A review of sampling and monitoring protocols related to radioactive elements in fractured rock aquifers

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An analysis of current available sampling and monitoring protocols related to radioactivity has been undertaken. The international best practice is outlined in order to better understand the available sampling as well as monitoring protocols related to radioactivity. Thereafter, the most relevant South African groundwater sampling and monitoring protocols are examined for their application to the subject matter. This piece of work highlights the need for more sampling and monitoring protocols related to fractured rock media in general and radioactivity in fractured rock media in particular.

Introduction

Groundwater monitoring can be defined as the scientifically-designed, continuing measurement and observation of the groundwater situation.[1] Ideally the design of network density and sampling frequency would be based on an optimisation of the cost of monitoring and of the accuracy of collected and derived data related to the objectives of the network.[2] In line with this, Netili *et al.* further propose that groundwater monitoring and sampling sites should be selected to be representative of geographic distribution, geology, groundwater use, land use and groundwater flow regimes, amongst other factors.[3]

Thus we can see that, ideally, the sampling programme for a groundwater investigation will collect the minimum number of samples required to have adequate three-dimensional spatial and stratigraphic coverage of the area being investigated. So, the fundamental task is to obtain samples that are representative, diagnostic and characteristic of the aquifer and to analyse them with minimal change in composition.[2] The data stemming from this knowledge should in turn lead to better groundwater management practices.

To effectively monitor and assess the radioactivity of uranium and its daughter elements in the groundwater, concentration analysis is often employed in the laboratory. This method requires appropriate *in situ* groundwater sampling, which can be influenced by device, selection of sampling network, quality and quantity of water sampled etc. Unfortunately, there is not yet a uniform groundwater sampling guideline which can be applied to the areas dominated by fractured rocks. Particularly, a single sampling manual and monitoring protocol is not available for the research of radioactive elements in fractured rocks.[1]

Background

Radioactivity sampling has normally been conducted in a similar fashion to sampling for heavy metals.[4] Fetter[5] argues that some of these radioactive elements behave in a similar manner to these heavy metals. Therefore it is justified to extract water samples utilising the same methodology.

In most cases a known area with uranium mineralisation is targeted for sampling. Thereafter, liaison with the laboratory is done in order to determine the volumes of sample required for analysis as well as the reagents, bottle types and storage and transport methods required in order to maintain sample integrity. Furthermore, initial work prior to sampling also includes the analysis of previous work completed in the area in order to determine the available data, data quality, gaps in data and well location amongst other factors. One of the most important factors is the nature of the sub-surface media, which will also be determined from desktop studies.

In primary porous media one is able to use multiple sampling methods due to the relatively uniform nature of the aquifer material. These methods include, but are not limited to:

- bailer (elongated plastic cylinder with a ball valve for containing the sample);
- groundwater pump (this could be attached or a mobile device);
- depth specific sampler (bailer with a control valve at the surface);
- windmill (at the end pipe a sample is normally taken).

Cook[6] has shown that, in fractured media, the spatial variability as well as the hydraulic conductivity can vary substantially. This is due to the fact that the fractures are isolated and not necessarily always water bearing or even interconnected (Figure 1).This poses problems for aquifer characterisation as well as groundwater sampling. Durrani and Ilic[7] have also stated that radioactive elements precipitate on fracture walls. The elements which precipitate depend on the pH, Eh as well as temperature of the groundwater, as shown by Ilani *et al.*[8] This means that spatial and temporal variability of radioactive elements in fractured rock aquifers is evident.[7] This poses an added complication to sampling for radioactivity in fractured rock media due to the temporal and spatial variability of the elements of interest in the groundwater sample.

Methodology

An online search was conducted for sampling and monitoring protocols developed all over the world. This search was then further refined in order to only include those manuals examining radioactivity and heavy metals. These manuals were intensively studied and the evolution of the sampling science within this specific field also analysed. Case studies related to sampling of radioactive elements in fractured rock media were also studied. These provided insights into applicable sampling methodologies and external factors to examine. These were all compared and best practice was examined for the specific application.

