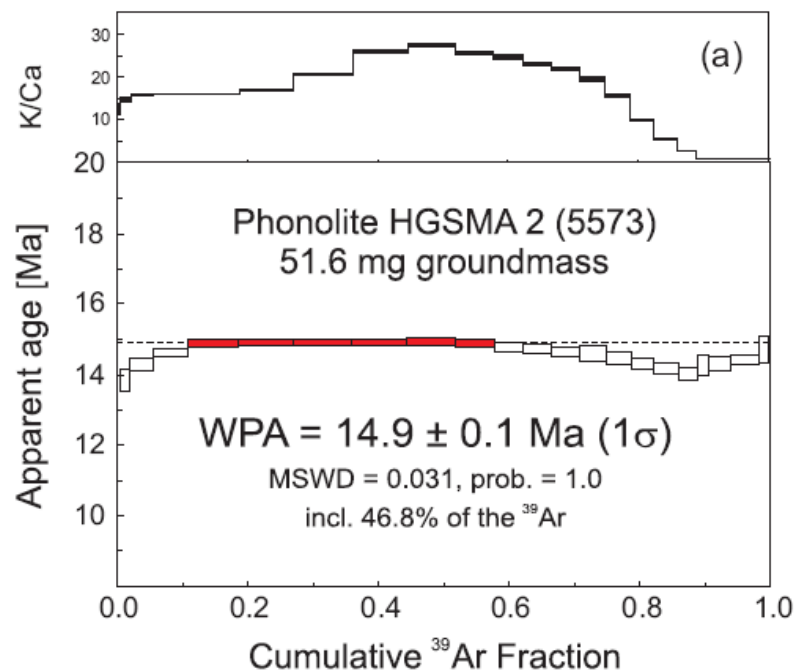


From: Woods Hole Oceanographic Institution (WHOI.edu)

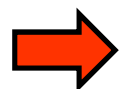
➔ E.g., to determine plate movement **velocities** (and hence to quantify mantle dynamics)

