U-Pb and Lu-Hf systematics of detrital zircon as a sedimentary provenance indicator

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ACKNOWLEDGEMENTS II
PREFACE III
CHOICE OF RESEARCH TARGETS IV
LIST OF PAPERS VI

INTRODUCTION 1

GEOLOGICAL SETTING 1
OSLO RIFT 1
SOUTH AFRICA 4
GEOCHRONOLOGY 5
THE LU-HF ISOTOPE SYSTEM IN ZIRCON 7
PROVENANCE STUDIES 8
NUMBER OF DETRITAL ZIRCON TO ANALYSE? 9
GRAPHICAL AND STATISTICAL TREATMENT OF DETRITAL ZIRCON DATA 11
CHOICE OF ANALYTICAL METHOD 16

INTRODUCTION TO THE PAPERS, FINDINGS AND CONCLUSIONS 19

PAPER I 19
PAPER II 20
PAPER III 21
PAPER IV 21
PAPER V 22
GENERAL CONCLUSIONS 23

REFERENCES 25

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Preface

This thesis is focused on the detrital zircon provenance *method*. The use of detrital zircon ages as a provenance indicator was made possible by the development of microbeam methods for uranium-lead isotope analysis (Secondary Ion Mass Spectrometry and Laser Ablation Inductively Coupled Mass Spectrometry) in the 1980s and early 1990s (e.g. Froude et al., 1983; Compston et al., 1984; Feng et al., 1993; Fryer et al., 1993; Hirata and Nesbitt, 1995), and has increased dramatically since then. At present (January 2017) a Google search on the combined terms "detrital zircon" and "U-Pb age" gives more than 42000 hits, suggesting that this is now a routine method in many research communities worldwide. Data obtained from the method are the basis of far-reaching conclusions regarding palaeogeography, oil well correlation, basin analysis etc. While there has recently been more emphasis on the quantitative treatment of detrital zircon data there are still some underlying assumptions inherent to the method, underlying all data interpretation, which needs questioning:

- Does "source to sink" work? i.e. can the detrital zircon population of a sediment or sedimentary rock always be tied back to its immediate precursor host?
- Are the isotopic signatures of potential source regions significantly distinct for them to be discernible in a detrital zircon data set?

The goal of this study is to present new detrital zircon data – mainly from South Africa, but also from the Oslo Region, Norway – and through this data test some of the underlying assumptions of the detrital zircon method, as well as to provide some recommendations on the graphical and statistical representation, and how to best treat and interpret detrital zircon data.

Since the main concern of this thesis is the detrital zircon method itself, the introduction includes a brief summary of the principles of the method – geochronology, LA-ICPMS, data interpretation etc., as well as a short summary of the geological background. For more thorough geological background on the various study areas the reader is referred to the individual papers included.

Five papers, of which four are published and one is prepared for journal submission, are included in the thesis. Each paper focuses on a different aspect of the detrital zircon method. The papers are presented chronologically after the introduction and are cross-referenced in the introduction with roman numerals I through V.

Data from all included papers (as well as supplementary material) are available from http://folk.uio.no/magnukr/thesis-data

Choice of research targets

Questions fundamental to the usefulness of the detrital zircon method such as "*How important has sedimentary recycling been for the accumulation of detrital zircon in a sedimentary basin?"* and *"Are potential source regions actually distinguishable?"* are best answered by targeting well-preserved sedimentary successions for which stratigraphy, tectonic setting and depositional environment are thoroughly know.

Geologically the Oslo Region is comprised of the on-shore half-graben segments of the Oslo Rift (Larsen et al., 2008). In this 10 000 km^2 area centred around the city of Oslo sedimentary successions of Cambrian-Silurian age, as well as rift-related, volcanic and sedimentary

successions of Late Carboniferous-Permian age are preserved (Larsen et al., 2008; Bruton et al., 2010) making the Oslo Region the youngest on-shore sedimentary basin in Fennoscandia. Previous research suggests that sedimentary recycling have taken place in Fennoscandia (Andersen et al., 2011). The Oslo Region with its Cambrian to Permian sedimentary history is therefore an excellent natural laboratory to test the extent of sedimentary recycling.

South Africa has a prolonged geological history, with preserved rocks from Archaean to Recent age. It has been part of several supercontinental cycles – notably Rodinia and Gondwana (e.g. Meert, 2003; Li et al., 2008). South Africa has particularly well-preserved Neoproterozoic to Triassic sedimentary successions which formed in a Gondwana setting (Catuneanu et al., 2005; Shone and Booth, 2005; Gresse et al., 2006). It has previously been suggested that there has been transportation of detritus to southern Africa and adjacent parts of Gondwana from its supercontinental neighbour Central Antarctica during the Triassic period (Veevers and Saeed, 2007). The question of the distinguishability of a potential source region, such as Central Antarctica, from any other source areas which have been adjacent parts of a supercontinent does, however, remain. With its prolonged sedimentary and supercontinental history South Africa proves an ideal candidate to test the extent of sedimentary recycling as well as the uniqueness of potential source terranes.

List of papers

Paper I

Kristoffersen, M., Andersen, T., Andresen, A., 2014. U–Pb age and Lu–Hf signatures of detrital zircon from Palaeozoic sandstones in the Oslo Rift, Norway. Geological Magazine 151, 816–829. https://dx.doi.org/10.1017/S0016756813000885

This paper is a continuation of my M.Sc. thesis. In the thesis entitled *Provenance of the Asker Group, Oslo Rift, Norway – A detrital zircon U-Pb and Lu-Hf study*, I analysed detrital zircon from the Carboniferous-Permian, Oslo Rift-related Asker Group sedimentary succession and found that sedimentary recycling was the likely cause of its age and ε_{Hf} signature. To test the extent of the recycling this work was expanded to include detrital zircon analyses from the latest Ordovician Langøyene Formation (Owen, 1981) as well as several samples, covering most of the succession, from the Silurian Ringerike Group (e.g. Davies et al., 2005a) which are both preserved in the Oslo Region.

Paper II

Andersen, T., Kristoffersen, M., Elburg, M.A., 2016. How far can we trust provenance and crustal evolution information from detrital zircons? A South African case study. Gondwana Research 34, 129–148. https://dx.doi.org/10.1016/j.gr.2016.03.003

Paper III

Kristoffersen, M., Andersen, T., Elburg, M.A., Watkeys, M.K., 2016. Detrital zircon in a supercontinental setting: locally derived and far-transported components in the Ordovician Natal Group, South Africa. Journal of the Geological Society, London. 173, 203–215. https://dx.doi.org/10.1144/jgs2015-012

It should be noted that U-Pb data from Natal Group detrital zircon have also been published by Vorster et al. (2016). Although late Mesoproterozoic and late Neoproterozoic to early Palaeozoic zircon fractions are found in both Paper III and Vorster et al. (2016), the distribution patterns differ. As discussed in Paper II this difference is likely caused by the abnormally large uncertainties reported by Vorster et al. (2016) masking significant lead loss. The Vorster et al. (2016) data is therefore not discussed further in this thesis.

Paper IV

Andersen, T., Kristoffersen, M., Elburg, M.A. Visualizing, interpreting and comparing detrital zircon age and Hf isotope data in basin analysis - a graphical approach. Basin Research (2017). https://dx.doi.org/10.1111/bre.12245

Paper V

Kristoffersen, M., Andersen, T., Elburg, M. A., Hicks, N. Provenance of the Mesoarchaean Pongola Supergroup: evidence from discordant and concordant detrital zircon. *Prepared for submission to Journal of African Earth Sciences.*

Software

detzrcr: An R-package for comparing detrital zircon samples. Available at CRAN (https://cran.r-project.org/package=detzrcr) or
github (https://github.com/magnuskristoffersen/detzrcr) (Paper IV).

