Sampling and analytical techniques

Fieldwork and sampling were carried out during field campaigns in 2008 and 2009. Dabbahu is remote and difficult to access, so the emphasis was on identifying and sampling as many lava flows as possible. In total 95 samples were collected and analysed (fig. 2a, table 1). The fieldwork was supported by the Natural Environmental Research Council Airborne Research and Survey Facility (NERC ARSF) aerial photographs obtained using a Leica RCD105 39 megapixel digital camera as part of a LiDAR acquisition. These data were used to obtain information on inaccessible areas, together with digital elevation model analysis in ArcGIS®. Whole rock major elements were analysed by X-ray fluorescence (XRF) using a PANalytical Axios-Advanced XRF spectrometer at Leicester University for 93 samples (table 2). In addition to the 10 standards analysed as part of each run, a total of nine additional ‘blind’ analyses were made of known standards (JR1, JR2, JG2 and BHV02) to ensure consistency and quality of data. Analyses of major elements were performed using fused glass discs made from ground rock powders mixed with 80:20 Li metaborate: Li tetraborate. Loss on ignition was performed in two stages at 750 °C and 950 °C in order to minimise alkali loss from these peralkaline rocks.

Analysed rocks included lava flows, obsidians and pumices. As the pumices are mostly aphyric, or contain rare, very small crystals, microanalysis was concentrated on crystal-bearing lava flow samples. Thin sections of 68 samples were carbon coated for initial analysis using a Hitachi S-3500N scanning electron microscope (SEM) at Bristol University. Major elements (SiO₂, TiO₂, Al₂O₃, FeO, MnO, MgO, CaO, Na₂O, K₂O) of identified individual minerals were analysed by electron microprobe using a CAMECA SX-100 instrument at the University of Bristol. Cr₂O₃, NiO, ZnO were also analysed in the mafic minerals, with SO₂, P₂O₅ and Cl in a subset of feldspars. Calibration was carried out on a range of silicate minerals, glasses and oxides. A 15kV accelerating voltage, 4 nA beam
current and defocused 10 µm beam was used for matrix glasses and feldspars, with Na
analysed first to reduce the effects of alkali migration (Humphreys et al., 2006). For minerals
other than feldspars, a 20 kV accelerating voltage, 10 nA beam current and focussed 1 µm
beam was used. Count times varied from 10 to 60s per element, and data were corrected
using the ‘PAP’ correction procedure (Pouchou and Pichoir, 1984).

68 samples were manually point-counted to ascertain the volume % of phenocrysts. The aim
was to count for 1500 points per sample, although some variation was due to the nature of the
thin sections. As a rough estimate this gives a reliability of ±2.5% (2σ) using the chart of Van
der Plas and Tobi (1965). Due to the scarcity of phenocrysts, it is likely there is significant
error in some samples. Similar limits are found using the 95% 2-sided confidence level of
Howarth (1998: average upper and lower limits <1.5%).

Samples for ⁴⁰Ar-³⁹Ar dating were collected during the field campaign of 2008. Samples were
reviewed for suitability to application of the ⁴⁰Ar/³⁹Ar dating technique based on minimum
alteration, vesicularity and amounts of interstitial glass (<5%). Many Dabbahu samples had
to be rejected as unsuitable for this technique due to a high % of glass content, or fine
groundmass material. From those which were suitable, 9 samples were chosen to give as wide
a compositional and spatial spread across Dabbahu as possible (locations of dated samples
are shown in fig. 2a). Three of the samples are basaltic trachyandesites , two are peralkaline
comenditic rhyolites and four are pantelleritic rhyolites. Groundmass separates were used in
the basaltic trachyandesite samples, and anorthoclase phenocryst separates were prepared for
the comendites and pantellerites. Obsidian samples were chosen according to the size (ideally
>5 mm), and freshness of the anorthoclase phenocrysts. Phenocrysts which were inclusion-
fee were chosen preferentially.

To minimise contamination great care was taken with regards to the cleanliness of all
equipment and work surfaces throughout the preparation process at the University of Bristol
and USGS Menlo Park. Samples were manually crushed to fine gravel using a hammer and steel plate. The chips were milled using a Retsch planetary ball mill and sieved using a stack of sieves to ensure the target size fraction was retrieved. The target fraction was then repeatedly cleaned ultrasonically (water, solvent and finally de-ionised water) which commonly improves radiogenic yield and minimises argon recoil by removing glass and any clay fraction which trap atmospheric argon, and cryptocrystalline groundmass. The samples were then dried at ~70 °C and re-sieved to remove any remaining fines released by the ultrasonic processing. Fine feldspar can cause problems during the irradiation as reactor-induced $^{39}$Ar moves ~0.1 µm (Villa, 1997), and if it is liberated from its crystal or moves to a more retentive phase, the radiogenic $^{40}$Ar is no longer correlated with the $^{39}$Ar. A magnetic separator was used to concentrate the 50-90% most magnetic portion. Finally around 150-200 mg was prepared for irradiation by hand-picking contaminants out of the split. The analysis was carried out using the method of Calvert and Lanphere (2006) at the facilities of USGS.

SUPPLEMENTARY FIGURES
Figure 1. A) Sampling localities. White circles indicate locations of samples at Dabbahu, black circles indicate samples dated by $^{39}\text{Ar}^{39}/^{39}\text{Ar}$. Some of the local place-names are also indicated. Hillshade image based on SPOT DEM courtesy of Sophie Hautot. B) Panchromatic SPOT image showing key textures such as the distinctive obsidian flows on the northern flanks of Dabbahu, and the prominent metaluminous rhyolite flow to the SW.
Figure 2. Backscatter electron images of representative textures found in the Dabbahu Rocks. A) sample 44, basalt, Fe-rich rimmed olivines in fine grained groundmass (cores Fo83-76, rims Fo80-57), B) sample 28, trachybasalt, ribbon-rimmed feldspar (core An10Or35, rim An13Or25), with plagioclase at left, C) samples 25 and 46, basaltic trachyandesites, skeletal olivine (Fo47, left) and magnetite (right), D) sample 048, trachyte, cristobalite crystal, E) sample 106, trachyandesite, sponge textured cpx, F) sample 071, comendite, euhedral zircon, G) sample 023, pantellerite, amphibole xenocryst – this is the only amphibole crystal found in the Dabbahu samples, H) sample 055, pantellerite, aenigmatite phenocryst with alkali feldspar and cpx.
Figure 3. Images of zircon located in comendite (sample 071). A) and B) backscatter images showing clusters of zircon in thin section. C) to H) cathodoluminescence images of zircons showing distinct cores surrounded by variable-width rims.
Figure 4. Ar-Ar age spectrum of dated Dabbahu samples