## Mixed phases IV: Volcanic rocks („fine grained“)

Comparison: Phenocryst vs. groundmass Ar-Ar age



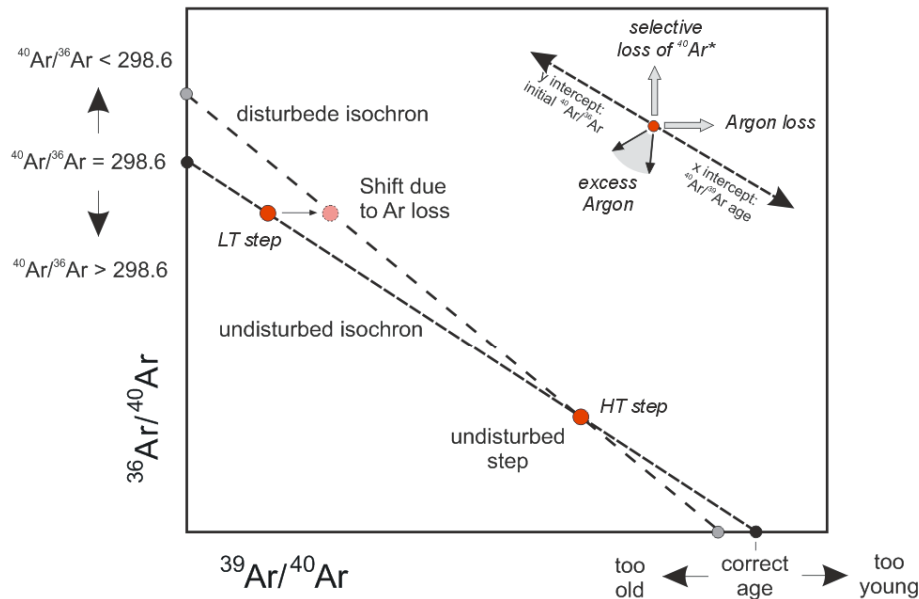
From Abratis et al., 2015



Whenever present, separate **phenocrysts** from **volcanic rocks!!!**

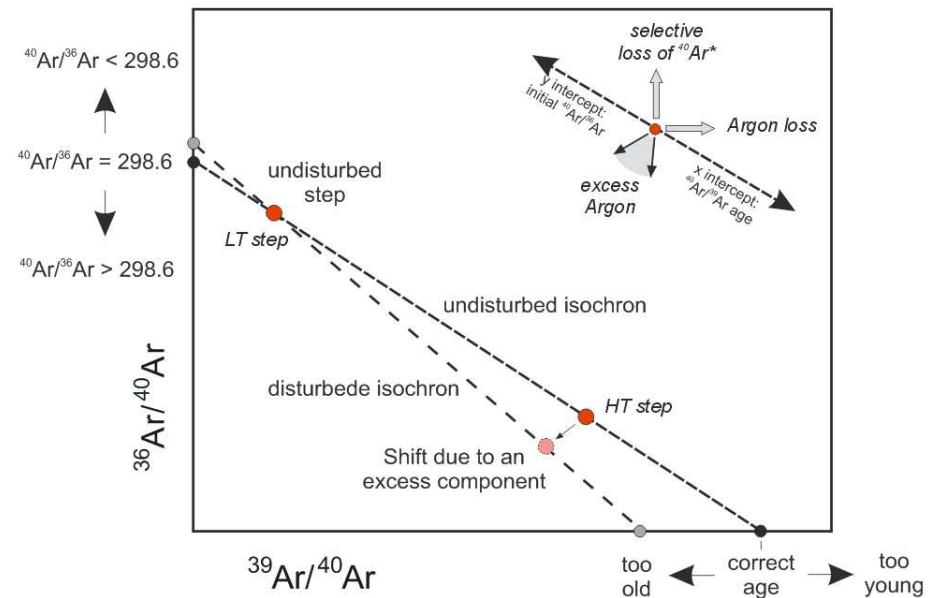
If no phenocrysts are present: High-resolution dating of groundmass

### Dating fine grained (volcanic) rocks: Problem

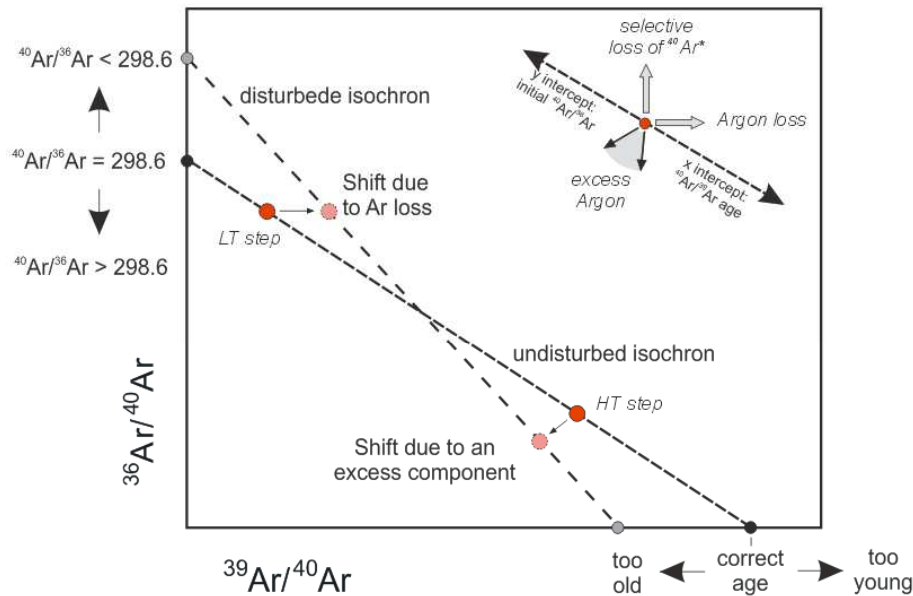


Effect of **argon loss** on a **LT step** in an inverse isochron diagram: age becomes too old, intercept becomes sub-atmospheric

Effect of **excess argon** in a **HT step** in an inverse isochron diagram: age becomes too old, intercept becomes sub-atmospheric

