

## Occurrence and source of radioactive lead in geothermal formation water, Denmark

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### ABSTRACT

The Margretheholm geothermal plant in Copenhagen experienced increasing injectivity problems during its first ten years of production due to deposition of lead in the injection well. Filters in the plant also had to be shifted more frequently due to deposition of lead. Furthermore, filters and other objects contaminated by lead were classified as NORM (Naturally Occurring Radioactive Material) and had to be stored in a protected area due to radiation. The radiation was mainly due to <sup>210</sup>Pb with a half-life of 22.2 years. Deposition of lead was caused by galvanic corrosion of steel by the warm (74 °C) saline water (Cl, 137 g/L) from the 2.6 km deep Bunter Sandstone Formation containing 0.4 mg/L dissolved lead (Pb<sup>2+</sup>). The prospects of exploiting the shallower (c. 1.8 km) Gassum Formation in the Copenhagen area without any costly precautions needed to avoid the lead and NORM issues were undertaken by comparing the content of lead and radionuclides in formation water of the Gassum formation with that of the Bunter Sandstone Formation.

Two other Danish geothermal plants exploiting the Gassum Formation had not experienced problems with lead and NORM having distinctly lower concentrations of lead (0.02 mg/L) and radionuclides. The likely source of radionuclides including <sup>210</sup>Pb in formation water was examined by analyses of uranium-rich minerals in various reservoir materials as well as the short-lived radionuclides in formation water. The source of radionuclides in formation water must be close to the production well due to the presence short-lived <sup>224</sup>Ra with a half-life of 3.6 days. Based on available data on geology and formation water chemistry from various wells, we consider the geothermal energy prospects for Gassum Formation to be good in the Copenhagen area.

### 1. INTRODUCTION

Initial problems with decreasing injectivity at the Margretheholm plant commissioned in 2005 were partially solved by lowering the pH of the injected water under normal operation adding hydrochloric acid for a short period of time, the so-called soft acidizing procedure. However, injectivity continued to deteriorate over the following years while the ability of soft acidizing diminished as a remedy. Camera inspection of the injection well in 2014 including bailer sampling gave no clear indication as to the cause of clogging. Several perforations were clearly blocked by indistinguishable solids. Flow-logging performed after the camera inspection showed that loss of injectivity was mainly due to blocking of the lower perforations in the injection well.

In 2015, filters clogged shortly after start of the pump, the reason being accumulation of mainly fine metallic lead as shown by XRD and chemical analysis (ICP-MS). Renovation of the plant two months later revealed large quantities of metallic lead in the flow lines and both wells. The accumulation of lead was assumed to be due to galvanic corrosion whereby lead deposition from water is associated with iron dissolution from installations. Radionuclide analysis of various solids confirmed that it had to be classified as NORM according to Danish authorities (SIS).

Resuming operation after the workover operation filters still had to be replaced regularly, though less frequently, which added to the amount of NORM wastes to be stored. It may be added that in addition to the Pb-NORM issue, radium may also accumulate, e.g. traces in barium sulphate/strontium sulphate deposits observed on heat exchanger plates a year later, which also caused a radiation issue.

As part of the licensing program, an analysis of the economy in an expansion with small geothermal plants was conducted based on using the Gassum Formation as the primary geothermal reservoir. The primary purpose of the present study is therefore to evaluate the

risk of whether geothermal production from the Gassum Formation reservoir in the Copenhagen area may include similar challenges with NORM as the Bunter/Skagerrak Formation does. Hence, the study is designed to:

1. Investigate the origin and extent of radionuclides in geothermal water from the Bunter/Skagerrak Formation reservoir in Margretheholm by comparing water chemistry with an assessment of possible radionuclide sources in the reservoir rock.
2. Assess the risk that similar radionuclide problems may occur for Gassum Formation by comparing the chemistry of geothermal water from Gassum Formation with the occurrence and amount of possible sources of radionuclides in the sandstone reservoir based on relevant locations, comprising the existing geothermal facilities at Margretheholm, Thisted and Sønderborg.

