

## 4. Materials and methods

### 4.1 Site selection and sampling design

The Harties 1-4 complex is located just south of Stilfontein and New Machavie is 22 kilometres west of Potchefstroom. Harties 1-4 was part of an initial feasibility study prior to the commencement of the main research project. Work on Harties 1-4 was discontinued as the gold price increased and Harties 1-4 had a relatively high gold content (compared to other TDFs in the area). The contractors responsible for re-mining focused their efforts on Harties 1-4, resulting in revocation of access to the site, the study was subsequently moved to New Machavie.

On Harties 1-4, a transect of boreholes was drilled using a hydraulic auger with a spiral drill bit (Photo 4.1) at 20 metre intervals from the northern day-wall to the penstock drain as shown in Figure 4.1.



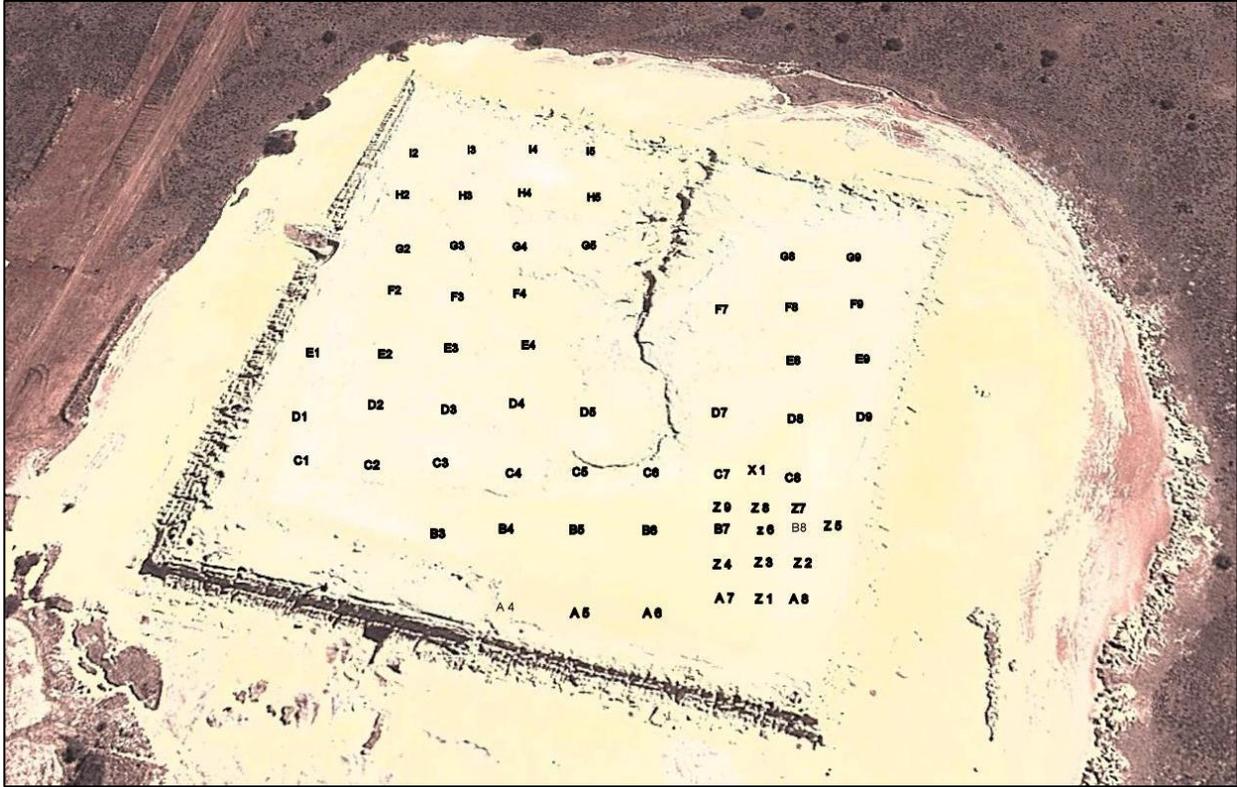
Figure 4.1: A Transect on Harties 1-4 (Google Earth C, 2013)

The Harties 1-4 TDF was constructed using an open-end paddock/day-wall deposition method which allowed differential settlement of particle sizes from the wall to the pool (Robertson, 2008). It has been suggested that radionuclides are associated with smaller particle fractions and thus one would expect an increase towards the pool of the TDF where the finest fraction was deposited (Pulford, 2010). Each borehole was drilled to a depth of 6 m with the same custom-made hydraulic auger with spiral drill rods. A composite sample was taken for each metre.