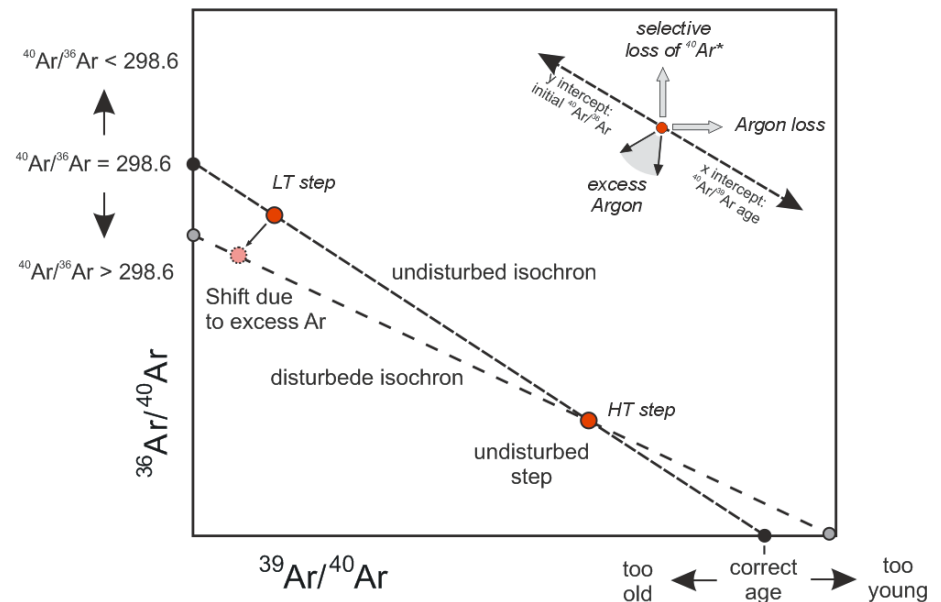


### Dating fine grained (volcanic) rocks: Problem

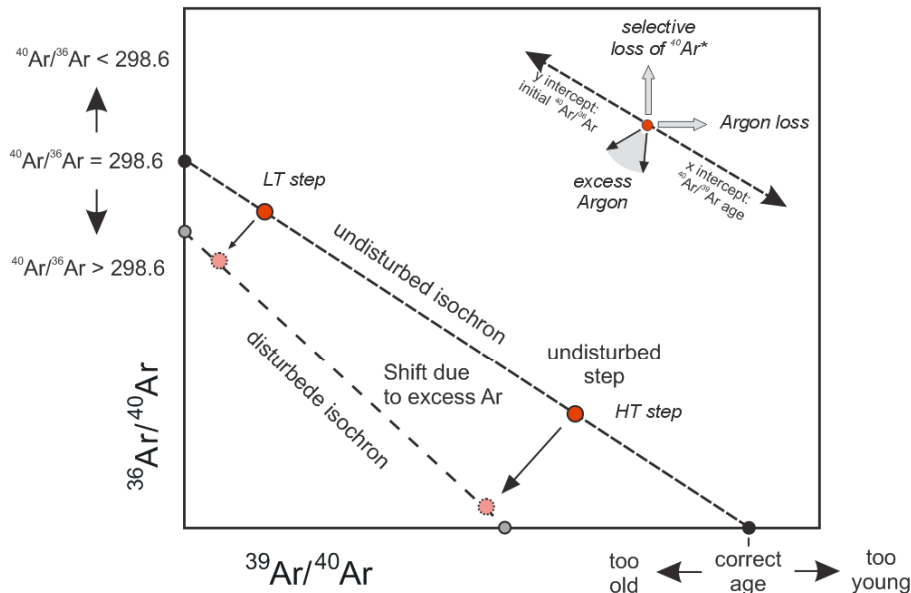


Effect of **argon loss** in a **LT step** and **excess argon** in a **HT step**: age becomes too old, intercept becomes sub-atmospheric

Effect of **excess Ar** in a **LT step**: age becomes too young, intercept becomes super-atmospheric