Introduction

Geological setting

Oslo Rift

Geologically the Oslo Region (Figure 1) comprises the onshore half-graben segments of the Oslo Rift (e.g. Larsen et al., 2008). It covers an area of c. 10 000 km^2, is c. 40–70 km wide and extends 115 km north and south of the city of Oslo (Bruton et al., 2010). The area is host to a preserved pre-rift Lower Palaeozoic succession as well as rift-related Palaeozoic sedimentary and magmatic rocks (Figure 2). An increased influx of siliciclastic material to the Oslo Region recorded in Middle Ordovician (Hansen, 2008; e.g. Candela and Hansen, 2010) and in the latest Ordovician period (Brenchley et al., 1979; Brenchley and Newall, 1980) has been interpreted to reflect sedimentary response to the growing Caledonian mountain chain to the NW of the Oslo Region (Bjørlykke, 1974; Bruton et al., 2010). During the Early Silurian period marine conditions prevailed with the deposition of the carbonates of the Steinsfjorden Formation (Figure 2). A transition to non-marine and red-bed facies occurred at or just below the Wenlock–Ludlow boundary (Bruton et al., 2010). The Old Red Sandstone sediments of the Late Silurian – earliest(?) Devonian Ringerike Group (Figure 2) are found discontinuously throughout the Oslo Region, and were deposited in the foreland basin to the rising Caledonian mountain range in the northwest (Worsley et al., 1983; Davies et al., 2005b). The Carboniferous Asker Group was deposited during the early phases of the development of the Oslo Rift (Larsen et al., 2008).

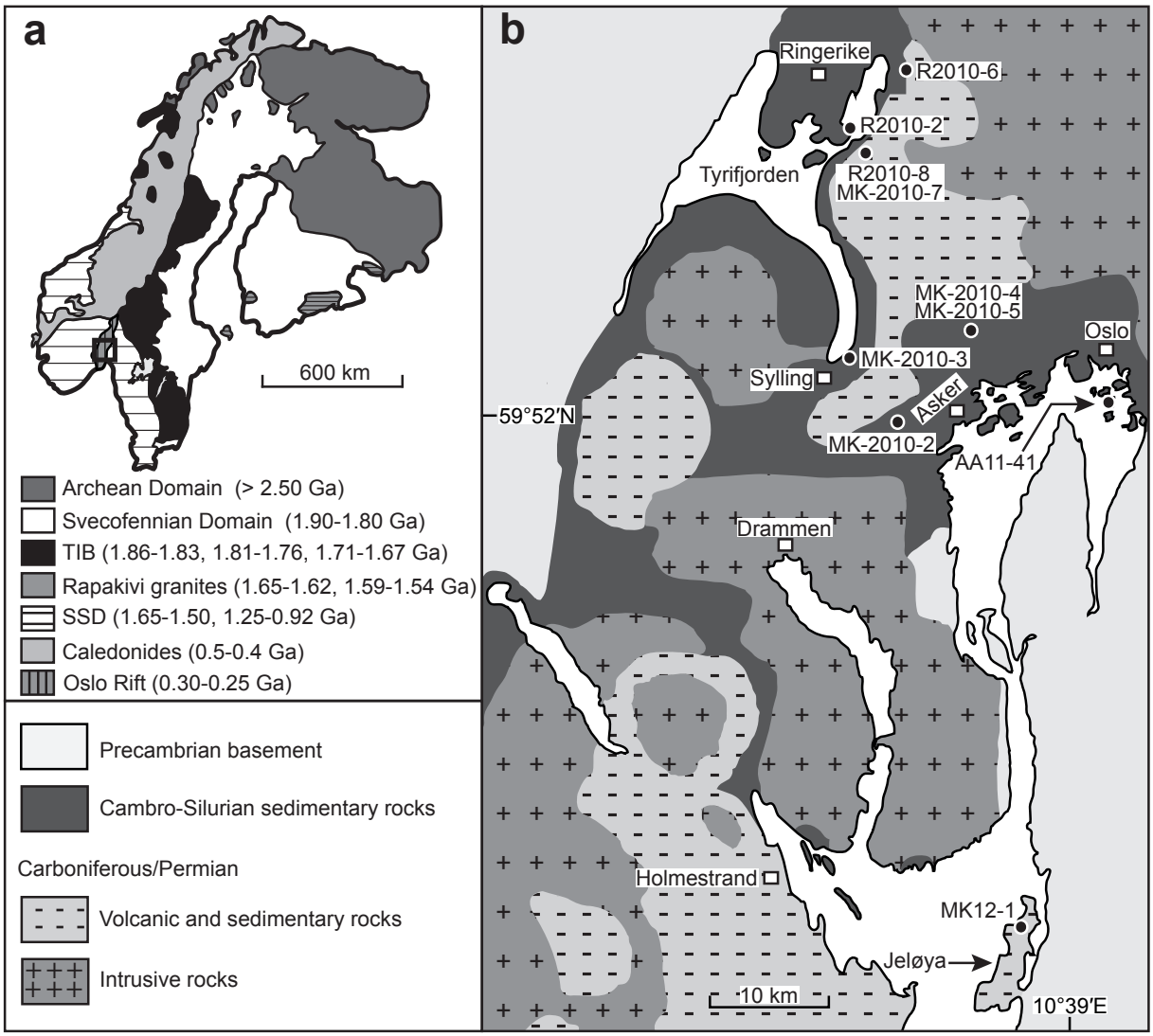

Figure 1. From Paper I. Overview of main crustal domains in Fennoscandia (modified after Gaál and Gorbatchev, 1987; Högdahl et al., 2004). The black box indicates the approximate position of (b). TIB – Transscandinavian Igneous Belt; SSD – SW Scandinavian Domain. (b) Simplified geological map of the central Oslo Graben (modified after Larsen et al., 2008) with Paper I sample locations indicated.

