DANMARKS OG GRØNLANDS GEOLOGISKE UNDERSØGELSE RAPPORT 2017/50

Provenance investigations related to the Xana-1 well

Zircon and rutile provenance in Xana-1, Gita-1, Amalie-1, Svane-1 and Marsvin-1

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GEOLOGICAL SURVEY OF DENMARK AND GREENLAND
DANISH MINISTRY OF ENERGY, UTILITIES AND CLIMATE

GEUS

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Released 31.12.2022

Content

1 Introduction

The present project was performed in co-operation with INEOS and Licence 4/98 partners Danoil and Nordsøfonden. It represents a continuation of the chemo-stratigraphic study by Weibel & Jacobsen (2017) which indicated an apparent good geochemical correlation between the Gita-1 and Xana-1 wells. Some similarities to sandstone intervals in the Svane-1 well was also found, whereas poor data quality from the Amalie-1 well hindered the comparison of sandstone composition.

In the Xana-1 well, the study indicated that the uppermost sandstone unit XA4 has a different geochemical composition than the underlying units. The sandstone interval XA4 in the Xana-1 well thereby resembles the overlying Kira unit geochemically by having similar Na/Ti, Ce/Na, Na/Al, Na/K, Mg/Ca, Mn/Ca ratios. Since the similarities are based on elements from unstable minerals, it may reflect lithological similarities and not necessarily sediment source similarities. This may however be investigated in further detail by applying zircon age dating and trace element analysis.

In order to further evaluate the nature of the Upper Jurassic depositional system in the Tail End Graben it was recommended to perform provenance investigation of the sandstone intervals in the Xana-1, Gita-1, Svane-1 and Amalie-1 wells to test if these are sourced from a single common provenance area towards the north or potentially show that a local sediment source area (e.g. the Ringkøbing-Fyn High) may have contributed to the sand-rich Upper Jurassic succession in the Tail End Graben.

In addition, the study will investigate if the geochemical differences observed between the lower J62/J63 sandstone units and the overlying J64 and Kira sandstone units may be related to a change in provenance area through time.

Identifying sand provenance areas (local vs more mature northern provenance areas) will increase the understanding of the depositional system and may help to predict reservoir quality and morphology of the submarine fan system in the Xana discovery.

The aim of the present study is therefore to test if it through analysis of U/Pb ages of detrital zircon and rutile combined with mineral chemistry of these minerals is possible to identify similarities between the sandy intervals in the wells studies. Further, it is the aim to qualify statements of the provenance of the sand by comparison to data from nearby wells penetrating Jurassic sandstones in the North Sea (Figure 1).

The study includes new analysis as well as the existing zircon age data from the area of interest. Until now only the shallow marine Heno Formation sandstones has been investigated and show a remarkable uniform age spectrum, which most likely represents continued reworking of the zircons. In this study, we will include the age equivalent deep marine sandstones from the Tail End graben to investigate differences in the provenance areas for the two depositional systems. The zircons will be age dated and their geochemical fingerprint investigated in order to try to obtain a more detailed understanding.

The Rutile analysis is a new provenance method introduced to the DK North Sea by GEUS. The rationale is that Rutile is formed during metamorphic events such as the Caledonian Orogeny and record this age whereas zircons survive metamorphism and events such as the Caledonian Orogeny in the source area is not easily detected among the detrital zircons. It should therefore be possible to differentiate rutile derived from the Caledonian areas, Norway and Scotland from the crystalline areas of Norway, Sweden and Germany. In addition, Rutile shows a larger geochemical variability which may help to fingerprint the areas of origin.

1.1 Samples

Cuttings samples from Xana 1X, Gita 1x, Svane 1A, Amalie 1 and Marsvin-1 were selected (Table 1, Figure 2):

Table 1

Well	Strat	Depth (m)
Xana-1X	J64	4622 m - 4703 m
Xana-1X	J63	4747 m - 4870 m
Xana-1X	J62	5014 m - 5114 m
Gita-1X	J64	4344 m - 4402 m
Gita-1X	J63	4440 m - 4566 m
Gita-1X	J62	4640 m - 4728 m
Svane-1A		5538 m - 5605 m
Svane-1A		5780 m - 5952 m
Amalie-1		4185 m - 4600 m
Amalie-1		4850 m - 5000 m
Marsvin-1		3776 m -3830 m