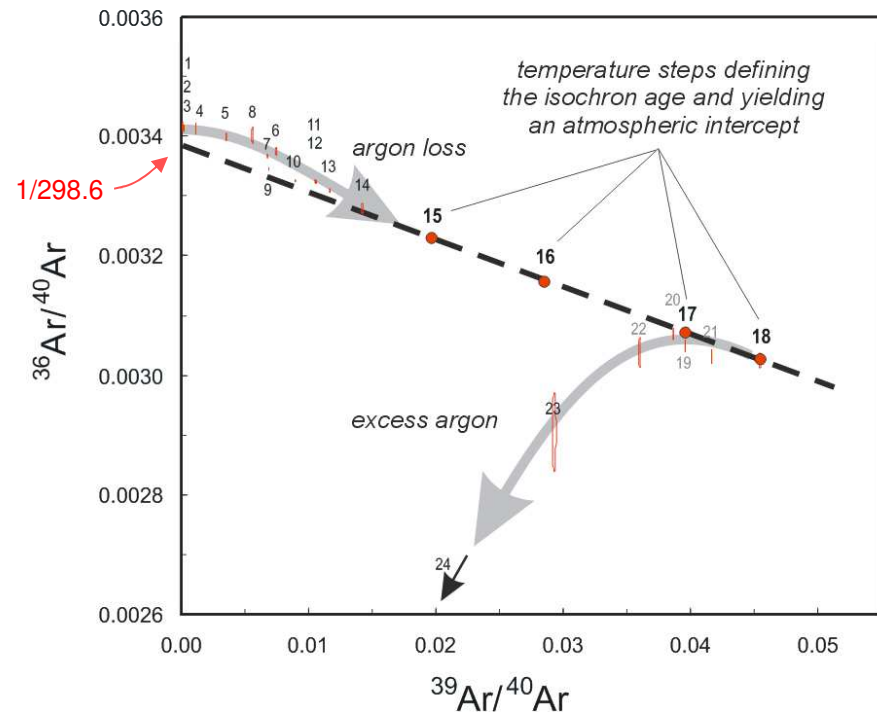


### Dating fine grained (volcanic) rocks: Problem

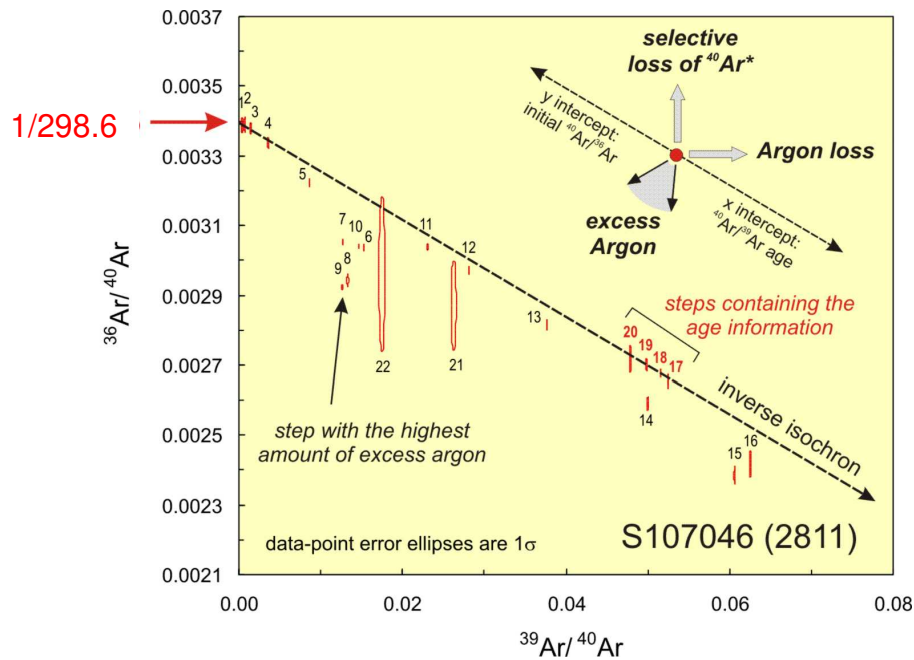


Effect of **excess Ar** in a **LT** and a **HT step**: age becomes too old, intercept becomes super-atmospheric

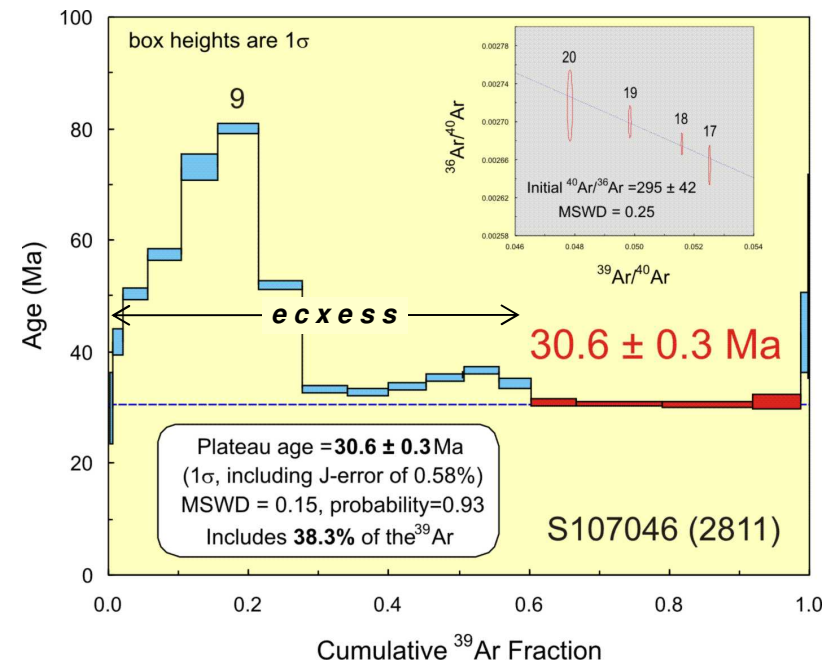
This is a real dataset: We obtain an **argon loss** signal in steps 1-14, then three virtually undisturbed steps that provide the **age** and an **atmospheric intercept**, before steps 19-24 were compromised by **excess argon**



### Dating fine grained (volcanic) rocks: **Example**



Inverse isochron diagram and age spectrum of a basanite sample from the Heldburg region, Germany



