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Sequence of construction of Ciomadul dacitic lava domes, East Carpathians, by unspiked K-Ar Cassinol-Gillot technique in comparison with other radiometric methods



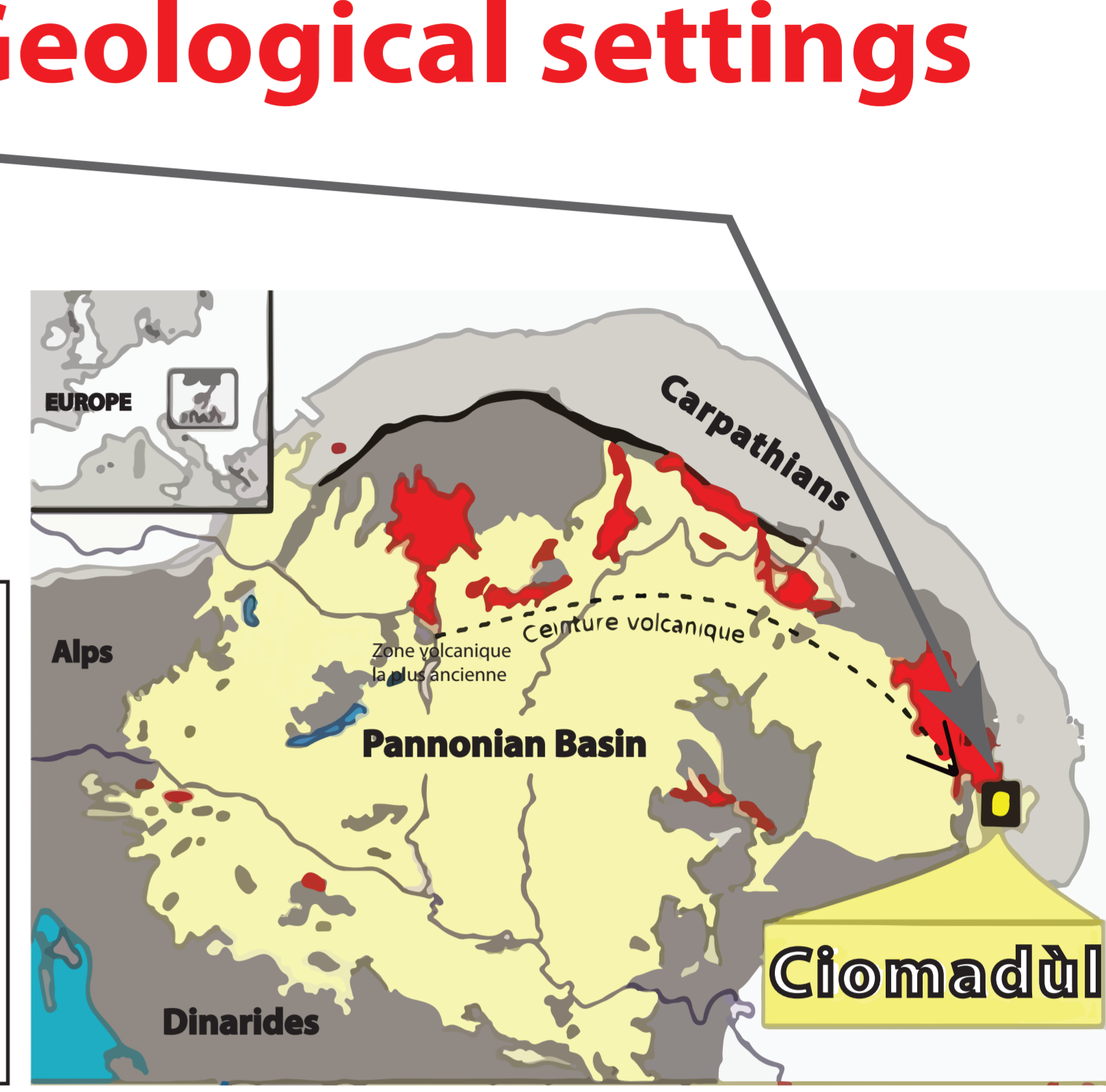
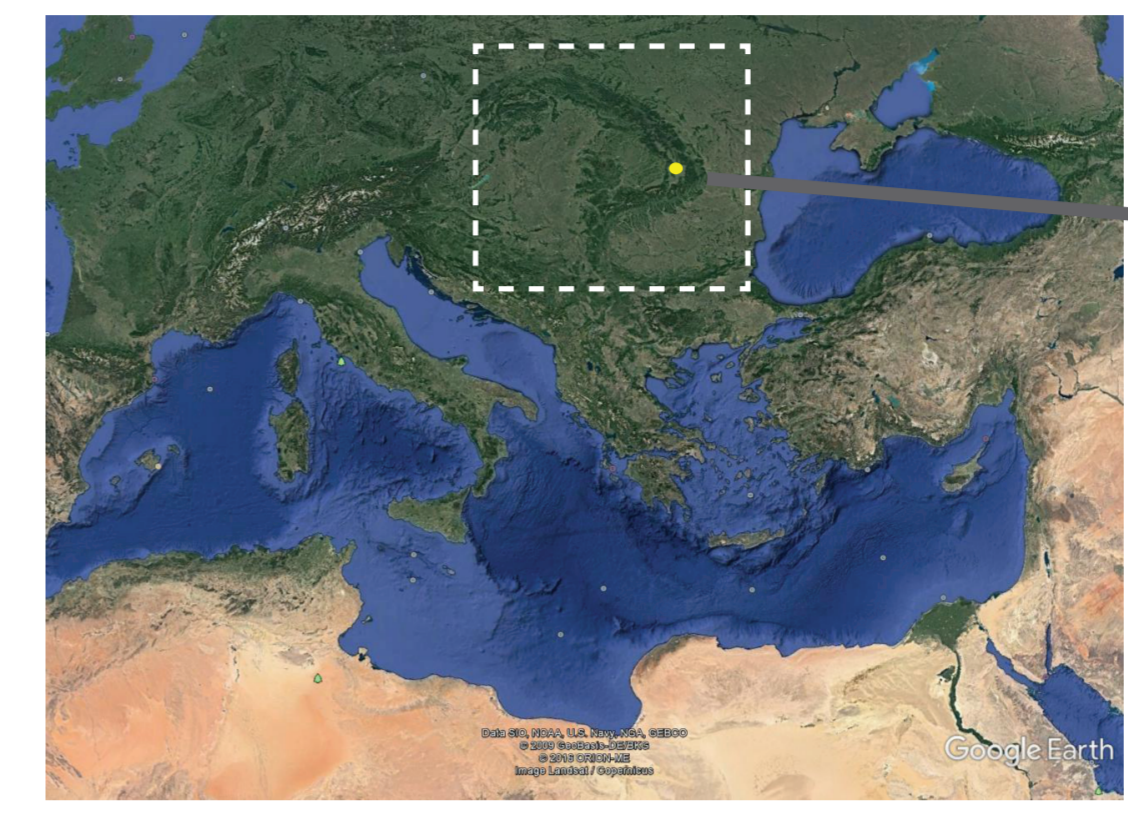
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Abstract