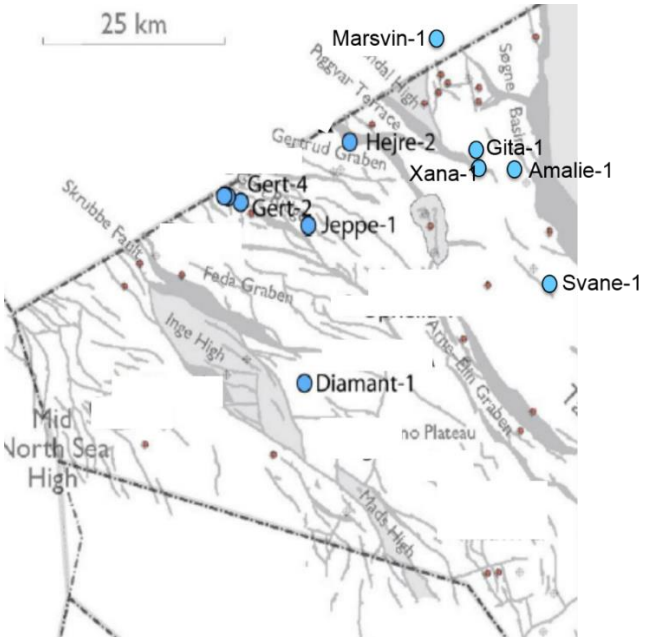


Figure 1 *Location of the wells mentioned in the report.*

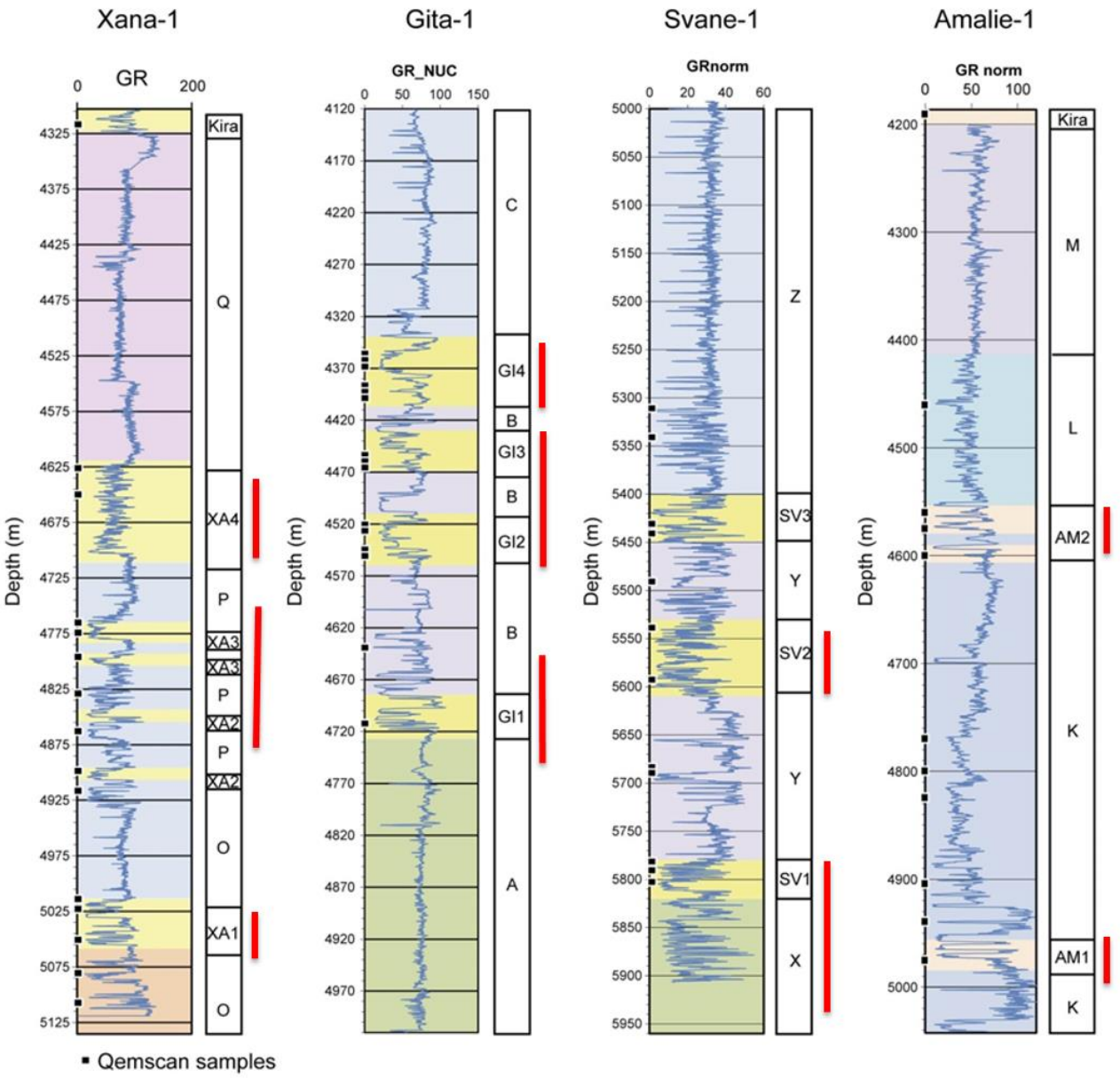


Figure 2 *Stratigraphic position of the samples analysed in Xana-1, Gita-1, Amalie-1 and Svane-1.*

1.2 Background

1.2.1 U-Pb dating of detrital zircon in Jurassic sandstones

Weibel and Knudsen (2007) conducted a study of the Heno Formation containing zircon provenance analysis of samples from Hejre-2, Gert-2, Gert-2, Jeppe-1 and Diamant-1. It was found, that the detrital zircon age spectra in the Heno Formation were rather similar being dominated by ages in the range between 1750 and 950 Ma (Figure 3) all with a bimodal distribution and prominent peaks around 1600 - 1450 Ma and 1100 – 1000 Ma and a minor population of Archean zircons with ages between ca. 3000 – 2500 Ma. However, there are some differences between the detrital zircon age spectra (Figure 3):

Ravn Member in the Gert-2 and Gert-4 wells are characterized by a fairly high proportion of younger zircons with ages in the three groups ca. 700-500 Ma (Avalonian), Ca. 470-400 Ma (Caledonian) and Ca. 350-250 Ma (Variscan). These ages are rare in the Gert Member in the Gert-2 and Gert-4 wells. The Caledonian zircons can be derived from Norway, but the Avalonian and Variscan ages have not been reported from bedrock (basement) rocks in Scandinavia suggesting that the provenance is at least partly to the South-west. The Diamant-1 well has a fairly clear Variscan peak which is in good accordance with the position of the well South of the other wells and closer to the Variscan Orogeny (Figure 3).
The bimodal distribution of the Meso- to Paleo-proterozoic zircons is seen in all the spectra (Figure 3) where the two peaks in general are approximately equally high.

