

Supplementary Information:

Noble gases in aquitard provide insight into underlying subsurface stratigraphy and free gas formation

Alexandra Kathryn Lightfoot*¹, Emiliano Stopelli², Michael Berg¹, Matthias Brennwald¹,
Rolf Kipfer^{1,3,4}

¹Eawag, Swiss Federal Institute of Aquatic Science and Technology, Department of Water Resources and Drinking Water, 8600 Dübendorf, Switzerland; ²International Services and Projects, Nagra, 5430 Wettingen, Switzerland; ³Department of Environmental System Sciences, Institute of Biogeochemistry and Pollutant Dynamics, ETH Zürich, 8092 Zürich, Switzerland; ⁴Department of Earth Sciences, Institute of Geochemistry and Petrology, ETH Zürich, 8092 Zürich, Switzerland.

**Corresponding author: Alexandra K. Lightfoot – lightfoot@eawag.ch*

AdvectAs team members

Karlsruhe Institute of Technology - Institute of Applied Geosciences: M. Schneider, Dr. E. Eiche, Prof. Dr. A. Kontny

Technical University of Berlin - Institute of Applied Geosciences: Prof. Dr. T. Neumann

University of Tübingen - Department of Geosciences: Dr. M. Glodowska, Dr. B. Rathi, Prof. Dr. A. Kappler, Jun.-Prof. Dr. S. Kleindienst, Prof. Dr.-Ing. O. A. Cirpka

Eawag, Swiss Federal Institute of Aquatic Science and Technology - Department of Water Resources and Drinking Water: A. Lightfoot, Dr. E. Stopelli, Prof. Dr. M. Berg, Prof. Dr. R. Kipfer, Prof. Dr. L. Winkel

Vietnam National University, Hanoi - Key Laboratory of Analytical Technology for Environmental Quality and Food Safety Control (KLATEFOS): Vu T. Duyen, Tran T. Mai, Vi M. Lan, Dao V. Nga, Dr. Pham T.K. Trang, Prof. Dr. Pham H. Viet

The University of Western Australia and CSIRO Land and Water: Prof. Dr. Henning Prommer

Sediment core: sample processing and unsaturated conditions

Fig. S1.1 shows the set-up of the sediment press used to squeeze sediments from the sediment core liners into the copper tubes. A piston (not shown) pushes freshly collected sediments contained in the liner, which is held in place by a cylindrical metal core holder, into a copper tube using a mechanical press. The copper tube is attached by Swagelok connectors. Once the sediments have flushed the copper tube sufficiently, the clamps are sealed, and the copper tube disconnected. Copper tubes are then transported back to Switzerland for further preparation and centrifugation to separate the pore water from the sediment matrix (see Methods).

Upon sample preparation, samples specifically at depths of 8.25, 8.75, 11.75, 12.75, 13.25 and 14.25 m showed features representative of unsaturated conditions. Gas pockets or bubbles were observed (Fig. S1.2) in the sampled sediments at the above-mentioned depths, and while the sediments felt damp, they did not give the appearance to be water saturated. These observations are clearly reflected in the noble gas ratio data of Fig 2., whereby all the aforementioned samples are situated in cluster U.