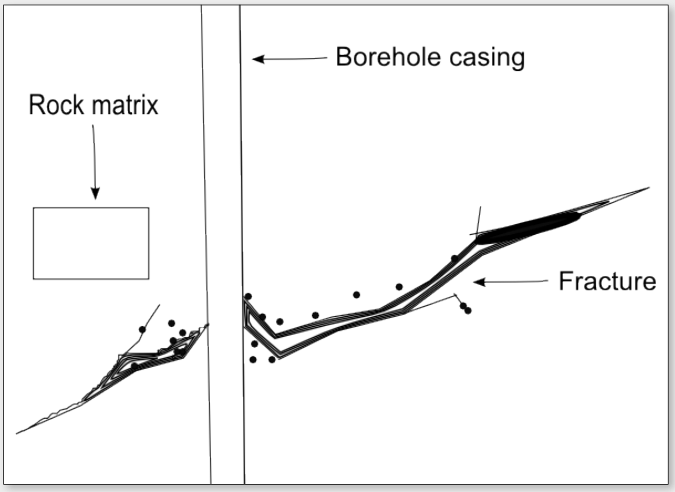

Figure 1. A typical example of a well intersecting a fracture, with alterations in the immediate vicinity of the fracture indicated by circular filled dots.

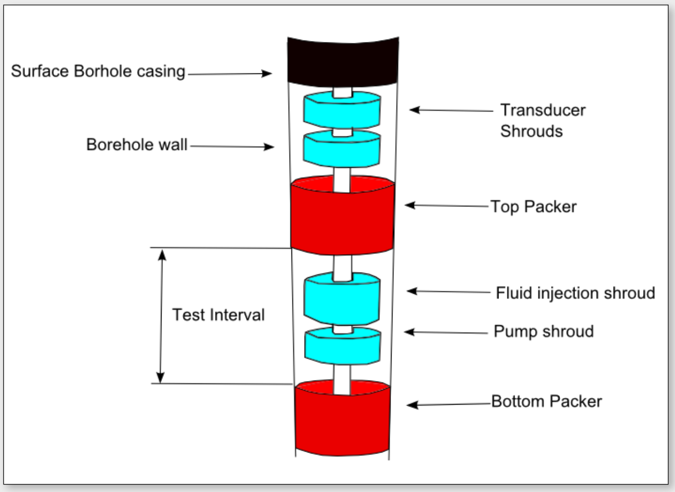

Figure 2. Multifunction BAT[3] in a bedrock borehole with borehole packers inflated to seal against the borehole wall (adapted from Reference 13).

International perspective

Jousma and Roelofson[1] have reviewed approximately 400 documents relating to sampling and monitoring groundwater and related aspects. The authors have not found a single document relating to radioactivity sampling in fractured rock aquifers. Furthermore, it was recommended that more research in hard rock aquifers is required.[1]

Despite the aforementioned point, the IAEA[9] have developed a manual specifically for radioactive monitoring of near surface waste facilities. Unfortunately, the manual does not deal with the intricacies of sampling, but instead refers the reader to various other manuals. One of these manuals was most probably the first groundwater sampling manual and was developed by Barcelona *et al.*[10] This document outlines a specific route to follow when sampling:

- the device should be simple to operate to minimise the possibility of operator error;
- the device should be rugged, portable, cleanable and repairable in the field;
- the device should have good flow controllability to permit low flow rates (=100 mL min^{-1}) for sampling volatile chemical constituents, as well as high flow rates (>1 L min^{-1}) for large-volume samples and for purging stored water from monitoring wells;
- the mechanism should minimise the physical and chemical disturbance of groundwater solution composition in order to avoid bias or imprecision in analytical results.

Freyer *et al.*[11] concur with the aforementioned recommendations. The authors have also shown that the low flow sampling devices, which are used for radon sampling, do not greatly affect the radon concentration. The low flow sampling devices used in this study were a membrane pump, a submersible pump and a bailer for purposes of comparing the effect of various instruments on degassing. Therefore, these devices are all aptly suited for sampling and fit the criteria previously mentioned by Barcelona *et al.*[10]

Furthermore, Barcelona *et al.*[10] systematically outline a general sampling protocol which could be used for any analyte which may be of major concern. The steps, goals and recommendations are shown in a tabular format in order to minimise confusion and simply explain the specifics relating to each systematic step (Table 1).

