1 A detrital record of the Nile River and its catchment.

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# **Abstract**

This research uses analyses from Nile catchment rivers, wadis, dunes and bedrocks to constrain the geological history of NE Africa and document influences on the composition of sediment reaching the Nile delta. Our data show evolution of the North African crust, highlighting phases in the development of the Arabian-Nubian Shield and amalgamation of Gondwana in Neoproterozoic times. The Saharan Metacraton and Congo Craton in Uganda have a common history of crustal growth, with new crust formation at 3.0-3.5 Ga, and crustal melting at c.2.7 Ga. The Hammamat Formation of the Arabian-Nubian Shield is locally-derived and has a maximum depositional age of 635 Ma. By contrast, Phanerozoic sedimentary rocks are derived from more distant sources. The fine-grained (mud) bulk signature of the modern Nile is dominated by input from the Ethiopian Highlands, transported by the Blue Nile and Atbara rivers. Detrital zircons in the Nile trunk are predominantly derived from Phanerozoic cover rocks. Most detritus from the upstream White Nile is trapped in the Sudd marshes and contributes little to the Nile trunk. Therefore, the White Nile downstream is dominated by locally-derived Phanerozoic cover. The White Nile proximal to the Gezira Fan is influenced by the fan's Blue Nile signature.

Modern river sediments can be used to efficiently sample large areas of upstream crust in order to help understand a region's geological history (e.g. lizuka et al., 2013). In this study, we use a range of provenance techniques, including U-Pb and Hf isotope analysis of zircon from river sands, and Sr-Nd-Hf tracer isotope analysis of river mud samples, in order to characterise the catchment of the modern Nile River. Our aims are to gain a broad overview of the geological evolution of NE Africa, and to constrain the influences on sediment composition to the Nile River and ultimately the Nile delta cone, an important depocentre for hydrocarbon reservoirs.

We report data for sand and mud samples from the Nile trunk, and its tributaries the Blue Nile, White Nile and Atbara, together with samples from dry wadis and aeolian dunes from the Red Sea Hills and Western Desert which represent possible sources of detritus to the river (Fig. 1). We have also studied sedimentary rocks from the Red Sea Hills and Western Desert that have draped NE Africa since the latest Precambrian. These rocks, and the modern wadi sands and aeolian dune sands which partly overlie them, which we have also studied, represent an important source of detritus to the river. We present zircon U-Pb and Hf-isotope data for gneissic basement within the Saharan Metacraton, which is poorly documented. These analyses help to characterise the nature of the Archaean crust of North Africa.

The Nile River is the longest river in the world, extending for more than 6,800 km, and draining an area of ~3.3 million km². The present-day Nile is made up of three main tributaries, the Blue Nile, White Nile and Atbara (Fig. 1). The mean annual water discharge during times of peak flow is dominated by the Blue Nile (68%), followed by the White Nile (10%) and Atbara (22%) (Williams and Adamson, 1982). Sediment supplied to the Nile trunk in Egypt is dominated by contributions from the Blue Nile (50-61%) and Atbara (30-42%) (Padoan et al., 2011). The vast majority of the White Nile sediment load is trapped in extensive swamps in South Sudan (the Sudd marshes), thus accounting for <3% of the total Nile trunk sediment budget (Garzanti et al., 2015).

The White Nile drains Archaean – Proterozoic rocks of the Congo craton, and extends through Precambrian rocks of the Saharan Metacraton (Abdelsalam et al., 2002). In its terminal tract, the White Nile has an extremely low gradient due to its positioning along the floor of an ancient lake that occupied its valley as long ago as 400 ka before present (Williams et al., 2003). The present flow regime of the White Nile was established in the late Pleistocene, at c.15 ka, when northwards flow was reinitiated due to intensification of the summer monsoon and resulting overflow of Lakes Albert and Victoria (Talbot et al., 2000; Williams et al., 2003).

The Blue Nile and Atbara, together with its tributary the Tekeze are sourced in the Ethiopian Highlands, where they drain Cenozoic flood basalts (e.g. Pik et al., 1998), Neoproterozoic Arabian-Nubian Shield (Arabian-Nubian Shield) basement rocks (e.g. Evuk et al., 2014; Johnson, 2014) and Phanerozoic cover sequences (e.g. Gani et al., 2009). Uplift of the Ethiopian Highlands in the Oligocene may have led to the initiation of flow in the Blue Nile. However, there is no consensus in published literature regarding the timing of initiation of such flow, as summarised below.

Proponents of Oligocene initiation of Nile River sediment supply from the Ethiopian plateau have used knick-point facies to infer a 3-phase incision in the Ethiopian Highlands at 29-10 Ma, 10-6 Ma and 6 Ma, which instigated erosion in the Tekeze, Atbara and Blue Nile catchments (Gani et al., 2007). Thermochronological studies using apatite and titanite He ages suggest that the elevated plateau physiography, which controls most of the present-day Nile hydrology, has existed since the Oligocene, ca.29 Ma (Pik et al., 2003). Authors favouring a Nile drainage originating in the Late Miocene cite sparse remotely sensed radar data to argue for a south-draining 'Qena System' dominating the Nile valley until the Messinian Salinity Crisis (Issawi and McCauley, 1992). Sediment volume calculations of the delta cone have been used to infer that a connection with the Ethiopian Highlands did not occur until the Pleistocene (Macgregor, 2012; Palacios, 2013).

The Red Sea Hills, to the east of the present day Nile trunk, consists of Arabian-Nubian Shield basement and Phanerozoic sedimentary rocks which are comprised of Palaeozoic and Mesozoic

clastic rocks and Eocene carbonates, partially overlain by modern wadi sands. Although not drained by the Nile at present, the Western Desert, to the west of the present day Nile trunk, is thought by Issawi and McCauley (1992) to have once contributed detritus to the main Nile trunk, and aeolian contribution occurs today (Garzanti et al., 2015). The Western Desert comprises reworked pre-Neoproterozoic crust of the Saharan Metacraton (Abdelsalam et al., 2002), and overlying Phanerozoic sedimentary successions. Modern day wadi sediment and aeolian dunes currently cover a large proportion of both these regions.

# **Geology of the Nile catchment**

The geology of east Africa has largely been shaped by the events of the Pan-African Orogeny when east and west Gondwana collided to form 'Greater Gondwana' at the end of the Neoproterozoic. The Pan-African orogeny in NE Africa involved the collision of Archaean cratons such as the Congo craton and the Saharan Metacraton with the Arabian-Nubian Shield, a terrane comprising Neoproterozoic, juvenile, oceanic island arcs. Phanerozoic cover sedimentary rocks blanket much of NE Africa, reflecting erosion from this mountain belt and subsequent Phanerozoic tectonics. The most prominent Phanerozoic tectonic events in the region involve rifting associated with opening of Neotethys, and later inversion associated with Africa-Mediterranean convergence. Uplift of the Ethiopian Highlands and surrounding area, and the eruption of continental flood basalts is thought to have had a strong influence on the hydrology of this area in the Cenozoic.

## Paleoproterozoic and Archean cratons

The cratons of Central Africa are formed of various blocks of Archaean and Palaeoproterozoic crust, flanked or truncated by Palaeoproterozoic to Mesoproterozoic orogenic belts. Together, these form the Congo Craton, defined as the "amalgamated central African landmass at the time of Gondwana assembly" (De Waele et al., 2008). The Congo Craton (Fig. 1) forms part of the West-African-Central-Belt which formed between 2.0 and 3.0 Ga ago and comprises orthogneiss, metasediments and granitoids of the Tanzania Craton, Gabon-Kamerun

Shield, Bomu-Kibalian Shield, Kasai Shield, and Angolan Shield (Cahen et al., 1984; De Waele et al., 2008; Goodwin, 1996; Tchameni et al., 2000; Walraven and Rumvegeri, 1993).

The term Saharan Metacraton refers to an area of pre-Neoproterozoic continental crust which has, in part, been highly remobilised during the Pan-African orogeny (Abdelsalam et al., 2002). It extends from the Arabian-Nubian Shield in the east to the Tuareg Shield in the west and the Congo Craton in the south. More than 50% of the Saharan Metacraton is overlain by Phanerozoic sedimentary rocks and desert sands (Abdelsalam et al., 2011). The poor exposure of the region means that the Saharan Metacraton, and its relationship to adjacent blocks, is poorly understood. The southern boundary is not well defined, but is taken to be marked by the Oubangides orogenic belt which separates it from the Congo Craton (Abdelsalam et al., 2002). Little modern geochronology has been carried out on rocks of the Saharan Metacraton. Legacy Rb-Sr and U-Pb data quoted by Abdelsalam et al. (2002) indicate a range of late Archaean and Palaeoproterozoic protolith ages, with significant cratonic reworking and addition of new crust in the Neoproterozoic. Bea et al. (2011) report SHRIMP U-Pb zircon ages as old as 3.22 ± 0.04 Ga for gneisses in the Uweinat and Gebel Kamil regions of the Western Desert in southernmost Egypt.