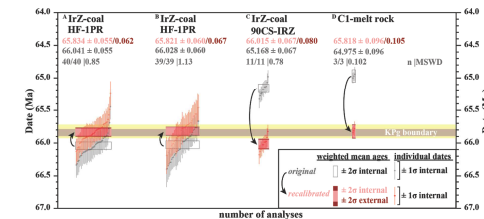
Four steps (comprising **38.8% of the <sup>39</sup>Ar**) contain the required age information at an atmospheric intercept, **the age seems reliable!**

### Beyond step-heating:

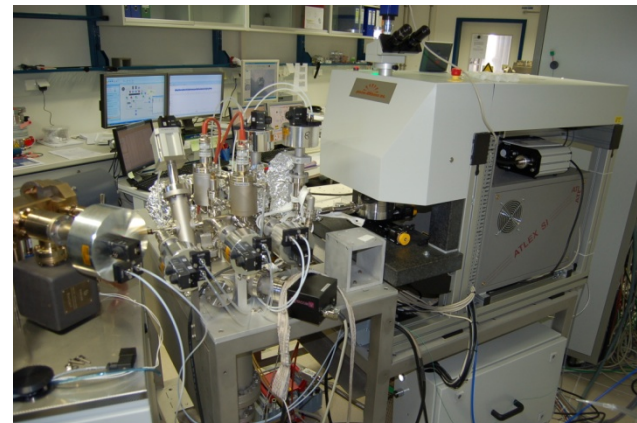
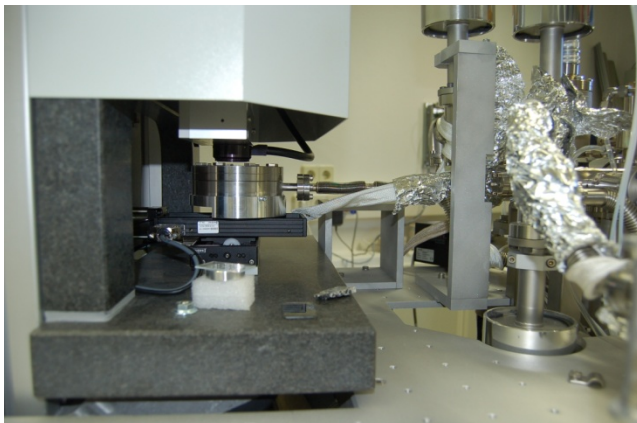
- Technical improvements
- Single grain total fusion dating
- In-situ dating using laser ablation