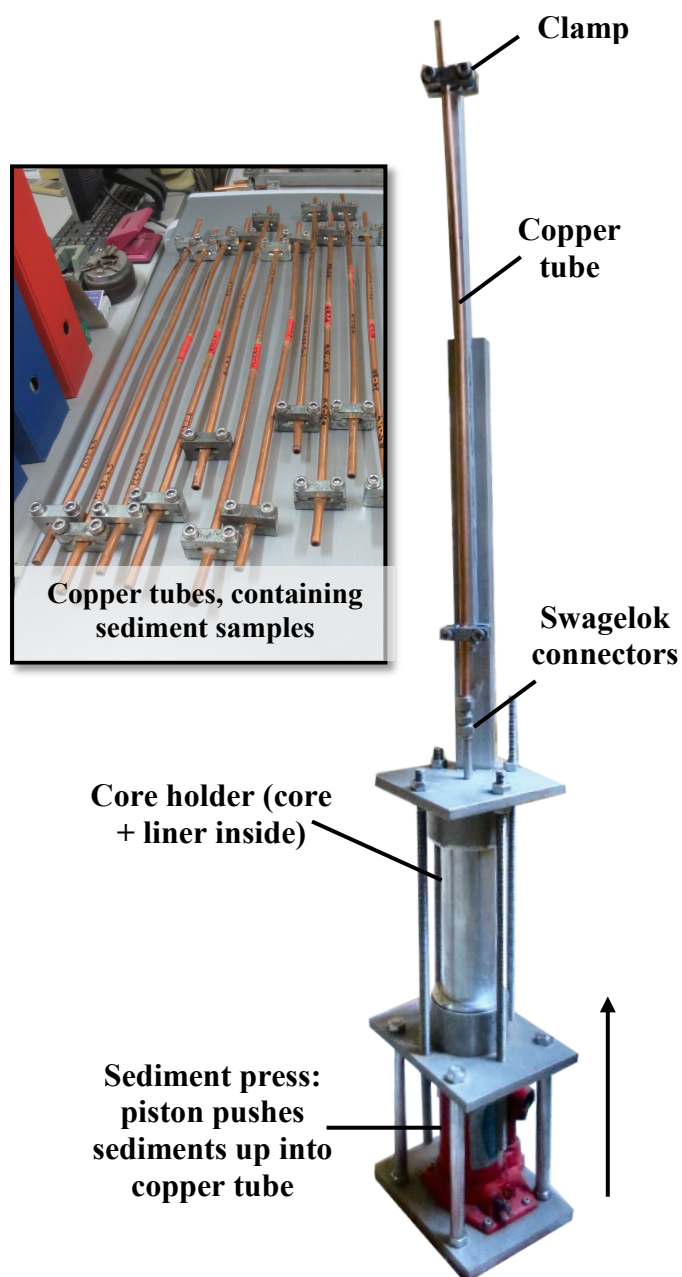


Figure S1.1: Sediment press with attached copper tube. Inset: copper tubes filled with sediments as collected in the field.

Such observations were noted from the discarded pieces of copper tubes containing sediments, during the preparation of cutting the larger 60 cm piece into smaller aliquots, and

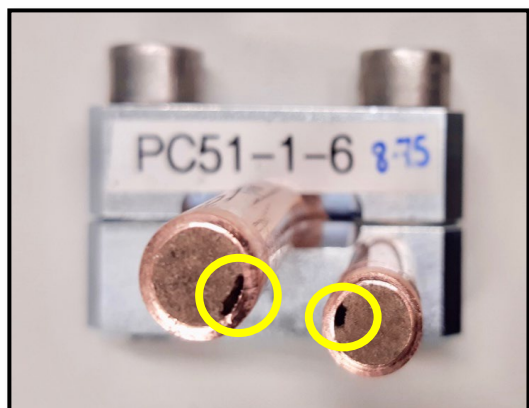


Figure S1.2: Slice through a leftover copper tube sediment sample (including clamp), showing an observable gas bubble in the sediments as circled.

prior to centrifugation. After centrifugation, a similar observation was made. For centrifugation, duplicate samples are routinely opened to find reasonably where the sediment-water interface is, and it is therefore known where to clamp the sample to be analysed such that only a water sample remains (Tomonaga et al., 2011). When opening the duplicate samples for those aliquots in cluster U, however, a sediment-gaseous interface was observed alongside only minute amounts of water or

moisture. It was therefore anticipated prior to analysis, that those samples might show noble gas ratios differing to those expected in ASW (e.g., more air-like).

Increased sand content and conductivity in unsaturated layers

To support the evaluation of saturated and unsaturated layers in the investigated sediment core, we compared our data to known sand content and hydraulic conductivity data for a sediment core taken around 200 m downstream (along the transect; Eiche et al., 2008). The sand percentile and hydraulic conductivity increase in the layers we identify in our aquitard sediment core (located at the village's health clinic) as unsaturated, with a small potential offset of around 1.5 m in the upper layer (Fig. S2). The contrary is also true, in which the layers with a reduced sand content (increasing silt-clay content) and conductivity at depths between 9 - 11.5 m, coincides with a lower $^3\text{He}/^4\text{He}$ ratio, as is expected in more consolidated sediments (see also Fig. 3).

We note that the hydraulic conductivity shows also a relatively smaller increase as compared to the sand content in the upper layer. However, given that we compare our data to a core situated some 200 m away, and that the stratigraphy of the layers in this aquitard are most likely subject to some spatial variation, our results are generally in good agreement with the data from Eiche et al. (2008).

