Some constraints on the modelling using the uranium isotopes $^{238}\text{U}$ and $^{234}\text{U}$ for dating groundwaters from Guarany aquifer, South America

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**Abstract**

Groundwaters from the Guarany aquifer located at the South American continent and sampled at four wells with described geological sections in São Paulo State, Brazil, were chemically and isotopically analysed with two aims: to evaluate the quality of this important hydrological resource and to investigate the possibility of using the natural uranium isotopes $^{234}\text{U}$ and $^{238}\text{U}$ as a chronological tool, since the $^{234}\text{U}/^{238}\text{U}$ activity ratio and dissolved U content data in groundwater systems have generated models for dating purposes.

**Introduction**

The natural radioelement uranium is widely distributed in crustal rocks, and under conditions present at the earth’s surface, it tends to be a mobile element. $^{238}\text{U}$ is the principal isotope of natural U (99.3% abundance) and is the parent nuclide in the mass number $(4n+2)$ decay series, the longest known series. The nuclide $^{234}\text{U}$ is radiogenic, being generated by $^{238}\text{U}$ in the decay sequence: $^{238}\text{U}$ (4.49 Ga, $\alpha$) $\rightarrow^{234}\text{Th}$ (24.1 days, $\beta'$) $\rightarrow^{234}\text{Pa}$ (1.18 min, $\beta'$) $\rightarrow^{234}\text{U}$ (248 ka, $\alpha$) $\rightarrow^{230}\text{Th}$ (75.2 ka, $\alpha$) $\rightarrow^{226}\text{Ra}$ (1622 a, $\alpha$) $\rightarrow...$

Secular equilibrium is established between $^{238}\text{U}$ and $^{234}\text{U}$ in all rocks and minerals which are closed systems for U which are more than 1.25 Ma old. The $^{234}\text{U}/^{238}\text{U}$ activity ratio (AR) is therefore unity within the bulk of most rock matrices. Dissolution processes at the rock-water interface frequently result in AR’s greater than unity for U dissolved in groundwater [1,2]. Alternative mechanisms which have been suggested for the generation of such enhanced AR’s
include preferential chemical dissolution of $^{234}\text{U}$ [3] and $\alpha$-recoil release of $^{234}\text{Th}$ at the rock-water interface [4]. Enhanced chemical dissolution of $^{234}\text{U}$ relative to $^{238}\text{U}$ may occur because of radiation damage to the crystal lattice in the vicinity of $^{238}\text{U}$ decay or because of oxidation of $^{234}\text{U}$ during the decay processes by which it is formed.

Uranium in solution may be depleted or enriched, depending on oxidation-reduction conditions. It is an element very sensitive to modifications on redox potentials, since if oxidizing conditions prevail, active solution of U occurs, and if the redox character changes towards reducing conditions (lower values of $Eh$), then, deposition of U takes place. Enhanced AR’s for dissolved U at more reducing conditions are favoured due to $^{234}\text{U}$ solution by the $^{234}\text{Th}$ recoil process, and, therefore, lower U concentrations and higher AR’s are generally expected at these aquifer zones.

In addition to uranium, other elements are also sensitive to redox processes, where of the 16 inorganic constituents that, for regulatory purposes, have recommended or mandatory concentration limits in drinking water supplies, 8 of these have more than one possible oxidation state in groundwater: As, Cr, Fe, Hg, Mn, Se, N, and S [5]. Some elements in the drinking water regulations that are relatively insensitive to the redox conditions are Cl, F, Ba and Ra, although in some systems even Ba and Ra are influenced indirectly by the redox conditions because of reactions with sulfate and iron, which are redox dependent [5].

Hydrological studies of groundwaters have utilized U contents and AR’s data with the purpose of dating. The change of AR with time for dissolved U in a groundwater which has evolved to a stable U chemistry in a reducing environment has been modelled [6-8]. The AR change after such a reduced zone has been entered is given by an equation dependent, among other factors, of the initial AR of the dissolved U in the groundwater on entering the reduced zone, and of the U content of the groundwater. In this study, it has been possible to utilize the data of the uranium isotopes $^{238}\text{U}$ and $^{234}\text{U}$ and a wide range of other constituents in groundwaters of the Guarany aquifer located in the South American continent with the aim of evaluating the modelling for dating commonly referred to in the literature [6-8].

