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Preliminary dating of the Viluy traps (Eastern Siberia): Eruption at the time of Late Devonian extinction events?

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ABSTRACT

In this short note, we report new age determinations from four samples of the Middle-Paleozoic Viluy Traps in Siberia, east of the more famous Permo-Triassic Siberian Traps. These samples, which were collected from three drill cores, have been analyzed in parallel and independently in the Orsay (France) and Berkeley Geochronology Center (BGC; USA) geochronology laboratories, using respectively the Cassignol-Gillot K-Ar and the $^{40}Ar/^{39}Ar$ techniques. Dating these samples as a concerted effort in two independent laboratories working jointly on their interpretation is a rather rare yet very valuable exercise. With the K-Ar technique, ages ranging from 338 to 367 Ma with uncertainties on the order of 5 Ma were obtained. With the $^{40}Ar/^{39}Ar$ technique, integrated ages range from 344 to 367 Ma, with uncertainties on the order of 1 Ma, and two samples yielded plateaus, i.e. the best determined ages, at 360.3 ± 0.9 and 370.0 ± 0.7 Ma. Three out of four ages yielded by the two separate methods are in agreement within uncertainties. One sample yields incompatible ages and could be from a later, altered dyke event. The $^{40}Ar/^{39}Ar$ plateau age of 370.0 ± 0.7 Ma (conventional calibration) or 373.4 ± 0.7 Ma (recalculated per Renne et al., 2010), the most reliable age obtained in this study, is compatible with recent determinations of the Late Devonian extinction events at the end-Frasnian (~376 ± 3 Ma). These results underscore a need for further work, in progress.

1. Introduction

In the course of a study of the rock- and paleo-magnetic properties of samples collected from volcanic outcrops related to (sometimes diamond-bearing) kimberlite pipes in the Viluy area, east of the Permo-Triassic Siberian Traps province, Kravchinsky et al. (2002) found two distinct paleomagnetic poles. One of these poles could unambiguously be correlated with the 250 Ma-old Siberian Traps (e.g. Pavlov et al., 2007) and therefore the magmatic samples could be assigned that age, corresponding to the Permo-Triassic boundary. The other pole was at a previously unknown location. Rather imprecise dating allowed its assignment to the period from 350 to 380 Ma, which Kravchinsky et al. (2002) noted included the Frasnian-Fammenian (FF) boundary, the largest mass extinction event prior to the Permo-Triassic and Guadalupian-Tatarian extinctions near the end of the Paleozoic. This led to the suggestion that the corresponding magmatism could have been related to a major flood basalt event, emplacing a large volume of magma at the FF boundary some 370 Ma ago. This would be an interesting indication of yet another such event related to diamond-bearing kimberlites, and would almost complete the list of Phanerozoic flood basalt/mass extinction correlations (summarized by Courtillot and Renne, 2003), with a single event at the time of the end-Ordovician mass extinction still unrecognized. Since that suggestion was made, the age of the Frasnian-Famennian boundary has been significantly revised. Based on the interpolation between two bentonites dated using U/Pb at 381.1 ± 1.3 and 363.6 ± 1.6 Ma (Tucker et al., 1998), the age of the FF boundary was interpolated at 376.5 Ma, some 10 Ma older than most previous estimates; as a result, Gradstein et al. (2005) placed the FF boundary in their new geological time scale at 374.5 ± 2.6 Ma. Using the same approach, a slightly older age of 376.1 ± 1.6 Ma has recently been proposed (Kaufmann et al., 2004) and subsequently revised to 376.1 ± 3.6 Ma (Kaufmann, 2006). In the debate on the causes of mass extinctions, meteorite or comet impacts have been the main candidate often opposed to volcanism. In their review, Courtillot and Renne (2003)

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noted that most mass extinctions had an associated flood basalt (or large igneous province – LIP), whereas only the Cretaceous-Tertiary extinction also had a reasonably well demonstrated impact. As far as the Frasnian-Famennian extinction event is concerned, association with the Siljan impact structure in Sweden, 65 to 75 km in diameter, has been proposed (see for instance review in Reimold et al., 2005). Reimold et al. have obtained new laser $^{40}Ar/^{39}Ar$ dates on melt breccias from the structure at 377 ± 2 Ma (95% confidence limits), making it coeval with the revised Frasnian-Famennian boundary age. However, these authors conclude that “it is unlikely that these events of relatively minor magnitude … would have resulted in a major global extinction event”. More recently, Gordon et al. (2009) have argued based on Osmium isotopic evidence against an impact at the FF boundary.