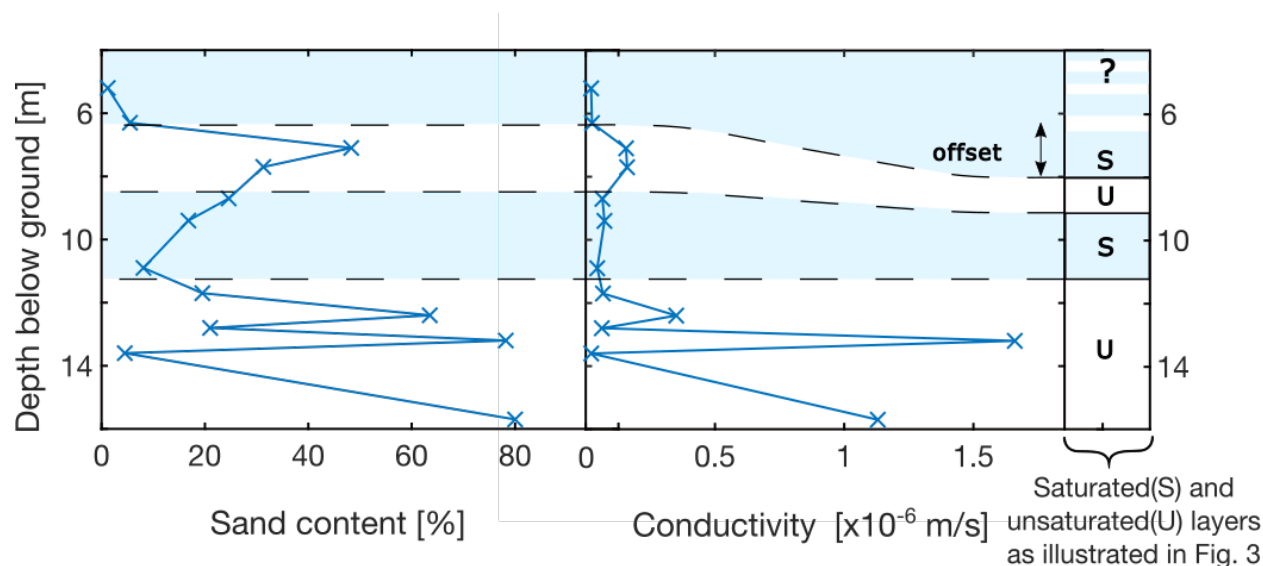


Figure S2: The sand content and hydraulic conductivity vs. depth through the aquitard of a sediment core taken around 200 m downstream from where the sediment core in our study was taken (data adapted from Eiche et al., 2008). The bar on the right indicates the U and S layers as defined from the sediment core in our study (Figs. 2, 3) and how these layers might correspond to the sand content and hydraulic conductivity data, showing also a small offset in the upper U layer.

Hydrostatic pressure and CH₄ saturation concentrations: a comparison with previous studies

Calculations from Stopelli et al. (2021) suggest an in-situ CH₄ saturation concentration in zone A of around $6.0 \times 10^{-2} \text{ cm}^3_{\text{STP}}/\text{g}$ (43 mg/L), for a well depth of 20 m and a hydraulic head at 8 m b.g.l (below ground level). In Lightfoot et al. (2022), saturation concentrations of $7.2 \times 10^{-2} \text{ cm}^3_{\text{STP}}/\text{g}$ (52 mg/L) were assumed for a well depth of 23 m and the same head depth, i.e., close to zone B. As previously described (see Results) our study suggests that the height of the overlying water column is anticipated to be reduced by approximately 4 m, leading to a reduced hydrostatic pressure of approximately 30%. Consequentially, the in-situ CH₄ saturation concentration is also lowered to around $5.2 \times 10^{-2} \text{ cm}^3_{\text{STP}}/\text{g}$ (37 mg/L) and is therefore a respective 15% and 30% lower than previous saturation concentrations applied in Stopelli et al. (2021), and Lightfoot et al. (2022). In the above calculations, the equation for the hydrostatic pressure has been applied: $P_{hyd} = \rho gh$; where ρ is the density of water (997 kg/m^3); g the acceleration due to gravity (9.8 m/s^2); and h (in m) is the height of the water column.