**General features of the studied aquifer**

The Guarany aquifer of Triassic-Jurassic age has continental dimensions, extending over some 950,000 km$^2$ within the Paraná sedimentary basin, South America, and comprising southern Brazil (states of Mato Grosso, Mato Grosso do Sul, Goiás, Minas Gerais, São Paulo, Paraná, Santa Catarina and Rio Grande do Sul), eastern Paraguay, NW Uruguay and the northeastern extreme corner of Argentina. The aquifer has an average thickness of 300-400 m, being composed of silty and shaly sandstones of fluvial-lacustrine origin (the Piramboia formation, in Brazil), and variegated quartzitic sandstones accumulated by eolian processes under desertic conditions (the Botucatu formation, in Brazil) [9]. A thick basaltic package of the Serra Geral formation (up to 1,500 m) overlies this aquifer,
reducing its exposed areas in non-continuous elongated strips, 10-100 km wide, along the edges of the proper Paraná basin, where the longest strip stretches between the states of São Paulo and Santa Catarina, in Brazil. The aquifer overlies previous formations ranging from the igneous basement to the Paleozoic sediments of the Passa-Dois and Tubarão Groups, being covered by Cretaceous sediments of the Bauru-Caiuá formations.

Situated within an intercratonic basin, the aquifer is almost unperturbed, the potentiometric surface of the water shows that about 70% of its total area has artesian conditions [10], and recharge occurs by direct infiltration of rainwater in the outcrop area, which is about 98,000 km² [11]. The percolating water moves from the phreatic exposed areas that surround the entire basin towards its central part, and, in spite of the great distances separating the existing exploitation centres, data obtained sporadically indicate hydraulic conductivity values of $10^{-4}$-$10^{-5}$ m/s, effective porosity values of 10-20%, storage coefficient values of $10^{-3}$-$10^{-6}$ and average transmissivity of $10^{-3}$ m²/s [11]. The yields of the wells vary from 10-150 m³/h in the phreatic exposed parts of the aquifer to more than 300 m³/h in the confined artesian wells [9].

Table 1: Characteristics of the tubular wells drilled at the Guarany aquifer.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Location</td>
<td></td>
<td>Monte Alto</td>
<td>Catanduva</td>
<td>Santa Cruz</td>
<td>Paraguacu</td>
</tr>
<tr>
<td>Latitude</td>
<td></td>
<td>21°15’13”S</td>
<td>21°07’36”S</td>
<td>22°54’56”S</td>
<td>22°25’21”S</td>
</tr>
<tr>
<td>Longitude</td>
<td></td>
<td>48°30’00”W</td>
<td>48°59’05”W</td>
<td>49°39’05”W</td>
<td>50°33’38”W</td>
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<tr>
<td>Altitude of the top of the well</td>
<td>m</td>
<td>585</td>
<td>496</td>
<td>440</td>
<td>474</td>
</tr>
<tr>
<td>Total length</td>
<td>m</td>
<td>581</td>
<td>860</td>
<td>124</td>
<td>3663</td>
</tr>
<tr>
<td>Top of the aquifer depth</td>
<td>m</td>
<td>121</td>
<td>39</td>
<td>326</td>
<td>500bsl</td>
</tr>
<tr>
<td>Geostatic pressure</td>
<td>bar</td>
<td>123.7</td>
<td>121.9</td>
<td>31.1</td>
<td>258.7</td>
</tr>
<tr>
<td>Potentiometric level depth</td>
<td>m</td>
<td>521</td>
<td>452</td>
<td>446</td>
<td>430</td>
</tr>
<tr>
<td>Flow rate</td>
<td>m³/h</td>
<td>2.74</td>
<td>25.49</td>
<td>5.9</td>
<td>6.28</td>
</tr>
<tr>
<td>Transmissivity</td>
<td>×10⁻³</td>
<td>m²/s</td>
<td>2.4</td>
<td>15.0</td>
<td>7.5</td>
</tr>
</tbody>
</table>

*bsl = below sea level.

**Sampling and analytical methods**

The study of the Guarany aquifer was performed at São Paulo State, Brazil, whose advanced stage of agricultural and industrial growth has caused a great diversity of problems related to the interaction between the society and the
environment, many of them important because can be responsible for the pollution of the underground hydrological resources utilized for public supply, inclusive the waters of the Guarany aquifer, that are extensively used for drinking purposes. Four pumped tubular wells drilled along two cross-sections AA' and BB' from the border of the Paraná basin up to its central part were sampled for physical, chemical and isotopic analyses, whose location and characteristics are described in Table 1 and Fig. 1.

![Geological cross-section](image)

Figure 1: Geological cross-section [12] from (a) Ribeirão Preto up to Três Lagoas-AA' and (b) Avaré up to Presidente Epitácio-BB' showing the direction of the groundwater flow in Guarany aquifer.