We have therefore undertaken a preliminary attempt to date four samples from the Viluy Traps which were collected in 2000 by one of us (DG). This preliminary attempt, undertaken in parallel and independently between two laboratories (XQ in Orsay and PR at BGC) in order to test the robustness of the first results and the methods they are based on, is reported in this short note.

2. Regional geological setting and geology of the sampling sites

A Middle Paleozoic LIP in the eastern part of the Siberian platform (Fig. 1) is well exposed along the Viluy, Markha and Lena rivers, and along some rift faults on the slopes of the Anabar and Aldan shields (e.g. Gusev and Shpount, 1987; Kiselev et al., 2007). This LIP has been referred to as the Yakutsk LIP by e.g. Ernst and Buchan (1997) and as the Viluy Traps by e.g. Kravchinsky et al. (2002). The intrusive rocks include dikes, sills and layered basalt breccias. Middle Paleozoic basalt outcrops have been mapped in several parts of the rifts, often interbedded with ash and tuffs. Volcanic products and sediments of Late Devonian – Early Carboniferous age cover 6–8 kilometers of sediments of Late Proterozoic and Early Paleozoic, up to Silurian age. LIP-derived conglomerates are known in sediments with ages younger than Tournaisian. Paleozoic dikes cut across Middle-Late

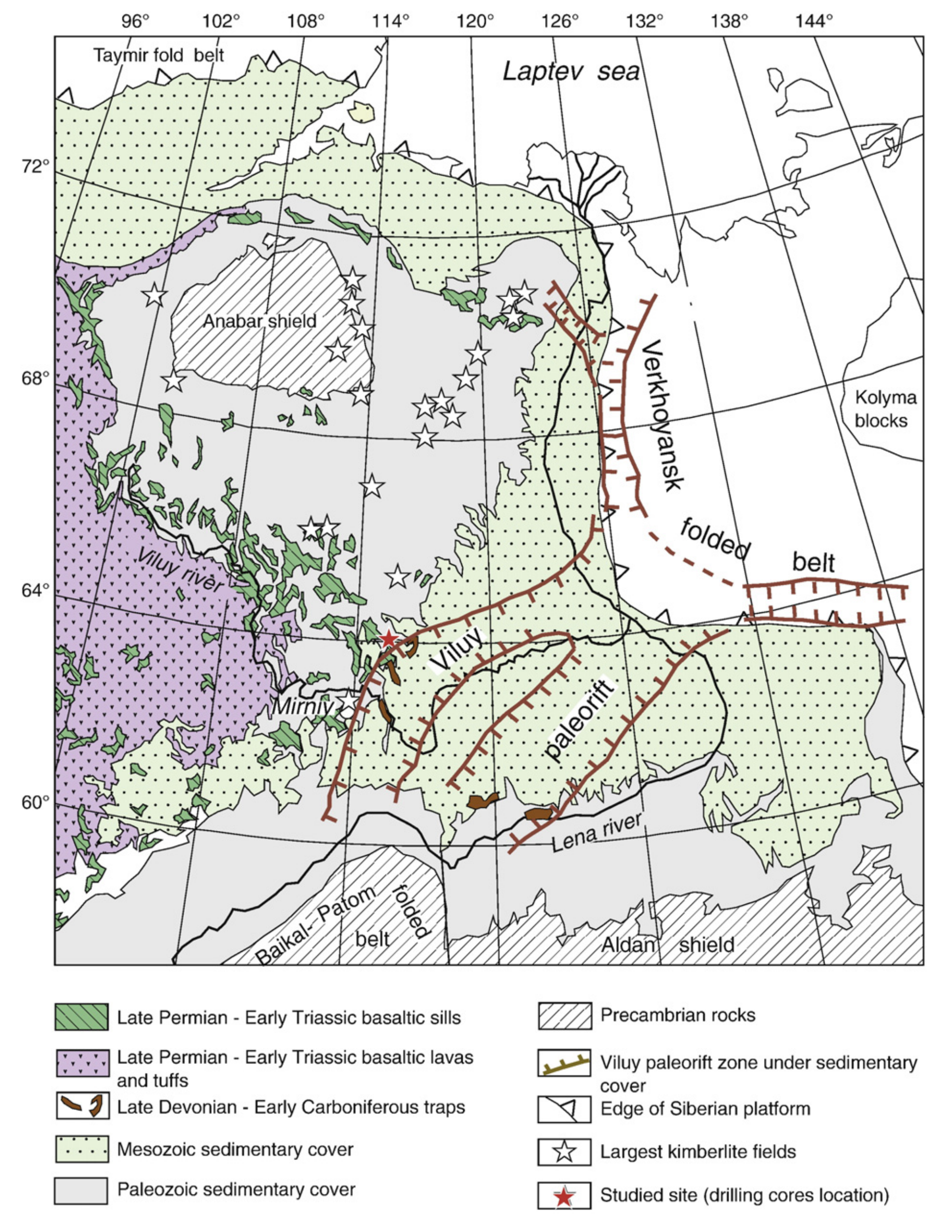

Fig. 1. General structure of the Siberian and Viluy Traps in Siberia and location of the three drill cores having yielded the four samples studied in this paper (red star).

