Comparative assessment of the concentration of radioactive materials in natural sources associated with mining activities in the Republic of Niger using two measurement techniques.

Abstract

In the present work, we report the results of radioactivity measurements carried out in soils from the COMINAK and SOMAIR mining areas, using normal mode gamma-ray spectroscopy (NMGS) and thermal neutron activation analysis (TNAA). Radio activities were determined for two series radionuclides (238U and 232Th) and one non-series radionuclide (40K) from measured gamma-ray spectra. The activity concentrations of 238-U and 232-Th were respectively determined from the average nuclide concentration [Pb-214 (295.2keV ; 19.20%), Pb-214(351.9keV ; 37.10%), Bi-214 (609.3keV ; 46.90%), Bi-214 (1120.2keV; 15.04%) and Bi-214 (1764.49 keV; 15.90%)] and [Pb-212 (238.6keV; 43.6%), Pb-212 (300.09 keV; 3.18%), andAc-228 (911.2keV; 25.8%), Ac-228 (968.9keV; 15.8%), Ac-228 (338.32 keV; 11.27%)]. The activity concentrations of 40-K are determined directly by measuring gamma-ray transitions at 1460.8keV (100%) using a high-resolution pure germanium detector with good efficiency from the Egyptian Atomic Energy Authority. The results obtained confirm that one of the samples (COMINAK soil) is more radioactive than the SOMAIR soil samples, and the results are similar for both NMGS and TNAA techniques, with the exception of thorium in the SOMAIR soil sample.

*Keywords: [*NORM, gamma-ray spectrometry, neutron activation, uranium mines*}*

1. Introduction

Uranium mining began in the Republic of Niger in the Monts Aériens region in 1968, with SOMAIR, a 50-70 m-deep open-pit mine located 7 km northwest of the mining town of Arlit. And the second, underground mine, located near the town of Akokan, and was created in 1974: COMINAK, with a depth of 250 m and over 250 km of galleries, is one of the largest underground mines in the world. The environmental impacts of uranium mining are as follows: Depletion of fossil aquifers, disappearance of vegetation cover, disturbance of flora and fauna, deformation of the landscape due to tailings formed by extracted materials, and deposition of radioactive waste and other radioactive materials. It is therefore clear that uranium mining in Niger over the past fifty years has had serious consequences for the environment and human health [1]. In order to properly assess the impact of uranium mining activities in Niger, an on-site mission was organized to gather the necessary information and carry out sampling at the COMINAK and SOMAIR sites. The aim of this work is to measure natural radioactive materials in the Agadez region, Republic of Niger, and to compare the results of two methods: normal mode gamma spectroscopy and thermal neutron activation analysis. The activity concentrations of 238U, 232Th and 40K are determined. Specific activities and mass concentration in ppm in soil samples were determined.

2. Methodology

Several techniques for measuring radioactivity, particularly uranium, have been developed in nuclear physics and radiochemistry laboratories around the world. Among these techniques are those developed at the Nuclear Physics Laboratory of the Faculty of Sciences in Rabat since 1977: gamma spectrometry using either a Ge(Li) or a NaI (Tl) well

detector, X-ray fluorescence spectrometry, solid nuclear trace detectors and neutron activation [6]. In this work, we used two methods to measure natural radioactivity: the normal mode gamma-ray spectrometry technique and neutron activation analysis. The activity of a radionuclide is determined from the number of events contributing to the total absorption peak per unit time. The corresponding count rate represents, to within a multiplicative factor, the radionuclide's activity. To do this, it is necessary to subtract the contribution of the continuum background, created by the interaction of higher-energy photons and ambient radiation. This operation constitutes the measurement of the net peak area. GENIE 2000 software is used to process spectra, from radionuclide identification to the calculation of characteristic γ-activities. Two methods can be used to calculate the net area of spectral peaks [4]:

2.1 Sample collection and preparation

The samples were taken at Arlit and Akokan in the north-east of the Republic of Niger. A 1 kg soil sample was also taken from the Arlit garden, and another 1 kg soil sample was taken from the Akokan garden. Once the samples had been collected from the sites, they were taken to the Centre National de Radioprotection (CNRP) in Niger for physical preparation. Soil samples were wrapped in plastic. Analysis of the samples was carried out in Egypt at the laboratory of the Egyptian Atomic Energy Authority.