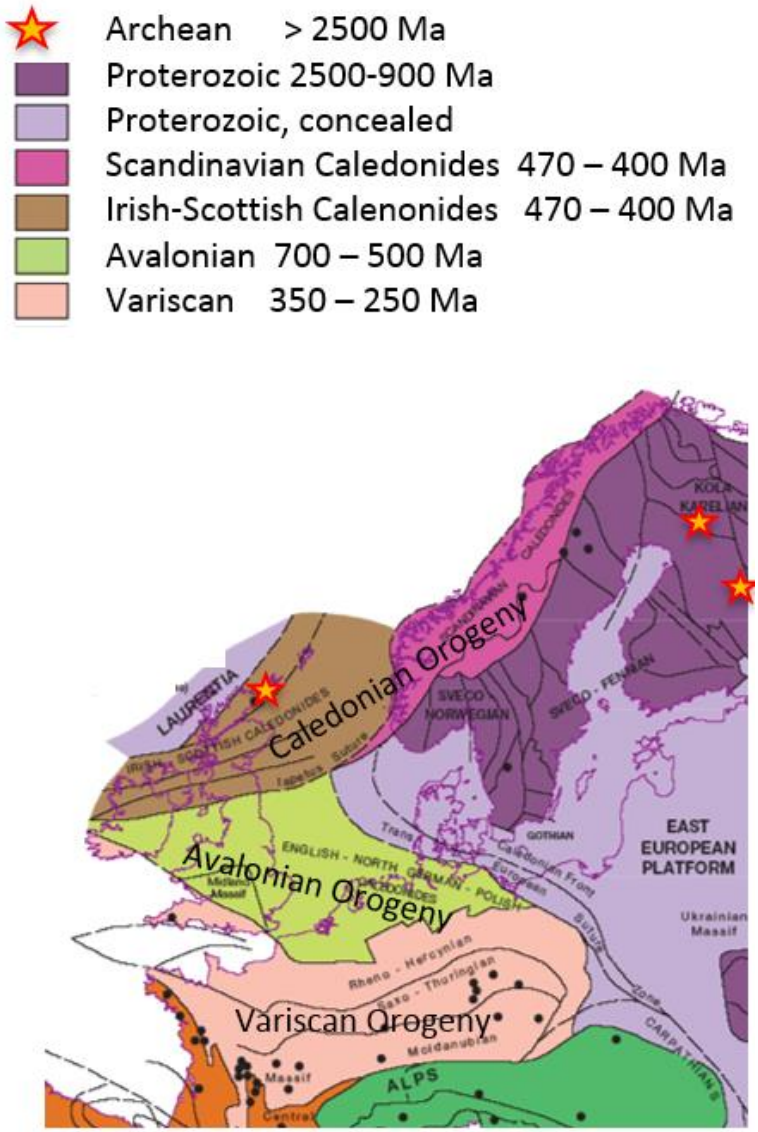
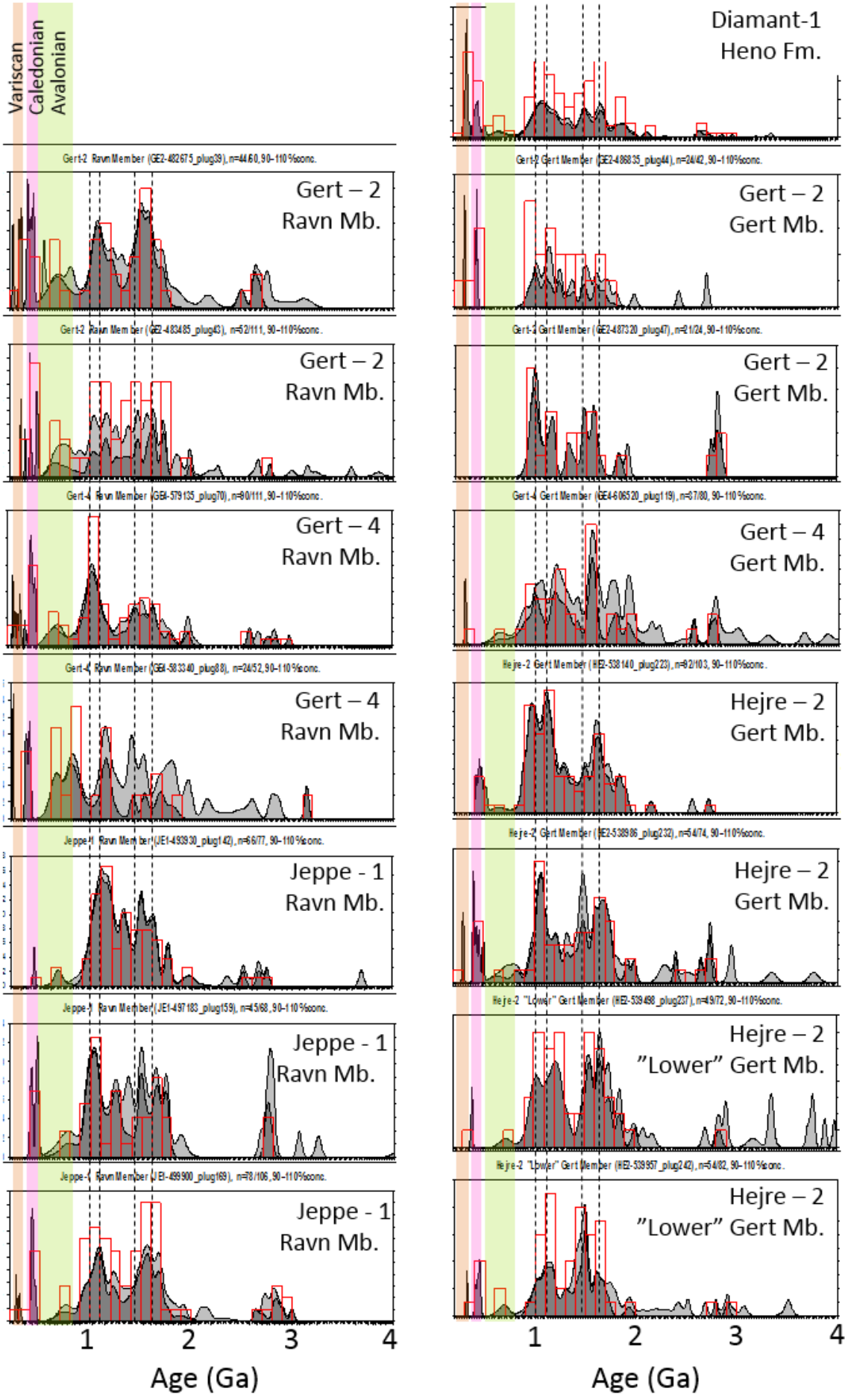


Figure 3 a *Ages of orogenic events in northern Europe.*

Figure 3b *Next page: Detrital zircon U/Pb ages from Diamant-1, Gert-1, Gert-4, Hejre-2 and Jeppe-1 (Weibel & Knudsen, 2007). Note that the x-axis start at 200 Ma. Stippled vertical lines show the most prominent detrital zircon ages in North sea sandstones: 1000 Ma, 1100 Ma, 1450 Ma and 1600 Ma.*


Variscan
Caledonian
Avalonian
Gert – 2
Ravn Mb.
Gert – 2
Ravn Mb.
Gert – 4
Ravn Mb.
Gert – 4
Ravn Mb.
Jeppe - 1
Ravn Mb.
Jeppe - 1
Ravn Mb.
Jeppe - 1
Ravn Mb.
Diamant-1
Heno Fm.
Gert – 2
Gert Mb.
Gert – 2
Gert Mb.
Gert – 4
Gert Mb.
Hejre – 2
Gert Mb.
Hejre – 2
Gert Mb.
Hejre – 2
”Lower” Gert Mb.
Hejre – 2
”Lower” Gert Mb.
1
2
3
4
Age (Ga)
1
2
3
4
Age (Ga)

1.2.2 U-Pb dating of detrital rutile in Jurassic sandstones

Rutile is a metamorphic mineral that records the age of the event that formed the mineral. This in contrast to zircon, that date the time of crystallization from the parent magma and as zircon is very robust and only to a very small degree is affected by the metamorphism. The effect of this is, that even if reworked e.g. in the Caledonian Orogeny, the zircon will keep its original age.

