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Natural U-Isotopes in Groundwaters Occurring at Campinas City, São Paulo State, Brazil

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ABSTRACT

This paper describes the characterisation of the natural U-isotopes (U-238, U-235, and U-234) in groundwaters occurring at Campinas city, São Paulo State, Brazil. The Alpha Spectrometry technique was successfully applied to the analysis of 6 samples collected in a field campaign realised on 27th November 2018 at previously defined points consisting of deep tubular wells drilled at the campus of UNICAMP-State University of Campinas, Barão Geraldo District. The analytical results indicated an accentuated presence of U-235 in some samples, which is a radionuclide of unusual occurrence in surface waters and groundwaters due to the low natural abundance of U-238 compared to U-235 in rocks, soils, and sediments, thus, pointing out the relevance of the data obtained for further isotope (hydro)geochemistry studies.

Keywords - natural radioactivity, uranium isotopes, groundwaters, Campinas city, Brazil

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I. Introduction

Uranium is a natural radioelement of common occurrence in crustal rocks, normally exhibiting lower abundance in intermediate, basic, and ultrabasic rocks if compared with the acid igneous ones [1]. ²³⁸U, ²³⁵U, and ²³⁴U are the three naturally occurring U-isotopes.

²³⁸U is the parent nuclide in the mass number (4n+2) decay series of radioelements, which proceeds as follows [2]: ²³⁸U (4.49 Ga, α) \rightarrow ²³⁴Th (24.1 d, β^{-}) \rightarrow ²³⁴Pa (1.18 m, β^{-}) \rightarrow ²³⁴U (248 ka, α) \rightarrow ²³⁰Th (75.2 ka, α) \rightarrow ²²⁶Ra (1622 a, α) \rightarrow ²²²Rn (3.83 d, α) \rightarrow ²¹⁸Po (3.05 m, α) \rightarrow ²¹⁴Pb (26.8 m, β^{-}) \rightarrow ...²⁰⁶Pb.

²³⁵U is the parent nuclide in the mass number (4n+3) decay series of radioelements, which proceeds as follows [2]: ²³⁵U (0.71 Ga, α) \rightarrow ²³¹Th (25.6 h, β^{-}) \rightarrow ²³¹Pa (34.3 ka, α) \rightarrow ²²⁷Ac (22 a, β^{-}) \rightarrow ²²⁷Th (18.2 d, α) \rightarrow ²²³Ra (11.1 d, α) \rightarrow ²¹⁹Rn (3.92 s, α) \rightarrow ²¹⁵Po (1.8 ms, α) \rightarrow ²¹¹Pb (36.1 min, β^{-}) \rightarrow ...²⁰⁷Pb.

Thus, ²³⁴U is radiogenic, i.e., a by-product of ²³⁸U in its radioactive decay chain. In a mixture of

natural uranium, the relative abundance of these Uisotopes is about 99.3% for 238 U, 0.72% for 235 U, and 0.0054% for 234 U [3].

Nuclear power plants employ ²³⁵U as fuel after ²³⁵U atoms split when neutrons from the uranium atoms clash with other ²³⁵U atoms, thus generating heat due to the chain reaction [4]. One ton of natural uranium typically produces 44 million kilowatthours of electricity [5].

The great majority of rocks contain U-minerals possessing an abundance of ²³⁵U compared to ²³⁸U corresponding to 1/137.8, with rare exceptions like in the case of the Oklo U-mine occurring at Gabon, Africa, where it was found a value of ²³⁵U significantly depleted relative to ²³⁸U [6]. Such an unusual finding was attributed to the *in situ* ²³⁵U destruction by fission processes, strongly resembling ²³⁵U/²³⁸U ratios in the spent nuclear fuel generated by today's nuclear power plants [6]. That site became well-known in the literature for possessing suitable geochemical conditions favoring the operation of a naturally occurring ²³⁵U-fission reactor [6].