2.1 .1 Preparation of samples for gamma spectroscopy measurement

A soil sample from the COMINAK region and another from SOMAIR, each weighing 0.5 kg, were placed in two cylindrical plastic bottles 11.5 cm high and 5.5 cm in diameter. For gamma-ray measurements, each of the two samples was placed in direct contact with the detector and counted for around 72,000 seconds (20 hours). The gamma-ray counting time was set at 72,000 seconds for both counting modes. A long counting time was used because the samples had very low activities and the measurements therefore required very slow counting. The analysis software is then able to match the energy of the spectrum peaks to the radionuclides potentially present from a library that references all known X and γ lines. Once the peaks have been identified, quantitative analysis can begin. The radionuclides contained in the samples were identified and the areas under the peaks were used to determine the activity concentrations of each nuclide. The activity concentrations of 238-U and 232-Th were determined from the average nuclide concentration [Pb-214 (295.2keV; 19.20%), Pb-214 (351. 9keV; 37.10%), Bi-214 (609.3keV; 46.90%), Bi-214 (1120.2keV; 15.04%) and Bi-214 (1764.49Kev; 15.90%)] and [Pb-212 (238.6keV;43.6%), Pb-212 (300.09 kev; 15.90%)] and [Pb-212 (238.6keV;43.6%), Pb-212 (300.09 kev; 15.90%)]. 3.18%), and Ac-228 (911.2keV;25.8%), Ac-228 (968.9keV; 15.8%),Ac-228 (338.32Kev; 11.27%)] respectively. Activity concentrations of 40-K were determined directly by measuring gamma-ray transitions at 1460.8keV (100%).

2.1.2 Sample preparation for neutron activation analysis (NAA) measurement

The measurement of this quantity, commonly practiced in the past on activation detectors placed between two fuel pellets, was then developed on fuel rods from 1995 onwards. The γ spectrometry bench equipped with a planar-type detector was specially designed to measure γ lines at 277.6 keV, emitted by neptunium 239 (produced by neutron capture on uranium 238), and at 293.3 keV, emitted by cerium 143 for normalization to the total fission rate. If the uranium and thorium content of the sample is low, determining the concentration of 238-U and 232-Th is difficult. Soil samples have low concentrations, so a conventional long irradiation NAA can be used to determine their uranium and thorium nuclide content by measuring the radioactivity of the Np-239 and Pa-233 wires respectively. In fact, the first results of neutron capture are 239-U and 233-Th, which have short half-lives. It is therefore impossible to use the relative method, as they would disintegrate in subsequent measurements of the gamma rays they emit. Four soil samples were prepared. Two samples from the Republic of Niger, an IAEA standard sample (IAEA-313) in which the activities are known, and a blank (IAEA Soil-7). Two measurements were made for each sample, one for a short irradiation time of 60 seconds and the second for a long irradiation time of 3600 seconds. The irradiation process was managed in accordance with the procedure that follows radiological safety rules, in which the samples to be irradiated were stored in an irradiation bar that was located inside an irradiation isolation room or cobalt room using robotic arms. At the end of each irradiation, the capsule was removed from the reactor and allowed to cool (decay) until the activity level was within acceptable limits for handling. The samples were then removed from the capsule for counting. Each sample was placed at the appropriate position on the detector and counts were accumulated over a predetermined time to obtain spectral intensities. For short irradiation, a count time of 600 seconds proved adequate, and for long irradiation, the count time was 3600 seconds.

2.1 .3 Measuring activity in the context of low-noise counting

The sensitivity of a system is defined as a detection limit at or close to background level. This is a useful benchmark, not an absolute level. The critical level (Lc), detection limits (DL) and minimum detectable activity (MDA) is three benchmarks. The critical level (Lc) is the net count rate that must be exceeded for the sample to present measurable radioactivity above background with a certain degree of confidence. The critical level values were compared with the corrected net counts recorded in the photographic peaks for each nuclide. If the number of counts exceeds the critical

level value, it is assumed that this line is emitted by the sample and the activity of this line has been determined; otherwise, it means that there is no measurable activity in the sample. The detection limit is based on the uncertainty of the results. In this work, the detection limit is calculated by Curie's formula of equation (1)

$$Dl = 2,71 + 4,65\sqrt{NB} \quad (1)$$

If the result is below the decision threshold, the radionuclide is not detected. This does not mean that the radionuclide is absent, but that the measurement method guarantees with a high probability (1-β) that if it were present, its activity would not exceed the detection limit. The result is expressed as < LD.