## 2. GEOLOGICAL BACKGROUND

The Margretheholm plant is located in the Copenhagen area in eastern Denmark (Figure 1). The sandstone reservoirs in the subsurface were deposited in the easternmost part of the Norwegian-Danish Basin, which was formed by crustal stretching followed by late Carboniferous–early Permian rifting and Mesozoic–Cenozoic thermal-dominated subsidence (Vejbæk 1997). Thereby a km-thick succession of sandstones, siltstones, mudstone, carbonates and evaporites accumulated in the basin. Of these, the

Triassic–Jurassic succession includes several potential geothermal reservoir sandstones such as the Gassum Formation and the Bunter/Skagerrak Sandstone Formation. In general, the sandstones from these formations are considered to be widespread with relatively good lateral continuity in areas unaffected by faults or salt structures.

The Lower Triassic Bunter Sandstone Formation is widespread in the North German Basin and in the southern part of the Norwegian–Danish Basin, where it is synchronous with the oldest part of the Lower–Upper Triassic Skagerrak Formation that accumulated in the northern part of the basin. The arid to semi-arid climate resulted in deposition of aeolian and fluvial sand interbedded with silt- and mudstones formed in shallow lakes with occasional evaporite deposition (Bertelsen 1980, Pedersen and Andersen 1980, Clemmensen 1985, Michelsen and Clausen 2002).

The Upper Triassic–Lower Jurassic Gassum Formation is present in most of the Norwegian–Danish Basin, except where major salt structures occur and on the basement blocks of the Ringkøbing–Fyn High. The formation was deposited in a humid climate and consists of marine and fluvial sandstones interbedded with marine and lagoonal mudstones, minor siltstones and thin coal beds formed during periods with recurrent sea-level changes (Michelsen et al. 2003, Nielsen 2003).