Only a few studies report ²³⁵U data in water resources due to its lower abundance compared to

²³⁸U in nature, with exceptions referring to investigations focusing on past U-mining activities or occasional releases related to accidents involving nuclear power plants as reported by [7].

²³⁴U was first identified in 1907 by McCoy and Ross, who named it U(II) [8]. The ²³⁴U/²³⁸U activity ratio (AR) is unity (condition of secular radioactive equilibrium between the U-isotopes ²³⁸U and ²³⁴U) in all minerals and rocks that are closed systems for U over a time-scale greater than 10^6 years [3]. On the other hand, AR values for uranium dissolved in waters are frequently above 1 when they interact with rocks and soils [3, 9]. Two major mechanisms have been pointed out for explaining the AR's values in solution exceeding the unity: 1) enhanced chemical solution of ²³⁴U due to radiation damage to crystal lattices or to autoxidation from U4+ to U6+ resulting from the decay of the parent ²³⁸U [10]; 2) alpha-particle recoil ejection of the ²³⁴U precursor (²³⁴Th) into solution [11]. AR's and U concentration data measured in groundwaters coupled to isotope dilution approaches have been utilized for deducing the proportions of groundwater masses mixing in different aquifer systems, among other applications, as reported by [9, 12-14].

The range of dissolved U concentration is often 0.1-10 μ g/L in rivers, lakes, and groundwaters elsewhere [3, 15], despite higher values having also been reported in the literature [7, 15-17]. There are health concerns about uranium ingestion over time in drinking water as this radioelement can affect the kidneys if its dissolved concentration surpasses the WHO guidance level of 30 μ g/L [18].

This paper describes a monitoring of the dissolved uranium concentration in groundwaters occurring at the urban area of Campinas municipality, São Paulo State (SP), Brazil (Fig. 1). The city is an important scientific and technological pole in Brazil with relevant research institutions developing their activities there.

II. Study area

Geologically, the area studied here lies dominantly within the Paraná Sedimentary Basin (PSB) in Brazil, whose total surface area is about 1.6 million km² distributed as follows: 62.5% in Brazil, 25% in Argentina, 6.25% in Paraguay, and 6.25% in Uruguay [20]. The PSB is intracratonic, possessing sedimentary sequences spreading from the SilurianDevonian times. The basin exhibits gentle dips towards the center of the basin, with the maximum thickness of sedimentary and volcanic rocks reaching up to about 8 km at the geometric center of the basin [21].



Fig. 1. Location of the study area at Campinas city, São Paulo State, Brazil [19].

Table	1. Simpl	ified Stratigraph	ic Co	olum	n of the PSB
since	Upper	Carboniferous	up	to	Cretaceous.
Accor	ding to [221			

PERIOD	GROUP	FORMATION	LITHOLOGY
Cretaceous	Bauru	Marília +	sandstones
То		Adamantina	siltstones
Jurassic			mudstones
		Caiuá	sandstones
			carbonatic
			nodules
	São Bento	Serra Geral	basalts
			diabase sills
		Botucatu	sandstones
Triassic		Pirambóia	sandstones
			shales
Upper	Passa	Corumbataí	mudstones
Permian	Dois		shales
			siltstones
		Irati	mudstones
			siltstones
			shales
			limestones
Mean	Tubarão	Tatuí	siltstones
Permian to			sandstones
Upper			limestone
Carboniferous			concretions
		Itararé	sandstones
			conglomerates
			diamictites
			tillites
			siltstones
			shales

The major rock types occurring at PSB are sandstones, conglomerates, diamictites, siltstones, shales, mudstones, limestones, basalts and diabases, as demonstrates its principal stratigraphic units as summarized in Table 1. CPRM [23] reported the occurrence of 1,053 deep tubular wells drilled at Campinas municipality for exploiting different aquifer systems of the PSB.