Located at the southern tip of the Intra-Carpathian Volcanic Range in Romania, the Ciomadul (Csomád) volcanic complex (CVC) is the youngest eruptive center of the Carpatho-Pannonian Region (CPR). It consists of a dozen of dacitic lava domes that encompass two youngest explosive craters. Whereas, in the last decade, the Ciomadul explosive history for the last 50 ka was well constrained by numerous studies, the chronology of the dome sequence still lacks robust chronological constraints, since it is based on biased K-Ar ages. Here, we provide a detailed K-Ar dating approach to refine the chronology of the domes, using the unspiked K-Ar Cassinol-Gillot techniques for the first time in Carpathian Range. New eruption ages were performed on groundmass, the only material the cooling of which is doubtlessly contemporaneous with the eruption. Each age obtained is found to be younger than those proposed on previous whole rock K-Ar datings. Our analysis shows that the K-Ar ages in the previous studies suffer from the unsuitability of the material used to obtain eruptive ages, since dating whole rock gives a mixed age due to various mineral fractions with different individual ages. A correlation between excess argon from these whole-rock measurements and the abundance of glomerocrysts is established. We also investigated the sequence of crystallisation of phenocrysts present in the lavas, with respect to the modification of the eruptive ages. We dated different phases containing potassium, i.e. feldspar, amphibole, biotite, plagioclase microlites, and groundmass. Ages obtained on feldspar, amphibole, biotite are systematically older than those obtained on the groundmass illustrating that these phenocrysts were formed before (or well before) the eruption, and brought up by the magma during the eruption. On the other hand, plagioclase microlites display a complex range of ages, from the age of groundmass to the age of plagioclase phenocrysts. The newly applied methodology, mainly consisting in a double full preparation first at larger grain size and next at < 100 micrometres, allows us to propose an appropriate procedure to separate the adequate material in order to obtain the K-Ar age of the eruption, i.e. the groundmass, in which there is no risk of presence of older, inherited phenocrysts.