There is a very limited literature database on detrital rutile ages in the North Sea area, and one of the few is Small et al (2013) who describes detrital rutile from the Beauly and Tay rivers in Scotland where samples have unimodal age distributions with peaks of ca. 420 Ma and ca. 470 Ma, respectively. These ages match the known ages of the last amphibolite facies metamorphic events (Baxter et al., 2002; Kinny et al., 2003).

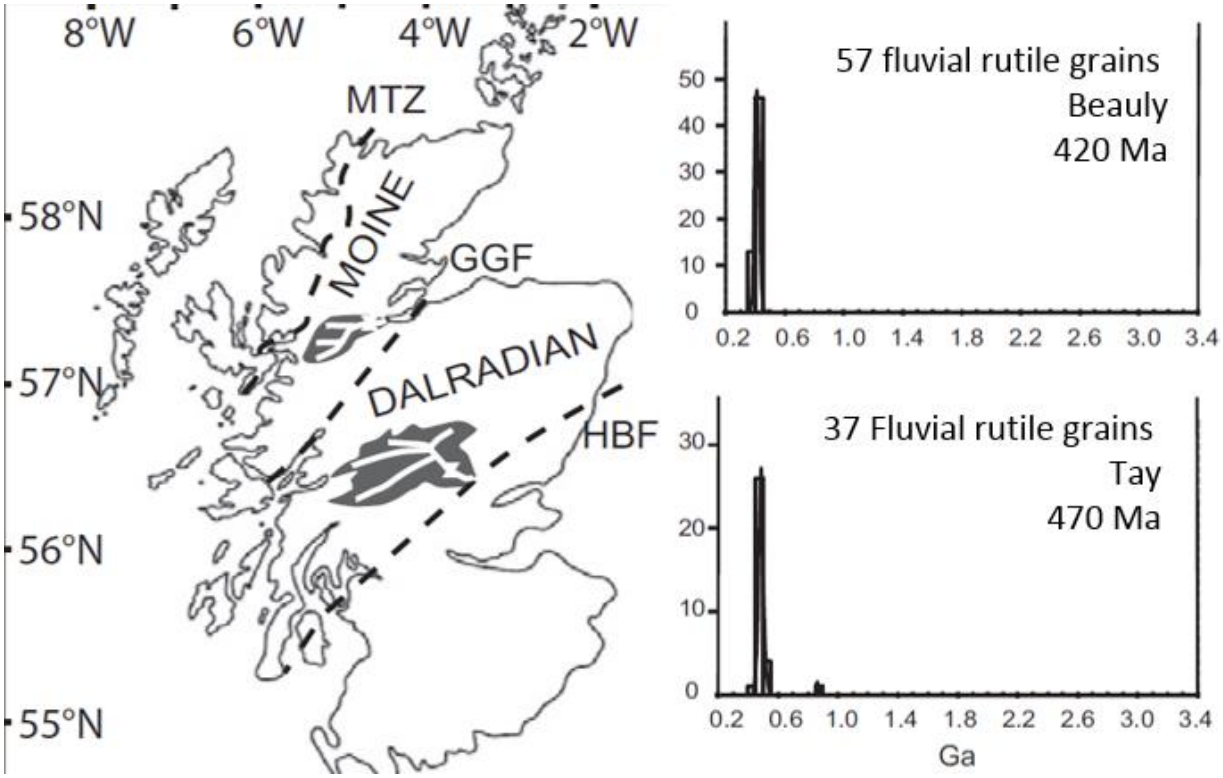


Figure 4 *Detrital rutile U/Pb ages two rivers in Scotland. The Beauly is draining the Moine area and the Tay is draining the Dalradian. From Small et al (2013).*

1.2.3 Trace elements in detrital zircon

When zircon crystalizes from a magma the content of trace elements will reflect the composition of the magma as well as the conditions under which the zircon crystalizes. Based on this, differences in the composition of the parent magmas as well as the conditions under which the zircon crystalized should be reflected in the detrital zircons derived from these terrains. This idea was tested by Hoskin and Ireland (2000) in sandstones from the North Sea, but they state that "zircon REE patterns and abundances are generally not useful as indicators of provenance." However, Wang et al (2012) found that it was possible to recognize differences in the composition of zircons from different geological environments.

2 Methods

2.1 Sample preparation

After crushing and milling the cuttings samples, zircon was separated using a water-shaking table, followed by hand-picking under a stereo-microscope from the separated fraction. Zircon and rutile grains were mounted on double-sided tape before casting into 2.5 cm round epoxy mounts which subsequently were polished.