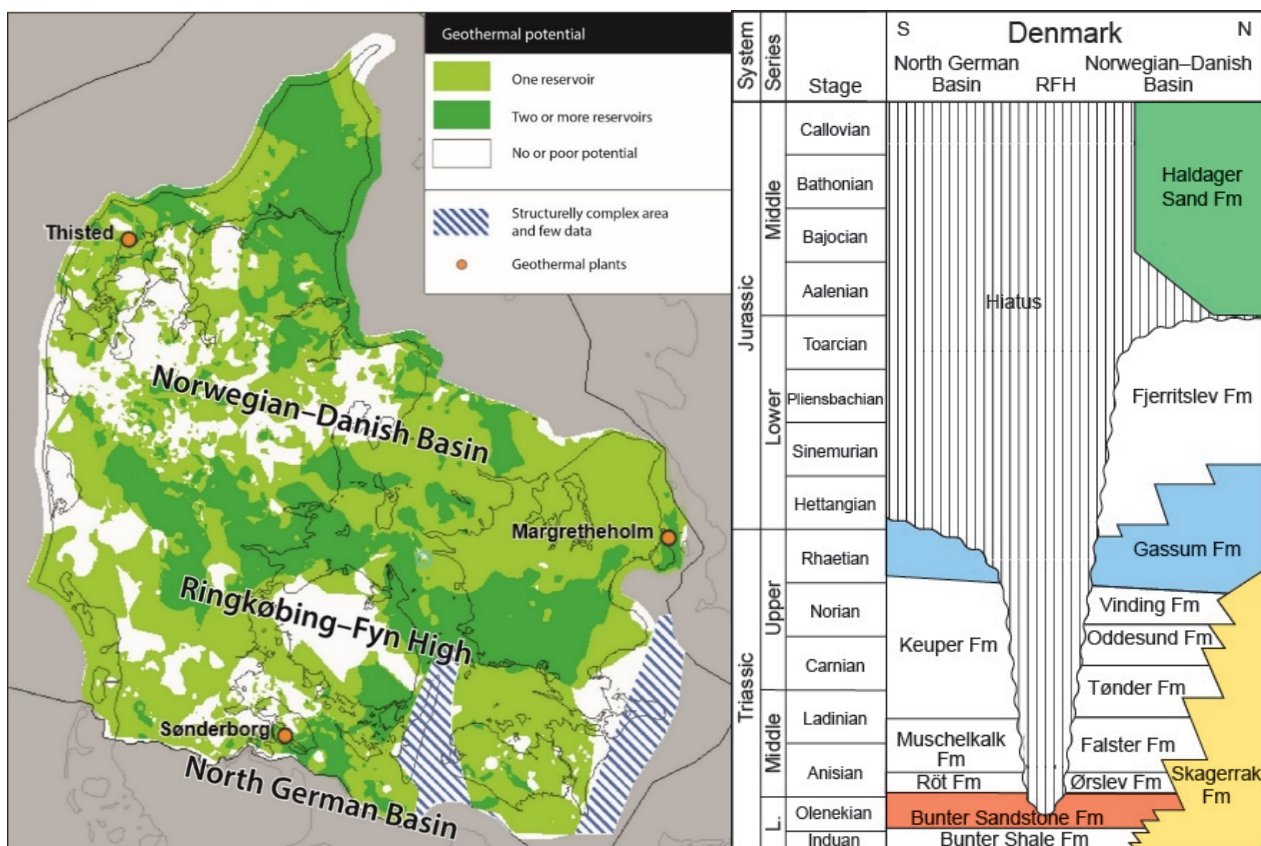


Figure 1: The map is from the Danish Geothermal WebGIS-portal (DybGeotermi.geus.dk) showing the geothermal potential in the Danish area and the location of the three existing geothermal plants (Vosgerau et al. 2016). The stratigraphic scheme depicts the Danish Lower Triassic – Middle Jurassic succession where the highlighted formations contain potential geothermal reservoir sandstones (simplified after Michelsen and Clausen 2002).

### 3. SAMPLES AND METHODS

Water samples for analysis of radionuclides were collected from the geothermal plants at Margrethholm, Sønderborg and Thisted during March-April 2018. Warm water of 46-70°C from the production wells were filtered directly through a 0.45 µm membrane filter into screw cap glass bottles of 1-5 liters. The samples were then brought to analysis within 5 hours after sampling. About 600 ml of water from each well was precipitated using Fe(OH)<sub>3</sub> and stable lead as yield monitor. The precipitate was dissolved in 2M HCl and <sup>210</sup>Pb was isolated using a slightly modified method based on Vajda et al. (1997). Stable lead recovery was determined using ICP-OES on an aliquot of the purified sample while the rest was used to determine <sup>210</sup>Pb on a Quantulus (Wallac/Perkin Elmer) ultra low-level liquid scintillator. Ra-isotopes were co-precipitated onto MnO<sub>2</sub> and the dissolved precipitate was gamma counted repeatedly for 2 weeks. Data treatment for <sup>224</sup>Ra were done in a similar way as Degering and Köhler (2011) to take into account the continuous ingrowth and decay.

Five samples were selected for analysis of U-rich minerals comprising two from the Bunter/Skagerrak Formation in Margrethholm-1 and three from the Gassum Formation distributed as one from each of the Margrethholm-1, Sønderborg-1 and Thisted-3 wells. This was done to measure the contents of U, Th and Pb in each grain. A core sample was used from Thisted-3 and cuttings were sampled from the other wells. The analyses were performed on hand-picked grains that were mounted in epoxy after being collected from crushed samples sorted on a water-shaking Wilfley table. Sufficiently zircon grains for analysis appeared in all samples, while rutile occurred more scarce, especially in the Bunter/Skagerrak Formation where only a single usable grain was found. All samples were also screened for other heavy minerals with high U-content such as monazite, apatite and titanite, but none of these occurred in any significant amount and thus were not analysed. The laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) was carried out using an Element2 magnetic sector-field mass spectrometer from Thermo-Fisher Scientific that is coupled to a NWR-213 laser ablation system from Elemental Lasers Inc. Analysis procedures basically followed as described in Frei and Gerdes (2009). Data were acquired from single spot analysis of 25 µm in diameter using an impulsed laser beam with a nominal laser fluence of 10 J/cm<sup>2</sup> and a pulse rate of 10 Hz. Total acquisition time for single analysis was max. 1.5 min., including 30 sec. gas blank measurement followed by laser ablation for 30 sec. and washout for 30 sec. in an air-tight chamber. About 200–300 nanograms of material was liberated and transported by helium gas through inert Tygon tubing to the mass spectrometer for isotopic determination. To minimize instrumental drift, a standard-sample-standard analysis protocol was followed, bracketing eight analyses by

three measurements of the standard zircon GJ-1 (Jackson et al. 2004). U, Th and Pb concentrations were calculated based on the content of these elements in the standard.