## The Arabian-Nubian Shield

The Arabian-Nubian Shield is a collage of Neoproterozoic (c. 870-670 Ma) continental margin and juvenile island arcs, cut by voluminous granitoid intrusions as young as latest Neoproterozoic, and overlain by younger sedimentary and volcanic basins (Johnson and Woldehaimanot, 2003; Kusky and Matsah, 2003; Stern, 1994; Stern and Hedge, 1985). Only minor outcrops of pre-Neoproterozoic crust are found. Formation of the Arabian-Nubian Shield began with the initiation of subduction and development of magmatic arcs at 870 Ma. Terrane amalgamation associated with closure of the Mozambique Ocean and amalgamation of East and West Gondwana took place between c. 780 Ma and c. 600 Ma (Johnson and Woldehaimanot, 2003). To the west of the Red Sea, the oldest arc terranes (> 800 Ma) of the Arabian Nubian shield occur in the south, in Ethiopia, Eritrea and Sudan

(Johnson and Woldehaimanot, 2003). Ophiolitic rocks of the Eastern Desert of Egypt range in age from 810 to 720 Ma (Ali et al., 2010), and are overlain by younger volcanic and sedimentary sequences (e.g. Breitkreuz et al. (2010).

Ediacaran alluvial sedimentary rocks of the Hammamat Formation overlie the Arabian-Nubian Shield basement in the Eastern Desert of Egypt. The Hammamat Formation clastic rocks comprise terrestrial conglomerates, sandstones and mudstones in the Red Sea Hills area, and have proposed correlatives overlying Midyan Terrane in northwest Saudi Arabia (Bezenjani et al., 2014). Previous workers, using a variety of approaches (e.g. maximum depositional age determined from detrital minerals, dating of cross-cutting igneous units), have proposed ages ranging from 630 Ma to 583 Ma for these rocks in Egypt (Bezenjani et al., 2014; Wilde and Youssef, 2002; Willis et al., 1988). The Hammamat Formation rocks' depositional environments, and their relationship to the Pan African Orogeny is of some debate (e.g. Abdeen and Greiling, 2005; Bezenjani et al., 2014; Eliwa et al., 2010; Johnson et al., 2011; Ries et al., 1983; Wilde and Youssef, 2002); the sediments are arguably (as per references above and refs therein) considered to be pre-collisional, syn-collisional or post-collisional, and either locally sourced and deposited in isolated basins, or of distant provenance deposited from a major fluvial system of continental proportions.

#### The Pan-African Orogeny

During the Pan-African Orogeny, the final closure of the Mozambique Ocean led to amalgamation of the Saharan Metacraton and Congo Craton with the Arabian-Nubian Shield and the formation of the 'Trans-Gondwanan Supermountains' in the region that now forms NE Africa (Meinhold et al., 2013; Squire et al., 2006). To the south, it has been proposed (Abdelsalam et al., 2002) that collision between the Saharan Metacraton and Congo Craton resulted in the formation of the Oubanguides orogenic belt or Central African Fold Belt.

## **Phanerozoic Sedimentary Rocks**

Erosion of the Trans-Gondwanan mountain belt and subsequent recycling of the eroded detritus during later inversion tectonics associated with various plate movements, resulted in the deposition of a thick cover of fluvial and marine sediments overlying the amalgamated terrains, from the Cambrian onwards. Sedimentation continued until Cenozoic times, influenced by periods of tectonism. The most important of these tectonic events is the Mesozoic opening of Neotethys and later the South Atlantic which resulted in the development of rift basins in the region, with Late Cretaceous-Early Eocene inversion of such basins due to the convergence of the Mediterranean with the African margin (Bosworth et al., 2008; Guiraud et al., 2005; Klitzsch and Squyres, 1990).

Previous studies of the cover sedimentary rocks in regions adjacent to the Nile catchment (i.e. Libya - (Altumi et al. (2013); Meinhold et al. (2011)), Jordan and Israel (Kolodner et al. (2006); Morag et al., 2011b) have put forward contrasting Phanerozoic palaeodrainage models for these regions during deposition of the cover. The textural and mineralogical maturity of the bulk of the cover, and the provenance of the sediments, has been ascribed to varying degrees of recycling, long distance transport, and/or intense chemical weathering. Particularly debated is the origin of the c.1000 Ma zircon population for which an obvious significant basement source in the Saharan Metacraton or Arabian-Nubian Shield has not been recognised (Kolodner et al., 2006; Meinhold et al., 2011).

# Cenozoic Uplift and Flood Basalts

Uplift of the Red Sea Hills associated with opening of the Red Sea rift, and uplift in Ethiopia associated with eruption of voluminous continental flood basalts, have had a major influence on Nile drainage.

Many studies have attempted to ascertain the timing of Red Sea Hills uplift, with authors suggesting that uplift of rift shoulders began around 24 Ma (Bosworth et al., 2005), 25-30 Ma (Ghebread, 1998), <29 Ma (Kenea, 2001) and 34 Ma (Omar and Steckler, 1995).

The continental flood basalts that dominate much of the Ethiopian Highlands are associated with East African rift-related magmatism and continental break-up (Buck, 2006; Ebinger, 2005). Prerift basaltic and silicic magmatism initiated around 31 Ma (Baker et al., 1996; Rochette et al., 1998; Ukstins et al., 2002). Magmatic upwelling resulted in the uplift, extensional stresses, and faulting within what is now the Ethiopian Highlands.

# The provenance signal of modern Nile River sediments

The provenance of sediments in the Nile River has previously been studied using heavy mineral distributions and petrography, Sr and Nd trace isotope studies, and U-Pb analysis of detrital zircons.

Sr and Nd ratios have proved useful when recording major changes in provenance along the course of the River Nile (Garzanti et al., 2013; Padoan et al., 2011). High  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios and low  $\epsilon_{Nd}$  values in White Nile muds from Archaean cratonic sources, contrast with lower  $^{87}\text{Sr}/^{86}\text{Sr}$  and higher  $\epsilon_{Nd}$  in the Sobat River, which receives most of its sediment load from the crystalline basement and Cenozoic volcanic rocks of the Ethiopian Highlands. Isotope signatures of the Blue Nile and Atbara rivers are dominated by Ethiopian volcanic detritus and show low  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios and higher  $\epsilon_{Nd}$  values.

Heavy mineral analysis and petrography have also been used to characterise the signature of the Nile catchment area (Garzanti et al., 2006). The Blue Nile, Atbara and Tekeze rivers drain predominantly flood basalts and rhyolitic ignimbrites in their upper courses until they reach the Neoproterozoic amphibolite-facies basement of the Arabian-Nubian Shield, consisting of granitoid gneisses, staurolite-bearing schists, and marbles (Tadesse et al., 2003), and Phanerozoic sedimentary rocks found in the pre-rift sedimentary succession of the Blue Nile Canyon. Despite both rivers draining similar lithologies in the Ethiopian Highlands, the Atbara contains significantly less quartz than the Blue Nile.

The Victoria Nile downstream of its Lake Victoria output carries feldspatho-quartzose sand, with feldspars derived from granitoid rocks exposed locally (Garzanti et al., 2006). Downstream of the Lake Albert outlet, Albert Nile sand is quartzose with few feldspars and poor heavy-mineral suites with epidote, hornblende, kyanite, rutile and zircon. Sediment composition changes across South Sudan, and White Nile sand of all grain sizes downstream of the Sudd marshes chiefly consist of monocrystalline quartz, becoming slightly enriched in plagioclase, volcanic lithics and clinopyroxene when downstream of the Gezira Fan. This fan deposit is ultimately derived from the Ethiopian Flood Basalts via a Pleistocene overspill from the Blue Nile (Garzanti et al., 2006).

The Red Sea Hills lie adjacent to the Nile River through much of its course through Sudan and Egypt. However, Red Sea Hills-derived sediment has not been considered as an end-member in modelling of the Sr and Nd isotope composition of sediments in the Nile delta cone (Krom et al., 2002; Revel et al., 2014). Although construction of the Aswan High Dam has clearly affected the trunk Nile sediment flux downstream, comparison of petrographic data from samples collected prior to and subsequent to construction of the dam indicate that sediment composition is minimally affected (Garzanti et al., 2015).

lizuka et al., (2013) present coupled U-Pb, Lu-Hf and O-isotope data for detrital zircons from a sample of modern Nile sand taken 'Near Cairo City'. They identified age peaks at 1.1-0.9, 0.85-0.7 and 0.7-0.55 Ga, with minor groups at c. 2.6 and 2.0 Ga. They noted that grains with ages between 0.85 and 0.7 Ga had positive  $\epsilon_{Hf}$  values, and were likely to have been derived from juvenile crust in the Arabian-Nubian Shield. Zircons with ages of 1.1-0.9 and 0.7-0.55 Ga, coinciding with times of supercontinent assembly, showed wide variations in O-isotope composition and  $\epsilon_{Hf}$ .