If the result is above the decision threshold, the probability that the radionuclide is present is high (greater than 1-α). The most probable result is A, and the probability is high (equal to 1-γ) that the result lies between the lower and upper limits of the confidence interval. The result is expressed as A ± b, where b is the expanded uncertainty. It is an exact value when the standard uncertainty exceeds 25% (because in this case the confidence interval is symmetrical), and an approximate value when the standard uncertainty does not exceed 25% (because in this case the confidence interval is not exactly symmetrical).

The MDA is the minimum quantity of radioactive nuclide that we can be sure of detecting. It is often equated with the activity equivalent to the detection limit, DL. From DL in equation (1), we can deduce the MDA, which then becomes the activity we can expect to detect in 95% of cases.

$$MDA = \frac{DL}{\varepsilon \times T_L \times I_\gamma} \quad (2)$$

Where Iγ is the gamma emission probability, T_L is the counting lifetime; ε is the photo peak efficiency.

The MDA per sample mass can be calculated using equation (3):

$$MDA = \frac{DL}{\varepsilon \times T_L \times I_\gamma \times m} \quad (3)$$

Where m is the sample mass in kg.

2.1 .4 ***Specific activity***

The activity per unit mass, i.e. the specific activity in Bq.kg-1, or the radiological concentration for each nuclide detected, is calculated by expressing Ai, for a nuclide i and for a peak at energy E, by equation (4). This expression ignores the limitations of both instrumentation and data analysis.

$$A_i = \frac{N_{in} - N_B}{\varepsilon_E \times T_L \times I_\gamma \times m} \quad (4)$$

Where, N_{int} - N_B is the net area of a peak at energy E, ε_E is the detection efficiency at energy E, T_L is the live counting time, I_γ is the gamma emission probability at energy E, and m is the sample mass in kg. If there is more than one peak in the energy analysis range for a certain nuclide, an attempt is made to average the activities of the peaks. The result is then the weighted average of the specific activity of the nuclide. On the basis of the measured photo γ peaks emitted by specific radionuclides in the 232Th and 238U decay series and in 40K, their radiological concentrations in the samples taken were determined. The calculations are based on the establishment of a secular equilibrium in the samples, due to the much shorter lifetimes of the daughter radionuclides in the 232Th and 238U decay series. Specifically, the concentration of 232Th was determined from the average concentrations of 212Pb and 228Ac in the samples, and that of 238U from the average concentrations of the decay products 214Pb and 214Bi. Thus, an accurate measurement of the radiological concentrations of 232Th and 238U was made, while an actual measurement of the 40K concentration was carried out [2]. The radiological concentrations of 232Th, 238U and 40K were then converted to total elemental concentrations of thorium, uranium and potassium, respectively, according to the following equation (5):

$$F_E = \frac{M_E \times C}{\lambda_E \times N_A \times I_\gamma \times f \times n} \times \sum_{i=1}^{n} A_i \quad (5)$$

where F_E is the fraction of element E in the sample or the concentration of the element, M_E is the atomic mass (g.mol-1), λ_E is the decay constant (s-1) of the parent radioisotope, N_A is the Avogadro number (6.023×10^{23} atoms. mol-1), f is the fractional atomic abundance of 232Th, 238U or 40K in nature, C is a constant (with a value of 100 or 1,000) that converts the ratio of element mass to soil mass into percent or ppm, and A_i is the radiological concentration of radionuclide i .