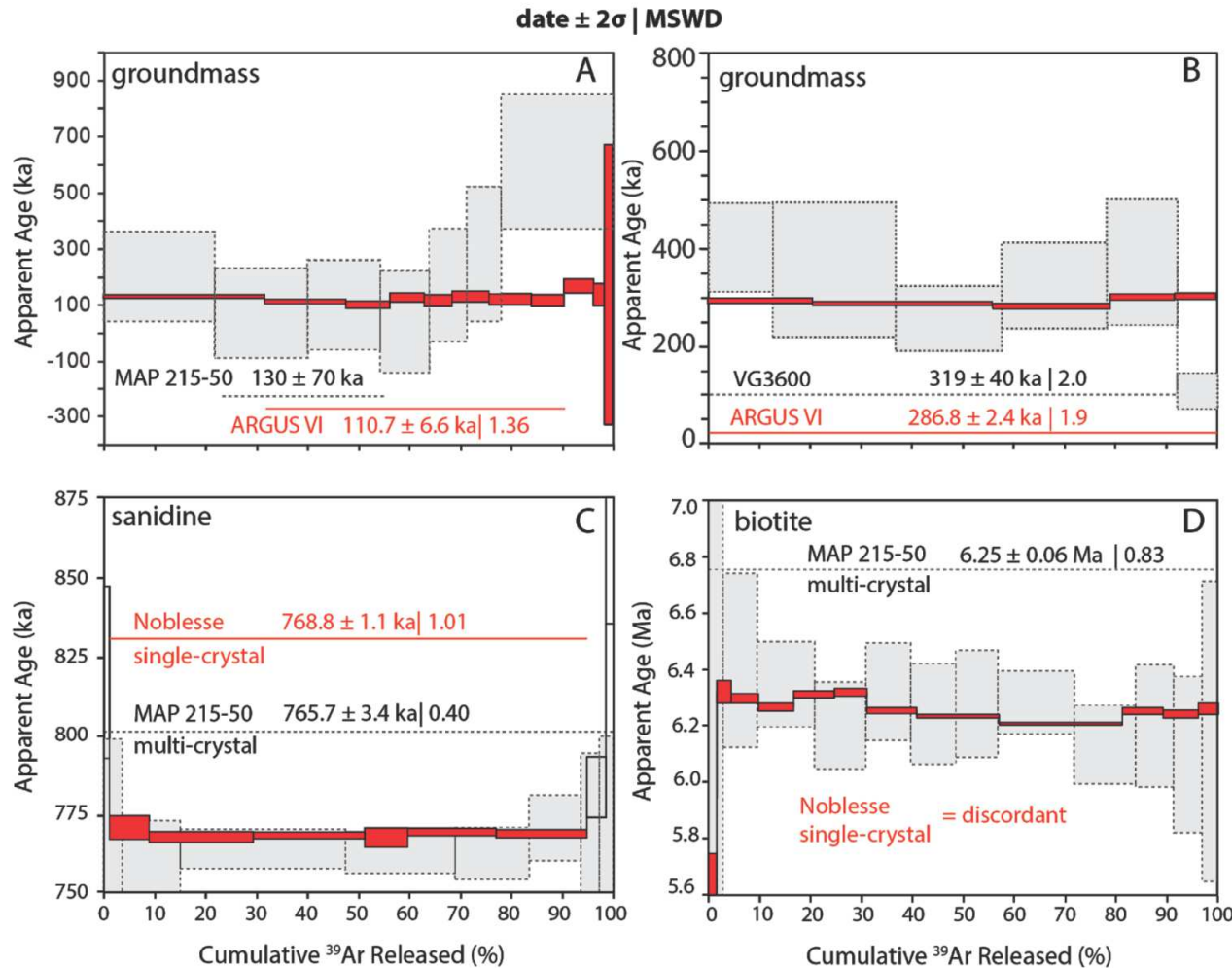
An ARGUS VI noble-gas mass spectrometer



An ArF excimer laser ablation system for in-situ Ar-Ar dating (LA-NGMS)