Be'eri-Shlevin et al. (2014) discuss U-Pb and Hf-isotope data for detrital zircons in Quaternary to Recent Israeli coastal sands, which are believed to have been derived by a combination of longshore drift and aeolian transport from the Nile delta. They conclude that the ubiquitous presence of 0.56-0.75 Ga detrital zircons with negative  $\epsilon_{\rm Hf}$  implies that the Arabian-

Nubian shield is not the main source of Nile sands. Rather, they believe that multiple recycling through Phanerozoic sedimentary rocks that blanketed North Africa explains the age and hafnium isotope composition of the detrital zircon populations, as well as the quartz budget of the system.

# **Analytical Methods**

Samples of medium sand and mud were taken from modern rivers, sedimentary bedrock and modern wadis and dunes. Sample locations are shown in Figure 1 and documented in Supplementary Item 1. A sample of gneissic basement was also collected from the Saharan Metacraton.

Detailed analytical methods for techniques used in this study are given in Supplementary Item 2.

Zircon and rutile grains were separated using standard methods, then hand-picked and mounted in epoxy disks and polished to reveal their interiors. All zircon grains were imaged using cathodoluminescence prior to analysis. U-Pb analyses for both zircon and rutile were carried out at the NERC Isotope Geosciences Laboratory (NIGL), using a single collector Nu-Attom mass spectrometer with one of three New Wave laser systems, typically using a 35µm laser spot. Hafnium isotope composition of zircons was measured at NIGL using a Thermo-Electron Neptune Plus mass spectrometer, coupled to a New Wave 193UC or 193FX Excimer laser. A 50µm spot was used, targeting previously dated zircon domains.

Plagioclase and white mica were separated from the light fraction remaining after zircon and rutile separation. <sup>40</sup>Ar/<sup>39</sup>Ar analyses were carried out at SUERC, East Kilbride, using a GVi instruments ARGUS 5-collector mass spectrometer using a variable sensitivity Faraday collector array in static collection (non-peak hopping) mode (Mark et al., 2009; Sparks et al., 2008).

Mud samples for Sr, Nd and Hf analysis were leached in 10% acetic acid to remove carbonate before spiking with <sup>149</sup>Sm-<sup>150</sup>Nd, <sup>176</sup>Lu-<sup>180</sup>Hf, <sup>87</sup>Rb and <sup>84</sup>Sr isotope tracers. Standard

dissolution methods and ion-exchange chromatography were used to separate elements of interest. Sr and Nd isotope compositions were measured at NIGL on a Thermo-Electron Triton mass spectrometer using dynamic multi-collection. Hf isotope composition was analysed in static mode on a Thermo-Electron Neptune mass spectrometer coupled to a Cetac Aridus II desolvating nebuliser.

XRF major and trace element analysis was carried out at the Open University in Milton Keynes using standard methods. Pressed powder pellets were used for trace element measurements, and fused discs for major elements.

Petrographic analysis and heavy mineral analysis of sands and muds were carried out at the Universita di Milano-Bicocca using methods modified from Garzanti et al. (2006) and Garzanti and Ando (2007). Assemblages were described in terms of transparent Heavy Mineral Concentrations (tHMC), Zircon-Tourmaline-Rutile Index (ZTR), Mineral Maturity Index (MMI) and Hornblende Colour Index (HCI).

# **Petrography and Heavy Mineral Analysis**

The analyses presented below are intended to provide data for regions which are not adequately covered by published datasets, summarised in Garzanti et al. (2015). Data are provided in Supplementary Items 3 and 4.

#### Red Sea Hills, Egypt

Modern wadi sands (RSH03A and RSH05A) in northern Egypt are sub-litharenites (Fig. 2), with carbonate clasts and minor chert and shale grains, and a poor to moderately poor tHMC of  $1.2 \pm 1.0$ . Epidote-amphibole-clinopyroxene transparent-heavy-mineral assemblages include minor garnet, zircon, rutile, staurolite and tourmaline (ZTR  $10 \pm 5$ ). Phanerozoic sandstone samples (RSH08A and RSH09A) from the Red Sea Hills are quartz arenites with an extremely high ZTR of 98-99 and tHMC between 12 and 20. The modern wadi sands contain slightly more (4-5%) feldspar than the bedrock

samples. This difference in mineralogical maturity between bedrock and wadi samples may reflect sampling bias as the modern wadi sands sample a larger area. By contrast, a sample from the Hammamat Formation (RSH14A) was found to be a sheared lithic-arkose volcaniclastic arenite dominated by epidote, with ZTR = 0 and tHMC = 73.

#### Western Desert, Egypt

Aeolian dune sands (WD03C, WD19C and WD20C) from the Egyptian desert west of the Nile are quartzose, with few feldspars (K-feldspar > plagioclase) and sedimentary rock fragments (limestone and subordinately siltstone/shale and chert). Very poor to moderately poor (tHMC of 0.8  $\pm$  0.4) transparent-heavy-mineral assemblages include epidote, zircon, amphibole, rutile, tourmaline, clinopyroxene, staurolite, garnet, kyanite and titanite (HCI = 11  $\pm$  1, MMI = 50, ZTR = 34  $\pm$  13).

#### Nile trunk, Sudan

Downstream of the confluence of the Blue Nile, White Nile and Atbara, (SD04A and SD05A), sediments are quartz-dominated with plagioclase feldspar more abundant than K-feldspar, and some volcanic detritus present. Both samples were found to be rich in transparent-heavy-mineral-assemblages (tHMC of 16.8 and 10.1) including clinopyroxene, epidote and hornblende (HCl = 6-7). The ZTR for both Nile trunk samples was very low (0-1).

# Major and trace element chemistry

Trace element results are presented in Figure 3, normalized to the trace element concentrations of Post Archean Australian Shale (PAAS), compiled by McLennan and Taylor (1985).

V, Cr, Co, Ni and Cu concentrations enable us to determine the relative amount and location of mafic material within the Nile catchment area. The full dataset is provided in Supplementary Item 5.

Values of V, Cr, Co, Ni and Cu are considerably higher in the Blue Nile and Atbara rivers, compared to the White Nile, reflecting the significant proportion of continental flood basalts in the Blue Nile and Atbara catchments. Similarity between the Blue Nile / Atbara values and those of the

Nile trunk in Sudan attest to the dominance of the Blue Nile / Atbara contribution compared to the White Nile to the trunk river downstream.

Western Desert values are similar to the White Nile values, reflecting their location in proximity to cratonic rocks. Red Sea Hills values show a greater mafic influence due to their closer proximity to the Arabian-Nubian Shield. Of note is the difference between Red Sea Hills wadi muds collected from locations overlying Red Sea Hills Phanerozoic bedrock, and Red Sea Hills wadi muds from locations directly overlying Arabian-Nubian Shield basement (samples RSH14B and RSH15B). The Ni spike in the latter most probably reflects significant derivation from the underlying ophiolitic ultramafic rocks of the Arabian-Nubian Shield.

# Zircon U-Pb geochronology and Hf isotope signatures

Zircon U-Pb and hafnium isotope data are shown in Figures 4 to 8. The locations of all analysed samples are shown in Figure 1. Data are documented in Supplementary Items 6-9.

#### **Cratonic sources**

## Western Desert Archaean Gneiss

The oldest bedrock sample analysed (WD16A) is a gneiss, mapped as Archaean crust of the Saharan Metacraton (Daumain et al., 1958), collected east of Uweinat in southernmost Egypt. Zircon crystals have oscillatory-zoned cores under cathodoluminescence (CL), with homogeneous CL-dark, possibly metamorphic overgrowths (Fig. 4 inset). Cores are commonly broken, with abrupt terminations, suggesting that the sample may have had a sedimentary protolith that has undergone high-grade metamorphism.

Fifteen U-Pb analyses of zircon cores have <sup>207</sup>Pb/<sup>206</sup>Pb ages ranging from 2924 to 3235 Ma, with a single analysis giving a statistically younger age of 2794 Ma. It is not possible to identify statistically valid populations within the zircon core dataset due to the small number of cores and rims found in the samples. This may reflect a detrital source for the zircons. The zircon cores have

an average  $\epsilon_{Hf}$  of -2.3, and a weighted average depleted mantle model age (TDM $_{Hf}$ ) of 3479 ± 47 Ma (MSWD = 4.1, n= 15/16).

Five analyses of zircon rims form a cluster with a weighted average  $^{207}$ Pb/ $^{206}$ Pb age of 2701  $\pm$  32 Ma. Three further rim analyses yield younger  $^{207}$ Pb/ $^{206}$ Pb ages, between 2524 and 2587 Ma. The zircon rims yield an average  $\epsilon_{Hf}$  of -10.6 and a weighted average TDM $_{Hf}$  of 3467  $\pm$  45 Ma (MSWD = 2.2, n=8), within error of the age derived from zircon cores, indicating that new zircon growth took place in a closed system during high-grade metamorphism (Flowerdew et al., 2006). The weighted average model age of all zircon analyses in this sample is 3474  $\pm$  31 Ma (MSWD = 3.3, n=23/24). It is notable that the Th/U ratio of cores (0.37 – 1.3) is considerably higher than that in the rims (0.03 – 0.14), supporting a metamorphic origin for the rims.