3. RESULTS AND DISCUSSION

3.1 Gamma spectrometry in normal mode

3.1.1 Critical level and detection limit:

The critical level and detection limit for the normal mode gamma-ray spectrometry method are, as shown in Table 1, between 19.74±12.3 and 125.53±20.41 counts and between 42.17±27.29 and 253.66±43.61 for Bi-214 (1764.49keV) and Pb-212 (238.63keV) respectively. This means that the soil sample must record values higher than these to be considered active. Analysis of the samples showed high concentrations recorded at COMINAK such as thorium (Th), Uranium (U) and Potassium (K). The present study has shown that the samples taken at the COMINAK site have high concentrations (see Table 2). This high concentration is thought to be due to the intensive uranium exploration carried out on the site over the years. The high presence of lead in the samples is certainly due to the use of fertilizers and household waste to improve market garden produce.

Table 1 Detection limits for gamma-ray spectrometry in normal mode

Radionuclides	Critical level(counts)	Detection limit(counts)
Pb-212	125.53±20.41	253.66±43.51
Pb-214	46.22±18.89	95.11±40.47
Ac-228	94.56±18.60	191.75±39.89
Pb-214	81.14±18.46	164.92±39.61
Pb-212	104.54±26.31	211.70±55.30
Bi-214	84.54±12.52	171.71±27.74
Ac-228	62.59±16.61	127.84±35.61
Ac-228	52.78±16.11	108.22±34.91
Bi-214	48.34±20.93	99.35±44.55
Bi-214	19.74±12.30	42.17±27.29
K-40	58.84 ±16.77	120.33±36.23

Table 2 activity concentration in the soil sample

Sample	Activity concentration in Bq/kg			Concentration in mg/kg		
	238U	232Th	40K	238U	232Th	40K
COMINAK	27.36± 0.02	47.21± 0.01	619.87± 0.01	2.32± 0.002	11.67± 0.001	19822± 0.001
SOMAIR1	15.7 ± 0.03	29.14± 0.02	542.48± 0.01	1.35± 0.003	7.2± 0.002	17350± 0.002

3.1.2 Estimating the accuracy and precision of NMGS measurements

Improving the quality of calculation forms therefore necessarily involves the use of controlled experiments and reduced uncertainties. However, given the harsh environment in which these measurement techniques are used, limiting phenomena are likely to introduce systematic errors that need to be assessed and quantified. Moreover, since the physical quantities of interest are never obtained directly, corrective factors must be applied to the raw measurement results. These are tainted by uncertainties that need to be assessed and propagated.

The precision and accuracy of the method are determined by the percentage, %P and En-score. Equations (6) and (7) were used to calculate the results shown in Table 5. A comparison was made between the U-238, Th-232 and K-40 activity concentration results obtained from samples from the Republic of Niger and the values specified by the IAEA for the IAEA-SOIL-7 reference sample. A certified reference material (IAEA-SOIL-7) was analyzed to confirm the accuracy of the methods. Results for all elements except K were within the certified concentration range.

$$\%P = \frac{x - X}{X} \times 100 \quad (6)$$

$$E_n = \frac{x - X}{\sqrt{U_x{}^2 + U_X{}^2}} \quad (7)$$

With x_ measured value for the sample, X _ IAEA certificate value, Ux-expanded uncertainty of the measured value and UX_ expanded uncertainty associated with the IAEA value. An En-score ≤1 indicates that x and X are in agreement

within their respective uncertainties. An En-score>1 indicates that x is different from X and therefore that the uncertainty associated with the result has been underestimated. The performance criteria for En-scores are therefore as follows:
If En ≤1 satisfactory (in line with IAEA value).
If En ≥ 1 unsatisfactory (disagrees with IAEA value

3.1.3 ***Concentration of activity***

Equation (6) was used to calculate the radionuclide activity concentration in mg/kg for the soil sample. The results are presented in Table 2. We conclude that Pb-212, Pb-214 and Bi-214 exist in measurable quantities only in the background environment of the detector and do not originate from the samples themselves.