The study area is located at Barão Geraldo District (BGD), in the center-north of the municipality of Campinas, east of the state of São Paulo, more specifically on the campus of the UNICAMP-State University of Campinas (Fig. 1). The region is located in a transition between the Atlantic Plateau and Peripheral Depression, in which occurs contact between the PSB the Crystalline Basement, as confirmed by the typical dominant lithologies verified at Campinas city [24]: (a) the PSB, covering the western portion of the municipality, and chiefly composed by sandstones, shales and rhythmites from Itararé Group, as well as diabase sills and dikes from Serra Geral Formation; (b) the Crystalline Basement, covering the eastern portion of the municipality, and characterized by the presence of granites from Itapira, Jaguariúna and Socorro Complexes, as well as granite gneisses, tonalitic gneisses, migmatites, paragneisses, mica schists and biotite gneisses from Varginha Guaxupé Complex.

The surface area of the UNICAMP campus is 0.45 km², circulating there about 50,000 people daily, which causes a demand of 54 million liters of water every month, with ~40% of this amount being provided by groundwater [19]. Three aquifer systems have been exploited at the UNICAMP campus for supplying this water demand [25, 26]: 1) Crystalline – discontinuous and semi-confined type, comprising rocks of the crystalline basement such as granites and gneisses; 2) Tubarão - locally discontinuous and free/semi-confined regime, constituted by conglomerates, diamictites, tillites, siltstones, shales, rhythmites, sandstones, and some rare coal layers; 3) Diabase - discontinuous and free/semi-confined regime, formed by intrusive basic rocks from Serra Geral Formation.

Six deep tubular wells have been sampled in 27th November 2018 at UNICAMP campus for Uisotopes analysis, whose location for monitoring points FEF, GM, IMECC, and IB is shown in Fig. 1. Table 2 describes all sampling points considered in this study. Fig. 2 and Table 3 show details of a typical deep (220 m depth) tubular well drilled there, which exploits the Crystalline Aquifer.

III. Experimental

In this study, the groundwater samples (20-25 L) were taken from taps installed in the pumped tubular wells drilled for exploiting the aquifers. The samples were stored in polyethylene flasks, acidified to $pH\leq2$ with HCl, and transported to LABIDRO-Hydrochemistry and Isotopes Laboratory, Geology Department, IGCE-UNESP-Rio Claro, for U-isotopes analysis.

Table 2. Groundwater Sampling Sites at UNICAMP Campus, Campinas City, São Paulo State, Brazil. Sampling Date = 11/27/2018.

Site	Code	Time of sampling
Multidisciplinary gym	GM	9:20 hs.
Institute of Mathematics,	IMECC	9:50 hs.
Statistics and Computation		
Institute of Biology (Zoology)	IB	10:25 hs.
Faculty of Physical Education	FEF	11:05 hs.
Charutão Reservoir	CHA	11:30 hs.
Carlos Drummond de Andrade	DRU	12:00 hs.
Square		

Well 3500007199 Campinas UNICAMP





Table 3. Lithologies of a typical tubular well (Well No. 3500007199) drillled at UNICAMP campus, Campinas city, São Paulo State, Brazil. According to [27].

FORMATION	DEPTH	LITHOLOGY
	RANGE	
	(m)	
Itararé	0-2	Sandy soil
	2-6	Fine sandstone
	6-11	Siltstone
	11-18	Fine sandstone
	18-54	Claystone
	54-61	Silty sand
	61-65	Fine sandstone
	65-69	Mean sandstone
Gneissic-	69-220	Granite
migmatitic		
Complex		

About 500 mg of known amounts of FeCl₃ (500 mg) and man-made uranium spike/tracer (232U, halflife of 69.8 years; activity = 4.4 dpm) were added to each sample. The natural U-isotopes (238U, 234U, and ²³⁵U) and artificial ²³²U were subjected to coprecipitation with Fe(OH)3 after increasing the pH to 7-8 by means of adding a solution of concentrated NH4OH. Afterwards, there was the supernatant discard and recovery of the deposited material, followed by its dissolution into 8M HCl and extraction of Fe³⁺ with an equivalent volume of isopropyl ether. Then, the acid solution containing the U-isotopes was purified through some anion exchange steps, firstly using a Cl-acidified column and, later, on a NO3⁻-acidified column of Dowex resin (50-100 mesh). The elution of the U-isotopes from the NO3⁻ column was finally done with 0.1 M HCl, whose solution was evaporated to dryness. The dry residue was dissolved in 10 mL of 2M (NH₄)₂SO₄ electrolyte and transferred to an electrodeposition cell. The pH was adjusted to 2.4 and electrodeposition of U on a stainless steel planchet was complete after 3 hours at a current density of 1 A/cm² [28].