## White Nile (Uganda)

Modern river sands were collected from the White Nile upstream of the Sudd marshes at Wadelai (N7-16S) and Murchison Falls (N7-17S) in northern Uganda. Both samples show a similar distribution of Archaean ages, with maxima at c. 2620 Ma (Fig. 5), and only a few older grains. Analyses with <sup>207</sup>Pb/<sup>206</sup>Pb ages of 2500 – 2570 Ma commonly occur as overgrowths on older zircon cores. Only 3% of grains in sample N7-16S give younger, Neoproterozoic to Mesoproterozoic ages. However, sample N7-17S contains 24% of such grains, with significant populations at c. 630, 800 and 900-1000 Ma

The c. 2620 Ma zircon population in sample N7-17S typically shows oscillatory zoning under CL, and has an average  $\epsilon_{Hf}$  of -7 and a TDM $_{Hf}$  of 3412  $\pm$  23 (MSWD = 2.3, n=68/74; Figs. 4 and 6). Eight grains with ages between 900 and 1000 Ma have an average  $\epsilon_{Hf}$  value of -24, a composition that is consistent with formation by melting of Archaean crust similar to that represented by the c. 2620 Ma zircon population. The 900 – 1000 Ma grains show oscillatory zoning with no evidence for core/rim relationships. Zircons with ages between 670 and 870 Ma have  $\epsilon_{Hf}$  between +3.5 and -15,

requiring input of juvenile material in addition to Archaean crust. These grains are typically CL-light, and weakly zoned or unzoned, with Th/U of 0.22 – 0.89.

Four analyses of rims on Archaean zircon cores give ages of c. 630 Ma, with an average  $\varepsilon_{Hf}$  of -30.5 (Fig. 4). The overgrowths show no zoning under CL, and have low Th/U (<= 0.03), suggesting a possible metamorphic origin. A single zircon rim, which formed on a c. 800 Ma grain, also gives an age of c. 630 Ma, with  $\varepsilon_{Hf}$  = -14, again consistent with growth in a closed system under metamorphic conditions (Flowerdew et al., 2006).

Figure 4 includes a field for North-East African Archaean Basement, which is derived using the composition of zircons in the Saharan Metacraton gneiss (WD16A) and the detrital zircon grains from the White Nile at Murchison Falls (N7-17S). The slope of the observed trend is consistent with an average <sup>176</sup>Lu/<sup>177</sup>Hf ratio in the sampled North African crust of c. 0.012. This field represents the likely composition of craton-derived grains in other sedimentary rocks of the Nile catchment, discussed below.

## Arabian-Nubian Shield cover – the Hammamat Formation

Sample RSH14A is a volcanic arenite collected within conglomerates of the Hammamat Formation, in Wadi Hammamat (El-Rahman et al., 2010). The Hammamat Formation in this area is folded and cleaved, prior to intrusion of the Um Had granite at  $596.3 \pm 1.7$  Ma (Andresen et al., 2009).

Sample RSH14A has a rich heavy mineral assemblage dominated by epidote. Zircon grains are dominated by a c. 630 - 780 Ma grains with juvenile hafnium isotope values (Fig. 6). 80% of the overall zircon population have positive, juvenile  $\epsilon_{Hf}$  values (Fig. 6). Grains in the region of 700-800 Ma show higher  $\epsilon_{Hf}$  values (+14) than seen in any other sample in the Nile catchment area. 2% of analysed grains gave Palaeoproterozoic or Archaean ages.

Only four grains give apparent ages younger than 620 Ma, the youngest having a  $^{206}$ Pb/ $^{238}$ U age of 596  $\pm$  18 Ma – within error of the age of the structurally much later Um Had granite. The

timing of deposition of the Hammamat Formation is unclear, with suggested ages ranging between 630 Ma and 583 Ma (Bezenjani et al., 2014; Wilde and Youssef, 2002; Willis et al., 1988). It is therefore critical to establish whether these young grains could be used to determine a maximum depositional age for the Hammamat Formation, or whether these grains may have been affected by Pb-loss. Several grains with ages younger than 645 Ma were therefore revisited, and five new analyses were carried out on each grain. Data and age calculations are presented in Supplementary Data File 8. All grains yielded ages of c. 635 Ma or older, confirming that the original analyses had been affected by Pb-loss (Fig. 7). The original analyses used a large laser spot size (35  $\mu$ m, cf. 20  $\mu$ m for the new analyses), and may have been more likely to intersect cracks and defects in the grains.

## Phanerozoic cover rocks

### Western Desert (Egypt)

Samples WD04A, WD12A and WD18A (Figs 5 and 6) are Phanerozoic sandstones. These samples have a dominant zircon population at c. 600 Ma, with > 60% of grains of this age showing negative  $\epsilon_{Hf}$ . Zircons between 700 and 900 Ma are dominated by more juvenile grains, with positive  $\epsilon_{Hf}$ . A population of c. 1000 Ma grains appears to fall into two distinct groups, with juvenile and more crust-dominated hafnium compositions. In addition, subordinate populations of Paleoproterozoic and Archaean grains (totalling c. 10%), and sparse Phanerozoic grains are present.

## Red Sea Hills (Egypt)

Sample RSH09A is a Phanerozoic sandstone from Wadi Kharit in the Red Sea Hills. Zircons are dominated by peaks at c. 620, 730-900 and 1000 Ma, with predominantly juvenile  $\epsilon_{Hf}$ . A minority of grains at c. 620 and 1000 Ma show more cratonic  $\epsilon_{Hf}$  values. 25% of c. 620 Ma grains and 42% of c. 1000 Ma grains show negative  $\epsilon_{Hf}$ . 8% of grains give Archaean or Palaeoproterozoic ages.

#### Modern wadi sands

Sample RSH07A is a modern wadi sand collected in Wadi Hammamat in the Red Sea Hills, deposited on Phanerozoic sandstone bedrock. The zircon age and  $\epsilon_{Hf}$  isotope distribution are similar to the Red Sea Hills Phanerozoic bedrock sample, with peaks at c. 620, 770 and 1000 Ma. 50% of 620 Ma grains and 42% of c. 1000 Ma grains have cratonic  $\epsilon_{Hf}$  values. 8% of grains give Archaean or Palaeoproterozoic ages.

#### Modern river sands

## Blue Nile

Sample ETH02A was collected from the Blue Nile Gorge in the Ethiopian Highlands. It shows two main zircon age populations at 660-720 Ma and 800-850 Ma.  $\epsilon_{Hf}$  values are dominantly juvenile, although sparse grains at 500-600 Ma yield  $\epsilon_{Hf}$  values as low as -10. Sparse c.1000 Ma ages are also present, but Archaean and Palaeoproterozoic ages are absent. Juvenile Cenozoic grains are present (Fig. 8). Sample SD03A was collected near Wad Madani in Sudan. It is dominated by 750-850 Ma zircons with juvenile  $\epsilon_{Hf}$  values. Only sparse c.600 Ma grains are found, and only one Palaeoproterozoic age was recorded.

# Tekeze

Sample ETH06A was collected from the Gibai River, a tributary of the Tekeze, while Sample ETH08A was collected further downstream at Malemin Bridge. Both samples show dominant peaks at c. 620 Ma and a number of peaks ranging back to c.1000 Ma. Sparse Palaeoproterozoic and Archaean grains make up c. 6% of each population. A single Cenozoic grain was identified in each sample.

#### Atbara

Sample SD06A was collected from the Atbara close to its confluence with the Nile trunk in Sudan. The most dominant population forms a peak at c.600 Ma, with a subordinate peak at c.800

Ma, both dominated by juvenile  $\epsilon_{Hf}$  values. Palaeoproterozoic and Archaean grains are sparse (3%). Cenozoic grains make up 6% of the population.

#### White Nile

Sample SD02A was collected from the White Nile downstream (north) of the Sudd marshes in Sudan, and south of the Gezira Fan. Zircons are dominated by c.600 Ma grains, with subpopulations showing both juvenile and more cratonic  $\epsilon_{Hf}$  values. Older age peaks at c. 850 and 1000 Ma are dominated by juvenile  $\epsilon_{Hf}$  values. Only two Archaean grains were identified (c. 2%), in marked contrast to sample N7-17S (see above) from upstream (south) of the Sudd marshes.

Sample SD01A was collected downstream of the Blue Nile-sourced Gezira Fan. It shares similar peak characteristics to sample SD02A, but with more dominant c.650 and 1000 Ma populations.

#### Nile trunk

Sample SD04A captures the signature of the Nile trunk in northern Sudan, downstream of the confluences of the Blue Nile, White Nile and Atbara, but upstream of the Aswan Dam. The detrital zircon age and  $\epsilon_{Hf}$  distribution are similar to the upstream Blue Nile sample (ETH02A), with peaks at c. 630, 680 and 830 Ma, all dominated by juvenile  $\epsilon_{Hf}$  values. Only two Archaean to Palaeoproterozoic ages are present (2%). Four Cenozoic ages were measured.