Table 3 Minimum detectable activity for nuclides present in the background

Radionclid	MDA(Bq/kg) COMI Soil	MDA(Bq/kg) SOMAIR Soil	MDA (Bq/l) SurpZI	MDA(Bq/l) COMI17	MDA (Bq/l) Surp1
Pb-212	0.026	0.0224	0.01124	0.0111	0.0113
Pb-214	0.055	0.0487	0.02678	0.0258	BDL
Ac-228	0.145	0.123	0.0454	0.045	0.0415
Pb-214	0.031	0.0322	0.0143	0.0149	0.0142
Pb-212	0.356	0.308	0.1564	0.149	BCL
Bi-214	0.022	0.0244	0.0113	0.0127	0.0126
Ac-228	0.042	0.0400	0.0222	0.0234	0.0219
Ac-228	0.064	0.0562	0.0402	0.0314	0.0326
Bi-214	0.123	0.0656	BDL	0.0419	0.0376
Bi-214	0.11	0.0902	0.0358	0.0414	0.024
K-40	0.121	0.123	0.0598	0.059	0.0634

Table 4 precision and accuracy for NMGS technique

Radionuclides	U-238	Th-232	K-40
COMINAK soil x(mg/kg)	2.32± 0.002	11.67± 0.001	19822 ± 0.001
SOMAIR soil x(mg/kg)	1.35 ± 0.003	7.2 ± 0.002	17350 ± 0.002
IAEA Sample 7, x (mg/kg)	2.6 ± 0.7	8.2± 0.5	12100± 600
%P COMINAK	10.8	18	63
In COMINAK	0.199	0.73	4.21
%P SOMAIR	43	12	85
In SOMAIR	0.45	0.999	8.63

When we consider each sample individually, looking at Table 4 considering each measurement for the COMINAK soil sample and the SOMAIR soil sample, it was found that the deviations of the uranium concentration results from the mean value of the IAEA certificate for uranium in the IAEA reference sample sol-7 are very high 48% and 10.8% in the

SOMAIR soil sample and the COMINAK soil sample respectively for the normal mode Gamma Spectrometry Technique. The respective En-score results are 0.42 and 0.199. As these results is less than 1: indicates that x and X are in agreement with their respective uncertainties. Also for thorium, the deviations are 12% and 18% respectively for the SOMAIR soil sample and the COMINAK soil sample for the normal mode gamma-ray spectrometry technique. The respective En-score results are 0.99 and 0.73. As these results is less than 1: indicates that x and X are in agreement with their respective uncertainties. We note that the maximum deviations of the IAEA soil potassium results are 85% and 63%, ~En=8.63 and En=4.21 in this method, indicating that the uncertainty associated with the measured value has been underestimated.

3.2 Thermal neutron activation analysis

3.2.1. Minimum detectable activity

The activities of U-238 and Th-232 (in Bq/kg) in the samples were estimated from the activities of Np-237 and Pa-233 formed after long irradiation. The samples were co-irradiated with the IAEA standard sample. Potassium K-40 activity was estimated from K-42 activity after short irradiation.

Table 5 Minimum Detectable Activity

Radionuclide	MDA for COMINAK soil on Bq/kg	MDA for SOMAIR Soil on Bq/kg	Probability of emission
Np-239	0.73	0.51	0.1438
Pa-233	0.43	0.27	0.386
K-42	1.7	1.36	0.18

3.2.2. Activity concentrations of radionuclides U-238, Th-232 and K-40

Measurements were taken from environmental samples where uranium and thorium are present in trace amounts (mg/kg). Equation (8) was used to calculate the activity concentration. The results are shown in Table 7.

$$C_{samp} = C_{st} \frac{W_{st} \times A_{sampl}}{W_{sampl} \times A_{st}} \quad (8)$$

Where "samp" and "st" refer to the Republic of Niger sample and the standard sample respectively

Table (6) Activity concentration for TNAA

Name of sample		COMINAK	SOMAIR	IAEA-soil 313
Activity concentration in Bq/kg	^{238}U(^{239} Np)	14138±0.06	6598.3±0.01	96154±0.02
	^{232}Th(^{232} Pa)	206.6± 0.23	114.09± 1.3	1200± 0.01
	^{40}K(K-42)	10925±0.06	6857.8±0.08	11942±0.04
Activity concentration in mg/kg	U-238	2.36 ± 0.03	1.34± 0.01	NA
	Th-232	11.72 0.13	7.87 0.12	NA
	K-40	NA	NA	NA

NA: Not Applicable

All deviations are less than 50% the method is therefore accurate.