2.2 Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS)

The *in-situ* zircon U-Pb geochronometry carried out in this study was performed at the Geological Survey of Denmark and Greenland (GEUS), employing a UP213 frequency-quintupled solid state Nd:YAG laser system from New Wave Research which was coupled to an ELEMENT 2 double-focusing single-collector sector-field ICP-MS from Thermo-Fisher Scientific. The mass spectrometer was equipped with a Fassel type quartz torch shielded with a grounded Pt electrode and a quartz bonnet. To ensure stable laser output energy, a laser warm-up time of at least 15-20 minutes were applied before operation, providing stable laser power and flat craters by a "resonator-flat" laser beam. The mass spectrometer was run for minimum one hour before analyses to stabilize the background signal. Prior to loading, samples and standards were carefully cleaned with ethanol to remove surface contamination. After insertion, the ablation cell was flushed with helium carrier gas for a minimum of 15-30 minutes to minimize gas blank level. The ablated material was swept by helium carrier gas and mixed with argon sample gas ca. ½ meter before entering the plasma. Prior to the analysis session the ICP-MS was optimized for dry plasma conditions through continuous linear ablation of the GJ-1 zircon standard. The signal-to-noise ratios for the heavy mass range of interest (from ^{202}Hg to ^{238}U) were maximized, while opting for low element-oxide production levels by minimizing the $^{254}UO_2/^{238}U$ ratio. The isotopes ^{202}Hg, Mass204 (^{204}Pb+^{204}Hg), ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th, ^{235}U and ^{238}U intensities were determined through peak jumping using electrostatic scanning in low resolution mode and with the magnet resting at ^{202}Hg. Data were acquired from 4 peaks per sample using a sampling and settling time of 1 ms for each isotope. Mass ^{202}Hg was measured to monitor the ^{204}Hg interference on ^{204}Pb, and if necessary, common Pb corrections were done through the interference and background corrected ^{204}Pb signal in combination with a model Pb composition (Stacey & Kramers 1975).

To minimize instrumental drift a standard-sample-standard analysis protocol was followed, bracketing 10 analyses by 3 to 6 measurements of the standard zircon GJ-1. To control the external reproducibility and the short-term precision for the ^{206}Pb/^{238}U, ^{207}Pb/^{235}U and ^{207}Pb/^{206}Pb ratios of the standard analyses (Frei et al 2006), a second zircon standard, Plesovice (Slama et al. 2008) was analysed several times during the analytical session, yielding an average age accuracy and precision of < 2% for the above-mentioned ratios. Data were acquired from single spot analysis of 25 µm using nominal laser fluence of ~10 J/cm2 and a pulse rate of 10 Hz, resulting in ablation crater depths of approximately 15-20 µm and ablated masses of ca. 65 ng. Total acquisition time for single analysis was max. 1.5 minutes, including 30 sec. gas blank measurement followed by laser ablation for 30 sec. and washout for 20 sec. Factory-supplied software was used for the acquisition of the transient data, obtained through automated running mode of pre-set analytical locations. Data reduction and calculation of ratios and ages were performed off-line through the software Iolite (Hellstrom et al. 2008, Paton et al. 2011), using the Iolite-integral VizualAge DRS (data reduction scheme) routine (Petrus & Kamber, 2011). Composite age-distribution curves for the detrital zircon grains presented as stacked probability-density diagrams were produced according to Thomsen et al (2016). All data obtained are stored in the GEUS zircon database.

3 Results

3.1 U/Pb ages in zircon

The yield of zircons from Xana-1, Gita-1 and Marsvin-1 was at acceptable level whereas the yield from Svane-1 and Amalie-1 was not as high as we would normally expect. The results are shown in Figure 5.

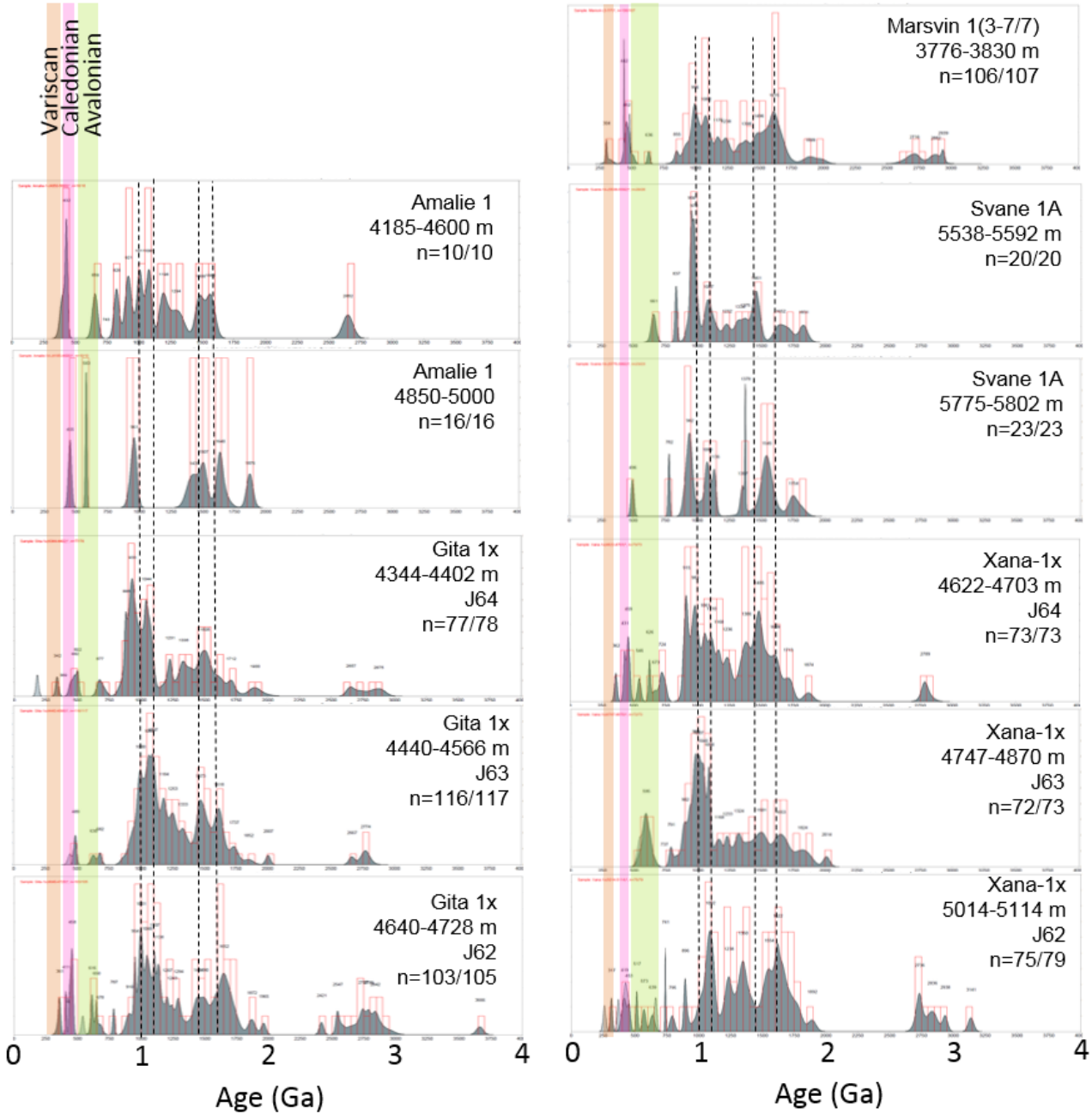


Figure 5 *Detrital zircon U/Pb ages from Gita-1, Xana-1, Amalie-1, Svane-1 and Marsvin-1 (3-7/7) this study.*