The EPA[12] also penned a protocol in a similar fashion to that of Barcelona *et al.*[10] The greatest attention was afforded to the physical aspects of groundwater flow and monitoring well design. Interestingly enough the use of packers is advocated in order to isolate a specific area of interest within the sub-surface.[12] These inflatable devices are placed above and below the fracture of interest, in order to isolate the area (Figure 2). Prior to this a pump is isolated within the structure. Shapiro[13] has designed the BAT3 (Bedrock Aquifer Transportable Testing Tool) specifically for sampling in fractured rock aquifers. Besides having packers and a pump it is also installed with three pressure transducers. One is located above the packers, one between the packers and the last is below the packers. These are utilised in order to monitor fluid pressure and correctly ensure that the packers are properly isolating the fracture of interest.[13]

Puls and Barcelona[14] strongly recommend that low flow sampling, in conjunction with packers, should be done in fractured rock aquifers. This approach should only be attempted after identifying the water bearing fractures and thus the sampling zone can be isolated.

EPA[12] promotes the hourly sampling of fractured aquifers for field determinands. This protocol was developed specifically for nuclear waste facilities and the parameters which would be measured on an hourly basis would include those which a data logger could determine. These include temperature, TDS and water level. This would aid in determining whether leakage has occurred from the storage facility, and also aid in determining anomalous inflows of contaminants in groundwater in a natural setting. The aforementioned could be inferred from a fluctuation in TDS, pH and temperature. It is an effective monitoring strategy and the aforementioned parameters would act as indicators for the contamination of groundwater.

OHIO EPA[15] have also developed a document specifically for groundwater sampling and monitoring. Once again there is not much difference between this technical manual and that of Barcelona *et al.*[10] and EPA.[12] An interesting component is the description of the use of statistics in order to assimilate data into information. Helsel

Table 1. Generalised groundwater sampling protocol (adapted from Reference 10).

Step	Goal	Recommendations
Hydrologic measurements	Establishment of static water level	Measure water level to approximately 0.3 cm (0.01 ft)
Well purging	Removal of stagnant water which would otherwise bias representative sample	Pump water until well purging parameters (e.g. pH, T, Eh) stabilise to approximately 10% over at least two successive well volumes pumped
Sample collection	Collection of samples at land surface and or in well-bore with minimal disturbance of sample chemistry	Pumping rates should be limited about 100 mL min⁻1 for volatile organics and gas sensitive parameters
Filtration/preservation	Filtration permits determination of soluble constituents and is a form of preservation. It should be done in the field as soon as possible after collection	Filter trace metals, inorganic anions/cations. Do not filter: TOC, TOX, volatile organic compound samples. Filter other organic compounds samples only when required
Field determinands	Field analysis of samples will effectively avoid bias in determination for parameters/constituents which do not store well, e.g. gases, alkalinity, pH etc.	Samples for determination of gases, alkalinity and pH should be analysed in the field if it all possible
Field blanks	These blanks and standards will permit the correction of analytical results for changes which may occur after sample collection, preservation and storage	At least one blank and one standard for each sensitive parameter should be made in the field on each day of sampling. Spiked samples are also recommended for good QA/QC
Sample storage	Refrigeration and protection of samples should minimise the chemical alteration of samples prior to analysis	Observe maximum sample holding or storage periods recommended by the Agency. Documentation of actual holding periods should be carefully performed

and Hirsch[16] have also shown the importance of utilising statistics as a tool for the interpretation of data. The document acts as a reference tool for hydrologists in the USGS and provides the basic, as well as advanced, statistical methods applicable to the hydrological sciences.[16]

OHIO EPA[17] have made the concerted effort to update their document, unlike Barcelona *et al.*[10] and EPA.[12] Specific chapters have been modified and/or added in order to make the manual more relevant. An extensive examination of sampling methodology was revisited and could prove to be useful, especially for the novice, due to its simplicity and applicability. Unfortunately, radioactivity is not focused upon and thus sampling protocol for radioactive elements is not covered. This must be due to the fact that OHIO EPA[17] was heading for a more generic sampling methodology and nothing specific was included in this updated version.