Geological settings

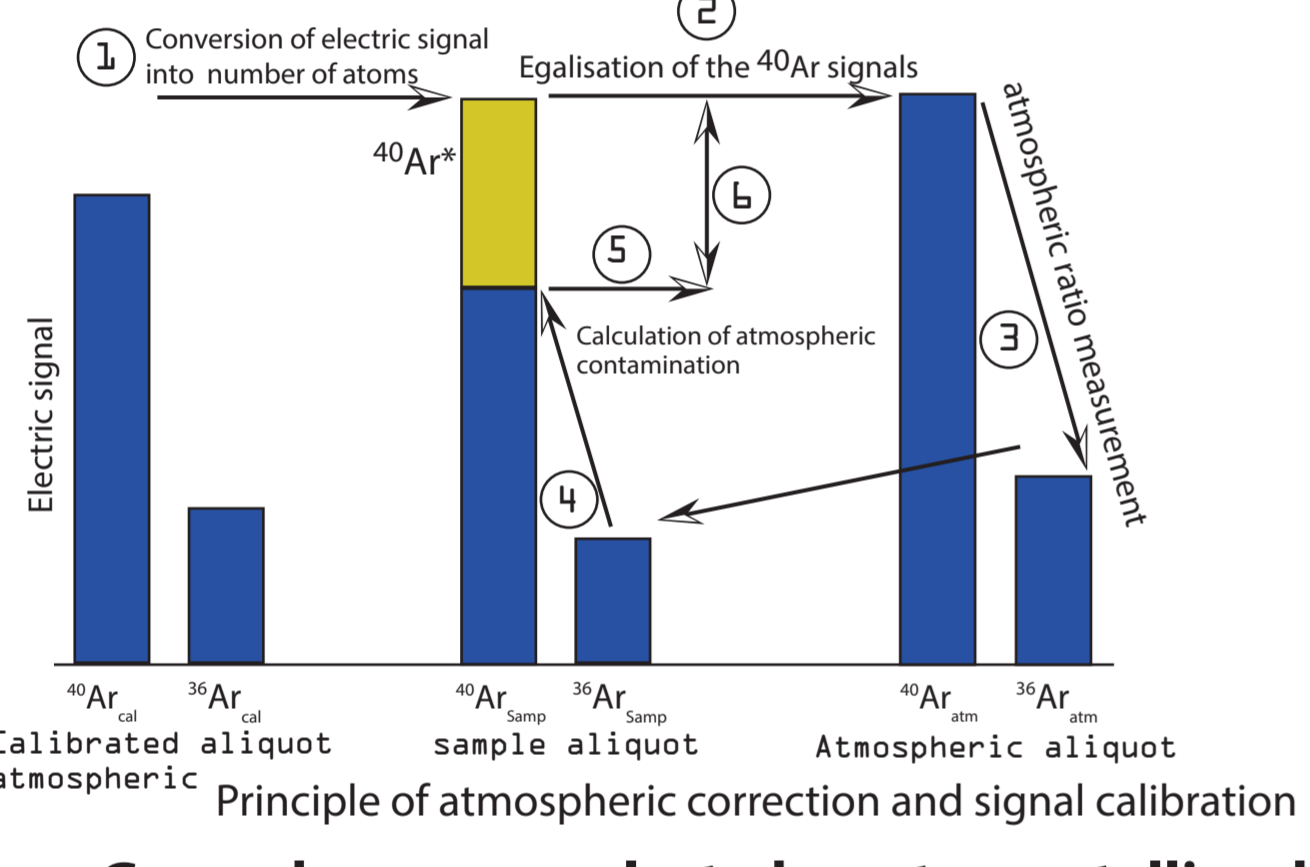
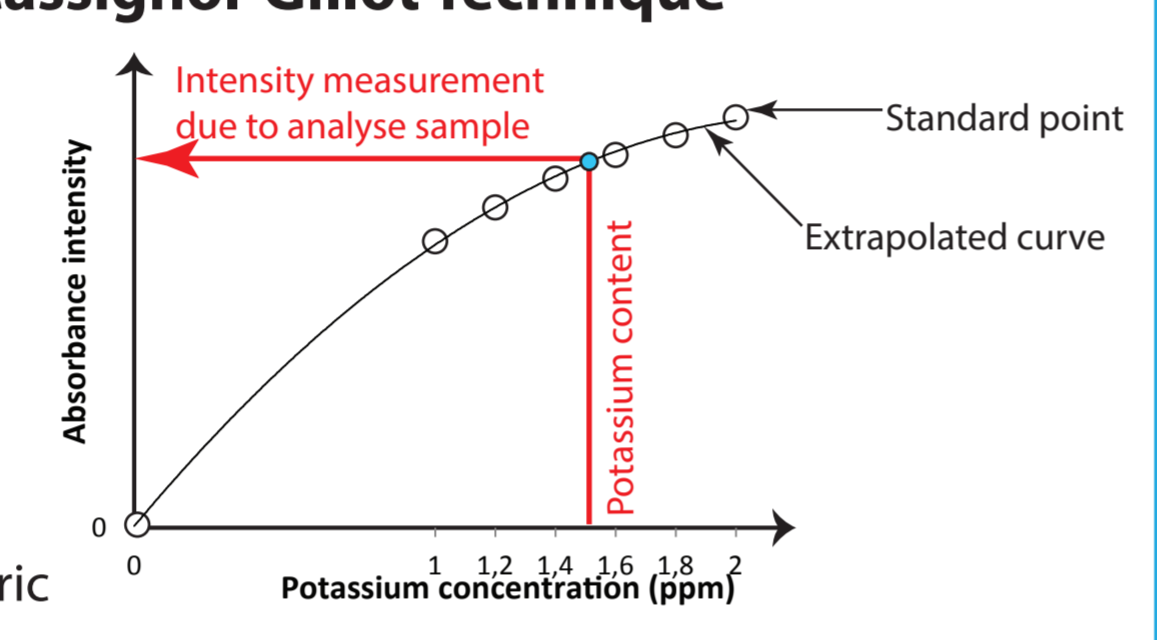


- Most recent volcanic activity in the Carpathian Range
- Consists of a dozen of dacitic lava domes
- Calc-alkaline to Shoshonitic volcanism

Methods

K-Ar dating Method with Cassinol-Gillot Technique

- Potassium content**
 - Measured by flame absorption spectrometry
 - Compared with extrapolated curve from different standard
- Argon**
 - Measured by mass spectrometry
 - Based on the double comparison of 36 and 40 isotopes of an atmospheric and a sample aliquot



- ### Why this geochronometer?
- Recent volcanic activity
 - Nature of Ciomadul rock: Cal-alkaline to alkalin rock
 - Very stable analytic condition reached by our mass spectrometer

Groundmass age = last phase to crystallize during eruption → Eruption age
Main issue is to separate by using heavy liquid in association with magnetic separator the groundmass from the important presence of inherited phenocrysts such as: **biotite, amphibole, feldspars (plagioclase)**.
Density range of groundmass in this study is between **2.40-2.51**; feldspar (**2.55-2.65**); Biotite/amphibole (**2.8-3.17**).
Observations
➤ Presence of mixed grains i.e. grains composed with groundmass/plagioclase and/or groundmass biotite, amphibole
➤ Groundmass density is to close to density of plagioclase: this could **biased** our results
For example grain with **2.50** of density could be composed of **2/3** of a 2.45 dense groundmass and a **1/3** of **2.60** dense plagioclase.

To avoid such problems, we proceeded to a double full steps separation
➤ **Double full steps separation**
Goals: separate from the groundmass any fraction of inherited mineral having an age not contemporaneous to the dome eruption. It also allows to minimize the potential risk to keep grain affected by fluid circulation.