**Photo 4.1: Hydraulic auger**

New Machavie was drilled following a grid pattern with 60 m intervals over the whole TDF which was decreased to 30 and 15 m intervals on the eastern half. Figure 4.2 below illustrates the drill locations on New Machavie TDF.



**Figure 4.2: Grid-based sampling on New Machavie (Google Earth D, 2013)**

The hydraulic auger was used with a core sampling drill bit (Photo 4.2) which allowed detailed sampling at 10 cm intervals. Boreholes were drilled to the underlying soil as far as possible; however, the eastern part of the TDF is underlain by hard rock dolomite with very little soil beneath it. As the hard rock dolomite tended to break the drill bits, the eastern part of the TDF was drilled just short of reaching soil.

Illustrated in Photo 4.2 is the core sampling procedure. Once the drill bit was extracted from the borehole, the external stainless steel casing was removed and the internal PVC sampling tube was opened. The core sample was then divided in 10 cm intervals, taking care to remove any fall-back material which may have contaminated the samples. Samples were then sealed in plastic bags for transportation and prepared for analysis as described under section 4.4.



**Photo 4.2: Core drill bit**

## **4.2 Down-hole natural gamma probing**

Initially, a natural gamma scintillometer was employed at Harties 1-4 as part of the project's feasibility study and to verify if significant variation in natural gamma radiation was detected to substantiate further research. For this reason, a transect was drilled on Harties 1-4. These boreholes were then probed using a sodium iodide natural gamma scintillometer. The detector crystal, which is doped with thallium, has a diameter of 25.4 mm and a length of 50.8 mm. It has a threshold value of 0.25 meV and was calibrated for equivalent  $U^{308}$  at the Pelindaba international calibration site. The probe itself has an outside diameter of 38 millimetres. All data were collected using AUSWIN software at 2 cm intervals at speeds less than 2 m/min. The data (cps) were then converted to ppm by stripping the spectral data using Spectra and calibrating the results using calibration constants supplied by Geotron as calibrated at the Pelindaba calibration site.

At New Machavie a down-hole natural gamma spectrometer was used with a 35 mm diameter and a 76 mm long sodium iodide crystal doped with Tl. The probe has a diameter of 48 mm. Logging speeds were kept below 2m/min to record a reading every 2 to 4 cm of the borehole. The spectrometer recorded counts in four energy windows: 400 – 3000 keV for total counts, 1360 -1560 keV for K, 1640 – 1880 keV for U and 2460 – 2760 keV for Th. After each hole, a calibration file was created using a Th reference, collecting data for 100 seconds. The data was recorded using G512 software for recording data and Spectra for data reduction, spectral alignment, calibration via the calibration file and exporting the data to an “Excel” operational format.

## **4.3 Surface natural gamma spectrometry survey**

New Machavie was surveyed using a RS 230 BGO (bismuth geranium oxide crystal) natural gamma spectrometer at 15 m grid line spacing. The spectrometer was kept at a constant height of 1 m. This meant that at any point a radius of 15 m was covered by the spectrometer and that the first 30 cm of the tailings formed part of the coverage area (Van Rensburg, 2011). The grid was completed on top of the TDF as well as around the TDF in order to relate movement of radionuclides from the tailings to the surrounding environment. The spectrometer was set to survey mode, with a setting of concurrent assays every 1 minute whilst walking the grid. Care was taken to keep walking speed constant to produce equally-spaced data points.

## **4.4 Laboratory natural gamma assaying of samples**

Samples taken from boreholes were air dried and weighed. One wt % of activated carbon was added to each sample to retain radon gas produced from the radioactive decay of U. These samples were then vacuum-sealed and left for 45 days to reach equilibrium with radon (Richards, 1981., Aswathanarayana, 1985 and IAEA, 2003). After the 45-day waiting period, each sample was assayed using a RS 230 BGO natural gamma spectrometer. Samples were placed in a 2 cm thick lead shielded container in direct contact with the spectrometer. Direct

