

**Rialtas na hÉireann** Government of Ireland







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## **APPENDIX D – RADIOMETRIC DATING**

#### **INTRODUCTION**

To assist the evaluation of the age of selected igneous rock units, 10 new radiometric dating analyses were carried out within this project. The wells and igneous rocks selected for this new analysis were chosen from the full database listed in Table A4 (Chapter A) with a focus on cases where no previous dating had been carried out, or where spurious and inconsistent results had been returned from historical analysis. The choice of rock units for new analysis was selected following suggestions from the project Steering Committee. The wells and boreholes chosen for the radiometric dating analysis are shown in Figure A6 (Chapter A). Of those igneous rocks analysed, those that are considered to be extrusive have been named and described in the appropriate section of chapter D, Stratigraphic Intervals, of this Atlas. The named rock units that have been analysed radiometrically are; Púca Member (?Galley Formation), Druid Formation (Stronsay Group), Merrow Member (Cot Sandstone Formation), Selkie Member (?Dursey Formation), Sheerie Member (Bradán Formation) and the Péist Member (Gweedore Formation). Photographs of the core samples and thin sections are provided as illustrative figures supporting the descriptions of these rock units in **chapter D** of the Atlas.

While the initial project scope required that K-Ar dating method be used, during the project the requirement was changed such that all analyses were carried out using the Ar-Ar method on the advice of the project steering committee, which expressed the opinion that this method was likely to yield better results. The igneous samples originally chosen for radiometric dating were described, including thin section analysis by Dr Carl Stevenson (University of Birmingham, UK) who also made recommendations regarding the feasibility of obtaining reliable dating results (Stevenson, 2017). The radiometric dating analyses were carried out by ActLabs, Ontario, Canada. Following the receipt of results, in February 2018 (Actlabs 2018), considerable discussion of the results, and their implications, ensued between the project stratigraphers, PAD/PIP, Dr Stevenson (email to P. Copestake) and Professor J. Stephen Daly (University College Dublin) who was recommended by the project steering committee to provide further independent professional opinion. The results were discussed in a conference call (summarised by ISPSG Secretariat, 2018) following which Daly provided an updated assessment of the results (Daly, 2018). Following this internal discussion, the project stratigraphers interpreted the results and those radiometric dates that are considered to be reliable have been incorporated into the overall stratigraphic evaluations carried out for this project. Summary comments on these Merlin consortium interpretations are provided in Table A.D. 1 and Table A.D. 2, together with a summary of key data from Stevenson (2017), Actlabs (2018) and Daly (2018) and details of all samples analysed radiometrically. These tables also summarise all the key results of the radiometric dating, with core photographs and thin section micrographs. Key reports from this work are provided as digital addenda to this atlas (see below).

The focus was on obtaining samples from cores; however, in one well 26/30-1, samples were taken from cuttings in order to try and obtain an age for crystalline basement. In addition, the samples analysed from the Peist Member are from ditch cuttings.

### PETROGRAPHY & DESCRIPTION OF VOLCANIC SAMPLES ANALYSED FOR **RADIOMETRIC DATING**

Candidate samples were first described and studied in thin section by an independent geochemical expert Dr Carl Stevenson (Stevenson, 2017, which is included in the Digital Addenda of this Atlas) and an example of a thin section is shown in Figure A.D. 1. A range of igneos rock types were encountered in wells drilled in the Irish offshore basins. Interpreted origins vary from extrusive (lavas and tuffs) to intrusive (sills and or dykes). Recommendations were made for new radiometric dating along with suggestions for the types of minerals to be analysed and potential risks that could affect the accurate radiometric dating, such as degree of mineral alteration. Alteration of primary minerals (principally feldspar to clay) was recognised as a significant limitation for at least five of the ten samples. Despite the results of the feasibility study, the ISPSG members requested that Ar-Ar analysis be carried out on all ten samples.