Groundmass Preparation based on double full steps separation

A Whole rock: crushed and sieved at **250-500µm**
200 g of this is cleaned and separated using heavy liquid in association with magnetic separator in order to isolate with typical narrow density range of density for the dacitic domes of **2.46-2.51** of groundmass but not only.

B This range of density (2.46-2.51) eliminates from **G.M.** (in black) most of grain made of pure minerals, particularly inherited phenocrysts present. Mixed grains of **Amp-Bte/G.M.** are mainly excluded because of high density of mafic mineral. Mixed **plg/G.M.** grain and peripheric part of some phenocrysts remain

C Remaining grain from step (B) was crushed and sieved again at: **63-125µm**
Grains are then separated by heavy liquid a second time in order to separate the pure **G.M.** fraction (C), typically (2.46-2.50 of density) from the remaining plagioclasic grains (D).

D **Plagioclasic grains** not excluded from **G.M.** during the 1st step but removed during 2nd step
G.M. = groundmass
Amp = Amphibole
Bte = Biotite
Plg = Plagioclase

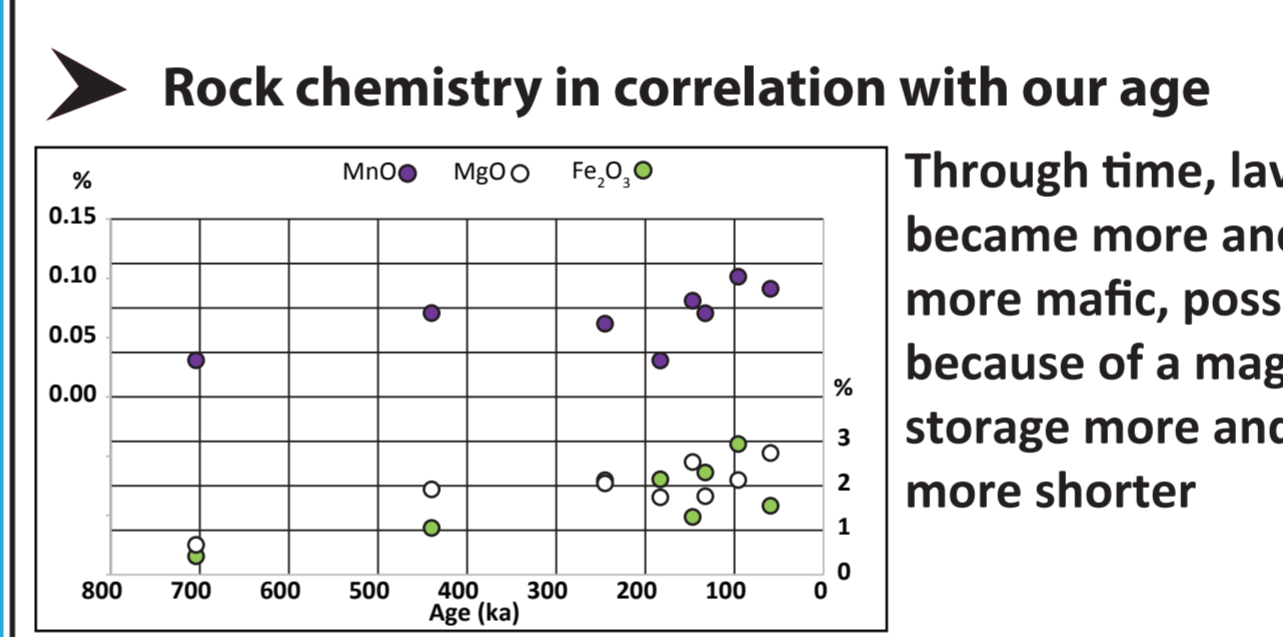
After purification of G.M. (+ magnetic separation), samples are hand-picked to check its 100 percent purity.