Sample BS01 was collected north of the Aswan dam at Al Wasta, south of Cairo. It is dominated by c. 600 Ma zircons, with smaller populations at c.830 and 1000 Ma.  $\epsilon_{Hf}$  values show dominantly juvenile values, but 11% of c. 600 Ma and 73% of c. 1000 Ma grains have negative, craton-influenced values. Archaean and Palaeoproterozoic grains make up 9% of the zircon population. No Cenozoic grains were observed.

# Ar/Ar Mica and Plagioclase Feldspar and U/Pb Rutile

Samples analysed include White Nile, Blue Nile and Atbara, Nile trunk, Red Sea Hills modern wadi and bedrock, and Western Desert aeolian samples. Data tables are provided in Supplementary Items 10 and 11.

The <sup>40</sup>Ar/<sup>39</sup>Ar mica and plagioclase data and the U-Pb rutile data all show an overwhelming Pan-African signature (c.600 Ma). This includes the White Nile samples upstream of the Sudd, despite having a strongly Archaean signature in U/Pb zircon analyses, and indicates strong overprinting by the Pan-African orogeny in this region. The Blue Nile also contains Cenozoic feldspars, derived from the Ethiopian Highlands and in agreement with the presence of Cenozoic zircons also recorded in this river (see above).

Although the number of white mica analyses is small, we observe a change in signal between White Nile sands south of the Sudd marshes, and the Nile trunk and Red Sea Hills to the north. The White Nile sample (N7-17S) is dominated by a single population with a mean age of c. 615 Ma. By contrast, both the Nile trunk in Sudan (Sample SD04A) and a Red Sea Hills wadi sand (RSH07A) show age peaks at c. 600 and 650 Ma.

# Sr, Nd and Hf isotope bulk analysis

Bedrock mudstone, and mud samples from the surface of flash-flood deposits, were collected in the Red Sea Hills. The samples show a limited range in isotope composition (Figs. 9, 10, Supplementary Item 12), with moderately radiogenic  $^{87}$ Sr/ $^{86}$ Sr (averaging 0.7096), and non-radiogenic  $\epsilon_{Nd}$  (averaging -6.1) and  $\epsilon_{Hf}$  (averaging -4.9). Mudstones, muds and aeolian sands were collected in the Western Desert. These show considerably more scattered results, with radiogenic  $^{87}$ Sr/ $^{86}$ Sr (0.708 to 0.716) and non-radiogenic  $\epsilon_{Nd}$  (-5.8 to -12.8) and  $\epsilon_{Hf}$  (-8 to -21.6).

Modern river mud samples collected from the Atbara and Tekeze rivers have Sr, Nd and Hf isotope compositions similar to average Ethiopian basalts (Figs. 9 and 10; Meshesha and Shinjo,

2010; Pik et al., 1999). Samples from the Blue Nile and the Nile trunk in northern Sudan plot on trends towards higher  $^{87}$ Sr/ $^{86}$ Sr, and lower  $\epsilon_{Nd}$  and  $\epsilon_{Hf}$ , consistent with incorporation of older sediment derived from sources other than Ethiopian basalt. Average values for Holocene Nile trunk sediments in Northern Sudan, and sand samples collected in Egypt before the construction of the Aswan High Dam also plot on this trend (Shukri, 1949; Woodward et al., 2015). A sample collected from the White Nile just south of its confluence with the Blue Nile at Khartoum plots close to the Nile trunk trend.

Three modern river mud samples have what, at first glance, appear to be anomalous isotope compositions, given their locations. A sample from the Uri River, Ethiopia, (ETH07B), a tributary of the Tekeze in Ethiopia, has values unlike those one might expect from the Ethiopian plateau continental flood basalt region. However, the Uri River's local catchment drainage basin geology is in fact Phanerozoic cover (Fig. 1), consistent with its isotopic signature. Samples SD01B and SD02B are White Nile samples, yet their Sr-Nd signatures are similar to the Blue Nile and Phanerozoic cover respectively. This is consistent with, in the case of SD01B, its location downstream of the Gezira Fan which is an overspill of Blue Nile detritus, and in the case of SD02B, its location downstream of the Sudd Marshes which acts as a sediment trap to the upstream White Nile and thus its provenance is locally derived, as discussed in more detail in following sections.

Two samples collected from the Albert and Victoria branches of the Nile in Uganda were also analysed. These have highly radiogenic  $^{87}$ Sr/ $^{86}$ Sr (0.718, 0.721), and non-radiogenic  $\epsilon_{Nd}$  (-26.3, -34.1) and  $\epsilon_{Hf}$  (-25.9, -55.4) consistent with derivation from local cratonic basement.

Excluding the three anomalous samples described above, for which local geological anomalies provide explanation, and the White Nile samples taken from Uganda, the range in isotope composition shown by the Nile and its tributaries is rather limited:  $^{87}$ Sr/ $^{86}$ Sr varies from 0.7043 in the Atbara to 0.7067 in the White Nile sample from Khartoum;  $\epsilon_{Nd}$  varies from 5.5 to -1.8; and  $\epsilon_{Hf}$  varies from 10.8 to -1.8.

# Discussion

Our data document early crust-forming events, Precambrian orogenies culminating in the Neoproterozoic assembly of Gondwana, subsequent erosion and deposition of voluminous sediments, and finally Cenozoic uplift of the Red Sea Hills and Ethiopian Highlands and eruption of continental flood basalts in Ethiopia. Additionally, our data track the influence of geomorphology and local geology on the progressive evolution of the Nile sedimentary signal downstream.

#### Palaeoproterozoic and Archaean cratons

The Nile traverses Precambrian rocks of the Congo Craton and Saharan Metacraton. The geology of the Saharan Metacraton (Fig. 1) is poorly understood due to the overlying sedimentary cover and desert sands, and as a result very little modern isotope data is available to help constrain its age and tectonic evolution; the U-Pb ages reported by Abdelsalam et al. (2002) are discordant pre-chemical abrasion ID-TIMS ages, or model ages derived by zircon evaporation techniques, which yield poorly constrained <sup>207</sup>Pb/<sup>206</sup>Pb ages only. However, Bea et al. (2011) report Archaean U-Pb SHRIMP ages as old as 3 Ga for gneisses at Uweinat, with evidence for crust as old as 3.22 Ga at Gebel Kamil in southern Egypt. Similar ages have been obtained in this study for gneisses from c. 80 km east of Uweinat, within the region mapped as the meta-igneous Gebel Kamil Series by Bea et al. (2011). The sample studied here (WD16A) has evidence for a metasedimentary protolith (broken pre-metamorphic zircon cores), with evidence for zircon growth between c. 3.2 and 2.9 Ga, and subsequent metamorphism at c. 2.7 Ga. Depleted mantle Hf model ages lie between 3.4 and 3.5 Ga. We see no evidence in our data for the c.2 Ga thermal event (the Eburnean orogeny) recorded by Bea et al. (2011).

Sample N7-17S, a modern river sand sample collected at Murchison Falls, Uganda, was sampled from within the Northeast Congo Block of the Congo Craton, close to the Aswa Shear Zone, which forms the boundary with remobilized crust of the Saharan Metacraton to the north (Appel et al., 2005; Katumwehe et al., 2015), adjacent to the Pan-African Central African Fold belt. Zircons

from this sample have similar Hf model ages (Fig. 4) to the Archaean gneiss sample from the Saharan Metacraton (WD16A). Zircons in the Murchison Falls sample record crystallisation at c.2.7 Ga, c.960 Ma and c. 600 Ma; the younger age population forms rims with Hf isotope compositions that are consistent with re-melting of similar Archaean crust. The fields defined by the Saharan Metacraton and modern river White Nile provides the best estimate of the composition of Precambrian cratonic basement in the Nile catchment. 600 Ma zircon rims, and similar aged rutile, plagioclase and mica indicate penetration of the effects of the Pan-African orogeny deep into the craton interior, in agreement with the previous work and conclusions of Appel et al. (2005) and Schenk et al. (2007).

#### The Hammamat Formation

The depositional and tectonic environment of the Hammamat Formation, which overlies the Precambrian Arabian-Nubian Shield basement, is disputed (e.g. Abdeen and Greiling, 2005; Bezenjani et al., 2014; Eliwa et al., 2010; Johnson et al., 2011; Ries et al., 1983; Wilde and Youssef, 2002). Proposed depositional ages lie close to the time of the Pan-African Orogeny, and both precollisional and post-collisional setting have been proposed. Local versus long distance provenance is debated. Previous work has recorded detrital zircon grains with ages as young as 585 Ma in the Hammamat Formation (Wilde and Youssef, 2002), which has implications for assigning a maximum depositional age to these rocks. The volcanic arenite sample from the Hammamat Formation studied here contains four zircon grains that give <sup>206</sup>Pb/<sup>238</sup>U ages younger than 620 Ma. In detail, these grains are slightly discordant, and repeat analysis (5 analyses for each grain) demonstrated that these grains had indeed suffered Pb-loss, and had crystallisation ages of 635-640 Ma (Fig. 7). This is consistent with the age of the youngest concordant zircon population with consistent hafnium isotope composition in the data set, which has an age of c. 640 Ma. In the light of this, we suggest that any maximum depositional ages for the Hammamat Formation based on sparse young grain ages should take into account the possibility that such grains have suffered Pb-loss.