The U-isotopes data were obtained by alpha spectrometry. The α -activities were measured with four Si (Au) surface barrier detectors each one possessing 450 mm² area and 0.1 mm depletion depth. They were inserted into vacuum chambers and the electronic signals were recorded on an EG&G ORTEC 919 Spectrum Master Multichannel Buffer, which converted them into spectra displaying the peaks associated to the recovered

natural U-isotopes and ²³²U tracer. One radioactive source possessing 54.6 dpm of ²³⁸U and 10 dpm of ²³²U as prepared at the *Centre de Faibles Radioactivités*, CNRS-CEA, Gif-sur-Yvette, France, was utilized to calibrate the alpha spectrometric system. This allowed to record the channels present in the alpha spectrum and the corresponding α particle energy, thus, permitting to plot the energy calibration curve of the alpha spectrometer that is a straight line suitable to identify the following alphaparticles energies associated to the U-isotopes [29]: ²³⁸U = 4.2 MeV; ²³⁵U = 4.4 MeV; ²³⁴U = 4.8 MeV; ²³²U = 5.3 MeV.

IV. Results and Discussion



Fig. 3. Expanded alpha spectrum obtained for Uisotopes analysis of the groundwater sample FEF collected at the study area. Volume = 20.6 L. Counting time = 23.2 hours.

Fig. 3 shows a typical alpha spectrum obtained during the data acquisition, which highlights a larger

²³⁴U peak compared to the ²³⁸U peak in the sample FEF, and also shows a small ²³⁵U peak compared to ²³⁸U and ²³⁴U peaks. Such an example illustrates the successful use of the analytical procedure adopted for measuring the natural U-isotopes in the study area. The results obtained in all alpha readings are reported in Table 4.

Table 4. Results of the Alpha Readings for the Groundwater Samples Analyzed in this Study.

Groundwater Sumples / maryzed in this Study.						
Sample	Volume	Counting	U-	U-	U-	U-
code	(L)	time (s)	238	235	234	232
GM	21.8	87,926	9914	490	19342	120
IMECC	22.0	83,530	1058	66	2155	183
IB	22.2	75,242	2074	85	4561	330
FEF	20.6	83,529	2560	93	4893	115
CHA	20.6	87,922	1614	69	3320	255
DRU	20.6	235.087	183	11	342	374

The data refer to the gross area (total counts) in the peak of each U-isotope in the alpha spectrum. Analytical uncertainty corresponding to ± 10 -15% at 1 σ standard deviation.

The results obtained in the alpha readings, as reported in Table 4, allowed us to calculate the dissolved U concentration in the groundwater samples, as well as the activity ratios $^{234}U/^{238}U$ (AR) and $^{235}U/^{238}U$ (AR₅) (Table 5). The dissolved U concentration in the groundwater samples analyzed ranged from 0.13-22.96 µg/L, surpassing the typical maximum worldwide value of 10 µg/L in rivers, lakes, and groundwaters [3, 9, 15] for sample GM, but lower than the maximum guideline reference value established by the WHO [18] for ingestion of this radionuclide in drinking-water, i.e. 30 µg/L.