Inorganic geochemistry was measured on 29 samples of sandstone and claystone. Quantitative geochemistry analysis with focus on trace element concentrations was performed on a PerkinElmer Elan 6100DRC quadrupole inductively coupled plasma mass spectrometer (ICP-MS). For each analysis, at least 5 g of material was crushed to < 100 µm in a tungsten carbide mortar prior to digestion. The analyses include 41 elements measured twice for each sample after subjecting it to different digestion techniques. The samples were dissolved in hydrofluoric acid after borate melting in order to dissolve the most difficult minerals like zircon that is relevant for this study because of its high U content. To ensure the data quality, reference standard samples were measured concurrently during the acquisition of the data series.

Automated mineral scanning of carbon-coated thin sections were made on a Zeiss Sigma 300VP field emission scanning electron microscope (SEM) coupled with two Bruker XFlash 6|30 129 eV energy dispersive X-ray spectrometers (EDS) to rapidly image and chemically map the samples using a step size of 10 µm. The level of backscatter electron (BSE) brightness is used to distinguish the rock from the epoxy. An X-ray spectrum was acquired for each measurement such that the chemical composition of each individual mineral phase is known. The Zeiss Mineralogic software platform was applied to determine the mineralogical composition of each analysis based on a mineral library adjusted to the current samples.

### 4. RADIOACTIVITY OF FORMATION WATER

Most intermediates of the decay chains <sup>238</sup>U → <sup>206</sup>Pb and <sup>232</sup>Th → <sup>208</sup>Pb have extremely low solubility and will not enter the formation water. Radium and possibly lead (depending on sulphide concentration, Kharaka et al. 1987) are exceptions and may be found in saline formation waters depending on the U and Th content in the rock (Fisher 1998). The Formation water from the geothermal plants at Margrethholm, Sønderborg and Thisted were analyzed as quickly as possible after sampling to be able to determine the content of short-lived radionuclides, which may help determine the migration path from their source. The half-lives of <sup>210</sup>Pb and <sup>224</sup>Ra are 22.2 years and 3.6 days, respectively.

Regarding migration of radionuclides, the source of radionuclides in the formation water must exist in the reservoir itself and probably quite close to the well bore, since the very short-lived Ra-224 is also present in the water (Table 1). This is in accordance with results of other investigations such as Hammond et al. (1988) and Degering et al. (2015).

**Table 1: Specific activity of radionuclides in formation water.**

Locality/Well	Ra-226 [Bq/kg]	±	Pb-210 [Bq/kg]	±	Ra-228 [Bq/kg]	±	Th-228 [Bq/kg]	±	Ra-224 [Bq/kg]	±
Margrethholm/MAH-2	13.5	1.5	2.46	0.74	35.1	2	2.4	0.2	49	7.4
Sønderborg/SG-2	6.0	0.5	0.042	0.01	5.8	0.5	0.66	0.05	11	1.7
Thisted/Thi-5	10.7	1	0.072	0.02	12.1	1	1.2	0.1	9	1.4

Operations involving radioactive material require approval by the Danish Health Authority, Radiation Protection (SIS). This includes Naturally Occurring Radioactive Material (NORM) as well. Any material containing one or more radionuclide exceeding the limits listed in BEK 85/2018 should be considered NORM. For mixtures of radionuclides, the summation rule applies:  $\sum_k \frac{c_k}{C_{U,k}} \leq 1$ , where  $C_k$  denote the activity concentration of radionuclide  $k$  and  $C_{U,k}$  denote its exemption limit in Bq/g ( $^{210}\text{Pb}=5$ ;  $^{226}\text{Ra}=1$ ;  $^{228}\text{Ra}=2$ ;  $^{228}\text{Th}=1$ ). By applying the summation rule for the radionuclides in the analyzed formation water, values from 0.009 to 0.032 are obtained, which is far below 1 that is the limit for NORM material (Table 2).

**Table 2: Sum of radionuclides in formation water relative to NORM limit including relative  $^{210}\text{Pb}$  content.**

Well	$\sum_k \frac{c_k}{C_{U,k}}$	$^{210}\text{Pb}/\text{Pb}$ (ppb)	Pb (mg/L)
Margrethholm-2	0.032	2.18	0.40
Sønderborg-2	0.009	0.74	0.02
Thisted-5	0.017	6.37	0.004

Using the half-life of  $^{210}\text{Pb}$ , the concentration can be calculated from its activity concentration (Table 1), which appears to constitute only 0.74–6.37 ppb of the total lead content (mg/L) in the water (Table 2). Radionuclide analyses previously performed on solid wastes from the Margrethholm plant show that the wastes should be considered TNORM (T for technical). The reason why the radioactivity exceeds the NORM limit for most of the solid wastes is the relatively high lead content, caused by deposition of lead from formation water due to galvanic corrosion. Lead is being deposited on iron due to galvanic corrosion according to equation [1].