Hammamat Formation zircon data analysed as part of this study (Figs. 5, 6 and 7) can be largely explained entirely by derivation from underlying local Arabian Nubian Shield bedrock, which is dominated by zircons with juvenile hafnium isotope compositions derived from 870-630 Ma oceanic arc rocks (e.g. Stern et al., 2010). A small number of Pre-Neoproterozoic detrital zircons are found in the Hammamat Formation. Wilde and Youssef (2002) suggest possible source areas for these Pre-Neoproterozoic grains could be the Central and South Eastern Desert, or further afield in the Arabian Nubian Shield in parts of Saudi Arabia where sparse zircons of this age have previously been recorded. However, we believe that such distal sources are not required to explain our data because it is possible that they have been derived from the more proximal Egyptian ANS (Ali et al., 2009). The Arabian Nubian Shield bedrock in Egypt incorporates some inherited pre-880 Ma igneous zircons with juvenile  $\epsilon_{\rm Hf}$  values indicating either incorporation of sediments during subduction along a passive margin or inheritance from the mantle source region, or both (Stern et al., 2011).

The volcanic lithic arenite composition and epidote-rich nature of the Hammamat bedrock sample (RSH14A) supports our interpretation that it is derived directly from Arabian-Nubian Shield arc rocks. Furthermore, the composition and zircon U-Pb ages of the sample are in stark contrast to the overlying post-collisional Phanerozoic cover sequences in the region, which are quartz arenites with ZTR indices as high as 99 and have diverse zircon U-Pb age spectra, including a typical 600 Ma peak as described below. We therefore see no requirement to invoke long-distance transport of detritus to form the Hammamat Formation.

### Phanerozoic Sedimentary Cover Rocks and overlying modern sediment

Our analyses provide the first isotopic data from the Phanerozoic sedimentary cover bedrocks in Egypt. Samples from Phanerozoic sandstone cover sequences in the Western Desert and Red Sea Hills of Egypt are mineralogically mature. They compare well to Phanerozoic cover successions in surrounding regions across North Africa and the Middle East in terms of both zircon age spectra (e.g. compare with North African data in Avigad et al. (2012); and Avigad et al. (2003)), and zircon  $\epsilon_{Hf}$ 

characteristics, namely the typical "double plunge" to negative values at c.600 Ma and c.1000 Ma (e.g. compare to data from Jordan; Morag et al., 2011a), first recognised as characteristic in Israeli coastal sands by Be'eri-Shlevin et al. (2014).

Red Sea Hills Phanerozoic cover rocks and modern wadi samples do not consist entirely of material recycled from the underlying Arabian-Nubian Shield basement. They are derived, at least in part, from out with the underlying Arabian-Nubian Shield basement. This is indicated by:

- the occurrence of >2000 Ma zircons, which are extremely rare in the Arabian-Nubian Shield (Stern et al., 2010);
- the increased mineralogical maturity and more complex zircon populations compared to the underlying locally sourced Hammamat Formation (see above);
- the presence of a significant 1000 Ma population with cratonic  $\epsilon_{Hf}$  values, which is incompatible with derivation from the juvenile arc of the Arabian-Nubian Shield.

A similar conclusion was reached by Morag et al. (2011b) who looked at  $\epsilon_{Hf}$  values of the 1000 Ma zircon population in cover sequences of Israel and Jordan. Although their data showed a preponderance of grains with negative  $\epsilon_{Hf}$  values, our data from Egypt shows two populations at c.1000 Ma, one with positive  $\epsilon_{Hf}$  values and one with negative values. Whilst the population with positive values would be compatible with derivation from an as yet unrecorded arc from within the Arabian-Nubian Shield, the provenance of the population with negative  $\epsilon_{Hf}$  values remains enigmatic. The cratonic c.1000 Ma zircons found in the Mesozoic cover of both Western Desert and Red Sea Hills samples can be most simply explained by recycling from underlying Palaeozoic cover sequences, but the original basement source is debated. As pointed out by Kolodner et al. (2009), long distance transport from suitable cratonic source areas far to the south are ruled out by the paucity of >2000 Ma grains which would also be present if derived from such a region. Avigad et al. (2003) proposed that the source could be material transported from East Africa towards the margins of Gondwana by Neoproterozoic glaciers. We suggest that the original source may have been from a

region since rifted off Gondwana, which may have also supplied zircons to, for example, the Neoproterozoic Arkenu Formation cover sediments of Libya, which contains grains of an appropriate age (Meinhold et al., 2011).

Phanerozoic bed rock samples and Red Sea Hills modern wadi sand which partially covers the bedrock, have similar zircon U-Pb age spectra and show a similar "double plunge" of  $\epsilon_{Hf}$  characteristics to negative values at c. 600 Ma and c.1000 Ma (Figs 5 and 6). This similarity suggests that the origin of the modern sand is broadly that of recycled material from Phanerozoic sedimentary rocks which the sand mantles. Some component of aeolian transport undoubtably contributes in the deposition of such modern sands. However, the extent of such transport distance is difficult to determine given the similarity of Phanerozoic sedimentary cover signature across vast distances of north Africa. Western Desert dune sands were not analysed for zircon U-Pb, and thus an assessment of far-field aeolian input distinct from that recycled from Phanerozoic sandstones cannot be assessed. The similarity in bulk rock data between the Western Desert mudstone and the aeolian dune sand leads us to tentatively suggest that, like in the Red Sea Hills, composition of the modern sand is broadly similar to Phanerozoic sedimentary bedrock.

## Young zircons derived from the Ethiopian Highlands

Cenozoic zircons were found in the Blue Nile (sample ETH2A), the Tekeze and Atbara rivers (ETH6A, ETH8A, SD06) and downstream in the Nile trunk in Sudan (SD04). Grains of such age were not recorded in the White Nile or modern wadi sediment from the Red Sea Hills (Fig. 8). The obvious source for these grains is the Cenozoic Ethiopian volcanic province in the Ethiopian Highlands (Fig. 1) which includes a variety of volcanics from flood basalts to shield volcanoes, and bimodal compositions with significant ignimbrites and rhyolitic airfall tuffs intercalated with the basalt lava flows (Kieffer et al., 2004; Prave et al., 2016; Ukstins et al., 2002). Our Cenozoic grain ages extend from ~23-33 Ma, with population peaks at 25 Ma and 30 Ma. These compare well with the timing of a major period of bimodal and silicic volcanism in Ethiopia, dated at ~25-30 Ma (Ukstins et al., 2002).

Furthermore, Hf analyses we undertook on both our samples and on grains from Lake Tana Rhyolites of the Ethiopian Highlands, previously analysed for U-Pb by Prave et al. (2016), show an excellent match (Fig. 8). Ukskins et al. (2002) documented pre-rift bimodal magmatism in Ethiopia from 31 to 24 Ma. They recorded a major decrease in volcanism between 25-20 Ma, which they associated with the transition from pre- to syn-rift volcanism triggered by the separation of Africa and Arabia. The zircons we have analysed would, by their definition, be of pre-rift origin.

# Effects of geomorphology, local bedrock geology and aeolian input on the isotope signal of the Nile River

North of Uganda, the White Nile passes through an extensive area of marsh land (The Sudd) which has trapped most of the detritus from the river since 2.7 Ma (Williams and Talbot, 2009). This is clearly reflected in the detrital zircon data. Zircons in Ugandan White Nile modern river sediments, south of the Sudd, are dominated by Archaean grains with evidence for crustal reworking and new zircon growth at c.960 and 600 Ma. A craton-dominated signature is also displayed by the highly negative  $\varepsilon_{Nd}$  values and high  $^{87}Sr/^{86}Sr$  values of the White Nile muds.

By contrast, north of the Sudd at Kosti, the detrital signature of White Nile zircons is much less cratonic. In sample SD02A, located north of the Sudd but south of the Gezira Fan, Archaean grains are almost completely absent, and instead the zircon populations show strong similarities to Blue Nile and/or Phanerozoic cover sediment signatures. A mud sample from the same location has Sr-Nd-Hf values similar to Phanerozoic cover (Figs 9 and 10). Potential contributing sources to this White Nile sample north of the Sudd could be the Sobat River, and/or alluvial fans draining into the river from the Nuba Mountains.

The River Sobat drains similar lithologies to the Blue Nile. Published data show it to have  $^{87}$ Sr/ $^{86}$ Sr values of 0.708-0.712 and  $\epsilon_{Nd}$  values of -1.6 to -9.1 (Padoan et al., 2011), spanning the range of our data for modern muds from Phanerozoic cover, as sampled in the Red Sea Hills However, the River Sobat flows through an extensive region of marsh land north of the Sudd (the Machar

Marshes) and thus was not considered to be a significant source of sediment to the Nile trunk downstream by Padoan et al., (2011).