Devonian sediments in this area. Sills are spread over the central part of the Viluy area. Intrusions under sedimentary cover are spread along the edges of the paleo-rifts, in association with large regional normal faults. Effusive volcanism is common in the lower parts of the sedimentary sections. The Viluy "aulacogen" extends over 600 km in a NE direction and plunges under the Verkhoyansk foldbelt (Fig. 1). Remaining volume of the province (much of which has most likely been eroded away or is still covered by the Siberian Traps to the West) is quite large – about 300,000 km^3 for magmatic rocks in the Viluy aulacogen alone (Kuzmin et al., 2010).

A system of grabens and minor depressions developed in the Viluy rift, accompanied by a large volume of basalt intrusions (sills and dykes) (Kiselev et al., 2006). A magmatic depression with a sediment thickness of 3-7 km formed in the western section of the rift at the same time. The intensity of tectonic movements and volcanic activity gradually declined during the Early Carboniferous period. A large amount of magmatic material (more than 300,000 km^3) was emplaced in the southwestern Viluy branch of the triple-junction system (Fig. 1). By extending this amount of magmatism to all three branches of the rift (western, eastern and northern), we may assign a volume of one million km^3 to the Viluy rift.

The age of this LIP was previously constrained by less than a dozen K-Ar ages with large scatter, obtained over the past 50 years and ranging from 450 to 320 Ma. Most ages are clustered near 340-380 Ma (Shpount and Oleinikov, 1987).

In order to attempt a pilot test of the hypothesis formulated by Kravchinsky et al. (2002), one of us (DG) provided samples from several available drill cores (Fig. 2). Dolerite samples were taken from drill cores in the Nakynsk kimberlite area during the summer of 2000. Coordinates of the sampling area are: 64.5°N, 116.5°E (the three drill holes are only a few km from each other). They belong to a 700 km-long dyke complex of the Viluy Middle Paleozoic Rift System. The rock samples comprise tholeites and sub-alkaline basalts, composed mainly of plagioclase and clinopyroxene. Olivine represents less than 5% of the total volume.

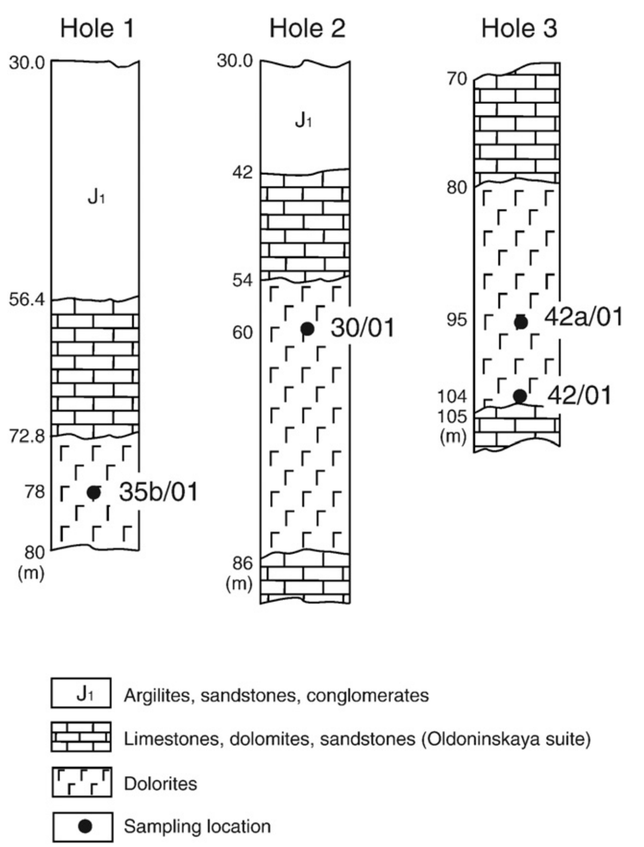

Fig. 2. Sample locations in the three drill cores as a function of depth in the core and lithology.