In the Marsvin-1 well the detrital zircon age distribution (total 107 zircons dated) is characterized by the two dominant peaks at 1000, 1100 and 1600 Ma with 9 Archean, 7 Caledonian and 2 Variscan zircons.

In Svane-1 (total of 20 and 23 zircons were dated in the two samples respectively) no Archean and Caledonian zircons were found but two Avalonian zircons. In Amalie-1, the total number of zircons dated is 26 which is a very low number. However, in both samples Caledonian as well as Avalonian zircons are detected.

In the Gita-1 well, the number of zircons dated is higher (78, 117 and 105 respectively) and apart from the bimodal distribution of the dominating Mesoproterozoic zircons it is seen that Archean (19), Caledonian (10) and Avalonian (10) zircons are present in all samples. It can further be noted, that in the uppermost sample (J64), the highest peak is at ca. 900 Ma, which is younger than the normally seen among the Mesoproterozoic zircons and the Mesoproterozoic population is skewed to the left in the upper two samples.

The detrital zircon age distributions in the Xana-1 share many features with Gita-1 such as the presence of both Caledonian and Avalonian zircons, that the uppermost sample (J64) has the highest peak at ca. 900 Ma and that the Mesoproterozoic population is skewed to the left in the middle sample (J63).

3.2 U/Pb ages in rutile

Only one sample – Marsvin-1 returned rutile that could be dated, and only 13 grains (Figure 6).

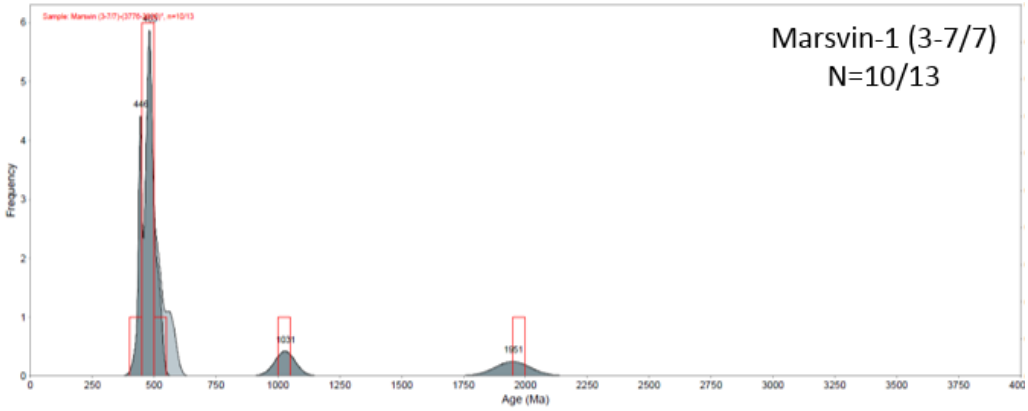


Figure 6 *Detrital rutile U/Pb ages from Marsvin-1 (3-7/7).*

The detrital rutile age spectrum show a majority of Caledonian ages. It is a bit surprising that the main peak is at ca. 483 Ma i.e. at the time of the Grampian episode – a forerunner of the main Caledonian orogeny at ca. 420 Ma.

3.3 Trace elements in zircon

The concentration of the following 22 elements were measured in all the detrital zircon grains: U, Th, Pb, P, Ti, Rb, Sr, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu and Hf.

The purpose of analyzing the trace elements in the zircon grains is as mentioned

To check the quality of the analytical results elements that are likely to be geochemically related were plotted on log/log scale (Figure 7). The data show good correlations suggesting that the data is of good quality.

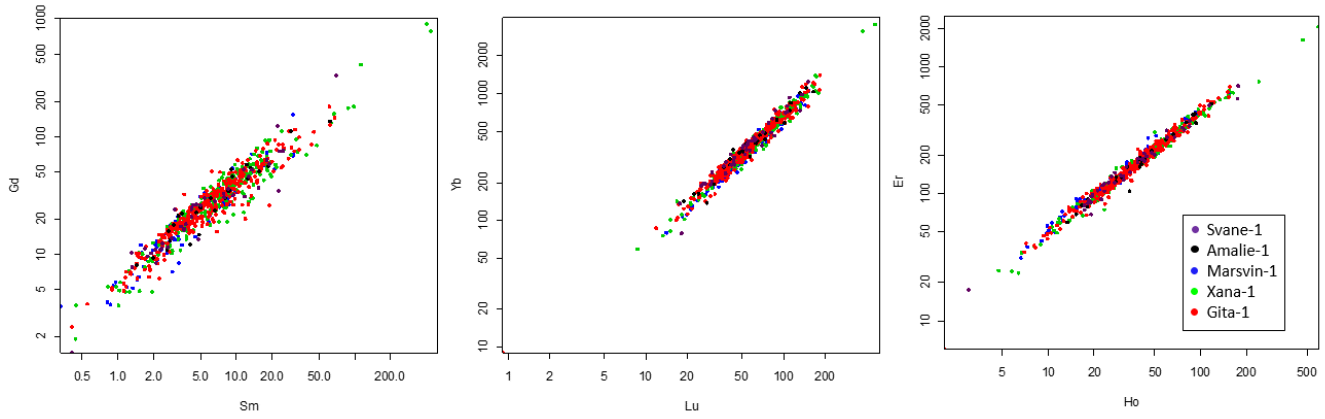


Figure 7 *Plot of ppm Sm versus ppm Gd, ppm Lu versus ppm Yb and ppm Ho versus ppm Er.*