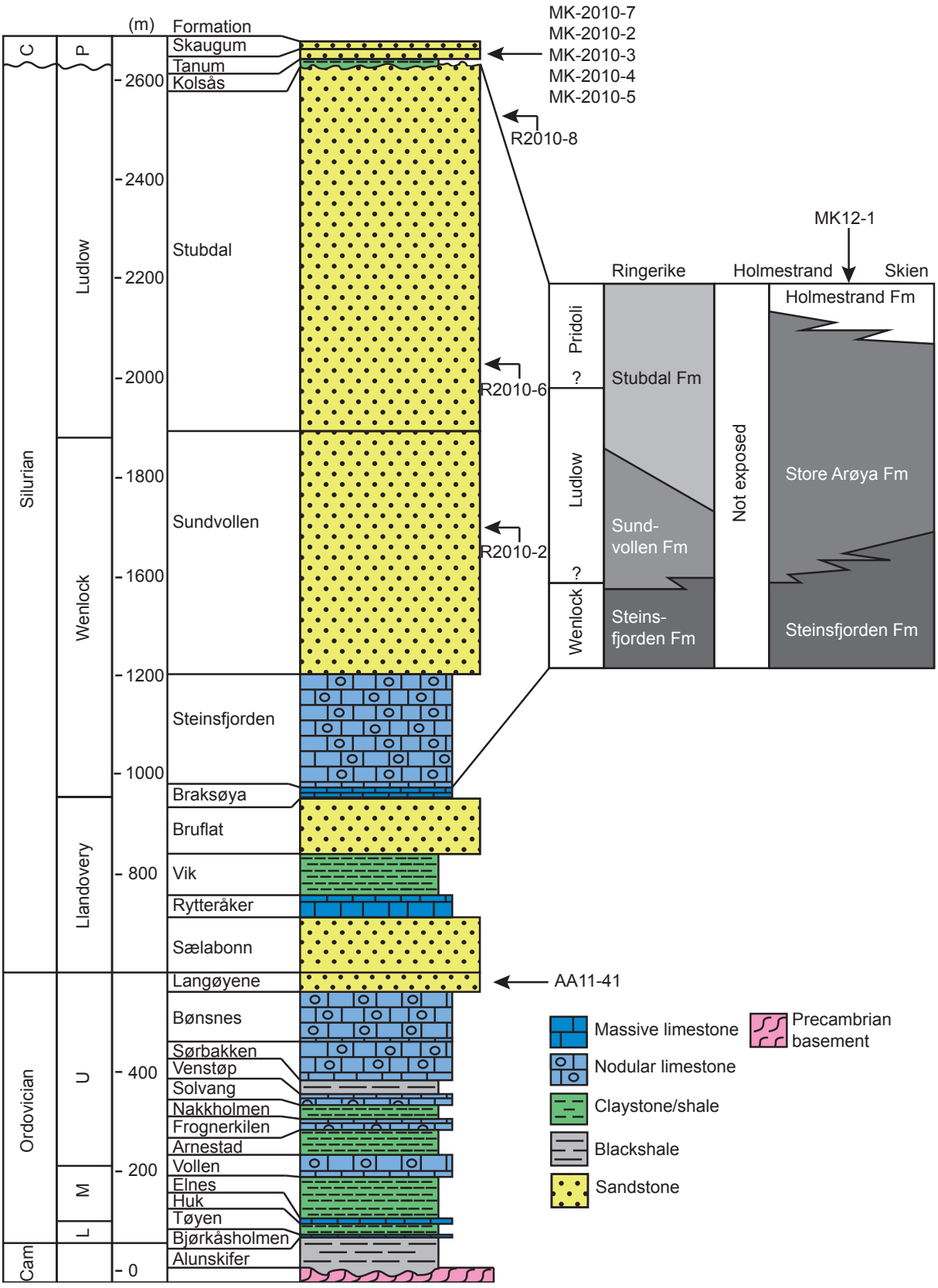

Figure 2. From Paper I. Simplified lithostratigraphic column of the Oslo Graben at Ringerike (modified after Henningsmoen, 1978; B. T. Larsen & S. Olaussen, unpub. field guide, The Oslo Region: a Study in Classical Palaeozoic Geology, Norsk Geologisk Forening, 2005), with approximate Paper I sample positions indicated. Inset shows the idealized stratigraphy of

the Ringerike Group and the Steinsfjorden Formation from Ringerike (Figure 1) in the northern part of the central Oslo Graben to Skien in the southern Oslo Graben (after Davies et al., 2005a). Cam – Cambrian; L – Lower; M – Middle; U – Upper; C – Carboniferous; P – Pennsylvanian; (m) – metres above the Precambrian basement.

South Africa

The geology of southern Africa is dominated by the Archaean Kaapvaal Craton, which preserves a record of *c*. 1000 myr of geological history spanning from *c*. 3600 to *c*. 2700 Ma (Hunter et al., 2006). It comprises largely granitoid rocks with a tonalite– trondhjemite–granodiorite composition (Hunter et al., 2006; Robb et al., 2006), infolded greenstone belts and their remnants, and volcano-sedimentary cover sequences (Hunter et al., 2006) such as the Pongola Supergroup (e.g. Gold, 2006), the Dominion Group (e.g. Marsh, 2006), and the Witwatersrand (e.g. McCarthy, 2006) and Ventersdorp Supergroups (e.g. van der Westhuizen et al., 2006). To the north the Kaapvaal Craton borders the Neoarchaean Limpopo Belt while at its southern and western margin it is bounded by the Namaqua–Natal Province (Cornell et al., 2006; Kramers et al., 2006). The Namaqua–Natal Province is a Mesoproterozoic orogenic belt related to the amalgamation of the supercontinent Rodinia (e.g. Cornell et al., 2006). It forms a continuous belt from the Namaqua Sector in southern Namibia and NW South Africa (Northern Cape province) to the Natal Sector in eastern South Africa (KwaZulu- Natal province), with the between-lying area covered by the Carboniferous to Triassic Karoo Supergroup (Cornell et al., 2006). Its southern boundary is formed by the Neoproterozoic to early Cambrian Saldania Belt (e.g. Cornell *et al*. 2006). From the end of the Pan-African orogeny in late Neoproterozoic time southern Africa remained within a stable supercontinental setting, Gondwana (Figure 3), until its breakup in Jurassic time (e.g. Torsvik and Cocks, 2013). In this setting continental cover sequences were deposited, ranging in depositional age from Neoproterozoic to Triassic. Deposition in the Cape basin lasted from

the Ordovician to the Early Carboniferous Period (Tankard et al., 2009); the Ordovician Natal Group is contemporaneous with at least some of this (Cape Supergroup) succession (Shone and Booth, 2005; Tankard et al., 2009).

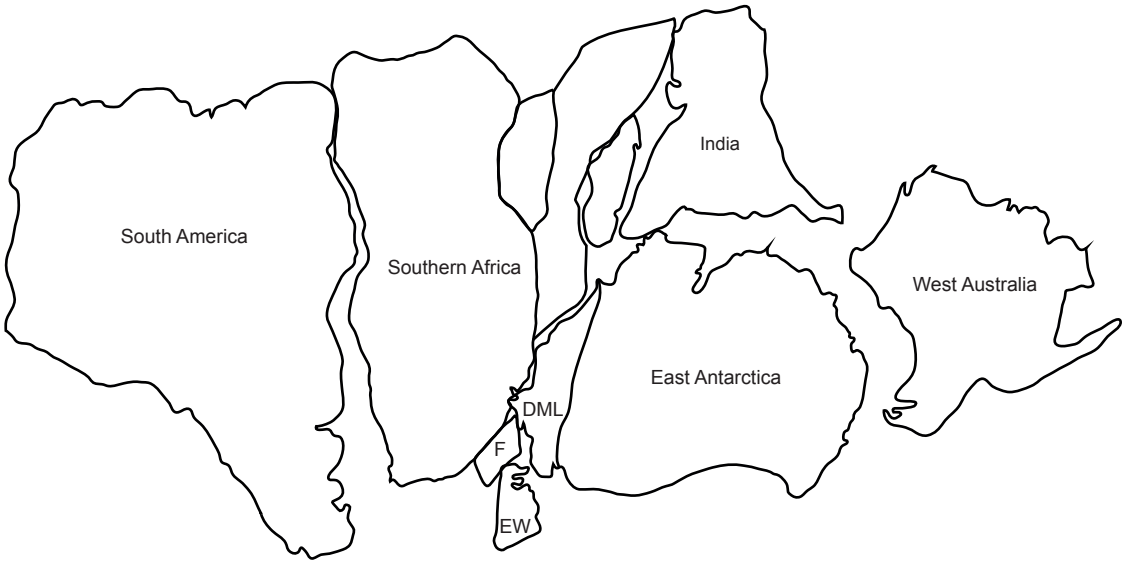

Figure 3. Southern Gondwana at 480 Ma (after Torsvik and Cocks, 2013). From Paper III. DML: Dronning Maud Land; F: Falkland Island; EW: Ellsworth-Whitmore Mountains.