Table 7 compares the activity concentration results for U-238, Th-232 and K-40 between those obtained from sample measurements and the values specified by the IAEA for the IAEA-SOIL-7 reference sample.

Table 7 Precision and accuracy for NAA technique

Radionuclides	U-238	Th-233	K-40
COMINAK soil x(mg/kg)	2.36 ± 0.03	11.72± 0.13	NA
SOMAIR soil x(mg/kg)	1.34 ± 0.01	7.87 ± 0.12	NA
IAEA-Soil 7 x (mg/kg)	2.6± 0.7	8.2± 0.5	12100 ± 600
%P COMINAK (%)	9.2	42.9	NA
In COMINAK	0.17	0.34	NA
%P SOMAIR	48.5	4.02	NA
In SOMAIR	0.89	0.35	NA

NA: Not Applicable

4. Comparative assessment: Thermal Neutron Activation Analysis (TNAA) and Normal Mode Gamma Spectrometry (NMGS)

4.2 Normal mode gamma-ray spectrometry offers the best detection capability and the lowest minimum detection limit.

For individual isotopes, when using NMGS, Th-233 is better detected than K-40, and U-238 is better detected with NMGS than with TNAA. This may be explained by the fact that uranium 238 has a higher radioactivity than thorium 232. On the other hand, Th-232 is better detected by the TNAA measurement technique than U-238. This may be explained by the fact that Th-232 has a high relative neutron activation property.

Table 8 - Comparison between MDA for NMGS and technical TNAA

Radionuclides	MDA (Bq/Kg) COMINAK with NMGS	MDA (Bq/Kg) SOMAIR with NMGS	MDA (Bq/Kg) COMINAK with TNAA	MDA (Bq/Kg) SOMAIR with TNAA
U-238	0.07	0.052	0.73	0.51
Th-233	0.13	0.11	0.43	0.27
K-40	0.121	0.123	1.7	1.36

The results obtained with NMGS for U-238 and Th-233 are less than one (En ≤1), which is satisfactory (in agreement with the results obtained with TNAA for 238U- and 232Th are smaller than 1 in the SOMAIR and COMINAK samples. This method is therefore satisfactory for these isotopes.

Table 9 Comparison between %P, E_n for NMGS and TNAA techniques

Radionuclides	En-score COMINAK with NMGS	En-score SOMAIR with NMGS	En-score COMINAK with TNAA	En-score SOMAIR with TNAA
U-238	0.199	0.45	0.17	0.89
Th-233	0.73	0.999	0.34	0.32
K-40	4.21	8.63	NA	NA

5. CONCLUSION

In the present study, natural radioactivity levels in soil samples collected in the Akokon and Arlit regions (COMINAK and SOMAIR sites in the Republic of Niger) were determined using normal mode gamma spectroscopy (NMGS) and thermal neutron activation analysis (TNAA). In terms of cost and availability, the neutron activation analysis method is more expensive and less available than the gamma spectrometry method.

For COMINAK soil samples using NMGS and TNAA techniques, the specific activities of u-238 are 2.32mg/kg and 2.36mg/kg respectively. The specific activities of Th-232 are 11.67mg/kg and 11.72mg/kg respectively. For SOMAIR soil samples measured using NMGS and TNAA, the specific activities for U-238 are 1.35mg/Kg and 1.34mg/Kg respectively. For Th-232, the specific activities are 7.2mg/kg and 7.87mg/kg respectively.

The specific activities determined using the NMGS and TNAA methods are fairly close, indicating that the values measured are reliable. The results confirm that the COMINAK soil sample is relatively more radioactive than the SOMAIR soil samples.

Furthermore, comparison of the results obtained with the known standard concentration of U-238 and Th-232 in the IAEA SOIL-7 sample shows that the NMGS technique is more accurate than the TNAA method. These results show that γ-spectrometry is a powerful, multidisciplinary measurement technique. Its practical implementation is straightforward, which explains its universal use for characterizing nuclear materials and irradiating objects. It does, however, require a good understanding of the main limiting phenomena that users are likely to encounter.

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