Groundwater samples (19-20 kg) were collected, stored in polyethylene bottles, and, depending on the requirements of the analysis, they were distributed as unfiltered and unpreserved (i.e. for temperature, pH, Eh redox potential, dissolved oxygen, and alkalinity determinations), filtered through 0.45 μm membranes and unpreserved (i.e. for major ions analyses) and filtered and preserved with different acids (i.e. for uranium isotopes and trace element determinations). Standard analytical techniques were utilized for obtaining the composition of the major and minor elements in the waters, for instance, potentiometry, methyl orange end-point titration, colorimetry, and ion selective electrodes.
Table 2. Chemical analyses of groundwaters from the studied part of the Guarany aquifer. The data for conductivity, dry residue and silica were provided by DAEE.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
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<th>3</th>
<th>4</th>
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<td>Temperature</td>
<td>°C</td>
<td>38</td>
<td>37</td>
<td>24</td>
<td>43</td>
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<tr>
<td>Dissolved O₂</td>
<td>mg/L</td>
<td>3.7</td>
<td>3.5</td>
<td>9.0</td>
<td>3.2</td>
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<tr>
<td>pH</td>
<td>-</td>
<td>7.5</td>
<td>8.9</td>
<td>8.3</td>
<td>9.6</td>
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<tr>
<td>Eh</td>
<td>mV</td>
<td>+149</td>
<td>-51</td>
<td>+457</td>
<td>-66</td>
</tr>
<tr>
<td>Conductivity (25°C)</td>
<td>μS/cm</td>
<td>171</td>
<td>280</td>
<td>112</td>
<td>605</td>
</tr>
<tr>
<td>Dry Residue (105°C)</td>
<td>mg/L</td>
<td>104</td>
<td>214</td>
<td>91</td>
<td>371</td>
</tr>
<tr>
<td>Silica</td>
<td>mg/L</td>
<td>14.9</td>
<td>29.0</td>
<td>24.0</td>
<td>49.0</td>
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<tr>
<td>Alkalinity (Bicarbonate)</td>
<td>mg/L</td>
<td>48</td>
<td>0</td>
<td>36</td>
<td>0</td>
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<tr>
<td>Alkalinity (Carbonate)</td>
<td>mg/L</td>
<td>0</td>
<td>4</td>
<td>0</td>
<td>72</td>
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<tr>
<td>Alkalinity (Hydroxide)</td>
<td>mg/L</td>
<td>0</td>
<td>58</td>
<td>0</td>
<td>22</td>
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<tr>
<td>Sulfate</td>
<td>mg/L</td>
<td>&lt;2</td>
<td>9</td>
<td>2</td>
<td>16</td>
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<tr>
<td>Chloride</td>
<td>mg/L</td>
<td>4.6</td>
<td>5.4</td>
<td>4.4</td>
<td>12.2</td>
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<tr>
<td>Fluoride</td>
<td>mg/L</td>
<td>0.21</td>
<td>0.74</td>
<td>0.16</td>
<td>4.05</td>
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<tr>
<td>Nitrate</td>
<td>mg/L</td>
<td>13.20</td>
<td>11.00</td>
<td>7.04</td>
<td>6.60</td>
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<tr>
<td>Ammonium</td>
<td>mg/L</td>
<td>&lt;0.04</td>
<td>&lt;0.04</td>
<td>&lt;0.04</td>
<td>0.8</td>
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<tr>
<td>Sodium</td>
<td>mg/L</td>
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<td>0.9</td>
<td>0.7</td>
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<tr>
<td>Potassium</td>
<td>mg/L</td>
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<td>0.7</td>
<td>0.7</td>
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<tr>
<td>Calcium</td>
<td>mg/L</td>
<td>&lt;0.1</td>
<td>0.9</td>
<td>0.2</td>
<td>0.7</td>
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<tr>
<td>Magnesium</td>
<td>mg/L</td>
<td>0.7</td>
<td>&lt;0.1</td>
<td>0.5</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>Barium</td>
<td>mg/L</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>1</td>
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<tr>
<td>Total iron</td>
<td>mg/L</td>
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<td>0.08</td>
<td>0.03</td>
<td>0.05</td>
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<tr>
<td>Iron (II)</td>
<td>mg/L</td>
<td>0.03</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
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<tr>
<td>Manganese</td>
<td>mg/L</td>
<td>0.13</td>
<td>0.11</td>
<td>0.02</td>
<td>0.10</td>
</tr>
<tr>
<td>Zinc</td>
<td>mg/L</td>
<td>0.14</td>
<td>0.10</td>
<td>0.11</td>
<td>0.26</td>
</tr>
<tr>
<td>Copper</td>
<td>mg/L</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Cadmium</td>
<td>μg/L</td>
<td>6</td>
<td>6</td>
<td>220</td>
<td>4</td>
</tr>
<tr>
<td>Total chromium</td>
<td>mg/L</td>
<td>0.03</td>
<td>0.04</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td>Radon</td>
<td>pCi/L</td>
<td>1728</td>
<td>1272</td>
<td>1240</td>
<td>1202</td>
</tr>
<tr>
<td>Radium-226</td>
<td>pCi/L</td>
<td>1.7</td>
<td>1.3</td>
<td>1.2</td>
<td>1.2</td>
</tr>
<tr>
<td>Radium-228</td>
<td>pCi/L</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Uranium</td>
<td>μg/L</td>
<td>1.183</td>
<td>0.593</td>
<td>1.143</td>
<td>0.393</td>
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<tr>
<td></td>
<td></td>
<td>0.574</td>
<td>0.684</td>
<td>0.114</td>
<td>0.314</td>
</tr>
<tr>
<td>234U/238U AR</td>
<td></td>
<td>1.173</td>
<td>2.913</td>
<td>1.943</td>
<td>2.743</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.304</td>
<td>2.234</td>
<td>2.244</td>
<td>1.774</td>
</tr>
</tbody>
</table>