### Beyond step-heating: Technical improvements

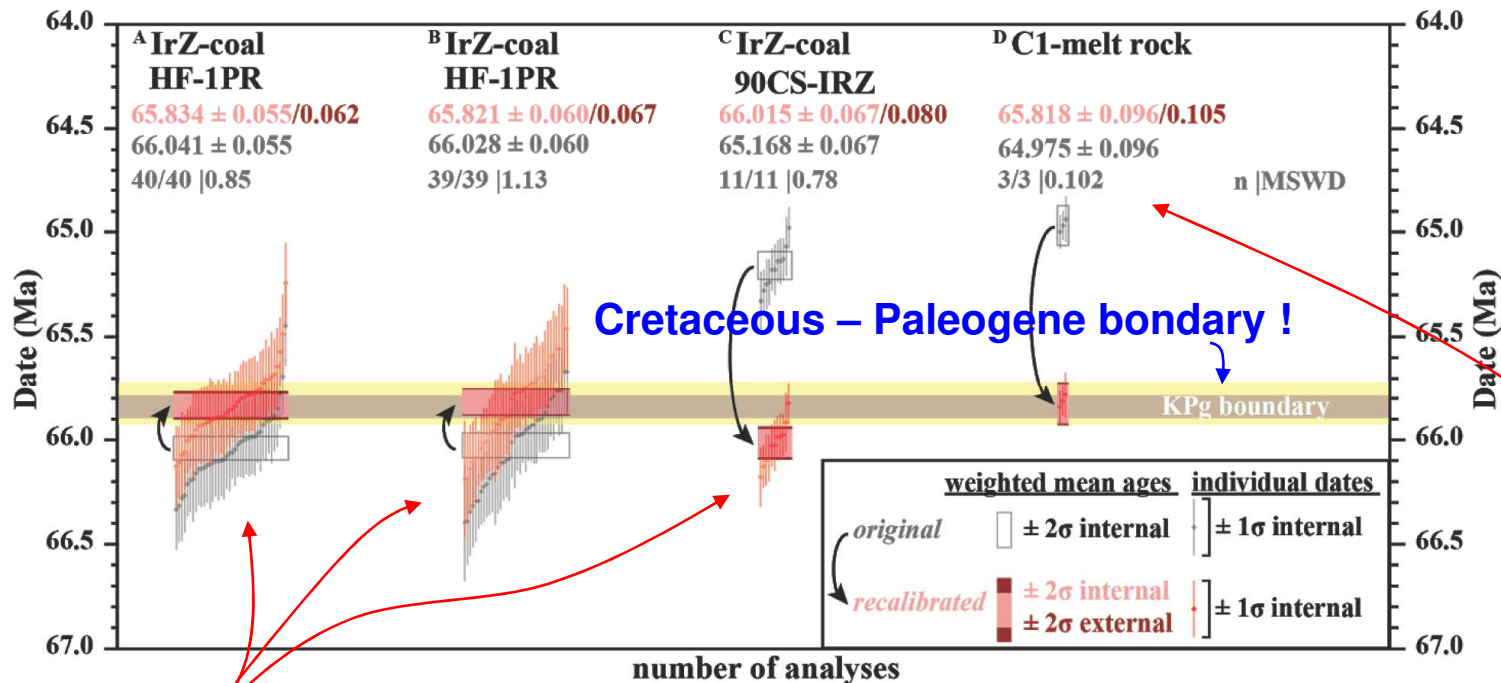


Comparison of data obtained on **different** noble-gas mass spectrometers.

The improvement in **data precision** between older single-collector and **modern** multi-collector noble-gas machines is by a **factor >10**.

Consequences that result are, for example, an increase of samples **showing overdispersion** (,geologic scatter').

### Beyond step-heating: Single-grain total fusion data

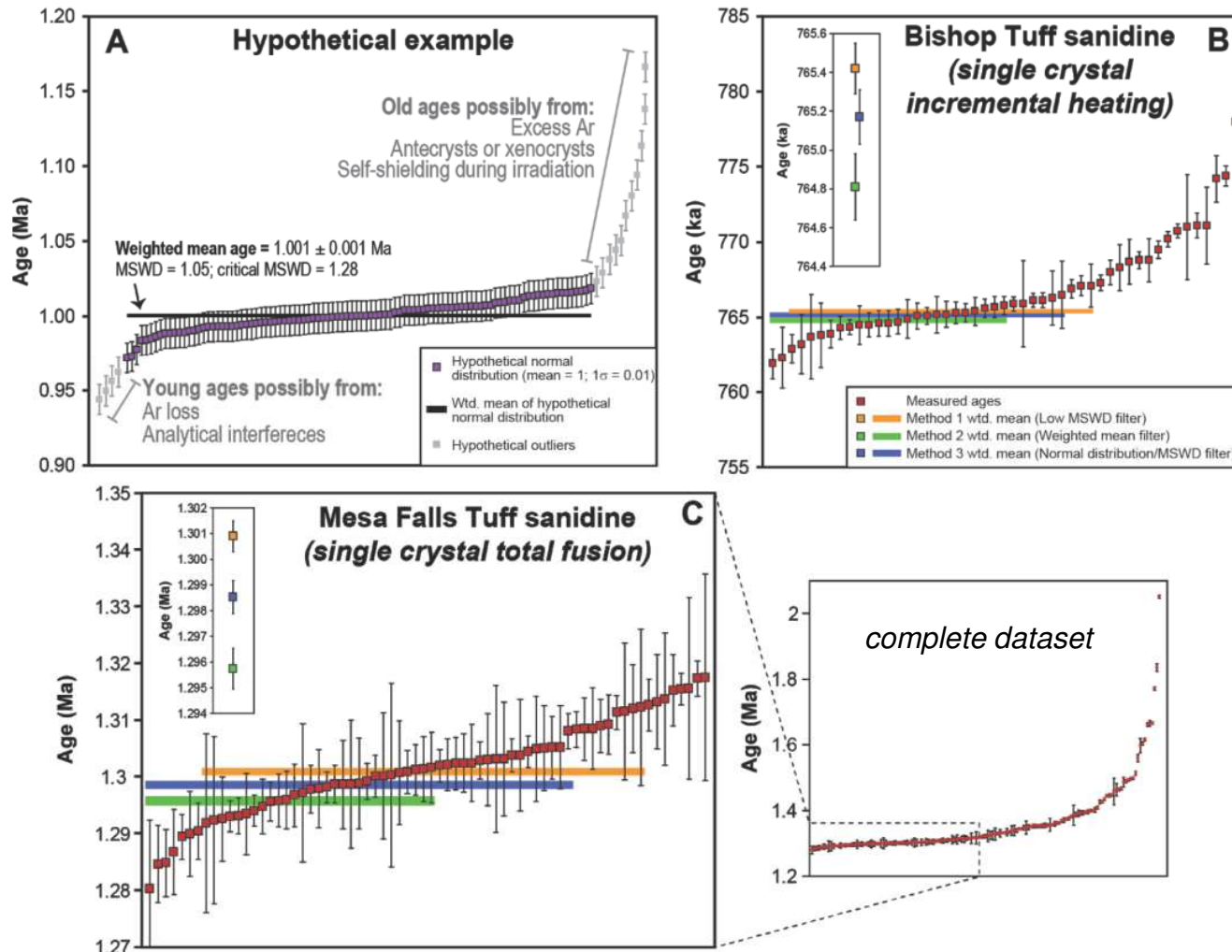


Schaen et al., 2020, GSA Bull., in press

Sanidine **single-grain total fusion ages** from **volcanic ashes** within a coal layer in NE Montana (USA), ordered by increasing age values.