Four samples were obtained, which were split in two each and analyzed for age in parallel and independently in the LGMT Orsay geochronology laboratory (XQ) using the K-Ar Cassignol-Gillot technique (e.g. Gillot and Cornette, 1986) and in the BGC laboratory (PR) using the $^{40}Ar/^{39}Ar$ laser stepwise heating technique (e.g. Renne, 2000). Results are given in Tables 1 and 2 and are illustrated in Figure 4.

3. K-Ar technique and results (Orsay laboratory)

Two main size fractions of plagioclase crystals are present in both samples 30/01 and 35b/01, while only one rather homogeneous size fraction is observed for 42/01 and 42/01a (Fig. 3). Being an incompatible element, K tends to remain concentrated in the melt or in the last crystallizing minerals, i.e. in the smaller plagioclase size fraction for the dolerites analyzed here. Therefore, we applied the K-Ar dating technique preferentially to the smallest size fraction. This required performing a careful double mineralogical separation to prevent mixing of the different size fractions. We followed the approach successfully applied to K-Ar dating of the deep-sea oceanic crust (Gillot and Hildenbrand, 2000). Samples were crushed between 0.5 and 1 mm size, then sorted by density using heavy liquids to separate the plagioclase phenocrysts from the heavier aggregates, which include mafic minerals and plagioclase microlites. Then, these aggregates were crushed again to a smaller size fraction (typically 0.125-0.250 μm) and plagioclase microlites were then isolated using heavy liquids. Finally, magnetic separation was performed to improve the purity of both plagioclase size fractions.

K was measured by flame emission spectrometry and compared with reference values of the MDO-G and ISH-G standards (Gillot et al., 1992). Ar was measured with a mass spectrometer identical to the one described by Gillot and Cornette (1986). The interlaboratory standard GL-O, with the recommended value of 6.679x10^{14} atom/g of ^{40}Ar (Odin et al., 1982), was used for ^{40}Ar signal calibration. Typical uncertainties of 1 % are achieved for the ^{40}Ar signal calibration and K determination. The uncertainty on the ^{40}Ar determination is a function of the radiogenic content of the sample. Since radiogenic yield measured here is high (Table 1), this uncertainty becomes negligible. Decay constants and isotopic ratios from Steiger and Jäger (1977) were used. Uncertainties are given at the 1 sigma level.

Table 1 shows the K-Ar ages obtained on microlites from samples 30/01 and 35b/01, and on phenocrysts from all samples. The plagioclase phenocrysts (0.5-1 mm) show a lower K content (by 2-3 times), as expected, but yield consistent ages (at the 1 sigma level) with the plagioclase crystals present in the microlite fraction. Phenocrysts and microlite ages have thus been combined and yield

Table 1
K-Ar ages (Orsay laboratory) based on Steiger and Jäger (1977) constants.

Sample	Plagioclase fraction	K%	% ^{40}Ar*	^{40}Ar* (at/g)	Age (Ma)	± 1
30/01	phenocrysts 0.5-1 mm	0.202	83.7%	8.67309E + 13	370.3	5.3
"	microlites 0.12-0.25 mm	0.355	93.4%	1.49132E + 14	363.1	5.1
"	"	0.429	93.6%	1.83185E + 14	368.5	5.2
				mean:	367.2	5.2
35b/01	phenocrysts 0.5-1 mm	0.330	90.3%	1.30939E + 14	344.8	4.9
"	microlites 0.12-0.25 mm	0.954	94.9%	3.76276E + 14	342.9	4.9
"	"	"	91.3%	3.72241E + 14	339.5	4.8
				mean:	342.4	4.9
42/01	0.12-0.25 mm	0.427	92.1%	1.65910E + 14	338.2	4.8
"	"	"	89.1%	1.65702E + 14	337.8	4.8
				mean:	338.0	4.8
42a/01	0.12-0.25 mm	0.262	92.7%	1.06881E + 14	353.6	5.0
"	"	"	82.8%	1.06358E + 14	352.0	5.0
				mean:	352.8	5.0

Table 2
$^{40}Ar/^{39}Ar$ ages (BGC) and comparison with K-Ar ages (Orsay).