Table 5. Analytical results of dissolved U concentration and U-isotopes ratios in groundwater samples analyzed in this study

samples analyzed in this study.				
Sample	U (μg/L)	²³⁴ U/ ²³⁸ U	²³⁵ U/ ²³⁸ U	
code		AR	AR ₅	
GM	22.96	1.95	0.049	
IMECC	1.67	2.05	0.062	
IB	1.72	2.20	0.041	
FEF	6.55	1.91	0.036	
CHA	1.92	2.07	0.043	
DRU	0.13	2.15	0.060	

Analytical uncertainty corresponding to $\pm 10\text{-}15\%$ at 1σ standard deviation.

The AR values found in the groundwater samples are higher than unity, between 1.91 and 2.20 (Table 5), but lower than the average range of 1.7-4.04 as reported by [7] for the water samples collected at the chapters Cove, Red Valley, Sweetwater, and Teec Nos Pos in the USA. However, in many rocks, minerals, soils, and sediments the AR is close to one, for instance, in bottom sediments from Nasser Lake, Egypt, Agha et al. [30] reported AR values of 0.903-1.088, which are lower than those found for the groundwaters analyzed in this study.

All the AR ratios greater than unity in the liquid phase are expected during the water-soil/rock interactions due to preferential chemical dissolution of ²³⁴U [10] and alpha-recoil release of ²³⁴Th at the rock-water interface [11]. The lithology, stratigraphy, hydrogeology, geochemical conditions, and extent of water-soil/rock interactions are among the factors responsible for the distinct dissolved U concentration and AR values in the studied site.

The AR₅ values found in the groundwater samples are between 0.018 and 0.062 (Table 5). In bottom sediments from Nasser Lake, Egypt, Agha et al. [30] reported AR₅ values of 0.0442-0.484, which are within the range of the values found in this paper, but, in both cases, deviating from the natural expected value of 0.0072 in U-mineralized rocks.

The highest AR₅ value reported in Table 5 (0.062) is almost 10 times higher than the natural expected value in U-mineralized rocks, possibly indicating a preferential ²³⁵U-transport into solution compared to ²³⁸U, in a similar way as verified for ²³⁴U. However, this is a subject for more detailed laboratory tests, which is beyond the scope of this paper.

The natural mass ratio ²³⁵U/²³⁸U of 0.72% was observed in only 16% of the water samples provided from the U.S. EPA (Environmental Protection Agency) studies involving the Uranium Mines Project Arizona, New Mexico, Utah-Navajo Lands [7] as the ²³⁵U/total U mass ratios generally ranged from 0.06 to 5.9%.

The higher AR₅ value found in this paper (0.062, Table 5) practically coincides with the maximum value of 5.9% as reported by [7] for groundwaters provided from the Uranium Mines Project Arizona, New Mexico, due to past U-mining activities, reinforcing the need of further experiments to find reasonable explanations for their occurrence in different aquifer systems elsewhere. This is because different from the sites focused on by [7], there are no records in the study area of this paper of anthropogenic impacts associated with

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the U-isotopes, such as the presence of depleted uranium or uranium fuel.

V. Conclusion

Campinas city, São Paulo State, Brazil, is an important scientific and technological pole in the country with important research centers installed there such as the UNICAMP-State University of Campinas, whose water demand is about 54×10^6 liters per month and is significantly supplied by groundwater exploiting different aquifer systems. This investigation involved the sampling of six tubular wells for analysis of the three natural Uisotopes 238U, 235U, and 234U. The dissolved U concentration ranged from 0.13-22.96 µg/L, whereas the ²³⁴U/²³⁸U activity ratio (AR) of dissolved U ranged from 1.91-2.20, and the 235U/238U activity ratio (AR₅) ranged 0.018-0.062. No groundwater sample surpassed the limiting value of 30 µg/L for ingestion of ²³⁸U in drinking water as proposed by the WHO. The obtained dataset is relevant for further investigations there by two major reasons: 1) to evaluate mixing volumes of groundwaters providing from different aquifer systems based on the reciprocal of the U concentration and AR data; 2) to promote laboratorial experiments to clarify the mechanisms responsible for the generation of the enhanced AR5 values in waters compared to those in the U-mineralized rocks.

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