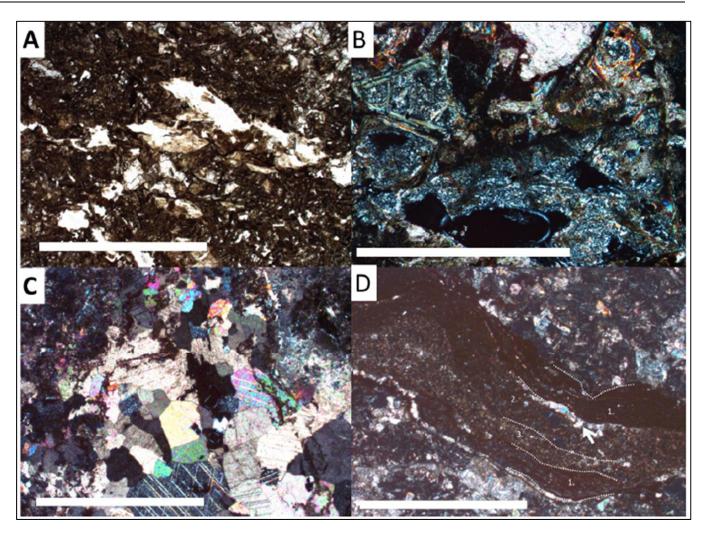


Figure A.D. 1 Example photomicrographs from sample 12/2-1z 3963.0m (Merrow Member) showing a range of altered textures and secondary mineralisation.

A) Plane polarised light, scale bar = 2 mm. Greenish brown sericitisation and chlorite dominating groundmass. B) Crossed polarised light, scale bar = 1 mm. Micro sphericular zeolite dominates view and mainly replaces primary igneous minerals. Calcite is present (top middle) and fibrous serpentinite veins remain where olivine has been replaced by zeolite and iddingsite. C) Crossed polarised light, scale bar = 2 mm. Sparry calcite replacing much of the rock. D) Plane polarised light, scale bar = 2 mm. Fine grained material in a vein is opaque and calcite. Multiple stages of vein growth (fine dotted lines and numbers) indicate multiple phases of hydrothermal fluid flow. White arrow highlights a fragment of calcite captured during early vein formation indicating early pervasive replacement alteration before veining, modified after Stevenson (2017).







#### **RADIOMETRIC DATING METHODOLOGY**

Ten samples were submitted to ActLabs, Ontario, Canada for Ar-Ar geochronology; four samples as mineral separates and the rest as whole rock samples.

The samples were pretreated for the Ar-Ar test; the  ${}^{40}$ Ar/ ${}^{39}$ Ar age was obtained by the incremental heating method using the Noblesse Noble Gas static mass spectrometer. The samples were irradiated in the nuclear reactor at McMaster University, along with the LP-6 flux monitors. Individual J-values for each sample were calculated by parabolic extrapolation of the measured flux gradient against irradiation height and typically give 0.2-0.3% uncertainties (1 $\sigma$ ). The Ar isotope composition was measured in a Noblesse Noble Gas static mass spectrometer (NU Instrument Ltd.). 1200°C blank of  ${}^{40}$ Ar did not exceed n\*10<sup>-11</sup> cc STP.

Where appropriate, Ar-Ar dating was undertaken on unaltered mineral separates (typically feldspars) to reduce the tendency for contamination in altered specimens and boost the concentration of key isotopes. Such an approach is best used when dealing with phenocrystic rocks with large feldspar crystals. It was therefore difficient in some cases to isolate ideal specimens, a problem most often encountered when dealing with lavas and rapidly cooled doleritic intrusions.

All ages were calculated using the corrected Steiger & Jäger (1977) decay constant of  $5.530 \pm 0.097 \times 10^{-10} \text{ l/yr} (2\sigma)$  as reported by Min *et al.* (2000). For all other constants used in the age calculations refer to Table 2 in Koppers *et al.* (2003).

*Note*: Plateaux were not observed in the majority of the samples. Also, isochrone could not be plotted. It could be attributed to the complex composition of the samples (see Ca/K ratio graphs) and to the likelihood of mineralogical alteration such as illitisation of feldspars". Probability Density Age Graphs were plotted for each sample. Using geological description, petrography study and probability density graphs the ages of main geological events can be concluded for this study.

#### **RADIOMETRIC DATING RESULTS**

The findings of the igneous rock analysis including petrography and radiometric dating are summarised in **Table A.D. 1** and **Table A.D. 2**. Additional details of the radiometric dating analyses are provided in the Actlabs (2018) end of job report which is included in the Digital Addenda of this Atlas.