Sample	Plateau age (Ma)	Integrated age (Ma)	K-Ar age (Ma)	Decay Constant
30/01	370.0 ± 0.7	367.0 ± 2.0	367.2 ± 5.2	Steiger and Jäger (1977)
	373.4 ± 0.7	370.4 ± 2.0	370.3 ± 5.3	Renne et al. (2010)
35b/01	none	344.3 ± 1.9	342.4 ± 4.9	Steiger and Jäger (1977)
	none	347.5 ± 1.9	345.3 ± 5.0	Renne et al. (2010)
42/01	none	349.4 ± 1.9	338.0 ± 4.8	Steiger and Jäger (1977)
	none	352.6 ± 1.9	340.9 ± 4.9	Renne et al. (2010)
42a/01	359.9 ± 1.9	356.0 ± 2.0	352.8 ± 5.0	Steiger and Jäger (1977)
	363.2 ± 2.0	359.3 ± 2.0	355.8 ± 5.1	Renne et al. (2010)

K-Ar ages of 367.2 ± 5.2 and 342.4 ± 4.9 Ma, obtained for 30/01 and 35b/01, respectively. Ages of 338.0 ± 4.8 and 352.8 ± 5.0 Ma have been obtained for samples 42/01 and 42a/01, respectively. They display a more homogeneous plagioclase size fraction, which prevented us from performing the double mineralogical separation for these samples.

Since 30/01 and 35b/01 were sampled in two different holes (Fig. 2), no stratigraphic relationship can be established between them. On the other hand, our K-Ar age of 338 ± 5 Ma suggests that 42/01 is a sill or dike intrusion, which was emplaced close to the contact between the Oldoninskaya suite sediments and the overlying dolerites, about 15 Ma after the volcanic stage dated at 353 ± 5 Ma (Table 1). Note that the age of 342 ± 5 Ma obtained for 35b/01 (Hole 1; Fig. 2) might also be interpreted as coming from that late stage volcanism.

4. $^{40}Ar/^{39}Ar$ technique and results (BGC)

Samples for $^{40}Ar/^{39}Ar$ dating were prepared independently from those for K-Ar analysis. Plagioclase phenocryst fragments from the 420-840 μm size fractions were separated from the matrix and selected for freedom from alteration and inclusions. Because of the high sensitivity of the experiment, no effort was made to concentrate the higher-K smaller plagioclase microlites. After irradiation with the FCs standard (28.02 Ma; Renne et al., 1998), 10-15 grains of each sample were analyzed by stepwise heating with a CO_2 laser using techniques described by Renne (2000). Ages are given with 1 sigma uncertainties, and are relative to the constants of Steiger and Jäger (1977). Results are given in the first three columns of Table 2.

Two of the samples (30/01 and 42a/01) yielded well-defined plateau ages of 370.0 ± 0.7 and 360.3 ± 0.9 Ma, respectively (Fig. 4). The integrated ages of all samples, which are analogous to K-Ar ages, are slightly lower than the plateau ages for the two samples yielding age plateaux. In addition, their low temperature disturbance could be consistent with minor ^{40}Ar loss. However, the two samples (35b/01 and 42/01) yielding more discordant age spectra (Fig. 4), which failed to define plateaux, are characterized by severely undulating Ca/K spectra (obtained from the neutron-activation products ^{37}Ar and ^{39}Ar),