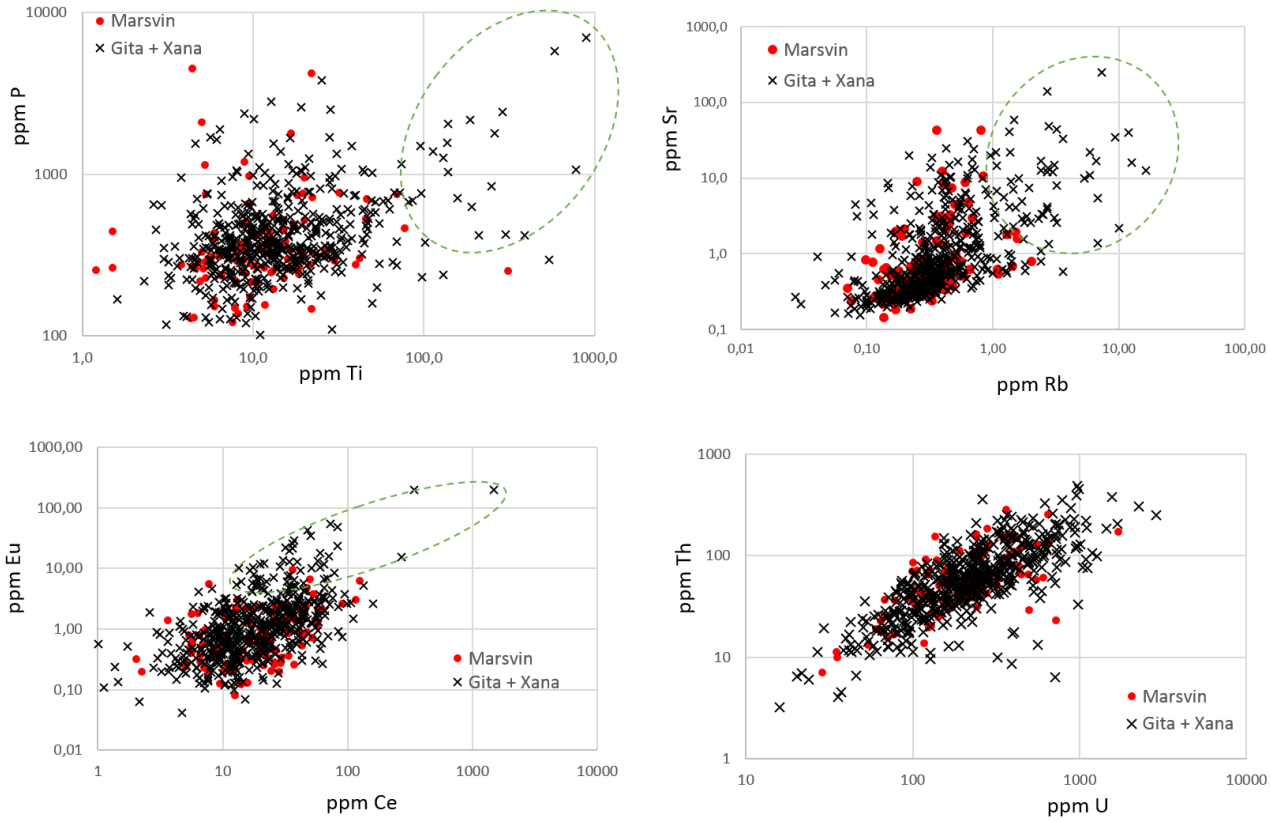


Figure 8 *Plot of trace elements in zircon. Gita-1 and Xana-1 are grouped in one group (not easy to discriminate using these elements).*

There is considerable overlap in the compositions of zircon from the different wells seen in Figure 8, which is not surprising as zircons from common rocks/geological environments such as e.g. grey tonalitic gneisses will have similar compositions. However, there are differences with higher frequency of zircon from Gita-1 and Xana-1 compared to Marsvin-1 with high P and Ti, high Sr and Rb high Eu as well as well as high U and Th (Figure 8).

4 Discussion

The overall picture in the samples analysed from Gita-1, Xana-1, Marsvin-1, Amalie-1 and Svane-1 is that the detrital zircon are dominated by 1750 to 950 Ma ages. Further, most samples have minor amounts of Archean and Caledonian zircons. This is not surprising and in line with what previously have been described from the Heno Formation (Weibel and Knudsen 2007). These ages are common in the basement both in Scandinavia and in Scotland. However, the detailed analysis of the distributions show, that there are significant variations in the Gert-2 and Gert-4 wells with influx of Variscan, Avalonian and Caledonian zircons in the Ravn Mb of the Heno Fm. That is not present in the Gert Mb showing that there was differences in the source with Ravn Mb being sourced from West and Gert from East. The Heno Fm. In Diamant-1 there is a large peak with Variscan zircons that are likely to be sourced from South.

The detrital zircon age distributions in the Xana-1 share many features with Gita-1 such as the presence of both Caledonian and Avalonian zircons and absence of Variscan zircons. The Mesoproterozoic population is skewed to the left in the middle sample (J63). This suggest a common source of the sand, and with Avalonian and Variscan zircons being present, there is a westerly component in the provenance.

In J64 section in both Gita-1 and Xana-1 the highest peak is at ca. 900 Ma (Figure 6). This is an exotic age for the Jurassic sandstones in the area (Figure 3), and an age not seen in the underlying J62 and J63 sands. This suggests a change in the provenance relative to the underlying sands. This 900 Ma peak has - so far - only been observed in Upper Paleozoic sandstones on the North Sea High (Figure 10, Lundmark *et al.* 2013). Could these sandstones have contributed to the J64 sands in both Gita-1 and Xana-1?

The trace element study of the detrital zircons show that there are significant differences in the fingerprint of the zircons and that with a population with high trace element such as Sr, Rb, Ti, P and Eu contents in Gita-1 and Xana-1 that is not found in Marsvin-1. This suggests that Gita-1 and Xana-1 may be source from a different provenance area than Marsvin.

The low number of zircons recovered and dated from Svane-1 and Amalie-1 makes it difficult to draw far-reaching conclusions on the provenance of the sands in these wells. However, it can be noted that even the total number of zircons dated there are Avalonian zircons present in both. Avalonian ages are not present in Scandinavia and these zircons must be derived from West.