Geochronology

Radiometric dating techniques are based on the principle that a radioactive parent nuclide, with known decay constant (λ), decays to a stable daughter nuclide either directly or through a series of intermediate (radioactive) daughter nuclides. For these dating techniques to function – i.e. the dating technique will yield a true age for the analysed mineral – the mineral dated should incorporate the parent nuclide during crystallization while excluding the daughter nuclide and the mineral must remain a closed system –i.e. the mineral has not lost nor gained any parent or daughter nuclide through any other means than radioactive decay – from the time of crystallization to the time of dating (e.g. Faure and Mensing, 2005). Under these conditions the entirety of daughter nuclide measured during analysis is radiogenic and the daughter to parent ratio will produce a true age for the dated mineral.

The uranium-lead isotope system is an oft used geochronometer which can yield highly precise ages using several methods: i.e. isotope-dilution thermal ionization mass spectrometry (ID-TIMS; e.g. Parrish and Noble, 2003), secondary ion mass spectrometry (SIMS; e.g. Ireland and Williams, 2003) and laser-ablation inductively coupled plasma mass spectrometry (LA-ICPMS; e.g. Košler and Sylvester, 2003). The U-Pb isotope system has the advantage that it contains two sub-systems – ^{238}U-^{206}Pb and ^{235}U-^{207}Pb – which behave chemically identical but decays at different rates (Table 1).

Table 1: Decay constants and half-lives of the U-Pb isotope system (Steiger and Jäger, 1977).

Isotope system	Decay constant (y^{-1})	Half-life (y)
^{238}U-^{206}Pb	1.55125×10^{-10} (λ_8)	4.468×10^9
^{235}U-^{207}Pb	9.8485×10^{-10} (λ_5)	0.7038×10^9

The equations used for the calculation of an U-Pb age are as follows (e.g. Faure and Mensing, 2005):

$$\frac{^{206}Pb}{^{238}U} = e^{\lambda_8 t} - 1 \quad (1)$$

$$\frac{^{207}Pb}{^{235}U} = e^{\lambda_5 t} - 1 \quad (2)$$

which can easily be solved for the age (t). Equations (1) and (2) can be also be combined to yield (e.g. Faure and Mensing, 2005):

$$\frac{^{207}Pb}{^{206}Pb} = \frac{^{235}U(e^{\lambda_5 t} - 1)}{^{238}U(e^{\lambda_8 t} - 1)} \quad (3)$$

Equation (3) can then be solved, iteratively or through interpolation, for *t* (using the constant $^{235}U/^{238}U = 1/137.88$ (Steiger and Jäger, 1977; note that a new $^{235}U/^{238}U = 1/137.818$ have recently been proposed (Hiess et al., 2012)), producing what is known as the ^{207}Pb-^{206}Pb age. The U-Pb isotope system thus yield three ages – the ^{206}Pb-^{238}U age, the ^{207}Pb-^{235}U age and the ^{207}Pb-^{206}Pb age. If the analysed mineral has remained a closed system all three ages will conform (i.e. the data are concordant) and if the system has been disturbed the dates will differ (i.e. the data are discordant). Because of the difference in decay constants for the two U-Pb decay systems the ^{206}Pb-^{238}U age will yield lowest uncertainty for young data while the ^{207}Pb-^{206}Pb age will yield the lowest uncertainty for older ages. There is no well-defined switch-over for at which age it is appropriate to use one age over the other, but a switch from the ^{206}Pb-^{238}U age to the ^{207}Pb-^{206} for dates older than 600-1000 Ma is in common use, as is using the age with the lowest uncertainty.

The Lu-Hf isotope system in zircon

Lutetium and hafnium are greatly fractionated during magma generation, because Hf is more incompatible compared to Lu (Kinny and Maas, 2003). This leads to a Hf-depletion of the mantle while the crust is enriched. It then follows that the Hf isotopic composition of the mantle and the crust have, over time, differentiated themselves from bulk unfractionated earth (Kinny and Maas, 2003). By comparing the Hf isotopic composition of an analysed sample to chondritic values (CHUR; used as a proxy for bulk unfractionated earth), expressed as

$$\varepsilon_{Hf} = \left[\frac{\left(\frac{^{176}Hf}{^{177}Hf}\right)_{sample}}{\left(\frac{^{176}Hf}{^{177}Hf}\right)_{CHUR}} - 1 \right] * 10^4,$$

the environment in which the sample formed can be ascertained, i.e. positive ε_{Hf}-values points to a mantle source, while negative ε_{Hf}-values points to a crustal source, values close to chondritic values can be indicative of a mixed source (Kinny and Maas, 2003).

Zircon always incorporates some hafnium, typically 0.5-2.0 wt% HfO_2 (Hoskin and Schaltegger, 2003), into its structure during crystallization (Kinny and Maas, 2003). ^{176}Hf is the radiogenic daughter of ^{176}Lu ($\lambda_{Lu} = 1.867 * 10^{-11}$; Söderlund et al., 2004), but since the zircon structure is less favourable for the incorporation of lutetium compared to hafnium (i.e. the Lu/Hf ratio is very low) the Lu-Hf isotope system in zircon cannot be used as a geochronometer. Because of this low Lu/Hf ratio the in-situ accumulation of ^{176}Hf will however be negligible, and the age-corrected ^{176}Hf (radiogenic) to ^{177}Hf (stable) ratio relative to the chondritic uniform reservoir (CHUR) will serve as a geochemical tracer for the nature of the melt from which the zircon formed (Kinny and Maas, 2003).

Provenance studies

In any provenance study of sediments or sedimentary rocks, no matter the method utilized – heavy minerals, palaeocurrents etc. – the aim is to unravel whence the detritus originated. In a detrital zircon provenance study a sub-set of zircon from a sediment or sedimentary rock are dated (through the U-Pb isotope system) and the distribution of ages are compared to potential source areas, in some cases additional provenance tracers – Lu-Hf, trace elements etc. – are also used.

Detrital zircon is an ideal candidate for provenance studies:

- it is very common, occurring in granitoid rocks and subsequently (through erosion) in sediments and sedimentary rocks
- it incorporates uranium, but very little of the uranium-daughter lead, in its structure during crystallisation (Kinny and Maas, 2003)
- it is highly durable both physically and chemically, and can remain a closed system in high-grade metamorphic environments (Williams, 2001; Hawkesworth and Kemp, 2006)
- additional isotope systems can be utilized, such as lutetium-hafnium, oxygen

There are, however, several downsides to the use of detrital zircon in provenance studies:

- zircon is mainly found in granitoid rocks which means that any basaltic source rocks will likely be missed
- its refractory nature makes zircon able to survive multiple episodes of sedimentary recycling, i.e. detrital zircon can only be traced back to its proto-source (the rock in which the zircon crystallized) and not the immediate source which is the goal of most basin studies

Number of detrital zircon to analyse?

In principle the detrital zircon method is a statistical method where the goal is to identify the different source components that have contributed to the total zircon population of the analysed sample and, ideally, quantify which components have made a more significant contribution than the others. Fedo et al. (2003) made a distinction between two different approaches to detrital zircon studies – namely qualitative and quantitative analysis. In a qualitative analysis an attempt is made to find age representations of source components in

the total population through analysing selected – based on morphology, colour etc. – grains disregarding their overall abundance (Fedo et al., 2003). In a quantitative analysis the goal is that the analysed data set is a one-to-one representation of the total detrital zircon population. Using the latter approach no heed is given to morphology, colour etc. during grain selection and the grain selection should be completely random (Fedo et al., 2003).