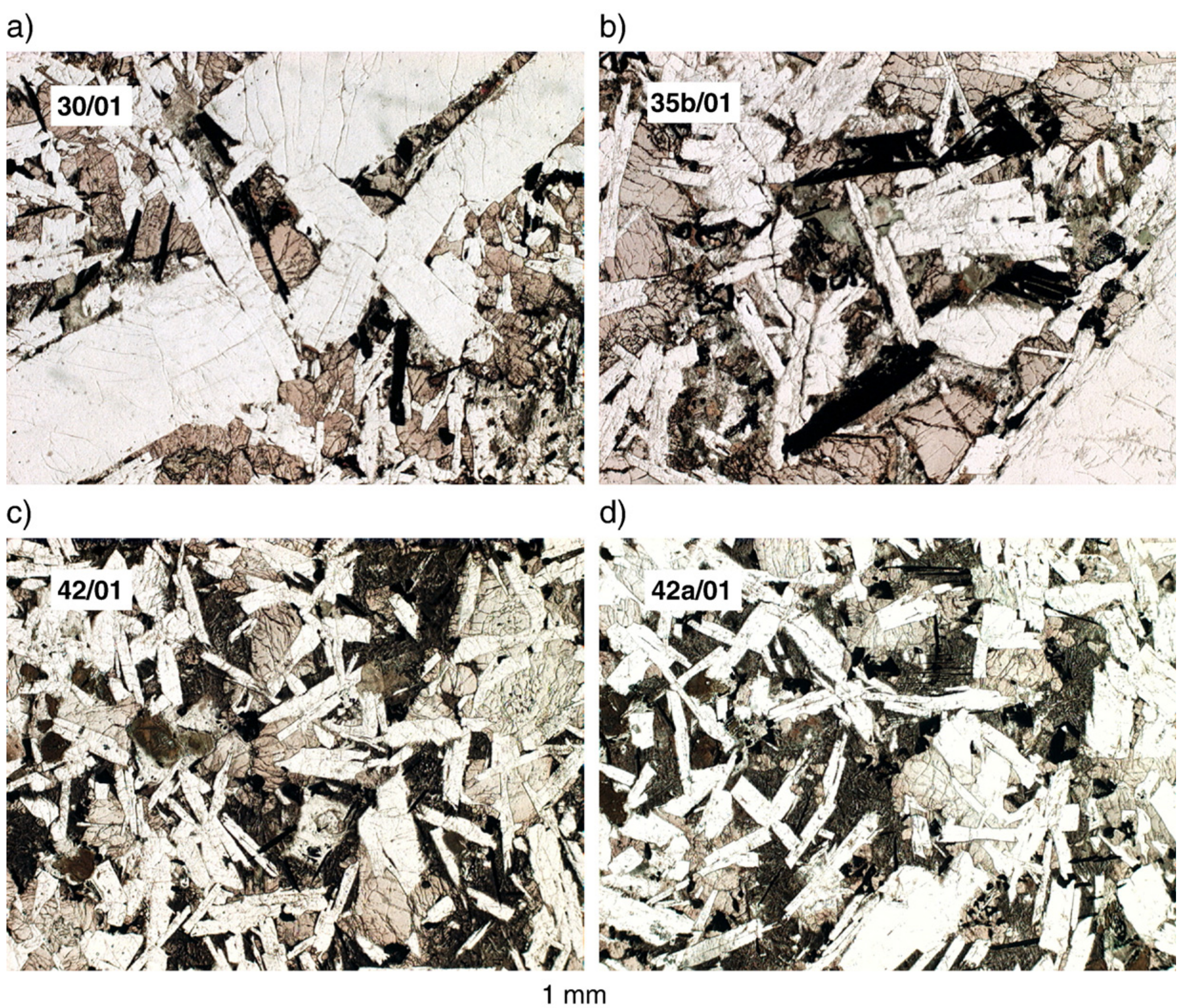

Fig. 3. Photomicrographs of thin sections from each sample in plane-polarized light. Note the presence of large plagioclase phenocrysts in samples 30/01 and 35b/01.