The USACE[18] has developed an engineering and design manual entitled *Monitoring Well Design, Installation, and Documentation at Hazardous, Toxic and Radioactive Waste Sites*. The manual unfortunately does not cover aspects of groundwater sampling. Once again this is not in line with the purpose of the document. Instead the purpose of the engineer manual is to provide the minimum elements for consideration in the design, installation and documentation of monitoring well placement and other geotechnical activities at projects known or suspected to contain chemically hazardous, toxic and/or radioactive waste.[18]

Wilde *et al.*,[4] on the other hand, have turned some attention towards the sampling of radioactivity. They suggest that radioactive elements should be sampled in a similar manner to heavy metals. This is a view shared by Weaver *et al.*[19] as well as Smedley *et al.*[20] Wilde *et al.*[4] suggest that a 1-litre polyethylene bottle be acid rinsed and then the sample should be preserved to $pH<2$ using HNO_3. It would also mean filtering the sample in order to remove suspended particles which could possibly lead to the precipitation of metals onto its surface. This manual is a major step forward, in terms of radioactivity sampling. We also find that each chapter of the manual is published separately and updated on a periodic basis.[4] Furthermore, corrections are posted on the website and these additions should be made to the respective chapters. This USGS manual is also quite generic and provides methods for surface as well as groundwater sampling.

Yeskis and Zavala[21] tackled methods of sampling as well as equipment and recommend low flow sampling, just like Puls and Barcelona.[14] This approach is justified because samples with elevated levels of turbidity are collected by high speed pumping. This results in the inclusion of otherwise immobile particles which cause an overestimation of specific analytes of interest.[14] Furthermore, with regards to radioactivity, we find that once there is a change in chemical environment there is also an alteration in the dominant radionuclide in the aqueous phase.[8] A good example of this is the fact that uranium dominates under oxidising conditions whereas radium prefers a reducing environment.[22] Thus Yeskis and Zavala[21] also advocate filtering, in order to differentiate between dissolved and non-dissolved species, therefore eliminating adsorbed radioactive particles.

DOE[23] as well as IAEA[9] developed a monitoring protocol for radioactive waste facilities. Aspects of monitoring network design, well placement and data management

Table 2. Comparison between sampling manuals and their relevance to radioactivity sampling. X—indicates that the topic is covered in the document; a blank cell shows that the topic was omitted from the document.

Reference	General sampling protocol	General sampling methods	General sample treatment	Monitoring protocol	Monitoring well design	Sampling frequency	Radioactivity sampling
4	X	X	X				X
9				X			X
10	X	X	X		X	X	
12	X	X			X	X	
15	X	X	X		X		
18		X			X	X	
19	X	X	X				X
21	X	X	X				
23				X			
24				X		X	

are examined.[23] IAEA[9] instead look at the monitoring of general environmental conditions, which includes soil, air, hydrology and hydrogeology. Both manuals are geared towards facilities management and DOE[23] provides a good systematic monitoring protocol. IAEA[9] on the other hand make many references to various other documents and is not as user friendly as DOE.[23]

Thus we see the natural progression of sampling manuals. This evolution involved a step towards the intensive examination of the physico–chemical parameters, as shown by Weaver *et al.*[19] Furthermore, we find specific manuals becoming all-encompassing guides and thus the need to constantly update as the knowledge base is widened, as in the case of Wilde *et al.*[4] and OHIO EPA.[15] It can also be seen that certain manuals, such as Jousma's guideline[24] on groundwater monitoring for general reference purposes, are very specific in their subject matter. Therefore, a simple comparison of the manuals examined in this review has been completed (Table 2). This was based on headings or sections in the document. Therefore if no section or subsection on the topic was present then the topic was regarded to be insufficiently covered or omitted.

A South African perspective

The most comprehensive groundwater sampling guide in South Africa at the moment is the second edition of *Groundwater Sampling*.[19] This manual outlines every aspect of sampling and even highlights what could go wrong. It is a practical approach to sampling and gives the user a systematic check list for field sampling.

Weaver *et al.*[19] have shown that prior to sampling it is necessary to liaise with the laboratory in order to ascertain which containers, preservatives and reagents are to be used when sampling for radionuclides. Levin,[25] who also developed a local sampling manual, states that sample bottles should be thoroughly rinsed with 10% HCl and then emptied and rinsed thrice with deionised water. Levin also states that the samples should be taken as follows:[25]

- 2 L for the determination of the trace elements such as Al, As, Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Ti, Si, Zn;
- 500 mL for the determination of U, V and NO_3^-;
- 250 mL for the determination of the major components SO_4^{2-}, Cl^-, F^-, Na^+, K^+, Ca^{2+} and Mg^{2+}.