contact removed “distance from the detector crystal” as a source of error. At regular intervals the background was measured after which a specific sample, to calibrate for background error as well as detector drift. A period of 5 min was allocated to each sample as the assay period. The spectrometer measured the K, Th and U channel every 2 s of this 5 min assay period and recorded the average reading as the concentration of K (in wt %), equivalent Th (ppm) and equivalent U (ppm). This data was then downloaded from the spectrometer using the RSAnalyst software received with the spectrometer. The concentration data was then corrected by subtracting background readings and correcting for mass of each sample and detector drift as described in IAEA (2003). Spectrometer assay results were then compared to ICP-MS results in order to quantify the degree of disequilibrium that occurs between the parent nuclides (Th and U) and their daughter elements, as the daughter elements are what the spectrometer actually measured. The spectrometer assay results were then corrected using the ICP-MS result and were then used in the modelling phase of this project.

#### **4.5 Elevation survey at New Machavie**

Included in the fieldwork at New Machavie was an elevation survey of the TDF that was drilled as well as the surrounding area, using a dumpy level with an accuracy of 2 cm. The elevation of each borehole was also measured. For the purpose of this study, the elevation data was normalised using a reference point to relative elevation which was sufficient for the modelling phase. The elevation data was used to create a digital elevation model (DEM) of the TDF upon which other data was projected.

A few steps are important when using a dumpy level (Bhalla & Larroquette., 2004):

- The tripod must be placed firmly and horizontally on the surface;
- The spirit level has to be used to make sure that the dumpy level is exactly horizontal;
- The reference site has to be defined in order to calculate elevations from and to calculate errors;
- For each location, measure and note latitude, longitude and elevation
- Three readings must be recorded for elevation (the top, middle and bottom readings of the three cross hairs as seen through the viewfinder on the measuring rod); and
- These readings must then be used to calculate relative elevation of each point relative to the reference point.

#### **4.6 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analyses of samples**

Samples were prepared at EcoAnalytica (Environmental laboratory associated with the North-West University) for analyses following the US EPA 3050B method (1996), where 1 gram of the sample was used with repeated additions of nitric acid and hydrogen peroxide in order to

dissolve most elements that could be released into the environment. Samples were then tested using ICP-MS as described in the US EPA method 6020A (2007).

#### **4.7 Calibration of down-hole data with laboratory results**

ICP-MS results were compared to the laboratory spectrometric assays (as discussed under Section 4.4) using Statistica 11 (Statistica 11, 2013). Initially the normality of the data was investigated by drawing histograms for each data set and plotting a normal distribution curve. Data found not to be normally distributed were discarded since normality underlies all further statistical analyses done on the data, including interpolation. Scatterplots were drawn for various combinations of data, including U (spectrometric assay) vs U (ICP-MS), Th (spectrometric assay) vs Th (ICP-MS), U (ICP-MS) vs Th (ICP-MS) and U (spectrometric assay) vs Th (spectrometric assay), as a means to assess potential relationships between these data sets. Correlation coefficients ( $r$ ) were calculated to quantify the linear relationship between variables and the coefficient of determination ( $r^2$ ) was determined in order to quantify the degree of variation within the data. The  $r^2$  can also be used to determine the ability of one variable to be used as an estimate of another. In this case the U (ICP-MS) concentration can be calculated from the U (spectrometric assay) concentration through the regression model as calculated in Statistica 11.

Down-hole spectrometric data was transformed from cps to ppm by calibration of various depth readings with ICP-MS results of samples taken at the same depths.

#### **4.8 Modelling of data**

Three dimensional models of New Machavie were created using the Rockworks 15 Software (Rockworks 15, 2013). To create distinct layers, the tailings and the soil below the TDF were modelled as different stratigraphic units. Elevation data from the elevation survey were interpolated using ordinary Gaussian kriging. Kriging was chosen as it has a greater versatility and more reliable statistical base from which to interpolate, although enough sample sites were present for any interpolation method to perform well. Kriging still presented the most realistic representation of New Machavie compared to IDW and other interpolation method. The variogram with the best fit was a Gaussian variogram without a nugget. The settings of the variogram are:

- Major Axis direction – 147.8
- Major axis range – 108.61
- Minor axis direction – 237.8
- Minor axis range – 84.09
- Nugget – 0
- Relative sill – 925.87

These settings produced a correlation of 0.99 and an anisotropy ratio of 0.77. This indicates that the stratigraphic model is a fairly accurate model of New Machavie.