The content of  $^{210}\text{Pb}$  is about 30–60 times larger in the Bunter/Skagerrak Formation in Margrethholm compared to the Gassum Formation in Thisted and Sønderborg. The formation water from the Gassum Formation in Thisted and Sønderborg shows a 20–100 times lower content of  $\text{Pb}_{\text{Tot}}$  compared to the water from Bunter/Skagerrak Formation in Margrethholm.

The concentration of Pb in formation water is controlled by the equilibrium with Pb-sulphide in geological deposits when there are reducing conditions. The concentration of Pb in the formation water is determined by the sulphide concentration, salinity,

organic constituents, including organic acids, and reservoir temperature. Low sulphide content, high salinity and high temperature promote the conditions for high concentration of Pb. Leaching from nearby formations (organic-rich shales) may also significantly increase the Pb content.

The ratios of the Gassum Formation sites are similar in terms of salinity and temperature (about 50°C) and probably also sulphide content to which organic content is of importance. The significantly higher Pb-content in the formation water from Bunter/Skagerrak Formation is probably associated with the less reducing conditions during deposition, which means lower sulphide content and thus a higher Pb-content in the water. In addition, the higher temperature (73°C) and higher salinity of the water in Bunter/Skagerrak Formation cause the solubility of Pb-sulphide to increase, enabling a higher Pb-concentration in the water. The significantly lower Pb-content that is found in the formation water in Gassum Formation relative to the content measured in Bunter/Skagerrak Formation reduces the risk of significant Pb precipitation and the associated problems such as clogging of the injection well with Pb.

## 5. URANIUM CONTENT IN HEAVY MINERALS

$^{210}\text{Pb}$  results from decay of U and must originate from the formation from which water is produced since there is only short time available to bring the radioactive isotopes to the surface after the decay in the U-containing minerals. Thus, most likely the radioactive isotopes originate from minerals in the reservoir itself and not from adjacent formations. The reservoir sandstones contain a number of U-rich heavy minerals that has the potential to constitute the primary sources of radioactivity. For example, zircons with a high U-content are often being completely or partly destroyed by the radioactivity that they carry (i.e. the process of metamictization). This process liberates or loosens radioactive daughter products so they can reach e.g. the formation fluid.

Analysis of the uranium content of a large number of heavy mineral grains from both Bunter/Skagerrak and Gassum Formations were carried out in order to investigate if specific sources of radionuclides could be identified. The LA-ICPMS analyses of zircon show that some of the grains from both the Bunter/Skagerrak and Gassum Formations have high contents of U, Th and Pb. However, the highest values are found in zircons from Bunter/Skagerrak Formation, which also have the highest average amounts of all three elements (Table 3).



**Table 3: U, Th and Pb concentrations in zircon and rutile grains calculated as average of all analyzed grains (number indicated by 'n').**

Mineral type	U (ppm)	Th (ppm)	Pb (ppm)	n
Zircon grains, <i>Bunter Fm</i>	443.7	190	805.7	155
Zircon grains, <i>Gassum Fm</i>	360.4	108.4	409.2	310
Rutile grains, <i>Bunter Fm</i>	48.8	0.2	8.6	1
Rutile grains, <i>Gassum Fm</i>	40.4	0.2	4.3	39

Rutile was the only other U-rich mineral that occurred in amounts large enough for analysis, however, only a single rutile grain was located in the Bunter/Skagerrak Formation, whereas more grains were present in Gassum Formation. The contents of U, Th and Pb are much smaller in rutile than in zircon (Table 3). Thus, rutile can be excluded as a possible source of radionuclides to the formation water.