1Values provided by DAEE; 2Uncertainty ±10% corresponding to 1σ standard deviation; 3Uncorrected value; 4Corrected value.

The aliquots for U isotopes analysis were acidified to pH less than 2 on using HCl, about 500 mg of FeCl₃ plus 3.39 dpm of ²³²U-²²⁸Th were added, and U was co-precipitated on Fe(OH)₃ by increasing the pH to 7-8 through addition of concentrated NH₄OH solution; the precipitated was recovered, dissolved in 8M HCl and Fe³⁺ was extracted into an equal volume of isopropyl ether. The acid
solution U-bearing was purified by anion exchange, first on a Cl⁻ and then on a NO₃⁻ column of Rexyn 201 50-100 mesh resin. U was finally eluted from the NO₃⁻ column with 0.1 M HCl and after evaporation to dryness 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 Acm⁻².

The U content and AR were measured by alpha spectrometry. The α-activities were determined with a 0.1 mm depletion depth, 200 mm² area silicon surface barrier detector. The spectra for natural U and ²³²U tracer extracted were recorded on a EG&G ORTEC 919 Spectrum Master Multichannel Buffer. The Decision Level Lₐ [13] for acceptance of a positive measurement in the ²³⁸U, ²³⁴U and ²³²U energy regions was 0.00082, 0.00154, and 0.00225 cpm, respectively. The concentration data were calculated by isotope dilution from the counting rates of ²³⁸U and ²³²U peaks and the AR data were calculated from the counting rates of ²³⁴U and ²³⁸U peaks. Analytical details for these measurements were reported elsewhere [2], and the results obtained in this investigation are shown in Table 2, inclusive those provided by DAEE (Department of Waters and Electricity of the State of São Paulo).

![Figure 2: Typical alpha energy spectrum of uranium extracted from groundwater, demonstrating the overlap of ²³²U and ²²⁸Th peaks.](image)

A very important aspect concerning to the generation of U-isotopes data corresponds to the alpha pulse height analysis, since ²²⁸Th is a daughter of ²³²U with an alpha energy that yields a peak not resolvable from that of ²³⁵U (Fig. 2). The contribution of ²²⁸Th in a typical alpha energy spectrum must be taken into account, and a way of doing this is to subtract the ²²⁴Ra peak from the apparent
$^{232}\text{U}$ peak [2]. The results for U concentration and AR reported in Table 2 refers to both, i.e. $^{228}\text{Th}$-corrected and $^{228}\text{Th}$-uncorrected values.

**Water quality and U-isotopes modelling**

With the exception of sample 3, the studied groundwaters are thermal, whose Eh-pH field indicates the three possible characters, i.e. oxidizing, transitional, and reducing (Fig. 3). The more thermal water (sample 4) also has higher values of conductivity and dry residue, related to the presence of the major anions carbonate, sulfate, chloride, and fluoride. When the obtained chemical data are plotted on a standard Piper diagram [14], it is possible to see that they are Na$^+$-K$^+$ dominated in terms of dissolved cations, and (bi)carbonate dominated in terms of dissolved anions, with the exception of sample 2 that is Cl$^-$-NO$_3^-$ dominated (Fig. 4).