#### **RADIOMETRIC DATING CONCLUSIONS, COMMENTS AND RECOMMENDATIONS**

Whilst other methods e.g. K-Ar and U-Pb were considered, Ar-Ar was considered to give the highest probability of yielding reliable radiometric ages given the limited number of samples and sample runs available. An acknowleded limitation of the study is therefore the inability to run repeat samples to ensure a reliable measure of accuracy in the outcomes.

Of the 10 samples analysed, several returned ages considered to be consistent with previous published analysis or inferred ages deduced from geological investigations and logical controls e.g. lava extruded onto a Paleocene sediment must be Paleocene or younger in age. The large numbers of reatively even peaks in the Probability Density plots for each sample suggest low confidence in the results. Ideally, such a plot should exhibit a single dominant peak corresponding to the correct age bracket. Multiple ages could represent distinct cooling and alteration events, however, there are several examples where peaks in the Actlabs plots do not correspond to logical or credible explanations. Third party expertise was sought to further establish the validity of the data and determine the relative credibility of any age assertions made by the consortium

The interpretation of the new data by independent geochemical expert, Professor J. Stephen Daly (Daly (2018); included in the Digital Addenda of this Atlas), indicates up to 7 of the 10 samples analysed are thought to have returned questionable or unsubstantiatable ages e.g. lava extruded onto a Paleocene sediment must be Paleocene or younger in age and cannot possess a crystallization age dating back to the Precambrian (unless crystals have been incorporated from an earlier cooling episode).

The results of new radiometric dating therefore demonstrate the difficulty in obtaining reliable dates for samples subjected to natural alteration. Whilst single samples have not, in some cases, yielded reliable ages, it is possible that more comprehensive follow-up studies involving multiple samples from the same rock could improve the reliability of the plateau ages and therefore improve the accuracy of the study. However, the generally highly altered nature of many of the samples will always



hamper the process of seeking reliable radiometric dates.

The final comments on the radiometric dating, included in **Table A.D. 1** and **Table A.D. 2** summarise the current Merlin consortium view of the age of the rock units that have been analysed. This takes into account all available stratigraphic data together with any inferences on stratigraphic position arising from the interpretation of seismic data near the samples in question. The latter approach has been quite revealing, particularly in the cases where the studied rocks lie beneath the Base Cretaceous seismic horizon as interpreted in this study.