Results

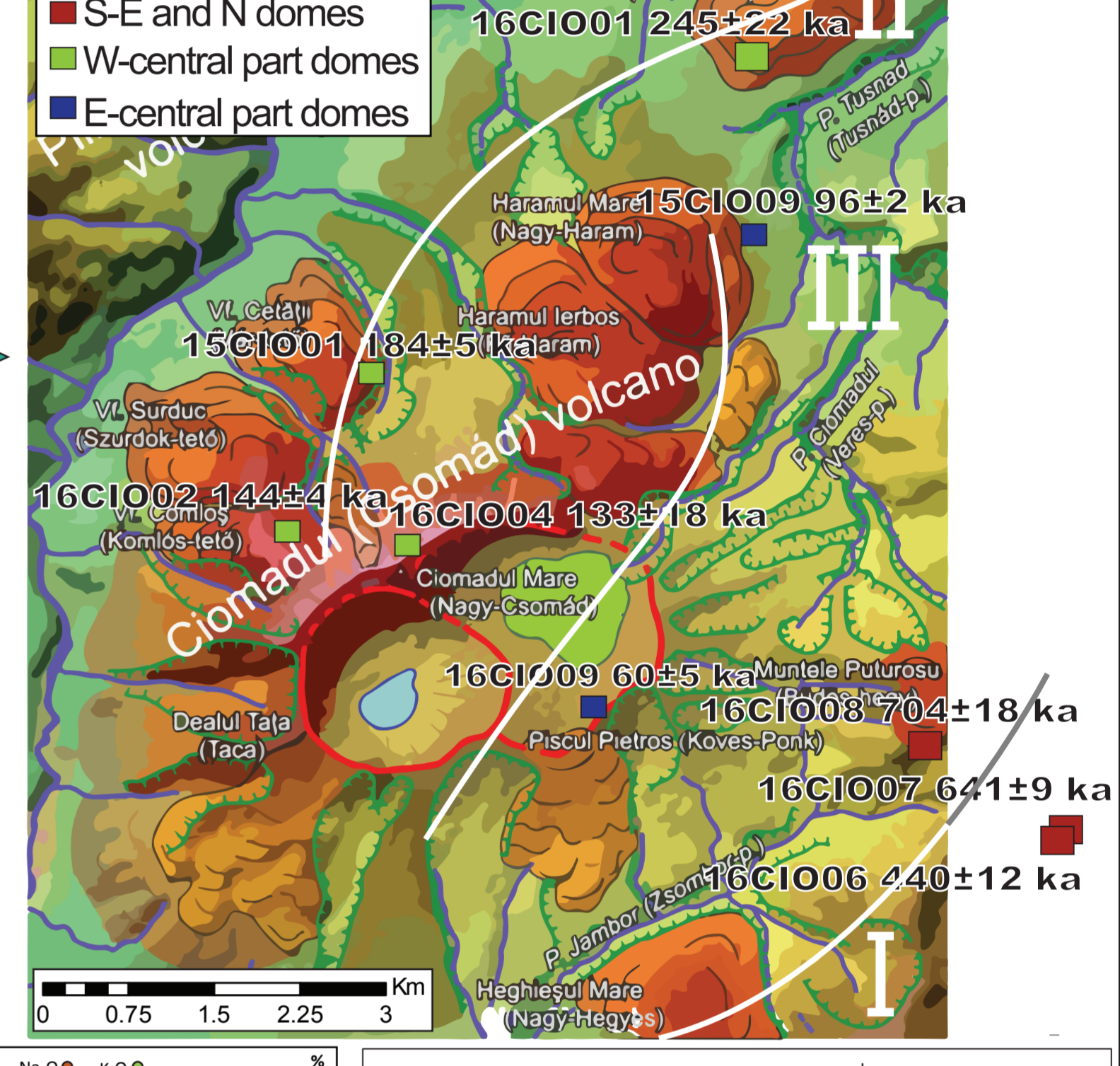
New K/Ar ages obtained in this study based on the Un-spiked Cassinol-Gillot technique

Official name	Dated phase	Method	Fraction size (µm)	K%	Mean age ± 1σ
16CIO08 Muntele Puturosu	G.M.	D.S.	63-125	3.226	704±18
16CIO07 Balvanos	G.M.	D.S.	63-125	3.585	641±9
16CIO06 Balvanos	G.M.	D.S.	63-125	3.449	440±12
16CIO00 Haramul Mic	Plag.	D.S.	63-125	1.336	245±22
15CIO01 Dealul Cetatii	G.M.	D.S.	63-125	3.722	184±5
16CIO02 Virful Comlos	G.M.	D.S.	63-125	3.471	144±4
16CIO04 Ciomadul Mare	µl Plag.	D.S.	40-80	1.391	133±18
15CIO09 Haramul Mare	G.M.	D.S.	63-125	3.668	96±2
16CIO09 Piscul Pietros	G.M.	D.S.	63-125	3.441	60±5