The number of grains to analyse for the data set to be a statistically significant representation of the zircon population has been discussed since the very beginning of detrital zircon provenance studies (i.e. Dodson et al., 1988). Dodson et al. (1988) argued, using binominal statistics, that at least 60 zircons must be analysed to ensure that the chance of missing one specific fraction is less than 5% if this fraction is less than 5% of the total population, i.e.

$$p = (1 - f)^k \tag{4}$$

where p is the probability, f is the fraction and k is the total number of zircon. Vermeesch (2004) suggested that 117 grains are needed to be 95% certain that no fraction smaller than 5% is missed. This critical limit number (n) was however based on a scenario in which 20 equally abundant, 5%, fractions. Andersen (2005) showed that in real world cases n must be very large ($n>>100$) for the analysed data set to be a true representation of the total detrital zircon population of the sample – i.e. for the analysis to be truly quantitative (Fedo et al., 2003). As a workable compromise he suggested that at least 35-70 randomly selected grains should be analysed along with selected fractions (based on morphology etc.). This approach has been used with some success (e.g. Pokki et al., 2013), but does not seem to have gained wide acceptance. While Andersen (2005) is frequently cited in detrital zircon literature there

is a tacit agreement in the community that at least 117 grains should be analysed for the data-set to be representative of the population.

Graphical and statistical treatment of detrital zircon data

Detrital zircon data should comprise statistically significant numbers of individual analyses. To visualize and interpret these data requires a method by which it can be graphically represented in a meaningful way. This can be done by plotting the empirical (cumulative) distribution function (ECDF; Wasserman, 2006) or its first derivative the probability density function (PDF; Wasserman, 2006). Since the infancy of detrital zircon geochronology (e.g. Dodson et al., 1988) the preferred method of comparing detrital zircon samples has been to visually compare peaks in the PDF-plot of U-Pb age data (Ireland et al., 1998; e.g. DeCelles et al., 2007). In detrital zircon studies the PDF has traditionally been estimated (Figure 4)

using what has been termed the probability density distribution (PDD; e.g. Sircombe, 2004).

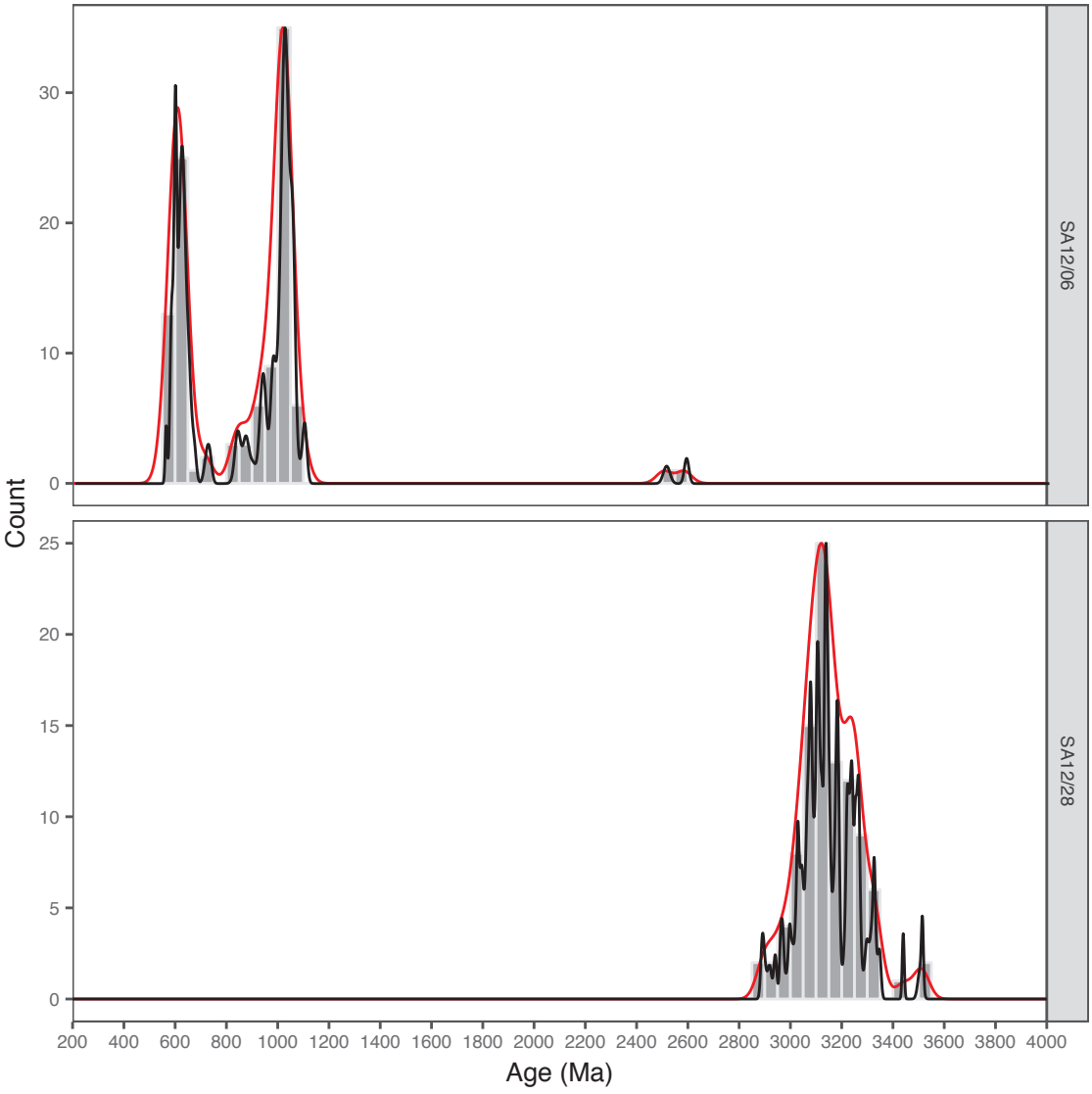

Figure 4. Combined PDD (black) and KDE (red; bandwidth=30) of the lowermost and uppermost parts of the Natal Group succession (from Paper III).

Using a Gaussian kernel the PDD is defined as

$$f(t) = \frac{1}{N}\sum_{i=1}^{N} \frac{e^{\frac{-(t-x_i)^2}{2\sigma^2}}}{\sqrt{2{\sigma_i}^2\pi}} \qquad (5)$$

where t is the range over which the density estimate is calculated, N is the number of analyses, x is the age and σ is the (1 sigma) uncertainty associated with a particular age. In

essence the PDD is calculated as follows: *t* is chosen as the total age range of the U-Pb data with constant (e.g. 1 Ma) steps, then the normal density of *t* is calculated with all pairs of U-Pb age and associated uncertainty as the mean and standard deviation, respectively, and these are then summed producing the density associated with each *t*. Since the PDD is a combination of several "local" probability density estimates it is a type of Gaussian finite mixture distribution (e.g. Forbes et al., 2011) which produces a pointwise density estimate. This is easily seen by setting sigma constant in equation (5) which reduces it to the formula for the fixed-bandwidth Gaussian kernel density estimate (KDE; e.g. Wasserman, 2006). Different kernels than the Gaussian (Epanechnikov, rectangular, triangular etc.) can be used for kernel density estimation, but as choice of kernel has minor effect on the estimated probability density (Vermeesch, 2012) only the normal kernel is in common use. The Gaussian kernel has the added advantage that it is continuous and simple to calculate as opposed to other kernels. Both PDD and KDE are valid probability density estimates, but the PDD has the disadvantage that it will produce narrow peaks for high precision data (i.e. low σ), making visual comparison of samples difficult. This will be especially pronounced in young material where the high precision ^{206}Pb-^{238}U age is generally preferred. To alleviate this problem some workers have used a fixed-bandwidth KDE which makes visual comparison between samples easier, but this method as the disadvantage that it disregards the uncertainty inherent to geochronological data. This can to some extent be corrected by calculating confidence bands for the probability density. But as bias reduction significantly complicates the calculation of confidence bands for KDEs (e.g. Fiorio, 2004) a preferable option is to plot the U-Pb age data as empirical cumulative density functions (ECDFs; Figure 5) for which confidence bands are easily calculated using the Dvoretzky-Kiefer-Wolfowitz

inequality (Dvoretzky et al., 1956; Wasserman, 2006).