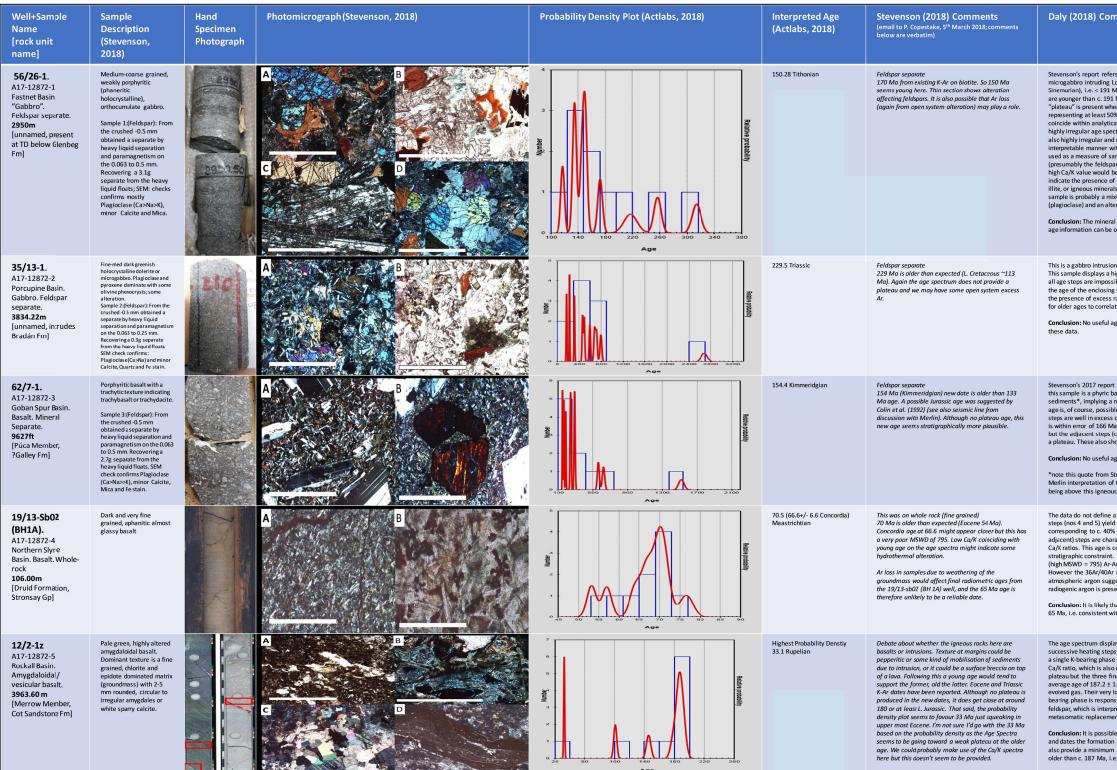


Table A.D. 1 Summary of radiometric Ar-Ar analyses carried out in this study, including sample descriptions and interpretations from this study, part 1.



mments	Conclusions (this study)
rs to this sample as an olivine Lower Jurassic sediments (Hettangian- Ma. Although the majority of age steps L Ma, no plateau age is defined (a en three consecutive age steps % of the 39Ar (39K) in the sample cla uncertainty). This sample dispays a ctrum. Moreover the Ca/K spectrum is d does not correlate in a readily with the ages. The Ca/K values can be ample purity. For plagioclase ar in this case is plagioclase), a uniform be expected. Lower Ca/K values could f a K-bearing alteration mineral such as lis such as biotite or homblende. This its ure of the intended target eration product e.g. illite. al separate is a mixture and no useful obtained from these data.	Dated by BP (Operator) and described by Caston et al. (1995) as Middle Jurassic. No reliable age information can be obtained from radjometric data. This also calls into question the Middle Jurassic dating of previous authors.
on cutting Lower Cretaceous sediments. highly irregular age spectrum in which sibly old, i.e. they significantly exceed g sediments, interpreted as indicating radiogenic argon. There is a tendency ate with the Ca/K ratio. age information can be obtained from	This is a gabbro intrusion cutting Lower Cretaceous (Late Albian Bradan Formation) and must be Early Cretaceous or younger in age. Considering this, it is concluded that the radiometric data are not reliable.
t (quoting Colin <i>et al.</i> , 1992) states that assalt overlain by Bathonian maximum age of c. 166 Ma. A younger Jei f the rock is intrusive. Most age of 166 Ma. One age step (169.9 ± 3.4) ta (i.e., the end of the Bathonian Stage, (c. 154 and 191. Ma) are far off defining now a significant variation in Ca/K ratio. age information can be obtained. Stevenson (2017) pre dated the final f the Base Cretaceous seismic horizon as rock in the 62/7-1 well.	The ActLabs Kimmeridgian age is consistent with the position of the rock (named as the new Picca Member herein), beneath the Base Cretaceous selimichorizon, which would suggest a Late Jurassic or oider age. The member is questionably allocated to the Galley Formation within the Hook Group, by correlation with the Fastnet Basin stratigraphy. The lava is certain by Hauterivian rocks (Gurnard Formation, Cromer Knoll Group) and is Immediately underlain by undated (but possible Late Jurassic) sediments. Various radiometric dates for the lava have been previously published by Esso (Colin et al., 1982; Bathonian), 130-137 Ma (Valanginian) (Tate & Dobson, 1988), 93.8 MG (Turonian) (BGS, 2009). The Actlabs age is the most plausible stratigraphically.
a plateau. However two successive d an average age of c. 65 Ma 6 of the gas evolved. These (and racterised by low but nearly constant consistent with the (admittedly limited) The data also define a very scattered Ar isodron age of 66.6 ± 6.6 Ma. r intercept value is similar to that of gesting that little if any excess sent. hat the age of this sample is close to c. tith a Palaeocene age	This sample is from the Druid Formation which is considered to be of Eocene age in this study. Constraining the age is difficult on stratigraphic grounds, as the lava is overlain by young Cenozoic section, typically Hebrides Margin Group but overlies Upper Cretaceous Chalk Group. Radiometric K/Ar dating carried out previously suggests early to middle Eocene ages; 40-43 Ma from the 13/13-sb02 (BH-1A) borehole (Dancer et al., 1999; Dancer et al., 2005).
ays a steady increase in age with ss. This is consistent with slow cooling of e and is supported by the near constant or ather low. The data do not define a nal steps, with a very define a weighted 1.9 Ma corresponding to c. 30% of the low Ca/K ratio suggests a single K- sible. This could correspond to K- orreted by Stevenson to be a ent of plagicolase. We that the c. 187 Ma age is meaningful n of metasomatic K-feldspar. This would a age for the basaltic host, it would be e., early Pliensbachian.	The member occurs within the Cot Sandstone Formation. Seismic interpretation places this formation below the Base Cretaceous Unconformity and above the Carboniferous. Lithologically, the Cot Sandstone is more similar to the Triassic Sherwood Sandstone Group than any other pre-Cretaceous ithorStratigraphic unit, hence the formation, and the Merrow Member, are tentatively placed within the Triassic in this study. Previously carried out radiometric K/Ar dating by Shell (2004) revealed an age of 250 Ma in the Early Triassic. This K/Ar age aligns with the overall stratigraphic favoured herein.