G.M. = Groundmass; D.S. = Double separation
µlplag = Plagioclase Microlite

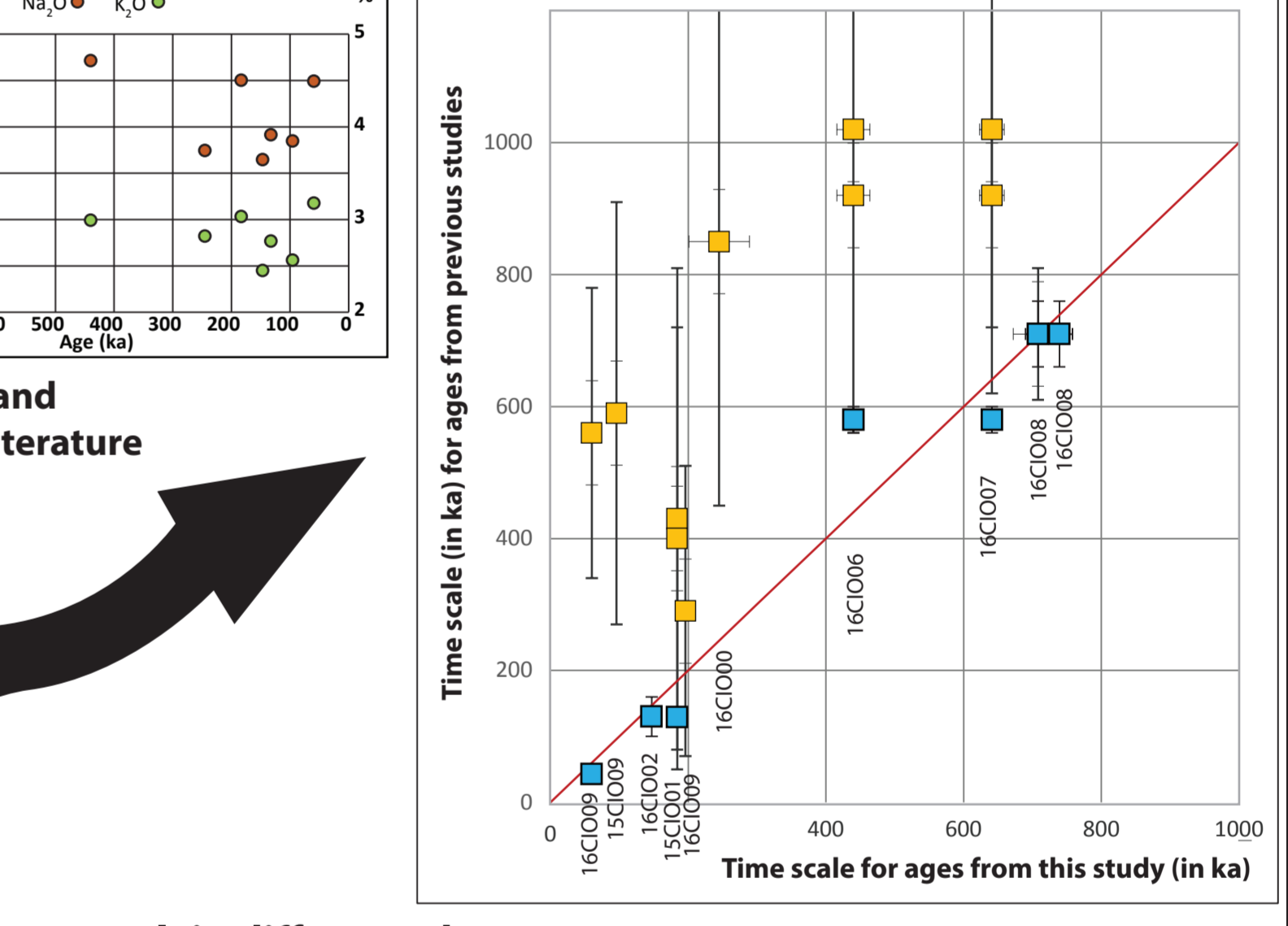


Spatial Evolution



Previous studies based on traditional K/Ar dating (Whole rock = WR) and dating performed on Zircon by (U-Th)/He method according to the literature

Official name	Dated fraction	W.R. age (ka)	Method	Dated fraction	(U-Th)/He age ± 1σ
16CIO08 Muntele P	W.R.	710±40	(U-Th)/He	Zircon	710±50
16CIO07 Balvanos	W.R.	1020±150	(U-Th)/He	Zircon	580±20
16CIO07 Balvanos	W.R.	920±180	(U-Th)/He	Zircon	580±20
16CIO00	W.R.	850	(U-Th)/He	Zircon	??
15CIO01 Dealul Cetatii	W.R.	400±160	(U-Th)/He	Zircon	129±13
16CIO02 Virful Comlos	W.R.	430±190	(U-Th)/He	Zircon	130±30
16CIO04 Ciomadul Mare	W.R.	??	(U-Th)/He	Zircon	??
15CIO09 Haramul Mare	W.R.	590±160	(U-Th)/He	Zircon	??
16CIO09 Piscul Pietros	Biotite	560±110	(U-Th)/He	Zircon	42.9±1.5



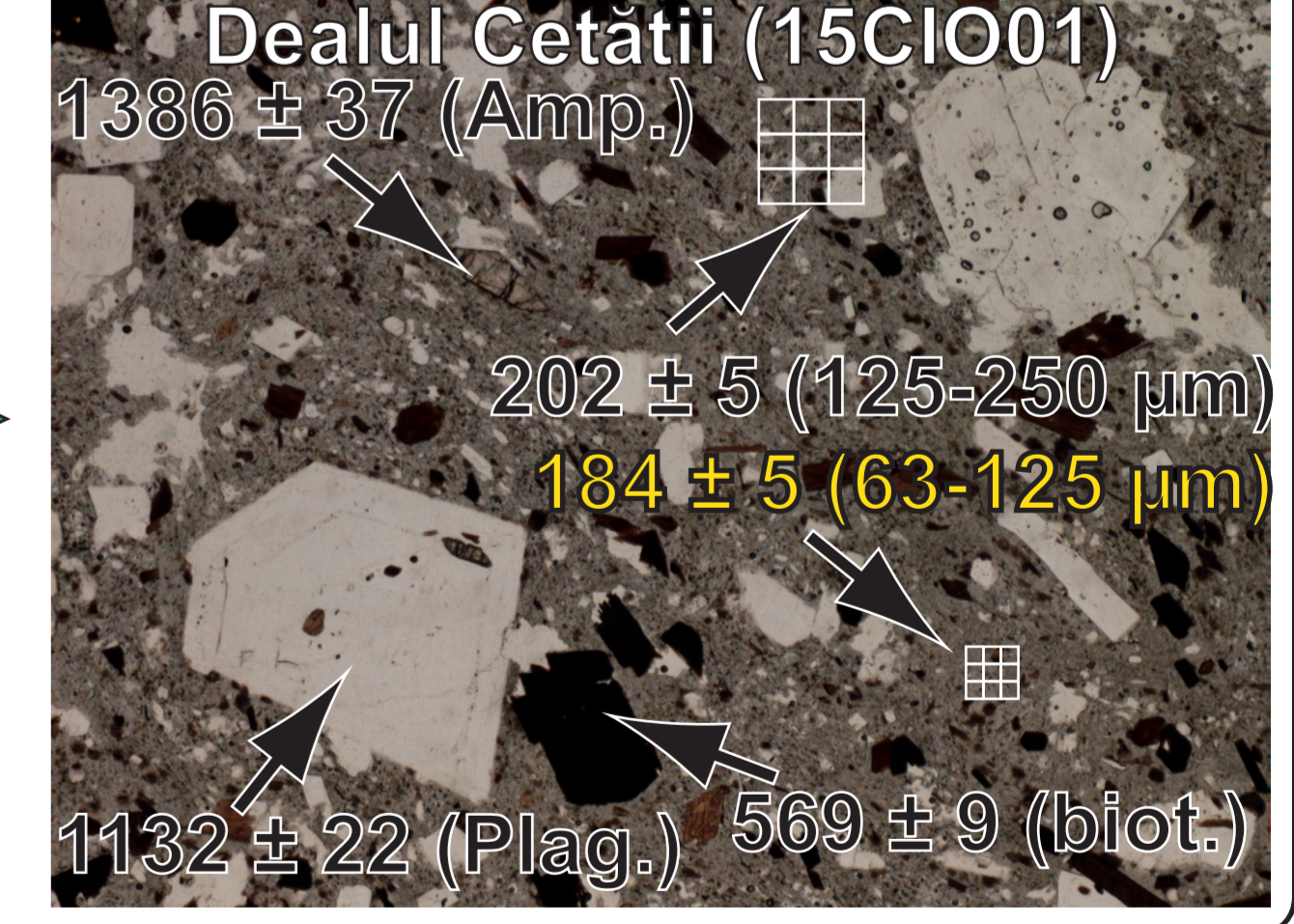
Analyse of the origin of the biasing of whole-rock age by presence of phenocrysts