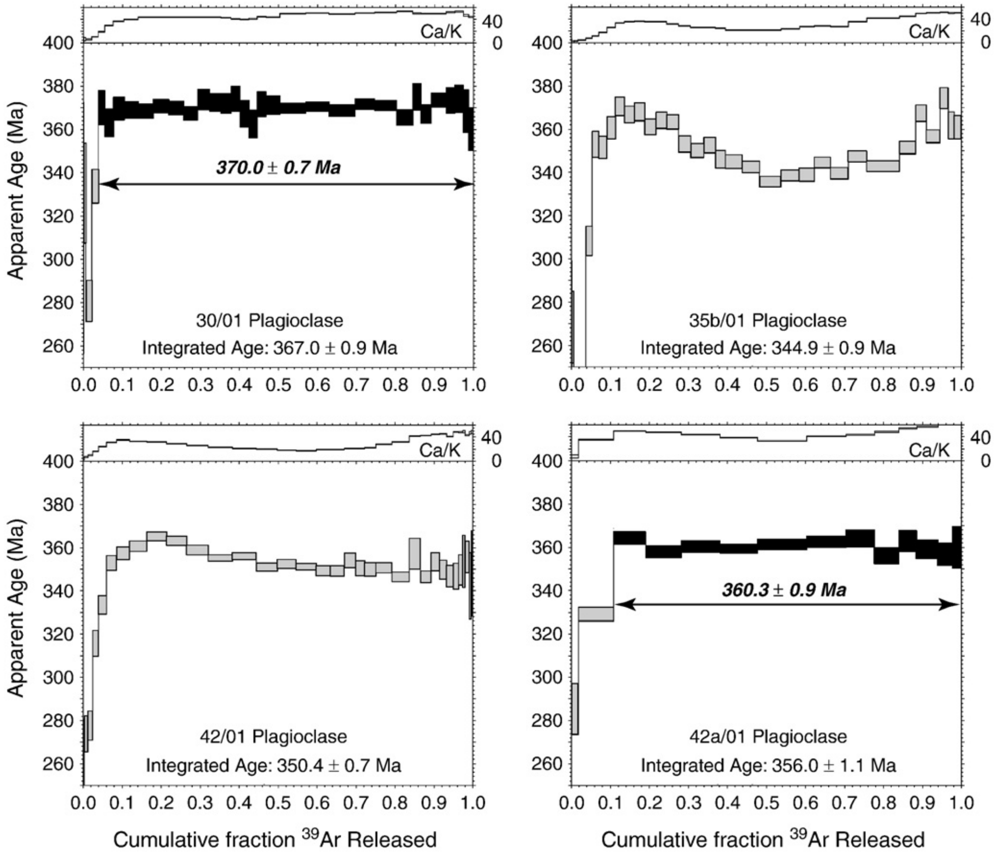

Fig. 4. $^{40}Ar/^{39}Ar$ apparent age spectra for the samples indicated. Ages shown are based on decay constants of Steiger and Jäger (1977) and 28.02 Ma for the FCs standard (Renne et al., 1998). Upper plot in each panel shows apparent Ca/K determined from Ar isotope data. All errors are 1 sigma. Plateau ages are denoted by arrows showing included steps. Integrated ages are calculated from summing total gas released in all steps.

which is tentatively identified with a submicroscopic scale antiperthite exsolution.

5. Electron microprobe analysis of plagioclase

Plagioclase phenocrysts from samples 42/01 and 42a/01 were analyzed by electron microprobe at the University of California (Berkeley) Dept. of Earth and Planetary Science. Methods and facilities were similar to those described by Cassata et al. (2009). In each case a core to rim traverse was analyzed. Both samples show normal zoning overall, with more calcic core and more sodic rims, but with some significant reversals. 42/01 ranges from An_{66} to An_{86}, and 42a/01 from An_{48} to An_{82}. Both of these compositions overlap the range in which the Huttenlocher exsolution gap can occur at An_{70} to An_{90}, and the latter also overlaps the Boggild gap which can occur at An_{35} to An_{65} (Wenk and Nakajima, 1980).

A second feature of interest is that the Ca/K values determined by microprobe reach much higher values than is suggested by the $^{40}Ar/^{39}Ar$ data (332 vs. 49 for 42/01 and 278 vs. 64 for 42a/01). This is easily attributed to the tendency of multigrained stepped heating to homogenize gas from different portions of broken zoned crystals. If this is indeed the explanation for the more restricted range of Ca/K implied by the Ar isotope data, then it suggests that the true amplitude in age variations, as with Ca/K, may be much larger in individual crystals.

6. Discussion and preliminary conclusion

Ages measured from the same samples by the two laboratories are in general in reasonable agreement. The most directly comparable results (Table 2, last two columns) are the $^{40}Ar/^{39}Ar$ integrated ages (BGC) and the K-Ar ages (Orsay), which in principle, if interfering Ar isotopes produced during irradiation were perfectly conserved, should be the same (except that K is measured by neutron activation in the former). Three of the four samples yielded indistinguishable ages on this basis, although it is noteworthy that the K-Ar results even in these three cases are systematically younger. However, when we compare analyses from comparable size fractions (plagioclase phenocrysts) for 30/01, indistinguishable mean ages of 370.3 ± 5.3 and 370.0 ± 0.7 Ma (Tables 1 and 2) have been obtained by the K-Ar and $^{40}Ar/^{39}Ar$ techniques, respectively (but with quite different uncertainties). The other sample (42a/01) apparently successfully dated by the $^{40}Ar/^{39}Ar$ technique at 359.9 ± 1.9 Ma has a K-Ar age of 352.8 ± 5.0 Ma, with a difference on the edge of statistical significance at the 1σ level.

The slight systematic discrepancy observed here between the two techniques (Table 2) could be related to the calibration of either the GL-O standard used for K-Ar or the FCs monitor used for $^{40}Ar/^{39}Ar$. Since GL-O is a glauconite, it cannot be directly compared to FCs by $^{40}Ar/^{39}Ar$ dating because of ^{39}Ar recoil that would result from neutron irradiation. However, dating of the MMhb-1 standard, for instance, provided K-Ar ages of 525 ± 2 Ma using the GL-O standard (Fiet et al., 2006), in agreement with the value of 523 ± 2 Ma obtained by $^{40}Ar/^{39}Ar$, when FCs at 28.02 Ma is used (Renne et al., 1998). Recent re-evaluations of the FCs age suggest slightly older ages of 28.201 ± 0.046 Ma (Kuiper et al., 2008) or 28.305 ± 0.046 Ma (Renne et al., 2010), which might superficially appear to further increase the difference between K-Ar and $^{40}Ar/^{39}Ar$ ages. However, the increase in the age of FCs is largely a consequence of inaccuracy of the electron capture decay constant of ^{40}K recommended by Steiger and Jäger (1977), which affects K-Ar ages as well (Renne et al., 2010). As can be seen in Table 2, recalculating both K-Ar and $^{40}Ar/^{39}Ar$ ages of this study for the calibration of Renne et al.