Well+Sample Name [rock unit name]	Sample Description (Stevenson, 2018)	Hand Specimen Photograph	Photomicrograph (Stevenson, 2018)	Probability Density Plot (Actlabs, 2018)	Interpreted Age (Actlabs, 2018)	Stevenson (2018) Comments (email to P. Copestake, 5 <sup>th</sup> March 2018;comments below are verbatim)	Daly (2018) Comm
<b>16/28-Sb01</b> A17-12872-6 Whole-rock. Basalt. <b>147.76 m</b> [Selkie Member, ?Dursey Fm]	Moderately veined with 1- 4 mm thick white (calcite or zeolite) veins. Veins are flanked by paler (alteration) rock. Away from veins but in patches are darker, fine grained but apparently holocrystalline, possibly primary basaltic rock. Sample 6(feldspar): From the crushed -0.5 mm obtained a separate by heavy liquid separation and paramagnetism on the 0.063 to 0.5 mm. Recovering a 0.04g separate from the heavy liquid floats. SEM check confirms mostly Calcite and low Mica+Plagioclase+Fe stain.	A A A A A A A A A A A A A A A A A A A	A B C C C C C C C C C C C C C C C C C C	Second states of the second st	Plateau 150.5 +/-1.2, Highest Probability Density 152.07 Concordia 149+/-10 Tithonian/Kimmeridgian	Whole rock Haughton et al. (2005) suggested a Cretaceous age for the lava. This is the only sample to praduce a plateau age of 150.5 M at + 1.12. Concordia age of 1494 + 10 less precise an MSWD (I'd go with plateau). Upper Jurassic may be stratigraphically jeasible depending on BCU pick from relevant seismic.	A plateau age of 150.5 ± 1.2 l steps and this is supported b Ma, which has a 36Ar/40Ar i atmospheric value. However variable. The first step is very that several K-bearing phases six steps used to calculate th within error. Steps 6 and 7 ar <b>Conclusion:</b> The relationship further evaluated, in particul- the sample was erupted into case and if the enclosing sed plateau age would have to be as it stands, the c. 150 Ma ag
<b>16/28-Sb01</b> A17-12872-7 Macdara Basin. Feldspar separate. Basalt. <b>148.25 m</b> [Selkie Member, ?Dursey Fm]	Basaltic, dark coloured breccia wth fine grained, holocrystalline aphantic matrix. Sample 74(Feldspar): From the crushed -0.5 mm obtained a separate by heavy liquid separate by heavy liquid separate mon the 0.063 to 0.5 mm. Sending a 0.1g separate from the heavy liquid floats. SEM: check confirms Plagioclase (Ca>Na,trace-K), lower Calcite and trace Fe stain	B IL /BS-SGL GSSGGGGS /LR-250		Petere property in the propert	Highest Probability Density 870.4 Pre-Cambrian	The unusually old age for 16/28-sb01 (148.25m) is puzzling. This was done on a mineral separate. The old date could be either the inclusion of some very old basement lithology as an inclusion in the sample (unlikely due to lack of a plateau and this is on a mineral separate), but is more likely due to excess Ar caused by hydrothermal alteration (pervasive alteration of plagioclase). No plateau but very old ages seem to coincide with low Ca/K ratio.	All age steps exceed 700Ma a canbe defined. The Ca/K rati lower than in the whole-rock is a plagicalcase separate, alth reported extensive alteration to incorporation of excess ra <b>Conclusion:</b> No useful age inf these data.
35/8-1 A17-12872-8 Porcupine Basin. Whole rock. Pyroclassic Tuff. 10668' [Sheerie Member, Bradán Fm]	Tuff comprising medium- fine grained ash, light grey, speckled, low density (rumaceous), quartz grains in feldspath c matrix, patchy alteration to clay.	all 1 are of	A B	Petro problem a region of the second	Highest Probability Density 40.38 Concordia 44+/-10 Bartonian	Tuff overlain by Albian-Aptian sediment so should be older than 113 Ma. New date suggests very young ca 40 Ma. Age spectra has no plateau but trying to get close around 40 (as per probability density plat). Isochron age with MSWD of 383 should be viewed with caution. Sample is altered and we co have a low Ca/K ratio coinciding with the most likely age steps on the age spectra so we could be dealing with a young hydrothermal event. Not quite as young though as the nearby dolerite (ca. 18 Ma).	Most of the age steps are clo canbe defined and the Ca/k Conclusion: No useful age in these data.
26/30-1 A17-12872-9 North Porcupine Basin. Whole rock. 5620' (Cuttings) [unnamed]	Washed cuttings (5610', 5620') include ca. 60% large clear mica flakes (some may be from drilling mud additive). Rock fragments 0.1-3 mm include Kfeldspar, quartz, biotite, ferromagnesian and plagioclase. Proportions seem to be consistent with a more alkali feldspar rich granitoid. Rock fragments at consistent with a monzogranite or possibly granodiorite	28/30-1 cuthes To restruction dating 5610' Matrix	No thin sections made.	Recovery of the property of th	Highest Probability Density 392.4 Mid Devonian	No plateau and age spectra a real mess. That said 392 Ma is consistent with basement.	It is not clear to me what wa defined and the Ca/K ratio is <b>Conclusion:</b> No useful age inf these data.
43/13-1 A17-12872-10 Porcupine Basin. Basalt. Whole rock. 2550-2560m cuttings [Péist Member, Gweedore Fm]	Very fine basalt (with some epidote), white specs (qu'te friable/crumbly – probably zeolite), bluish very fine-fine silt siltstone, red brown friable mudstone. Basalt and pale (bluish) silt predominate.	NA	No thin sections made.	And the second s	330.08 Mississippian (E.Carb)	Whole rock – basalt fragments (cuttings) Expected to be Early Paleocene* but coming out as 330 Ma (Missassippian). Reported to be heavily altered and no plateau so I would not read too much into this. * At the time the radiometric dating work was done, this basalt was thought to be most likely Early Paleocene in age. This age interpretation has since changed based on new seismic and biostratigraphic interpretation in this study.	The data define very variable extremely variable Ca/K ratio calculated from these data. <b>Conclusion:</b> No useful age inf these data.