Aquitard pore space: further noble gas analysis

Fig. S3 illustrates a plot of the $^3\text{He}/^4\text{He}$ ratios vs. the Ne/He ratios of the aquitard pore space. A similar clustering of the data is observed as was illustrated with the Ne, Ar and Kr noble gas ratios in Fig. 2, although the clusters are not so distinct given the close proximity of the data points. For the samples situated in the unsaturated cluster (U), the respective $^3\text{He}/^4\text{He}$ ratios are close to, or above the $^3\text{He}/^4\text{He}$ ratio in air and ASW (with the exception of one sample at a depth of 11.75 m). Interestingly, the U cluster samples seem to exhibit a small accumulation

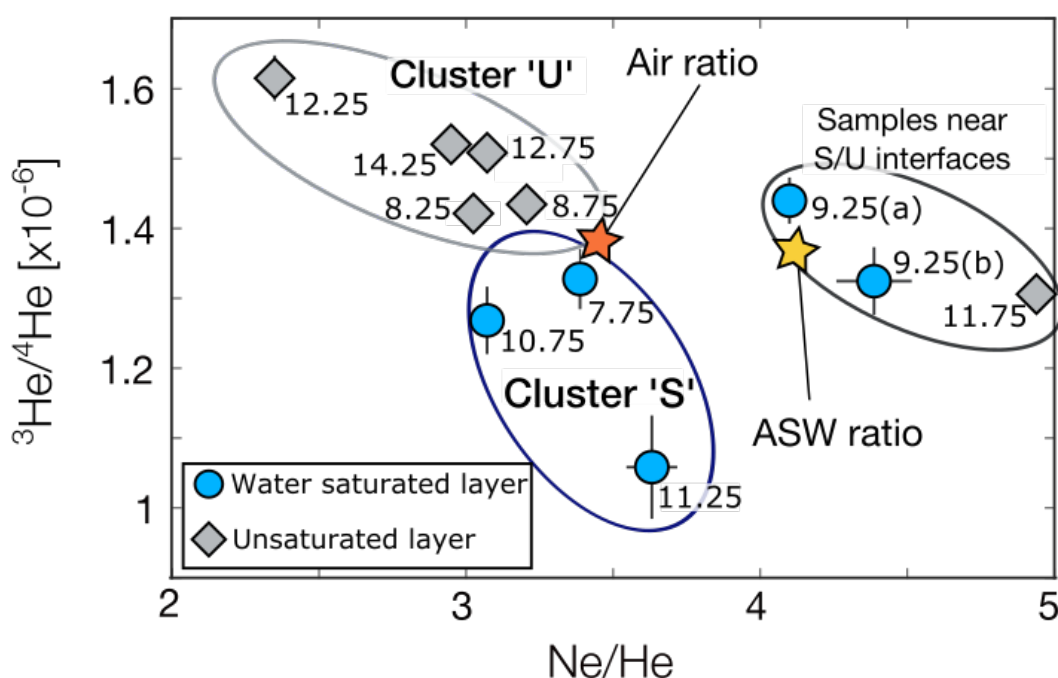


Figure S3: $^3\text{He}/^4\text{He}$ ratio vs. Ne/He ratio of the pore space samples through the aquitard. Numbers indicate sample depths in meters. Some similar groupings (as with the Ne, Ar and Kr ratio data in Fig. 3) between the saturated (S) and unsaturated (U) clusters are observed. While the U samples in general show a slightly higher $^3\text{He}/^4\text{He}$ ratio than air, several of the corresponding Ne/He ratios are actually lower than the air ratio, indicating an aged-air or gaseous phase.

of radiogenic ^4He , as observed only when considering the Ne/He ratios, which are below the Ne/He ratio of air. As such, the U cluster samples seem to exhibit aged air or gas in the unsaturated pore space.

Samples situated in the water saturated cluster (S), generally show both $^3\text{He}/^4\text{He}$ and Ne/He ratios to be lower than the respective ratios in ASW, which confirms accumulation of radiogenic ^4He . A third grouping in this data set has been established for those samples close to a S/U interface. The two samples at a depth of 9.25 m have concentrations close to ASW, as may be expected near the outer most (top) part of a confined layer, where water begins to

saturate the pore space, but ^4He has not yet had enough time to accumulate. The following sample in the saturated layer, at a depth of 10.75 m, contains considerably more radiogenic ^4He , thus lowering both the $^3\text{He}/^4\text{He}$ and Ne/He ratios.

The sample at depth 11.25 m, close to the lower S/U interface (see Fig. 3), shows a slightly higher Ne/He ratio while the $^3\text{He}/^4\text{He}$ ratio decreases, relative to the above sample at 10.75 m. This observation is somewhat conflicting. However, given the proximity of sample 11.25 (m) to the underlying unsaturated layer, these observations could be explained by hypothesizing that the rate at which He diffuses into the underlying layer (without fractionation of the He isotopes), is greater than the rate of radiogenic accumulation in the saturated layer i.e., implying a net loss of He from the saturated layer. A net loss of He to the lower unsaturated layer, is also supported by the $^3\text{He}/^4\text{He}$ ratio observed in sample at 11.75 m (see also Fig. 3), where the effective $^3\text{He}/^4\text{He}$ ratio is significantly lower than for other samples in the same unsaturated layer, thus indicating ^4He input from the above saturated layer.