Age obtained on pure separated minerals

Sample code	Dated phase	Fraction size (µm)	K%	Mean age ± 1σ
15CIO01	Amp	125-250	0.931	1386±37
15CIO01	Plag.Gl	125-250	0.822	1132±22
15CIO01	Biot.	125-250	6.532	569±9

Influence on the groundmass age of the single and double steps separation methods

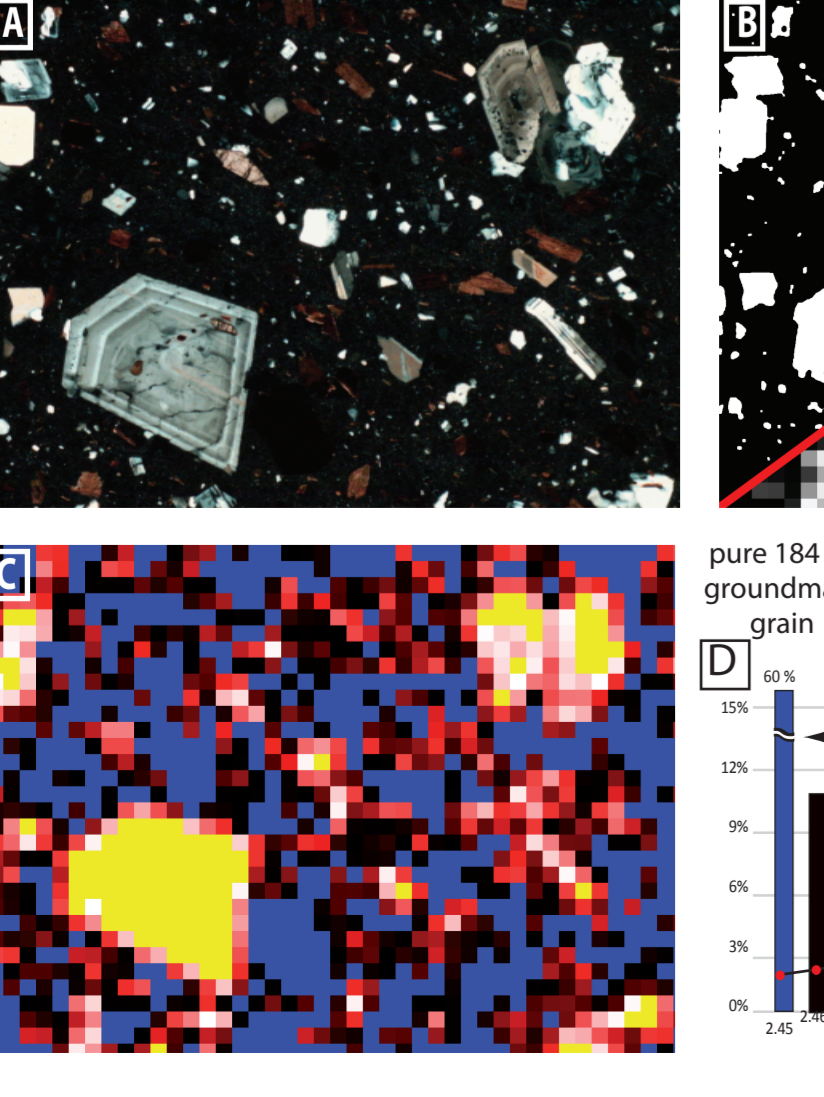
Sample code	Dated phase	Fraction size (µm)	K%	Mean age ± 1σ
15CIO01	Single step	125-250	3.612	202±6
15CIO01	Double step	63-125	3.612	184±5



Discussion

Known parameters
Pure groundmass
Age (a_g): 184±5ka
Proportion (P_i): 60%; K% (K_g): 3.72
Pure Plagioclase
Age: 1132±22 Ma
Proportion: 11%; K% (K_g): 0.82%
Mixed grains
Proportion (P_i): 29%