 Table A.D. 2 Summary of radiometric Ar-Ar analyses carried out in this study, including sample descriptions and interpretations from this study, part 2.



nments       Conclusions (this study)         ± 12 Ma has been calculated from six for by the inserter construct with the were the CAY values are highly saves are responsible. In addition the tase wery different for the rest suggesting haves are responsible. In addition the tase processes the possibility that if no wets adjunct on the obtained from       Extrusive basaltic laws, Late Jurassic, Tithonian sile works and a set on the obtained from of law below Base Cretaceous seismic horizon.         OMa and are highly variable. No plateau age can be considered valid.       Extrusive basaltic laws, Late Jurassic, Tithonian sile works and one rest and point disage are to be sendement is of Cretaceous seismic horizon.         OMa and are highly variable. No plateau rest as major. This is unexpected if this ratio. negation during alteration, ge information can be obtained from       Extrusive basaltic laws, Late Jurassic, Tithonian sile Momber (Dursey from, supported by position of law below Base Cretaceous seismic horizon.         at was analysed but no plateau age Ca/cratio is highly variable.       The reck is tamed as the new Sheerie Momber filtered neith in his study, of law to Middle Ablain age, based on biotratignephy.         at was analysed but no plateau can be this is externely variable.       The sample represents granitic basement, of unknown age, but Which is likely to be are Calconing age hub the plateau can be this externely variable.         at was analysed but no plateau can be this is externely variable.       The sample represents granitic basement, of unknown age, but Which is likely to be are Calconing age to the which is likely to be are Calconing age to the which is lawly to are can be ablained from         risels age info		
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