The most likely sediment source for a sandstone is re-deposition of another sandstone. However, apart from the Upper Paleozoic sandstones on the North Sea High mentioned above, there are very little data available on the provenance of the sandstones that could be the immediate source for the Jurassic sands in Xana-1 and Gita-1. Olivarius et al. (2016 and 2017) have analysed Triassic sands in the Danish, Norwegian and North-German Basins as well as basement from the Ringkøbing Fyn High (Figure 10). Most of these sandstones have detrital zircon age distribution patterns characterized by large populations around ca. 1500 Ma and are a likely source for the Triassic rocks. Western Sweden and Eastern South Norway are also dominated by these ages (Orange and red on Figure 10, Olivarius & Nielsen, 2016). This suggests that the provenance of the sands in Xana-1 is not from the east.

The finding of rutile with ages of ca. 480 Ma in Marsvin-1 and not in Gita-1 and Xana-1 was surprising and support that the provenance is different. The Grampian 480 Ma age of the rutile is also a bit surprising. This age known from the Scandinavian Caledonides but far to the north in Lapland. In Scotland this age is well known from studies of recent sediments (Small et al 2013) in the Dalradian. This suggests a source in Scotland for these rutiles.

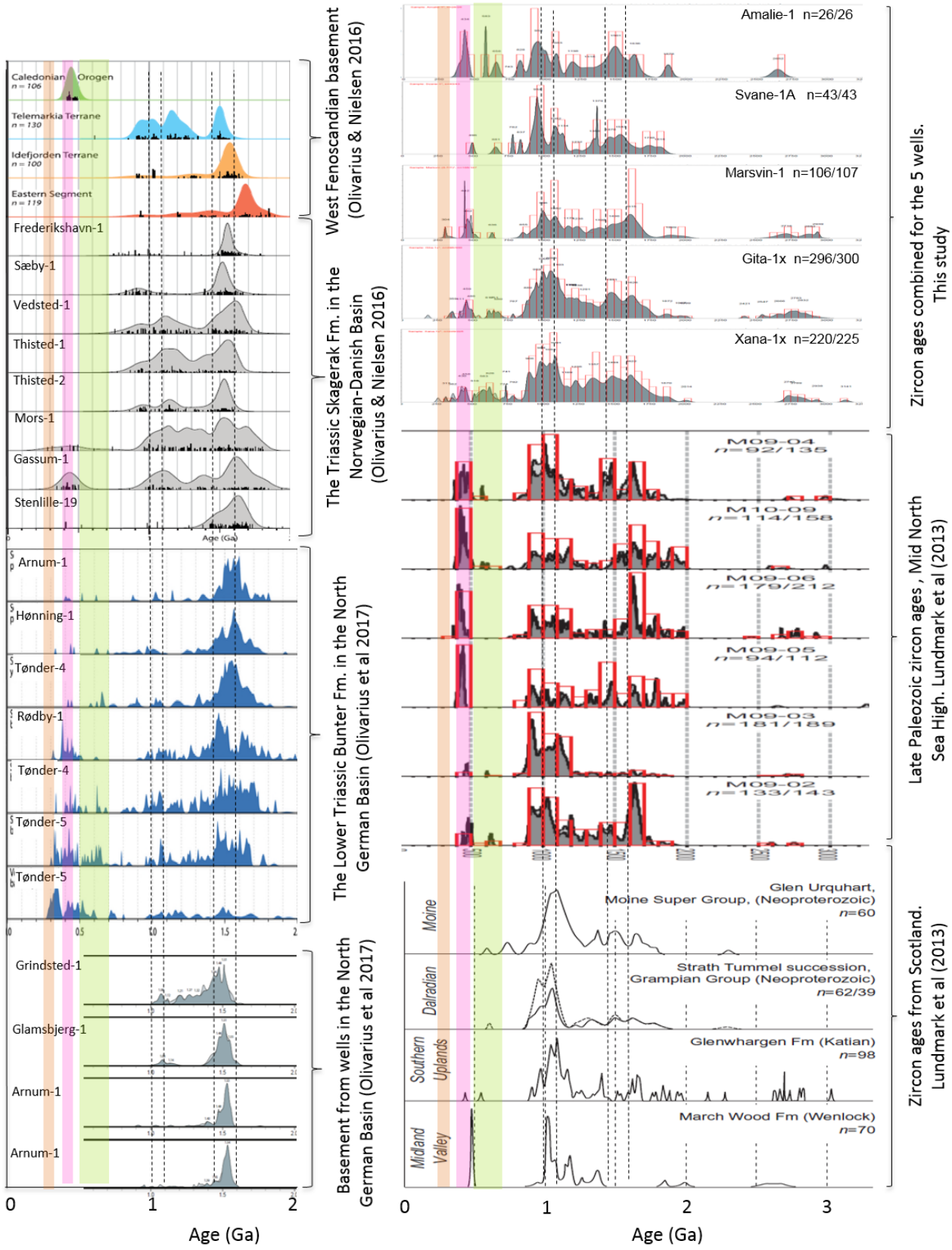


Figure 10 *Age distributions in the potential source areas.*

5 Conclusions

The uppermost sandstone unit XA4 (J64) has a different zircon provenance signature than the underlying J62 and J63 sandstone units in Xana-1 seen by the presence of a 900 Ma peak in the detrital zircons, which is not present in the underlying units. This is a feature that Xana-1 share with Gita-1 where the 900 Ma peak is also present in J64 but not in the underlying J63 and J62 sandstone indicating that there was a shift in the source from J63 to J64. The 900 Ma peak in the detrital zircon population is not known from other Jurassic sandstones in the area nor from the Ringkøbing-Fyn-High, but is known from Paleozoic sandstones on the Mid North Sea High.

The provenance fingerprint in the Xana-1 and Gita-1 wells share most features and could be sourced from a single common provenance area. The presence of Avalonian zircons suggests a westerly component in the source area most prominent in the Xana-1 well. The provenance fingerprint of the sandstones in Marsvin-1 to the North is different which is seen both in the trace-element fingerprint in the zircon and in the presence of Scottish rutile in Marsvin-1. The available data from basement rocks on the Ringkøbing-Fyn-High are dominated by ca. 1500 Ma zircons and this is not a likely source.

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