These aforementioned authors definitely took cognisance of the fact that the trace metal content of water could be altered in storage. Therefore, the use of acid has been recommended in order to reduce the possibility of precipitation of heavy metals, which includes radioactive elements. Taking cognisance of the fact that radioactive elements are heavy metals one then has to filter the sample, once it has been extracted from the aquifer.[25] Typically a 0.45 µm filter paper is utilised. Levin also suggests that the filter paper should be kept, if the suspended particles are to be analysed.[25] This is important, taking into consideration that radium precipitates under oxidising conditions.[8] Other than these specific methods, relating to sampling for radioactivity, we find that all other aspects are completely generic within these two locally developed manuals.

Weaver *et al.* advise that approximately two well volumes should be purged in order to remove stagnant water.[19] Levin on the other hand says that the pump should be run for 10 minutes before a sample is taken.[25] Prior to this a water level should be measured. Furthermore, it is suggested that field parameters be measured *in situ*.[19,25] These include temperature, total dissolved solutes, electrical conductivity, pH, Eh and oxidation reduction potential. This is done for the following reasons:[19]

- to check the efficiency of purging;
- to obtain reliable values of those determinands that will change in the bottles during transport to the laboratory;
- to obtain some values that may be needed to decide on the procedure or sampling sequence immediately during the sampling run.

Weaver *et al.* also advise the use of a flow-through cell in order to maintain the *in situ* condition of the sample and thus gain an actual representation of the conditions in the sub-surface.[19]

Vogel *et al.* were visionary in their use of packers for sampling in the Beaufort West area.[26] Even though their study is not strictly classified as a protocol, it is interesting to take note of the methods used. A submersible pump mounted between two inflatable rubber packers, approximately 1.8 m apart was utilised.[26] This equipment allowed multi-level sampling within boreholes. At the depth of interest the packers were inflated with nitrogen and the pump then delivered water to the surface from the aforementioned fracture.

Future outlook

Unfortunately, there is not yet a uniform groundwater sampling guideline which can be applied to areas dominated by fractured rocks. Particularly, no single sampling manual and monitoring protocol is available for the effective sampling of radioactive elements in fractured rocks.[1] Xu *et al.* have recently developed a pre-cursor to such a document,[27] specifically for a South African context in fractured rock aquifers. This protocol formed part of a larger study funded by the Water Research Commission (WRC) of South Africa related to uranium in groundwater.

In the interim, the use of the same sampling protocol as for heavy metals has been employed in most instances. This is common due to the nature of the radioisotopes being similar to heavy metals.[5]

Case studies such as Yucca Mountain in North America as well as the Nagra project in the Swiss Alps are good examples of multi- and interdisciplinary work in order to understand fractured rock aquifers and the unsaturated fractured zone. These are not manuals or protocols in the strictest sense of the definition according to Jousma.[24] They do, however, provide a blueprint for similar studies in order for a complete site characterisation and an understanding of sub-surface processes at various scales.

Further research is required as well as the large scale implementation of the developed protocol in order to ascertain the applicability thereof. Thus the continued understanding of hard rock aquifers could be fostered. This is of the utmost importance if the scientific community is to continue to advance and solve problems such as water supply from these saturated geological units.

The data stemming from the studies should be put to good use and aid in the development of an effective monitoring programme. It is useless if the data is not utilised to its maximum capacity.[2] This can only be done if statistical analysis is brought into play. Also an effective database management system would be needed in order to maximise the use of data.

One major factor which needs to be included in future sampling manuals is the contextualisation of the work within the framework of the Theory of Sampling (TOS). It seems as if most major works have merely just looked at best practice in line with the latest knowledge of the contaminant to be sampled. These manuals have not included the science of sampling or looked at aspects of representative, unbiased sampling, as outlined by Petersen *et al.*[28] This is especially true in water science as hydrologists are infamous for grab samples. Furthermore, the fact that measurement uncertainty needs to be further examined will aid in alleviating issues around data quality.

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