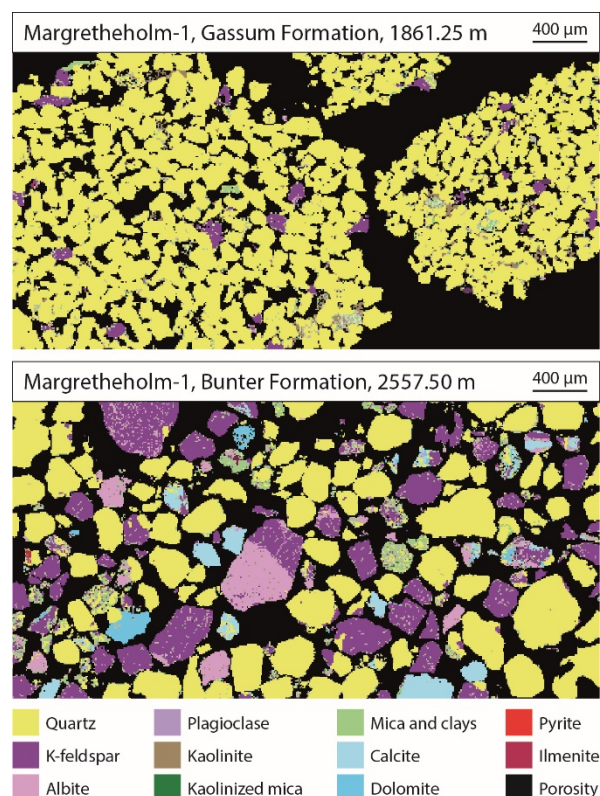
The geochemical results show that the total amount of Zr and hence zircon is comparable between the Bunter/Skagerrak and Gassum Formations (Table 4). However, the generally higher U-content in zircon grains in the Bunter/Skagerrak Formation indicates that metamict zircons occur in this formation and, although not abundant, that the metamict zircon grains might constitute a potential significant source for the supply of radionuclides into the formation water.

**Table 4: Geochemistry of bulk samples of sandstones and claystones calculated as average of all analyzed samples (number indicated by 'n').**

Lithology	U (ppm)	Th (ppm)	Pb (ppm)	Zr (ppm)	n
Sandstone, <i>Bunter Fm</i>	2.27	9.91	16.53	323.45	5
Sandstone, <i>Gassum Fm</i>	2.27	8.66	12.21	357.93	16
Claystone, <i>Bunter Fm</i>	2.77	12.04	15.75	200.97	3
Claystone, <i>Gassum Fm</i>	3.12	12.45	18.56	254.31	5

The sediment in Bunter/Skagerrak Formation in eastern Denmark was supplied from a local source area as evident by its narrow zircon age distribution, whereas the higher mineralogical maturity of the sediment in the Gassum Formation is caused by longer transport distance and higher-energy depositional environments (Nielsen 2003, Olivarius and Nielsen 2016, Weibel et al. 2017). This is seen by the smaller content of unstable grains in the Gassum Formation (Figure 2). Thus,

fragile metamict zircon grains would have had the best chance of avoiding mechanical breakdown in the Bunter/Skagerrak Formation.

**Figure 2: Mineralscans made by SEM of cuttings samples of sandstone intervals from the Margrethholm-1 well.**

## 6. CONCLUSIONS

Lead solids accumulate in pipes and filters at the geothermal facility at Margrethholm due to galvanic corrosion. Content of radioactive lead ( $^{210}\text{Pb}$ ) in lead-contaminated objects exceeds the limit for NORM decided by the Danish authorities (SIS), which add to the cost of handling of such wastes. The content of  $^{210}\text{Pb}$  and other radioactive isotopes in the geothermal water is far below the limit for NORM material, meaning that the NORM problem occurs only when lead accumulates. Whether lead deposits will form as a result of galvanic corrosion depends on the lead content in the geothermal water, together with the type of materials used in the installations. Initially, the lead content in the water depends upon reservoir temperature, organic constituents, salinity and sulphide concentration. The Bunter/Skagerrak Formation is generally buried deeper than the Gassum Formation and therefore reach higher temperature and higher salinity. The  $^{210}\text{Pb}$  originates from uranium ( $^{238}\text{U}$ ) within the reservoir, as evident from presence of the very short-lived  $^{224}\text{Ra}$  in the formation water. Zircon is the only mineral in the formations that has both high uranium-content and occurs in amounts large enough to be a possible source of the radionuclides in the water. Zircon grains with very high uranium-content become

degraded by the radioactivity so the decay products including  $^{210}\text{Pb}$  can more easily escape to the water and such zircons are more abundant in Bunter/Skagerrak Formation than Gassum Formation. This is consistent with the fact that the sand in Bunter/Skagerrak Formation has been transported significantly shorter from source area to deposition than the sand in Gassum Formation, so it is likely that a higher number of fragile zircons with extra high uranium content have survived the transport.

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