(2010) yields an offset similar to that obtained using Steiger and Jäger (1977).

An alternative explanation could be that these samples were affected by slight (1-2%) Ar loss, which could explain a discrepancy between K-Ar and $^{40}Ar/^{39}Ar$ plateau ages but not between K-Ar and $^{40}Ar/^{39}Ar$ integrated ages. However, over the whole Ar release interval (Fig. 3), the Ca/K spectra for all but one (30/01) of the $^{40}Ar/^{39}Ar$ experiments display a more or less marked undulating pattern, which parallels the age spectrum. This may suggest recoil redistribution of ^{37}Ar and ^{39}Ar. The initial increase in apparent age, correlated with increasing Ca/K, observed in all four $^{40}Ar/^{39}Ar$ experiments, suggests Ar loss from grain-marginal sites whose lower Ca/K is consistent with normal igneous compositional zoning. However, the integrated $^{40}Ar/^{39}Ar$ ages, which are systematically older than K-Ar ages for samples displaying undulatory Ca/K spectra suggest that recoil loss of ^{39}Ar has occurred, in addition to internal redistribution.

It is noteworthy that in the one sample (30/01) for which we have a K-Ar age older than the $^{40}Ar/^{39}Ar$ integrated age (although the two are indistinguishable within uncertainty), we also have a well-defined $^{40}Ar/^{39}Ar$ plateau age and a relatively flat Ca/K spectrum. Thus we can exclude ^{39}Ar bulk recoil loss from this sample, and the well-defined plateau in both age and Ca/K spectra indicates that there are no complications arising from internal recoil redistribution. The $^{40}Ar/^{39}Ar$ plateau age of 370.0 ± 0.7 Ma (conventional calibration) or 373.4 ± 0.7 Ma (recalculated per Renne et al., 2010; Table 2) appears to be the most reliable age obtained in this study. The plateau age for 42a/01, though meeting most criteria for a plateau, is viewed with caution due to the undulating Ca/K. This sample was analyzed in relatively few steps due to limited material, and the resulting imprecision of step ages may mask complexities in the age spectrum.

Because of the higher intrinsic ability of the $^{40}Ar/^{39}Ar$ technique to recognize Ar loss, and more generally to detect any disturbance of the K-Ar chronometer, we will concentrate on this approach in future efforts in dating the Viluy Traps. However, the Cassignol-Gillot K-Ar technique, which has been shown to give reliable ages for undisturbed basalt samples (e.g. Chenet et al., 2007; Coulié et al., 2003), can be useful in checking whether Ar recoil due to neutron irradiation performed for $^{40}Ar/^{39}Ar$ dating affects the results.

In conclusion, with this exceedingly limited collection, we have indications that Viluy magmatism could have been restricted around 370 Ma, which would indicate proximity (but certainly neither demonstrate nor preclude temporal coincidence) between this magmatism and the age of the Frasnian-Fammenian mass extinction now considered to be at ~376 ± 3 Ma and/or the end-Devonian at ~361 ± 3 Ma (Gradstein et al., 2005; Kaufmann, 2006). At present, the best fit obtained is between the former at 376 ± 3 Ma and our plateau age of 373.4 ± 0.7 Ma for 30/01 (calibrated per Renne et al., 2010; Table 2). Viluy magmatism and the LIP it emplaced could have occurred as a series of several main pulses, as now found in several flood basalt provinces (e.g. Courtillot et al., 1999), most clearly for the Deccan Traps (Chenet et al., 2007, 2009). Pirajno et al. (2009) quote the fact that "*Masaitis* (2007) discussed two pulses of magmatism: the *Appaya pulse* (*Givetian–Frasnian*) and the *Emyaksin pulse* (*Frasnian–Famennian*)". However, an earlier pulse at the Givetian-Frasnian boundary, i.e. ~384 ± 3 Ma is apparently too old to fit any of the ages we find. Nevertheless, a much larger collection of samples is necessary in order to determine the actual age and duration of magmatism and its relation to the extinctions. Dating these rocks would also provide guidelines in the identification of older (and in the discovery of new) magmatic sites of potential economical interest (e.g. diamond related). Particular care needs to be exercised in describing the exact locations, field relations and characteristics (tectonic, stratigraphic, volcanological, i.e. pipes, dykes, sills) of the samples. We are about to undertake such a larger study, based on the encouraging preliminary results reported here. In closing, we note that dating geological samples as a concerted effort by two independent laboratories working jointly on their interpretation is a rather rare yet very valuable and revealing exercise.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at doi:10.1016/j.epsl.2010.09.045.

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