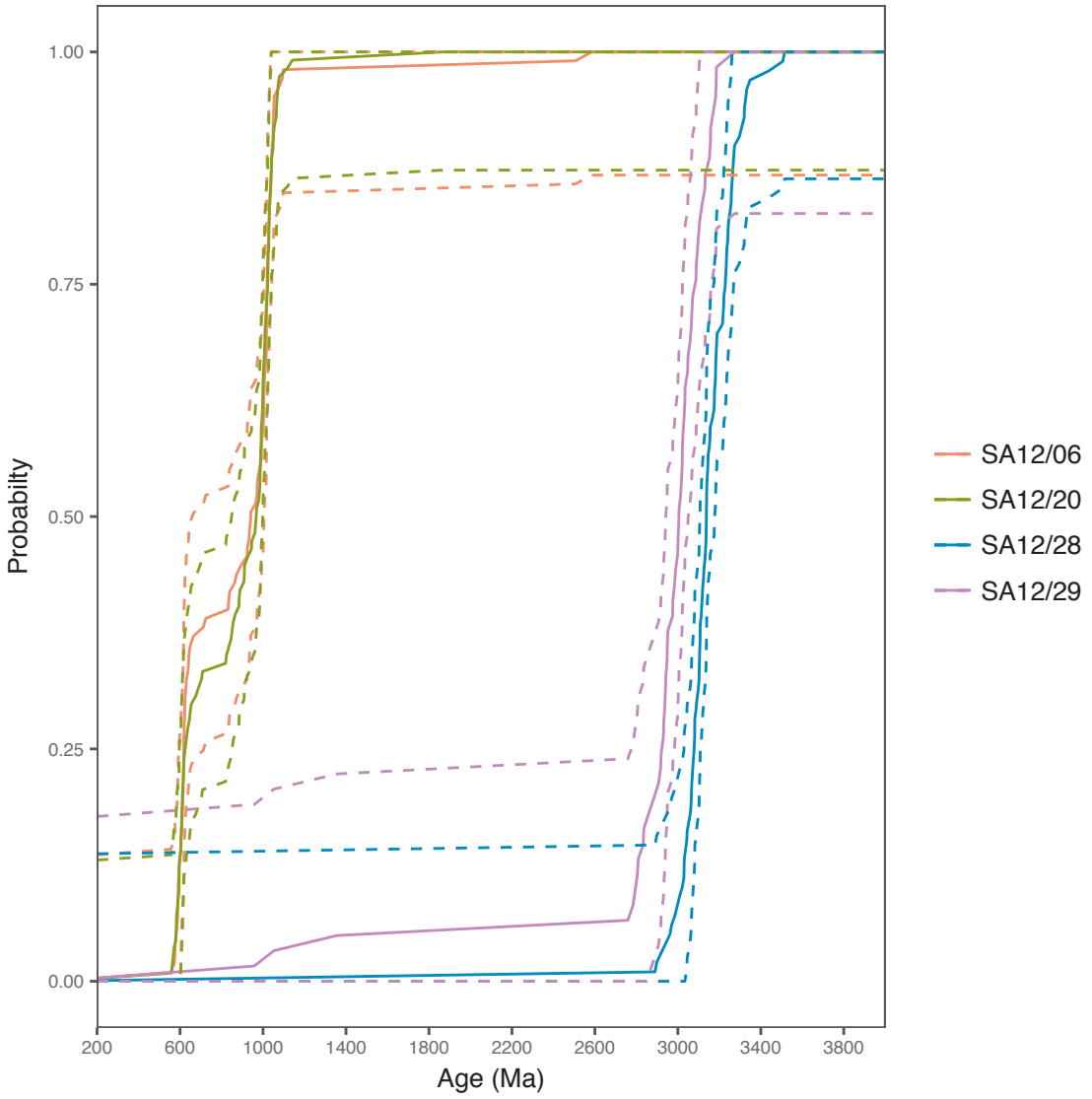

Figure 5. Plot of the empirical cumulative distribution functions with associated 95% confidence bands (Dvoretzky et al., 1956) of the lowermost and uppermost part of the Natal Group succession (from Paper III).

Visual comparison of ECDFs with associated confidence bands can then give a good indication on the similarity/dissimilarity of the samples and if apparent dissimilarity is real or just an artefact of sampling bias.

The primary mode of applying statistical tests to univariate detrital zircon data has been to compare samples by the pair-wise Kolmogorov-Smirnov test (KS-test; e.g. Berry et al., 2001; Beranek et al., 2013), in some cases applying a ”smoothing”, analogous to PDD smoothing, to the calculated ECDFs (e.g. Anfinson et al., 2012). In a pair-wise KS-test the maximum

distance in the y-direction between two ECDFs (D) is found and a probability (p-value) is calculated for D. If the p-value is larger than some predetermined threshold α (typically α=0.05 in detrital zircon studies) it is deemed unlikely that the sample pair come from different sources. It should be pointed out that the KS-test does not tell you if two samples are identical, but how unlikely it is that the samples are from different sources. The KS-test is, however, not a good similarity measure between detrital zircon samples, largely because of its sensitivity to sample size (Vermeesch, 2013). To rectify this issue a new similarity measure – similar to previously published measures (Gehrels, 2000; Amidon et al., 2005a, 2005b) – was recently introduced by Satkoski et al. (2013):

$$L = 1 - (\sum_{i=1}^{N} |a_i - b_i|/2) \quad (3)$$

where a and b are the estimated probability function of the two samples being compared and N is the number of ages in the range the PDF was calculated (the samples must be unitized). This likeness parameter is not as dependent on the number of analyses, giving L=0.61 for 50 grain subpopulations and L=0.79 for 150 grain subpopulations (Satkoski et al., 2013). Another recently introduced measure of similarity is the O-parameter of Andersen et al. (2016) which is defined as the fraction of the probability domain of the ECDFs over which the confidence bands of two ECDFs overlap. 1-O is then a measure of similarity where 1-O=0 means that the samples are indistinguishable within their confidence intervals.

Whereas detrital zircon studies have traditionally focused on U-Pb age data there has been an increase in studies combining age data with Lu-Hf isotope analysis. This combined approach to detrital zircon provenance studies is especially useful in areas where possible source areas

have entirely overlapping U-Pb age signatures but vastly different Lu-Hf signatures as for instance the North China and South China Cratons (Zhang et al., 2012; Zhao et al., 2013).

The most common way of comparing bivariate detrital zircon data have been to plot the data as scatterplots of either initial ε_{Hf} or initial $^{176}Hf/^{177}Hf$ vs. age, and in some cases applying contours of comparison data in the background (e.g. Andersen et al., 2011).

A two-dimensional variety of the pair-wise KS statistical similarity test have been applied to detrital zircon data (e.g. Paper III). Unfortunately, this test comes with the same caveats as the one-dimensional variety and has the added disadvantage that it is not distribution free when two or more dimensions are present, i.e. there is no unique way of sorting the multivariate data and the unreliability of the resulting statistic will be difficult to ascertain (Babu and Feigelson, 2006). The likeness parameter of Satkoski et al. (2013) can, however, be easily extended to two-dimensional data (Andersen et al., 2016) and is, as such, a good likeness measure of bivariate detrital zircon data although giving somewhat lower values compared to the univariate variety (Andersen et al., 2016; Paper III).