Collectively, some of the unusual observations in the lighter noble gas ratios at or close to the S/U interfaces, could be attributed to both the potential radiogenic input (of He), and changes in the gas dynamics between the water and gaseous phases at those specific locations within the sediment core. Thus, the exchange of gases between the saturated and unsaturated layers seems necessary to reasonably explain some of the above observations.

Groundwater samples: comparison with a previous in-situ analysis

Fig. S4 compares the Ar and Kr data in this study, to that from our previous study in Lightfoot et al. (2022), where noble and reactive gases in the groundwater at Van Phuc was analysed in the field with a portable mass spectrometer (also called miniRUEDI; see Brennwald et al., 2016). As the online measurements from the miniRUEDI are highly robust in terms of minimising degassing artefacts on sampling, the aim in comparing data sets is to elucidate the possibility of degassing upon sampling with the copper tubes, specifically for Ar and Kr.

While there is indeed a slight divergence (as seen in the polynomial fits) between the data set taken with the miniRUEDI and the data from our study, the overall conclusions of Ar and Kr data in groundwater remains clear: atmospheric noble gases are highly depleted in response to CH_4 oversaturation and subsequent gas removal (Lightfoot et al., 2022). We are therefore

confident that the arguments presented for in-situ degassing for this aquifer, particularly in terms of Ar and Kr concentrations, are well justified.

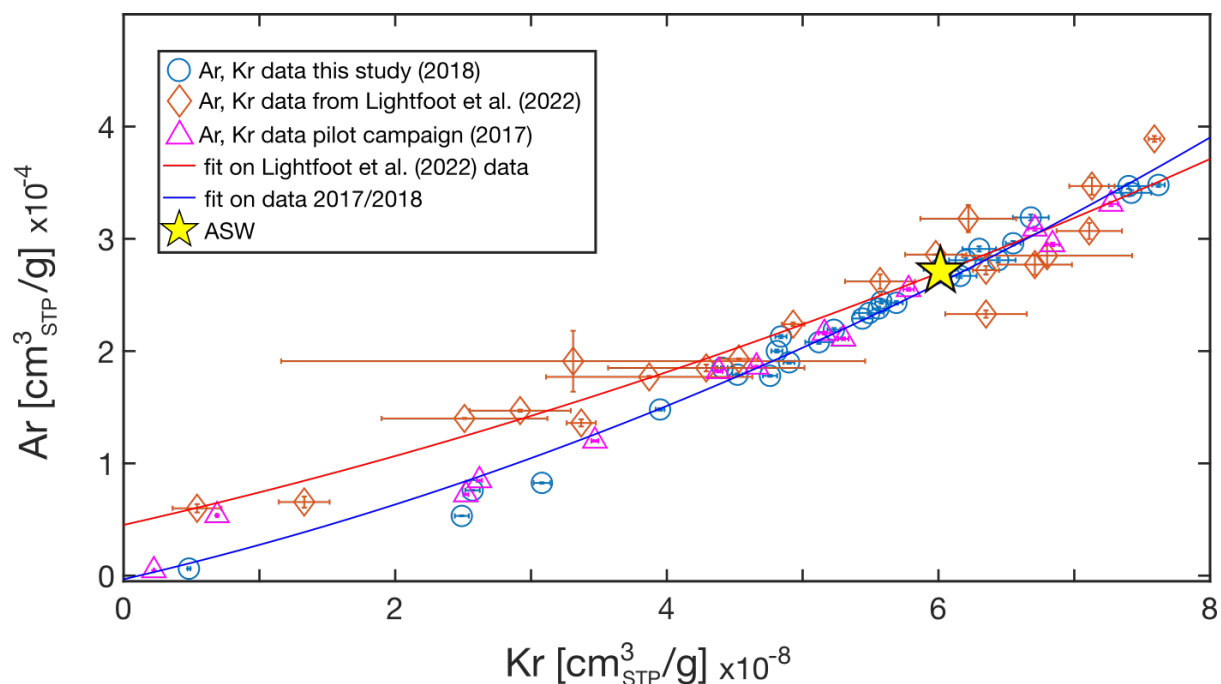


Figure S4: Comparison of Ar and Kr concentrations in groundwater at the study site (as collected in copper tubes; blue circles and pink triangles), with data from a previous study (Lightfoot et al., 2022; red diamonds), which analysed the noble gas concentrations of Ar and Kr directly in the field using a portable mass spectrometer. The year in parenthesis on the data in this study indicates sampling year, whereby some samples in copper tubes were taken from a pilot field campaign carried out in 2017. Air saturated water (ASW) is identified as a yellow star.