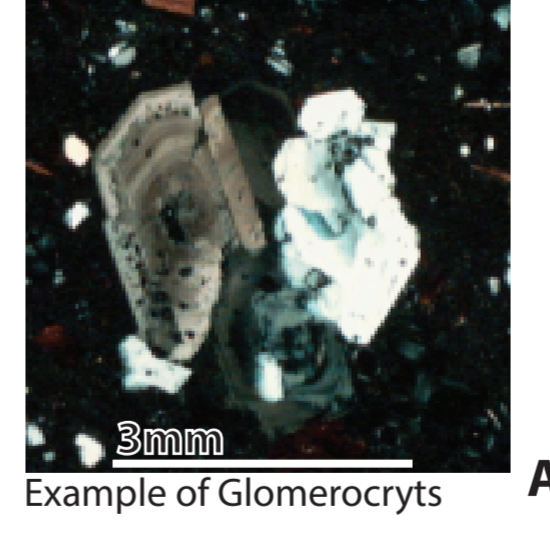
Whole-rock age simulation from Image analysis of thin section
In order to test the whole rock incidence and show that ages obtained on whole rock are biased. An image simulation of grain composition has been done from the thin section of 15CIO01 sample coming from Dealul Cetatii (A) in different steps:



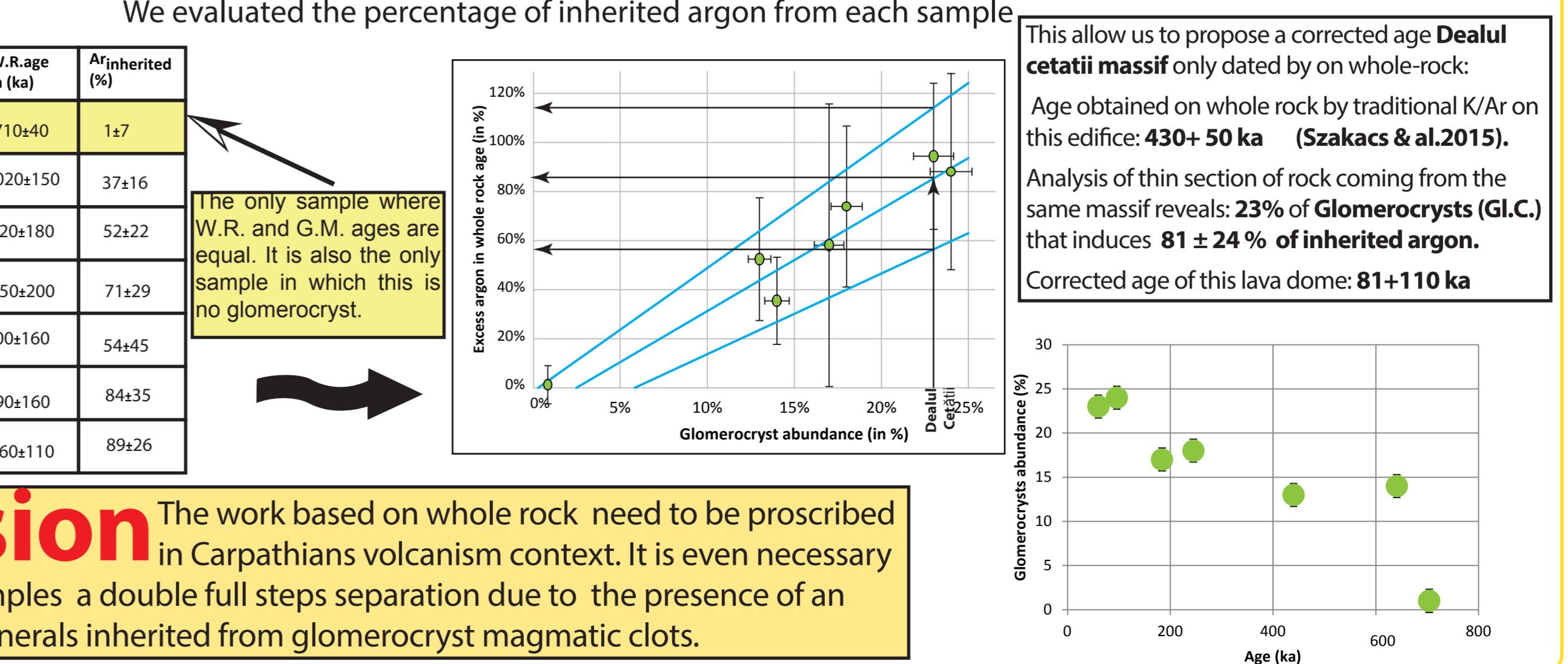
- Thin section image (A) is binarized on gray scale range (B1) and pixelated (B2) to simulate a 200 µm size grains
- Determination of each grain composition from their color (C): Blue (pure G.M. grain), Yellow (pure plagioclase grain), Range of red (Mixed grain)
- Estimation of age of whole rock and for each grains (D):
 $Age = \sum a_i \cdot p_i \cdot K_i / \sum p_i \cdot K_i$
Where a_i, p_i and K_i are the age, the proportion and K content of each phase present in each grain i obtained age:
320 ka → 74% elder than groundmass age
- Generally, fractionation occurs during rock crushing with a larger proportion of groundmass than phenocrysts being crushed at lower size than 200 µm. For instance if a third of G.M. is removed during crushing and no Plg is lost, the resulting proportion between G.M. and Plg would induce an age of **405 ka**

Quantification of the abundance of glomerocrysts on whole-rock age

Glomerocrysts are aggregates of phenocrysts (> 1 mm and up to 5 mm in size), with cumulate texture containing mainly plagioclases and amphibole and secondarily Fe-Ti oxides, apatite, biotite and zircon.



- **Mineralogical analyses**
Evaluation of percentage of glomerocrysts in each rock (thin section) dated on both whole-rock (previous studies) and pure separated groundmass (this work)
- Assuming that the principal reason of difference of our age with traditional K/Ar dating is due to inherited argon.



Conclusion

The work based on whole rock need to be proscribed in Carpathians volcanism context. It is even necessary to abandon for such samples a double full steps separation due to the presence of an abundant amount of minerals inherited from glomerocryst magmatic clots.