Sample SD02 was collected north of Kosti, close to the southern limit of the Gezira Fan, so significant input of Blue Nile material from the fan is not thought to be likely, and furthermore, such input would not be compatible with the bulk sediment Sr and Nd data. However, from the Last Glacial Maximum (c. 20 ka) onwards, a major alluvial fan (the Khor Abu Habl alluvial fan) has drained into the river from the Nuba Mountains to the west (Williams et al., 2000). Ephemeral rivers feeding the fan drain Proterozoic basement rocks and overlying Phanerozoic siliciclastic cover sequences, thus compatible with the isotopic signatures observed in this sample.

Further downstream, a mud sample at SD01B, collected just to the south of Khartoum has an  $\epsilon_{Nd}$  of -1.8 and  $^{87}\text{Sr}/^{86}\text{Sr}$  of 0.7067, a signature with affinity to the Ethiopian Continental Flood Basalts. This is due to the sample location being adjacent to the Gezira Fan (Williams 2009), which formed during the Late Pleistocene by overspill from the Blue Nile, and contributes today to White Nile sediment load. Significant contribution from the palaeo-Blue Nile sourced Gezira Fan is also reflected in the zircon age spectrum of sample SD01A (Fig 5) where the dominant peak at 800 Ma strongly resembles the modern Blue Nile signature of sample SD03A in Sudan.

Further downstream to the north, the Blue Nile, and then the Atbara, join the Nile trunk. Both the Blue Nile and the Atbara (with its tributary the Tekeze) drain, from south to north downstream: Cenozoic volcanic rocks, Phanerozoic cover, Arabian-Nubian Shield, and Proterozoic basement with Pan-African mineral cooling ages (Mock et al., 1999). Sr-Nd-Hf isotope data indicate that the Atbara has a higher proportion of volcanic detrital contribution to the river sediment compared to the Blue Nile. This is also reflected in petrographic data showing a higher proportion of clinopyroxene and olivine (Garzanti et al., 2006), and the higher proportion of V and Cr in the Atbara compared to the Blue Nile (Fig. 3). By contrast, a sample collected from a tributary of the Tekeze (the Uri River) in an area of Phanerozoic cover rocks has an isotope composition typical of Phanerozoic

cover successions (Figs. 9 and 10). Variation in the contributing bedrock lithology is also illustrated by the significant change in proportions of major zircon populations in the Blue Nile downstream. Notably, the region of Arabian-Nubian Shield cut by the Blue Nile in NW Ethiopia contains granitoids with U-Pb zircon ages in the ranges from 650 - 700 and 800-880 Ma, and juvenile  $\epsilon_{Nd}$  values (data summarised in Johnson (2014)). These populations are clearly represented in our Blue Nile dataset (Fig. 5), with prominent peaks at 800-830 Ma in samples ETH02A and SD03A, persisting into the Nile trunk at Dongola (sample SD04A). 680 Ma zircons are also abundant in sample ETH02A from the Blue Nile, and in the southern Nile trunk (SD04A).

The Nile trunk sample downstream of the White Nile confluence with the Blue Nile and Atbara, at Dongola in Sudan (SD04A), shows a U/Pb zircon signature much the same as the Atbara and Blue Nile combined. Garzanti et al. (2006) suggested a greater input to the trunk Nile from the Blue Nile compared to the Atbara, based on petrographic data. The greater similarity in Sr-Nd and Cr and V values between the Blue Nile and Nile trunk, compared to the Atbara and Nile trunk (Figs. 3 and 9) are consistent with this proposal.

By the time the Nile River reaches northern Egypt (sample BSO1A; Figs 5 and 6), its signature has changed to more closely resemble that of typical Phanerozoic sedimentary cover in terms of zircon age spectra, with more Palaeo-Proterozoic to Archaean grains, and the double plunging  $\epsilon_{Hf}$  to negative values at c.600 Ma and c.1000 Ma (also clearly observed in a Nile sample collected near Cairo by lizuka et al. (2013). This downstream change can be explained by the Nile's route in this stretch of the river, which flows across the Phanerozoic sedimentary bedrock cover and overlying modern sediment of the Red Sea Hills to the East, and Western Desert to the west. Any such input to the Nile from the west would more likely be transported to the river by aeolian processes, and from the east would include flash flood transportation, given the higher topography of the Red Sea Hills compared to the Western Desert.

Overall, when considering the entire length of the Nile, undoubtably aeolian transportation plays a part in delivering detritus to the river. However, such input does not mask the dominant bedrock and geomorphological controls which we have demonstrated to effect the changes in the river's signature downstream, for both sand and mud grade material.

Finally, we consider that the contribution of the White Nile to the Nile delta is small, the result both of its significantly lower mean annual discharge compared to the Blue Nile, and sediment trapping in the Sudd Marshes. Both Sr-Nd and trace element values of the trunk Nile are more similar to those of the Blue Nile / Atbara than the White Nile (Figs 3, 9 and 10). Additionally, the white micas of the trunk Nile show similarity in Ar-Ar age distribution to grains collected from the Red Sea Hills, and dissimilarity with the age spectra from the White Nile south of the Sudd. Whilst Pre-Neoproterozoic zircon grains make up to 97% of sediment in the White Nile south of the Sudd, such aged grains in the Nile trunk are not required to be delivered by the White Nile. Pre-Neoproterozoic grains are in fact extremely sparse in the White Nile north of the Sudd (average 2%). Pre-Neoproterozoic grains are relatively common in the Phanerozoic cover sedimentary rocks and modern wadi sediments sampled in the Red Sea Hills (average 9%) and the Western Desert (average 12%) and are also found in the Blue Nile (average 1%) and in sediments from the Atbara and Tekeze (average 6%) (Fig. 5). Any of these regions could have supplied the Neoproterozoic zircons recorded in the Nile trunk.

### Supercontinent Assembly and the Pan-African Orogeny

Figure 11 shows all of the U/Pb and Hf isotope data derived from Nile hinterland samples as part of this study. The largest peak at c.600 Ma relates to the assembly of East and West Gondwana, when Neoproterozoic juvenile intra-oceanic island arcs of the Arabian-Nubian Shield accreted, and finally collided with the Saharan Metacraton and Congo Craton (Johnson et al., 2011). This can be seen in samples derived from as far afield as the White Nile in Uganda.

The influence of the Pan-African Orogeny was also seen in U/Pb rutile, and Ar/Ar plagioclase and mica ages which reflect post orogenic cooling. This is in agreement with hornblende, muscovite, amphibole, biotite and sericite <sup>40</sup>Ar/<sup>39</sup>Ar data from Arabian-Nubian Shield bedrock (Johnson et al., 2011).

In detail, the zircon U-Pb / Hf isotope data show two maxima at c. 840-780 and 700-600 Ma with juvenile  $\epsilon_{Hf}$  values, which represent two major episodes of arc development within the Arabian-Nubian Shield. The reduction in data density between the two episodes may relate to reduced subduction-related magmatism at this time, perhaps resulting from collisions between arcs within the Arabian-Nubian Shield. Arc-collision is documented by the emplacement of ophiolites at this time (Ali et al., 2010), and may be represented in our dataset by c. 750 Ma detrital zircons from the Hammamat Formation, which have the highest (most juvenile)  $\epsilon_{Hf}$  values in the entire data set.

The final closure of the Mozambique Ocean is documented by the marked switch to more negative  $\epsilon_{Hf}$  values in the c.600 Ma population, due to the intrusion of post-collisional granites derived by melting of continental crust within the suture zone.

A notable feature of the data is the discontinuity of the field of c.1000 Ma grains with juvenile  $\epsilon_{Hf}$  versus those with more cratonic influence (i.e.  $\epsilon_{Hf}$  < 0). Whilst the ultimate source of these grains remains unknown, our data suggest two distinct ultimate sources, one from an oceanicarc setting and a second involving re-melting of a cratonic source.

The most cratonic (i.e. lowest  $\epsilon_{Hf}$ ) grains within each age population in our dataset plot close to the 3500 Ma evolution line for North African crust derived using our basement data from the Saharan Metacraton in Egypt and Congo Craton in Uganda.

The supercontinent cycles which led to the creation of Gondwana (650-500 Ma) are represented in all of the samples collected as part of this study (Fig. 11). The isotope signatures of the samples analysed from the Nile River and its hinterland contain Mesoproterozoic and older zircons that may be related to earlier supercontinent cycles. However, the location of the Congo

craton in reconstructions of Rodinia (De Waele et al., 2008) is a matter of debate and the lack of exposure and reliable geological data from the Saharan Metacraton means that its location in Rodinia and earlier supercontinent configurations is unknown. Although our data contain some c.2000 Ma zircons, these cannot be directly related to the formation of the supercontinent 'Nuna' (aka. Colombia) because Nuna did not involve North African cratons (Rogers and Santosh, 2002).

# **Conclusions**

Gneissic basement of the Saharan Metacraton in southern Egypt, and detrital zircons derived from rocks of the Congo Craton in northern Uganda, both indicate an age of crust formation between 3.0 and 3.5 Ga, with subsequent melting and/or metamorphism at c. 2.7 Ga.