As the O-parameter of Andersen et al. (2016) compares pairs of ECDFs with associated 95% confidence intervals it can also be used for depleted mantle model ages, which themselves are a combination of U-Pb age and Lu-Hf data, thereby giving a measure of the similarity of bivariate detrital zircon data.

Choice of analytical method

While the highest precision U-Pb zircon data are obtained by ID-TIMS analysis (e.g. Parrish and Noble, 2003) the technique is too laborious to use for the relatively large number of zircon analyses (~100) required in a detrital zircon study. Rapid data acquisition techniques in common use in detrital zircon studies are secondary ion mass spectrometry (SIMS) and laser ablation inductively coupled mass spectrometry (LA-ICPMS). Both techniques deliver

comparable levels of accuracy and precision, but differ in mass of sample needed (SIMS requiring less) and speed of data acquisition (LA-ICPMS being faster).
A skilled LA-ICPMS operator can, during a typical 8-hour work-day, analyse ~200 zircon grains. This has made the LA-ICPMS the most commonly used data acquisition technique in detrital zircon studies.

Prior to LA-ICPMS analysis individual zircon grains have been picked from a mineral-separate, cast in epoxy and polished. This epoxy puck is then, typically, imaged – backscatter imaging or cathodoluminescence – using a scanning electron microscope. These images are used during an analyses session for the optimal placement of the laser spot – identify core-rim relationships, avoid cracks, only analyse homogenous zones etc. When the laser is fired an aerosol of the sampled material is produced (some is also ionized) and transported to the plasma region of the instrument by a carrier-gas (typically helium) (Košler and Sylvester, 2003).

The inductively coupled plasma is generated in argon which flows through the torch (Figure 6) – three concentric glass tubes in which the sample flows through the innermost tube. To produce the plasma a few electrons are fed to the argon, these start to oscillate in a radio frequency field generated by a copper coil surrounding the torch which is in turn connected to a radio frequency power generator (Košler and Sylvester, 2003). The oscillating electrons collide with Ar atoms thus ionizing the argon gas, and subsequently increasing the temperature and producing the plasma.
The inductively coupled plasma reaches a temperature of 8000-10000 K making it an excellent ionizer (Košler and Sylvester, 2003).

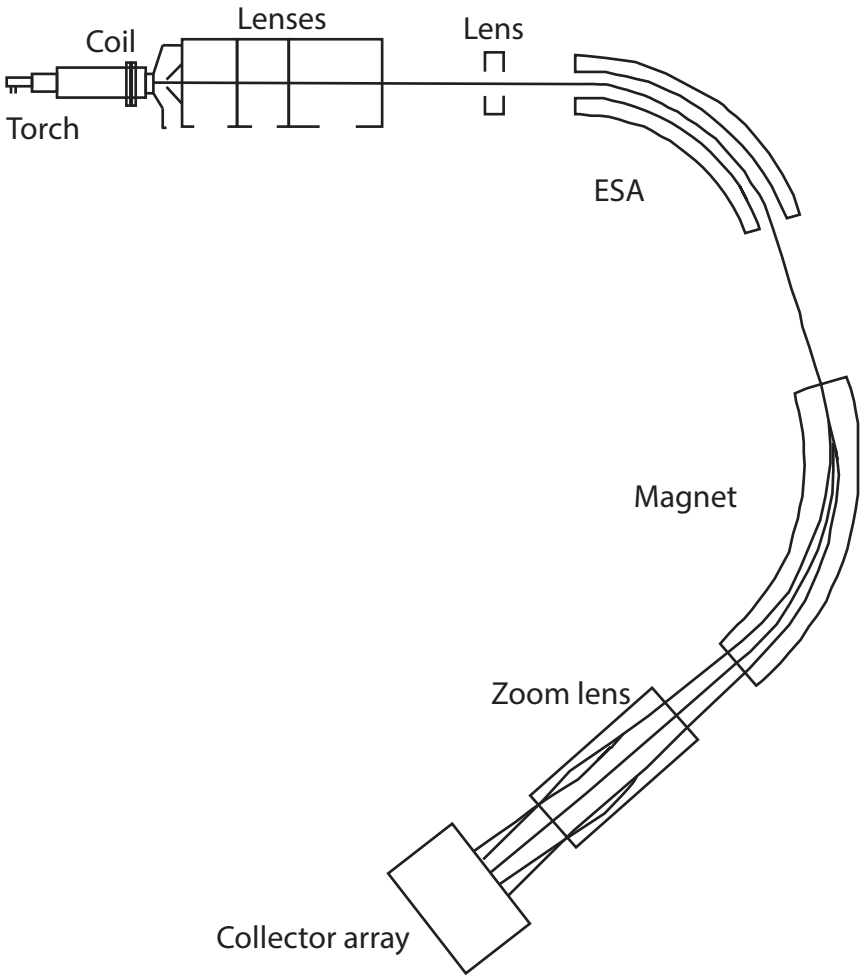

Figure 6. Simplified instrument schematic of magnetic sector ICPMS. ESA: Electrostatic analyser. After Nu Plasma HR instrument brochure.

Once ionized the sample is transferred to the analyser region of the instrument through a pair of cones (sampler and skimmer) at the ICPMS interface before reaching the detector (Košler and Sylvester, 2003). Depending on the instrument the analyser region consists of a mass and/or kinetic energy analyser. A type of instrument commonly used in detrital zircon studies is the double focusing magnetic sector ICPMS (Figure 6) which uses a magnet and an electrostatic filter to separate the ions and provides high precision simultaneous collection of several ions which are detected in Faraday cups and/or electron multipliers (Košler and

Sylvester, 2003). The mass analyser of another commonly used instrument, the quadrupole ICPMS (Figure 7) consists of four metal rods where each two opposing rods are connected with an AC and DC potential. By applying a specific setting to these rods only ions with a specific mass to charge ratio can pass through the analyser region to the detector while other ions are drawn to the rods and are removed from the system via turbo pumps (Košler and Sylvester, 2003).

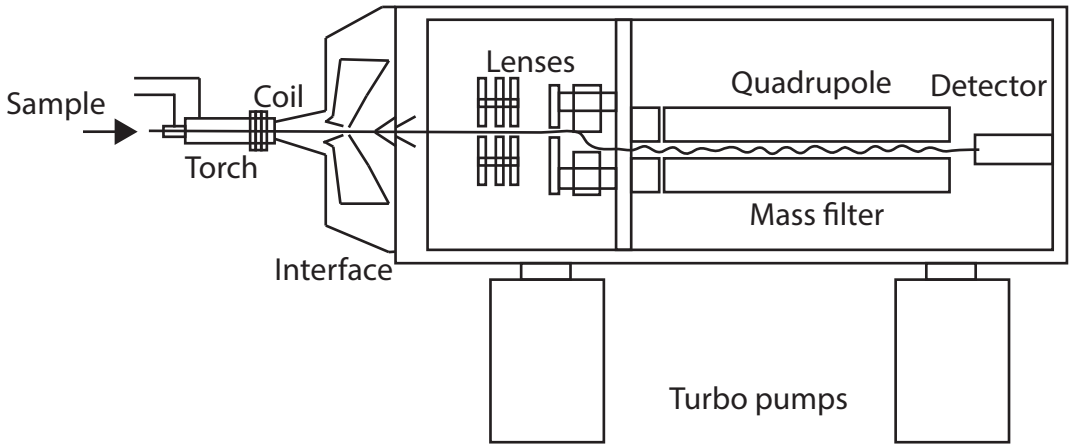

Figure 7. Quadrupole ICPMS instrument schematic (from Košler and Sylvester, 2003).