Step-heating data from a melt-rock formed during the **impact formation of the Chicxulub Crater** in Yucatan, Mexico, whose age defines the **Cretaceous-Paleogene boundary**

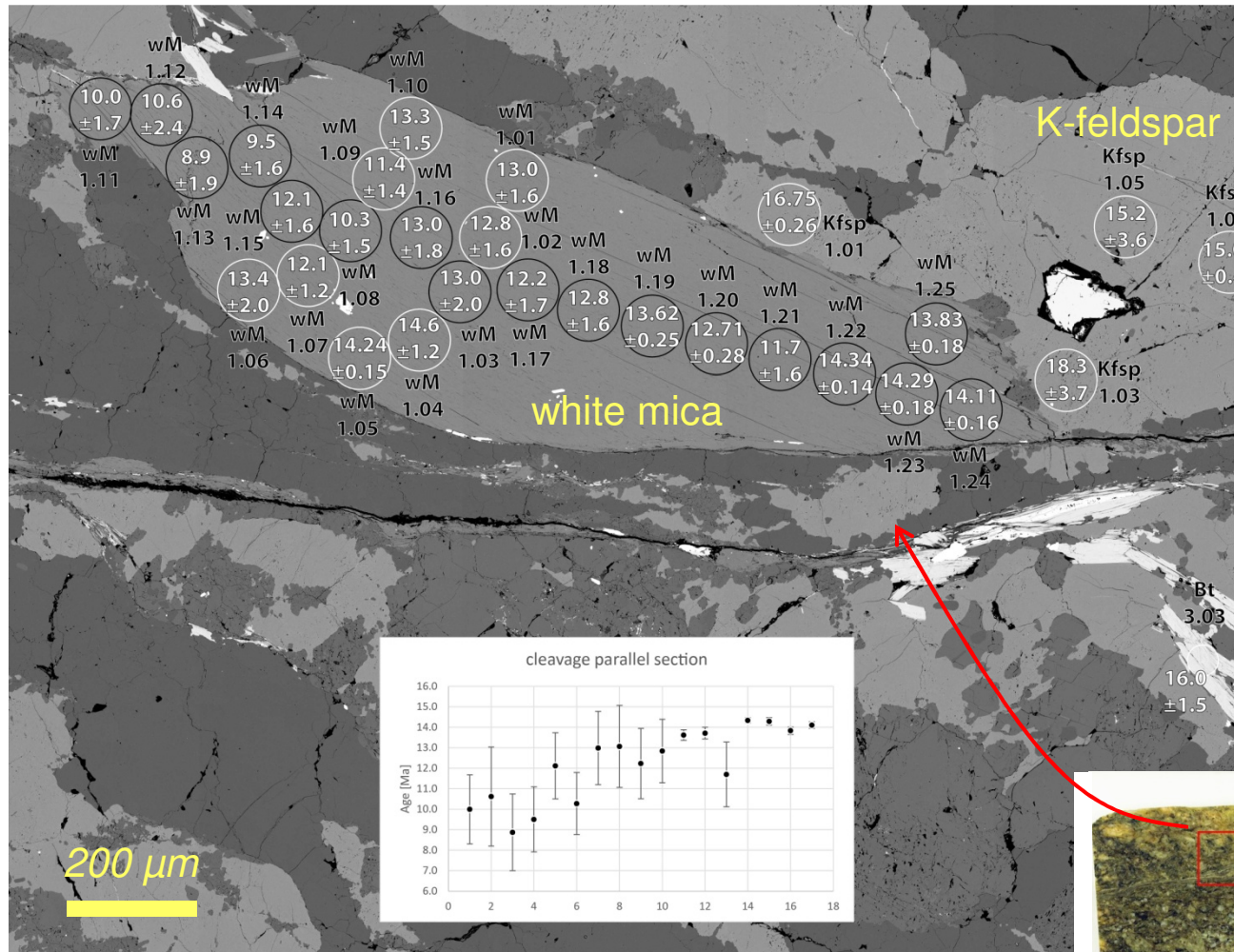
### Beyond step-heating: Single-grain data



Different means in interpreting single-grain age data, which is either **single-grain total fusion data (A)**, or **single grain step (incremental) heating data (B)**.

Distinct deviations to lower and higher ages are commonly observed, why **filtering** by either the MSWD or weighted mean is applied.

### Beyond step-heating: In-situ dating using laser ablation



**That's it !**