Groundwater: degassing trend supported by general noble gas analysis

Overall, the noble gases Ne and Kr follow a similar pattern to Ar in terms of excess and degassing of the noble gas concentrations in the wells relative to ASW concentrations (Table 3). Generally, for the degassed wells, Ne is more strongly depleted than Ar, relative to ASW concentrations, while Kr is slightly less depleted than Ar. This discrepancy from degassing is anticipated due to the differences in diffusion coefficients for the different noble gas species (i.e., lower diffusion coefficients for heavier gas species).

Unfortunately, Xe concentrations were only obtained for a few of the samples, due to experimental problems during analysis. However, for those samples successfully analysed, Xe concentrations are in general agreement with the noble gas patterns throughout the transect e.g., an excess air component is observed in well 5, while well 3b shows clear signs of depletion relative to ASW concentrations. He concentrations were also measured; however, since the Ne, Ar and Kr data in general offer a more straightforward interpretation of excess air and depletion

in response to degassing (as they do not require disentangling of a potentially significant radiogenic component on both ^3He and ^4He isotopes), only those atmospheric noble gas components are here considered.

DATA TABLES

Table 1. Concentrations of noble gases in air (in ppm), and in air saturated water (ASW) at the local prevailing conditions: $T = 24^{\circ}\text{C}$, $S = 0.1 \text{ g/kg}$, altitude = 10 masl. Solubility data to calculate ASW for He, Ne and Ar are taken from Weiss (1970, 1971) and for Kr, solubility data from Weiss & Kyser (1978), was applied. For Xe, solubility data is taken from Clever et al. (1979)

Standard concentrations	$^3\text{He}/^4\text{He}$ ratio (no units)	He	Ne	Ar	Kr	Xe
Air [ppm]	1.38×10^{-6}	5.2	18	9340	1.1	0.09
ASW [$\text{cm}^3_{\text{STP}}/\text{g}$]	1.36×10^{-6}	4.32×10^{-8}	1.76×10^{-7}	2.82×10^{-4}	6.18×10^{-8}	8.26×10^{-9}

Table 2.1. Sediment porewater amounts in cm^3_{STP} . Amounts as opposed to concentrations are quoted due to an incomplete separation between the sediments and water (or gas) phase upon centrifugation. As a consequence, only noble gas ratios should be applied for data interpretation. Notably, gas amounts are generally higher in the unsaturated layers than in the water saturated layers. The error on each value is given in parenthesis following the obtained result and applied to the least significant digit(s) e.g., $0.65(1) = (0.65 \pm 0.01) \times 10^{-7} \text{ cm}^3_{\text{STP}}$. Over-all, standard 1σ errors for all individual gases are $< 3\%$, while for the $^3\text{He}/^4\text{He}$ ratio the error is $< 7\%$. Errors here tend to be higher than for the usual analysis of water samples, namely because of the small amount of gas measured.

Depth [m]	S/U	$^3\text{He}/^4\text{He}$ ratio $\times 10^{-6}$	He $\times 10^{-7} \text{ cm}^3_{\text{STP}}$	Ne $\times 10^{-7} \text{ cm}^3_{\text{STP}}$	Ar $\times 10^{-4} \text{ cm}^3_{\text{STP}}$	Kr $\times 10^{-8} \text{ cm}^3_{\text{STP}}$	Xe $\times 10^{-8} \text{ cm}^3_{\text{STP}}$
7.75	S	1.33(4)	0.65(1)	2.21(2)	1.68(1)	2.96(3)	0.36(7)
8.25	U	1.42(2)	8.08(3)	24.45(20)	12.71(3)	14.13(12)	1.03(2)
8.75	U	1.43(2)	10.47(5)	33.58(27)	18.67(4)	22.29(17)	1.69(2)
9.25(a)	S	1.44(3)	1.09(1)	4.46(4)	6.38(1)	11.99(10)	1.37(3)
9.25(b)	S	1.32(5)	0.75(1)	3.28(8)	5.56(2)	10.80(10)	1.22(2)
10.75	S	1.27(5)	0.41(1)	1.26(1)	1.60(1)	3.09(3)	0.41(1)
11.25	S	1.06(7)	0.15(1)	0.55(1)	1.38(1)	3.41(4)	0.50(1)
11.75	U	1.31(3)	2.73(7)	13.49(11)	8.11(2)	9.79(8)	0.70(1)
12.25	U	1.62(3)	19.38(7)	45.50(36)	23.70(5)	28.11(23)	2.16(3)
12.75	U	1.51(2)	12.85(5)	39.44(32)	20.88(4)	24.33(20)	1.85(3)
14.25	U	1.52(2)	6.90(3)	20.36(16)	11.40(2)	13.61(12)	1.12(2)