Introduction to the papers, findings and conclusions

Paper I

U–Pb age and Lu–Hf signatures of detrital zircon from Palaeozoic sandstones in the Oslo Rift, Norway

M. Kristoffersen, T. Andersen and Arild Andresen (2014)

Published in Geological Magazine 151, 816-829.

Paper I presents detrital zircon U-Pb and Lu-Hf data from sedimentary rock sequences from the Oslo Region, Norway (Figure 1). The rocks studied cover a large age range – from the

Ordovician Langøyene Formation, through the Silurian Ringerike Group to the Carboniferous, Oslo Rift-related Asker Group (Figure 1 and Figure 2). All samples have U-Pb and Lu-Hf signatures compatible with virtually every possible Fennoscandian primary zircon source. The detrital zircon isotopic signatures found are not compatible with a single source terrane but must have been sourced from a sedimentary, immediate precursor rock which itself is likely a result of sedimentary recycling. This confirms that there must have been extensive sedimentary recycling in Fennoscandia and that these cycles must, at least, have lasted from Ordovician to Carboniferous time.

Paper II

How far can we trust provenance and crustal evolution information from detrital zircons? A South African case study

T. Andersen, M. Kristoffersen and M. Elburg (2016)

Published in Gondwana Research 34, 129-148.

Paper II presents detrital zircon U-Pb and Lu-Hf data from the Cape Supergroup, the Msikaba Formation and the Karoo Supergroup, South Africa. All studied samples share large Mesoproterozoic and Neoproterozoic age fractions with overlapping ε_{Hf} signatures among the samples and a Permian age fraction is found in the uppermost Karoo Supergroup samples. Archaean zircon grains (i.e. derived from the underlying Kaapvaal Craton) are virtually absent. This data show that the Phanerozoic sedimentary rocks of South Africa record several cycles of sedimentary recycling, in which significant events in the geological history (i.e. the Kaapvaal Craton) is not recorded and only the Permian age fraction found in the uppermost Karoo Supergroup samples have any indication of (Gondwanide) provenance. In such cases detrital zircon is a recorder of sedimentary processes and not of provenance.

Paper III

Detrital zircon in a supercontinental setting: locally derived and far-transported components in the Ordovician Natal Group, South Africa

M. Kristoffersen, T. Andersen, M. Elburg and M.K. Watkeys (2016)

Published in Journal of the Geological Society, London, 173, 203-215.

This paper presents detrital zircon U-Pb and Lu-Hf data from the Ordovician Natal Group, South Africa (Figure 3). With the exclusion of the basal Ulundi Conglomerate samples which contain exclusively Archean detrital zircon and must be derived from a proximal Kaapvaal Craton source, the samples from the Natal Group have virtually identical Meso- and Neoproterozoic U-Pb signatures and Lu-Hf signatures, i.e. juvenile Mesoproterozoic material, and juvenile Mesoproterozoic material which has been reworked during the Pan-African orogeny. This signature is shared with samples from several sedimentary sequences from former Gondwana with depositional ages ranging from Neoproterozoic to Permian. It is highly unlikely that a single source could have fed samples with such a vast temporal and geographical distribution and a likelier explanation is that several different source terranes with virtually identical isotopic signatures have fed these sedimentary sequences. This is thus an example of it being impossible to ascertain a provenance history for sedimentary sequences from detrital zircon because of source terrane signature non-uniqueness.

Paper IV

Visualizing, interpreting and comparing detrital zircon age and Hf isotope data in basin analysis - a graphical approach

T. Andersen, M. Kristoffersen and M. Elburg (2017)

Basin Research, https://dx.doi.org/10.1111/bre.12245

In this paper a novel approach – upper quantile vs. lower quantile U-Pb age and Lu-Hf model age plots – of visualizing, interpreting and comparing detrital zircon data is introduced. This approach uses the 75th and 25th percentile of the empirical cumulative distribution function as basis for plotting detrital zircon data, hence reducing complex data to simple points in two-dimensional space while keeping a direct, understandable relationship to the underlying data. The paper also contributes a user-friendly, freely available, open source, software package for R which implements this method as well as several other methods in current use, for visual and statistical comparison of detrital zircon samples.

Paper V

Provenance of the Mesoarchaean Pongola Supergroup: evidence from discordant and concordant detrital zircon

M. Kristoffersen, T. Andersen, M. Elburg and N. Hicks

Prepared for submission to Journal of African Earth Sciences.

This paper presents detrital zircon U-Pb and Lu-Hf data from sedimentary rock samples covering most of the exposed Pongola Supergroup, South Africa. A large portion of the analysed zircon grains have discordant U-Pb ages, but numerical modelling shows that a recent lead-loss event is likely for most samples. It is therefore acceptable to use these zircon grains as a provenance indicator. Four samples, however, record U-Pb ages incompatible with the depositional age for the Pongola Supergroup.

The data show that while one sample most likely has been fed directly from an areally restricted source, now covered or eroded away, with a similar signature to the Ancient Gneiss

Complex, the other samples are a mix, to various degrees, of detritus from an Ancient Gneiss Complex-like source and the surrounding Eastern Domain of the Kaapvaal Craton.
The data also illustrate the need to assess discordant data before discarding (zircon with discordancy > 10% are commonly discarded in detrital zircon studies) them outright, if not there is a genuine risk of losing valuable data. Additionally, the data shows that numeric modelling of lead-loss age should not be applied indiscriminately to discordant detrital zircon data without the prior, independently determined, knowledge of the depositional ages of the host-rocks.

General conclusions

The detrital zircon provenance method is used to its advantage when there is a clearly defined primary (proto) source, or a sedimentary source with a unique isotopic signature for the analysed detritus. In many cases (e.g. Papers I and II) the innate nature of zircon, i.e. its chemical and physical durability, often seen as a great boon for the method, is actually – through sedimentary recycling – masking the relationship between the sample and its source. In these cases the detrital zircon grains are not fed from a primary, magmatic source, but are recycled from a sedimentary, immediate precursor rock which themselves are likely the result of sedimentary recycling. If there is a likely precursor rock available for analysis this problem is easily rectified, but often such a rock is not available and in areas with extensive sedimentary recycling the isotopic signatures tend to homogenise (e.g. Papers I and II), thus making a provenance identification impossible.
There is also a problem in detrital zircon studies of uniquely identifying a possible source region. This was shown to be impossible for a large region of former Gondwana where recycling and source terrane non-uniqueness led to isotopic signatures carrying no actual provenance information (Papers II and III).

With these difficulties inherent to the method in mind it is important to treat detrital zircon data from a quantitative approach. The novel method of visualizing, interpreting and comparing detrital zircon samples introduced in Paper IV, alongside a user-friendly computer implementation, will hopefully be adopted by the detrital zircon community.

If the mentioned caveats are accounted for detrital zircon can be a useful method in the geoscientists arsenal. The method is however not a panacea, and should not be treated as one.

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Paper I

U–Pb age and Lu–Hf signatures of detrital zircon from Palaeozoic sandstones in the Oslo Rift, Norway

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Paper II

How far can we trust provenance and crustal evolution information from detrital zircons? A South African case study

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Paper III

Detrital zircon in a supercontinental setting: locally derived and far-transported components in the Ordovician Natal Group, South Africa

M. Kristoffersen, T. Andersen, M. Elburg and M.K. Watkeys (2016)

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Paper IV

Visualizing, interpreting and comparing detrital zircon age and Hf isotope data in basin analysis - a graphical approach

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Paper V

Provenance of the Mesoarchaean Pongola Supergroup: evidence from discordant and concordant detrital zircon

M. Kristoffersen, T. Andersen, M. Elburg and N. Hicks

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