Our combined U-Pb and Hf-isotope zircon dataset document the evolution of the North African crust, in particular highlighting phases in the development of the Arabian-Nubian Shield and its collision with the Saharan Metacraton. The data show two phases of arc magmatism, at c. 840-780 and 700-600 Ma, with a reduction in magmatism at c. 750 Ma, during a period of arc collisions within the Arabian Nubian Shield.

The Hammamat Formation, sampled in Wadi Hammamat, has a maximum depositional age of 635 Ma, based on reanalysis of anomalously young zircon grains, which are shown to have suffered Pb-loss. Provenance analysis indicates that the Hammamat Formation is locally-derived. By contrast, the Phanerozoic cover sedimentary rocks overlying the Arabian-Nubian Shield are derived, at least in part, from distal sources beyond the Arabian-Nubian Shield. Phanerozoic cover sedimentary rocks which blanket much of North East Africa represent an important source of detritus to the Nile River and are characterised by the presence of a major zircon population with both juvenile and crustal  $\epsilon_{\rm Hf}$  values reflecting the Pan African orogenic event, and a significant (9-12%) population of pre-Neoproterozoic zircon grains. The original source of c.1000 Ma grains in the Phanerozoic sedimentary cover rocks remains enigmatic, but we have identified that there are two distinct populations: one indicating a juvenile ultimate source and one a cratonic source.

In the modern Nile drainage, there is considerable evolution downstream, controlled predominantly by changes in local geology and geomorphology. The provenance signature of the White Nile is dramatically different upstream and downstream of the Sudd marshes as a result of sediment trapping. South of the Sudd, White Nile sediments are craton-derived. North of the Sudd, at Kosti, the signature of the White Nile is dominated by material derived from Phanerozoic sandstones supplied via alluvial fans to the west of the river. Further north, south of Khartoum, White Nile sediment composition is affected by its proximity to the Pleistocene Blue-Nile sourced Gezira Fan. The Blue Nile's and Atbara's signatures are influenced predominantly by input from the Ethiopian Flood Basalts in terms of their bulk rock signature, and by proximity to the Arabian-Nubian shield in terms of zircon characteristics. A further shift in sediment signature in terms of zircon characteristics is seen by the time the Nile reaches northern Egypt, reflecting the river's passage through Phanerozoic cover sedimentary rocks and overlying modern sands of the Red Sea Hills and Western Desert through this stretch of river. By contrast, the bulk isotopic Sr-Nd-Hf data show little downstream evolution and remain dominated by mafic input from the Ethiopian Highlands as far south as northern Egypt.

Acknowledgements: This work was funded by a NERC Open CASE PhD studentship award NE/I018433/1, BP Egypt, and awards from the NERC Isotope Geoscience Facilities Steering Committee (IP-1248-0511, IP-1299-0512). We gratefully acknowledge the expertise of Dr. Frank Darius at the Freie Universitat, Berlin, Germany, and Mr. Mahmoud Abo Elwfa Muhammad for our Egyptian fieldwork, Tadesse Berhanu of Addis Ababa University in Ethiopia, and Mahdia Ibrahim and family, along with our guides Midhat and Moez in Sudan. At NIGL, we thank Carlyn Stewart, Vanessa Pashley and Nick Roberts for valuable lab assistance. This paper benefitted from careful reviews by William Bosworth and Robert Stern.

# **Figure Captions**

- Figure 1. Modern river samples and hinterland geology of Nile River source areas. Inset shows
   location of map as boxed area. Map modified from (al-Miṣrīyah, 1981; Johnson, 2014; Kazmin, 1972;
   Ministry of Energy and Mines, 1981).
  - Figure 2. Petrographic variability of detrital modes in modern river sands, bedrock and aeolian sands from the Nile trunk and hinterland, illustrating the proportion of quartz (Q), K-feldspar (Kfs) and plagioclase feldspar (Pl). Samples are modern river sands unless otherwise stated. Data from Garzanti et al. (2006)(\*) and Garzanti et al. (2015) (\*\*).
  - Figure 3. Selected trace element concentrations from each of the Nile source areas and the main Nile trunk, normalized to trace element concentrations of Post Archean Australian Shale (PAAS: McLennan and Taylor (1985). All samples are modern muds or mudstones, except for samples from the Nile trunk in Egypt collected prior to construction of the Aswan Dam (Shukri, 1949), which are modern sands. \* Data from Garzanti et al. (2013). The field for Red Sea Hills samples encompasses eleven samples. Other distributions represent single analyses, or averages of small groups of samples (See Supplementary Item 5).
  - **Figure 4.** U/Pb age and hafnium isotope composition of complex zircons in Archaean gneiss within the Saharan Metacraton (WD16A), and detrital zircons in White Nile sand draining Archaean craton (N7-17S). Two-sigma uncertainties are smaller than the symbol size, typically c. 20 Ma on the age determinations, and 1-2  $\epsilon_{\rm Hf}$  units.
  - **Figure 5.** Detrital zircon U-Pb age frequency and relative probability plots for samples from the Nile catchment and surrounds.
  - **Figure 6.** Detrital zircon U-Pb age frequency and relative probability plots with  $\epsilon_{Hf}$  vs U-Pb age. The blue line at 600 Ma marks the approximate time of the Pan-African orogeny and the collision of the Arabian-Nubian Shield with the Saharan Metacraton and Congo craton. The blue line at 1000 Ma highlights the dual juvenile and cratonic populations of the enigmatic 1000 Ma population. The

reference line for 3500 Ma crust is calculated using  $^{176}$ Lu/ $^{177}$ Hf ratio of 0.017 which is derived from the apparent crustal evolution trend displayed in Fig. 4.

**Figure 7.** Tera-Wasserburg plot illustrating the effect of Pb loss on an apparently 'young' Hammamat Formation zircon grain. Replicate analyses of the same grain indicate a true age c. 40 Ma older.

**Figure**: **8.** Cenozoic zircon data from the modern Nile catchment. (a) Histograms for Cenozoic detrital zircons from the Nile and its tributaries, together with zircons from Lake Tana rhyolites from the Ethiopian Highlands (ages from Prave et al. (2016)). Probability density plot is shown for combined data. (b) Plot of initial  $\varepsilon_{Hf}$  values against age for detrital zircons from the modern Nile and its tributaries, together with zircons from the Lake Tana rhyolites. Numbers in parentheses in the key indicate the number of Cenozoic grains detected in each sample relative to the total number of grains analysed. Note that zero Cenozoic grains were recorded in the modern White Nile and Red Sea Hills modern wadi sediments.

**Figure 9.** <sup>87</sup>Sr/<sup>86</sup>Sr versus  $\varepsilon_{Nd}$  isotope data for Nile river muds; Egyptian Nile trunk sands taken prior to the construction of the Aswan dam (samples from Shukri, 1949); Nile hinterland mudstones, wadi muds and aeolian sands; and Sudanese Holocene Nile trunk samples (Woodward et al., 2015). Also shown for comparison are average data from the Ethiopian Contintental Flood Basalts (Pik et al., 1999).

Figure 10  $\epsilon_{Hf}$  plotted against  $\epsilon_{Nd}$  for the same samples as shown in Fig 8. \*Ethiopian basalt data from Meshesha and Shinjo (2010). Symbols as in Fig 9, except as noted.

**Figure 11.** Probability density plot illustrating the Hf isotope – time evolution of the Nile source regions supplying detrital zircons discussed in this study. The density of the data distribution is calculated using a modified version of the MATLAB implementation of the Kernel Density Estimation procedure supplied by Botev et al (2010) using bandwidths equivalent to the typical analytical uncertainty of the U/Pb ages (±20 Ma) and epsilon hafnium values (± 1 epsilon units). Contours are generated by the MATLAB *contour3* function and plotted using the *plot3d* function. Also shown is

829 the compilation of African detrital data from Condie and Aster (2010), and the modern African river 830 data of Iizuka et al (2013) filtered using the discordance criteria applied in this paper. **Supplementary information** 831 832 Supplementary Item 1: Sample information 833 Supplementary Item 2: Methods Supplementary Item 3: Petrography data 834 835 Supplementary Item 4: Heavy mineral data 836 Supplementary Item 5: Major and trace element data. 837 Supplementary Item 6: U-Pb zircon data 838 Supplementary Item 7: Lu-Hf zircon data 839 Supplementary Item 8: additional U-Pb and Hf data from the Hammamat Formation 840 Supplementary Item 9: Cenozoic zircon age calculations. 841 Supplementary Item 10: U-Pb rutile data 842 Supplementary Item 11: Ar-Ar mica and plagioclase data 843 Supplementary Item 12: Tracer isotope data. References 844 845 Abdeen, M.M., Greiling, R.O., 2005. A quantitative structural study of Late Pan-African compresssional deformation in the Central Eastern Desert (Egypt) during 846 847 Gondwana Assembly. Gondwana Research, 8: 457-471. Abdelsalam, M.G., Gao, S.S., Liégeois, J.-P., 2011. Upper mantle structure of the Saharan 848 849 Metacraton. Journal of African earth sciences, 60(5): 328-336.

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