Table 2.2. Sediment porewater or pore-gas ratios. Comparing $^{20}\text{Ne}/^{22}\text{Ne}$ ratios to $^3\text{He}/^4\text{He}$ ratios confirm radiogenic and tritogenic components: $^{20}\text{Ne}/^{22}\text{Ne}$ ratios have a maximum deviation from ASW or air values by -3.6% and +2%, respectively, whereas for the $^3\text{He}/^4\text{He}$ ratios, this deviation is -22% and +15%. Such large deviations from ASW and air ratios in the He ratios, confirm the presence of a radiogenic component. Nevertheless, $^{20}\text{Ne}/^{22}\text{Ne}$ ratios do deviate more than might be expected from their air and ASW ratios, which is most likely a result of the rather small initial gas amounts measured (see table 2.1). Ne/Kr, Ar/Kr and Xe/Kr all clearly indicate fractionation change between the S and U layers. Ne/He ratios have the additional influence of radiogenic ^4He , as described in the SI, Fig. S3.

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Depth [m]	S/U	$^{20}\text{Ne}/^{22}\text{Ne}$ ratio*	Ne/He	Ne/Kr	Ar/Kr $\times 10^3$	Xe/Kr $\times 10^{-2}$
7.75	S	9.74(4)	3.39(3)	7.46(9)	5.67(5)	12.01(22)
8.25	U	9.98(1)	3.02(3)	17.30(20)	8.99(8)	7.27(16)
8.75	U	9.88(1)	3.21(3)	15.07(17)	8.38(7)	7.60(12)
9.25(a)	S	9.64(4)	4.10(4)	3.72(4)	5.32(5)	11.39(26)
9.25(b)	S	9.77(2)	4.4(12)	3.04(8)	5.15(6)	11.29(21)
10.75	S	9.70(4)	3.07(3)	4.08(5)	5.18(6)	13.32(37)
11.25	S	9.43(10)	3.63(7)	1.62(2)	4.05(4)	14.71(32)
11.75	U	9.78(1)	4.94(4)	13.78(16)	8.28(7)	7.13(11)
12.25	U	9.98(1)	2.35(2)	16.19(18)	8.43(7)	7.67(12)
12.75	U	9.91(1)	3.07(3)	16.21(19)	8.58(7)	7.61(13)
14.25	U	9.89(1)	2.95(3)	14.96(18)	8.38(8)	8.24(18)

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* $^{20}\text{Ne}/^{22}\text{Ne}$ ASW = 9.78; in air = 9.80. Additional ratios can be determined from Table 1 (also see Figs. 2 and S3).

Table 3. Sampling (well) coordinates are given alongside the original well name and noble gas concentrations in groundwater. Notably, Xe data is missing for some of the samples due to experimental problems during analysis. The error on each value is given in parenthesis following the obtained result and applied to the least significant digit(s) e.g., 6.21(12) = (6.21 ± 0.12) x10⁻⁸ cm³_{STP}/g. Standard 1σ errors for individual gases are < 2% for Ar, Ne, Kr and Xe. The error on ³H measurements is generally < 15%, with a few outliers as discussed in the caption of Fig. 5. CH₄ results are from our publication of Stopelli et al., 2021. Tritium concentrations quoted are at the time of sample collection.

Simplified well name	Original well name	Depth to screen ±1.5 m	Location Lat/Long DMS	Ne x10 ⁻⁷ cm ³ _{STP} /g	Ar x10 ⁻⁴ cm ³ _{STP} /g	Kr x10 ⁻⁸ cm ³ _{STP} /g	Xe x10 ⁻⁹ cm ³ _{STP} /g	³ H TU	CH ₄ mg/L
1	AMS12	24	20°54'50.9" N 105°54'21.0" E	1.66(3)	2.81(2)	6.21(12)	-	1.34(5)	<1
2a	VP56	20	20°54'57.9" N 105° 54'11.9" E	0.87(1)	2.00(2)	4.81(4)	7.24(8)	1.17(4)	13
2b	VP57	30	20°54'57.9" N 105° 54'11.9" E	1.20(1)	2.67(2)	6.08(5)	6.53(7)	1.29(4)	10
2c	VP58	40	20°54'57.9" N 105° 54'11.9" E	1.21(1)	2.44(2)	5.58(5)	7.62(10)	1.18(4)	6.9
3a	PC51	20	20°55'04.1" N 105°53'54.3" E	0.18(1)	0.53(1)	2.49(5)	-	1.64(11)	40
3b	PC52	28	20°55'04.1" N 105°53'54.3" E	0.49(1)	1.48(1)	3.95(3)	5.24(6)	1.16(5)	48
3c	VP55	40	20°55'04.1" N 105°53'54.3" E	1.13(1)	2.38(2)	5.56(5)	7.78(9)	1.45(8)	21
4	Household#2	45	20°55'08.6" N 105°53'50.7" E	1.13(2)	2.34(2)	5.49(11)	-	0.93(27)	-
5	VP59 [†]	20	20°55'15.0" N 105°53'46.2" E	1.99(1)	3.48(2)	7.62(5)	10.01(9)	1.07(8)	11
6	VPNS3	27	20°55'14.9" N 105°53'46.1" E	2.17(3)	3.19(3)	6.68(13)	-	1.08(16)	2.3
7a	AMS5	24	20°55'17.4" N 105°53'41.7" E	0.037(1)	0.062(1)	0.48(1)	-	1.09(12)	58
7b	VPNS5	35	20°55'17.3" N 105°53'41.8" E	2.15(3)	3.47(2)	7.40(15)	-	1.81(12)	6.1
8a	AMS11-25	24	20°55'18.4" N 105°53'38.3" E	0.248(4)	0.76(1)	2.57(5)	-	1.43(18)	37
8b	AMS11-32	33	20°55'18.4" N 105°53'38.3" E	1.38(2)	2.81(2)	6.44(13)	-	1.00(5)	<1
8c	AMS11-47	48	20°55'18.4" N 105°53'38.3" E	1.70(3)	2.91(2)	6.30(12)	-	0.13(14) [‡]	<1
9	PC44	38	20°55'18.5" N 105°53'38.2" E	1.83(3)	3.41(2)	7.42(15)	-	0.52(4)	<1
10	AMS31	25	20°55'18.5" N 105°53'38.2" E	0.87(1)	2.08(2)	5.12(10)	-	1.28(11)	16
11	PC43	28	20°55'18.7" N 105°53'38.2" E	1.14(1)	2.43(2)	5.69(5)	8.04(9)	1.19(4)	14
12	AMS32 [†]	25	20°55'18.9" N 105°53'37.6" E	0.56(3)	1.78(1)	4.76(5)	6.90(7)	1.70(5)	19
13	AMS36	27	20°55'19.6" N 105°53'37.6" E	1.39(2)	2.67(2)	6.16(12)	-	0.94(21)	<1
14a	AMS4	23	20°55'19.38" N 105°53'36.17" E	1.51(2)	2.73(2)	6.01(12)	-	1.21(19)	<1
14b	VPNS4	38	20°55'18.9" N 105°53'36.7" E	1.77(2)	2.96(2)	6.55(6)	8.69(10)	0.73(45)	<1
15a	VPMLA-22 [†]	21	20°55'23.7" N 105°53'31.1" E	1.05(1)	2.29(1)	5.44(8)	-	0.06(15)	<1
15b	VPMLA-37	37	20°55'23.7" N 105°53'31.1" E	0.91(1)	1.85(1)	4.41(4)	6.67(7)	0.02(3)	<1
15c	VPMLA-52	53	20°55'23.7" N 105°53'31.1" E	1.25(1)	2.13(1)	4.84(4)	6.88(8)	0.03(3)	<1
#1	Household#1	45	20°55'08.3" N 105°54'07.6" E	0.71(1)	1.79(1)	4.52(4)	7.25(8)	-	<1
#2	AMS15	24	20°55'35.8" N 105°53'51.7" E	0.78(1)	1.90(1)	4.90(4)	7.32(8)	0.13(4)	<1
#3	AMS13	24	20°54'39.4" N 105°53'36.7" E	0.21(1)	0.83(1)	3.08(6)	-	1.10(13)	3

[†]Concentrations are an average of two duplicate samples.

[‡] Tritium concentration result is from a sample taken in a pilot campaign 1 year earlier, 2017.

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