![Figure 3: Data for groundwaters from the studied area plotted on an Eh-pH diagram.](image)

If the chemical data obtained for the groundwaters are compared with the maximum (minimum in the case of dissolved oxygen) permissible concentration limits in drinking water established by the national standard NTA-60 (São Paulo State Register 12486 published on 20 October 1978), it is possible to verify that some parameters are above the permissible levels, for instance, nitrate (samples 1 and 2), fluoride (sample 4), and cadmium (sample 3). Unfortunatelly, there is no
brazilian regulation until now concerning to radon originally present in drinking water, but if the USEPA's reference value of 300 pCi/L [15] is taken into account, then, it is possible to verify that all the obtained data exceeded this limit. In some of these cases, anthropogenic inputs may be responsible for the contamination, whereas, in other, natural water-rock/soil interactions can justify the observed enhancement, that are not related to the occurrence of any environmental impact due to the human activities.

![Figure 4: Chemical data for the studied waters plotted on a Piper diagram.](image)

In terms of the redox conditions, modification of transitional towards reducing character occurs along the groundwater flow direction in profile AA' (Fig. 1), as well of the oxidizing towards reducing character along the groundwater flow direction in profile BB'. Oxidizing conditions generally prevail in the recharge zone of an aquifer and the most active etch solution of U occurs in this zone. A groundwater which has dissolved U close to recharge may undergo further change in its U hydrochemistry due to additional etch solution or, if its redox character changes towards reducing conditions, due to U deposition and $^{234}$U solution by the $^{234}$Th recoil process. The AR change after such a recoil-dominated (reduced) zone has been entered, $\text{AR}_t$, is given by the equation [6-8]:

$$\text{AR}_t = 1 + (\text{AR}_i - 1) e^{-\lambda t} + 0.235 \rho \text{SR} \left[1 - e^{-\lambda t}\right] \frac{[U]_r}{[U]_s};$$

where: $\rho =$ rock density (g cm$^{-3}$); $\text{AR}_i =$ the initial AR of the dissolved U in the groundwater on entering the reduced zone; $R = ^{234}$Th recoil range in the rock matrix ($\sim 3 \times 10^6$ cm); 0.235 = fraction of $^{234}$Th recoil atoms from within the recoil range of the surface that enter solution; $[U]_r =$ U content of the rock (ug g$^{-1}$); $[U]_s =$ U content of the groundwater (ug cm$^{-3}$); $\lambda =$ decay constant of $^{234}$U ($2.79 \times 10^{-6}$ a$^{-1}$); and $S =$ the extent of the rock surface in contact with unit volume of groundwater (cm$^2$ cm$^{-3}$), which is related to the specific internal surface area, $s$ (cm$^2$ g$^{-1}$), and the aquifer porosity, $\phi$, by the equation $S = \rho s/\phi$. The second term in the equation represents the decay of excess $^{234}$U which was acquired by preferential etch solution before the groundwater encountered reducing
conditions, whereas the third term evaluates the alpha-recoil enhancement of the AR for a groundwater which has entered a reducing zone.

The redox conditions and uncorrected U content and AR values of the analyzed groundwaters are favorable for applying the modelling, and, in order to perform this, the following parameters were utilized: $\phi = 20\%$ [16]; $S = 1829$ cm$^{-1}$ evaluated from the average diameter of the sandstone grains (0.164 mm) [17]; $\rho = 2$ g cm$^{-3}$ and $[U]_s = 1.58$ $\mu$g g$^{-1}$.

According to the uncorrected values of $[U]_s$, AR, and AR, for dissolved uranium, it was possible to find ages corresponding to 106,750 and 32,276 years for the groundwater flow in profiles AA and BB, respectively, where the higher value exceeds the timescale of the $^{14}$C method.

The available data for transmissivity, thickness of the aquifer and hydraulic gradient allow to use the Darcy’s law for the groundwater flow in porous media for obtaining the velocity of the groundwater flow, and, since the distance between the wells is known, it is possible to estimate ages corresponding to 36,150 and 113,957 years for the groundwater flow in profiles AA and BB, respectively.

Such higher value also exceeds the timescale of the $^{14}$C method, but the Darcy’s law-ages are opposite of the U-modelled ages, and, thus, contrary practically coincident ages were verified by the two independent methods. It is interesting to consider that the $^{14}$C ages for the same samples [12] corresponded to 8,300 and 18,900 yr BP for the groundwater flow in profiles AA and BB, respectively, which also differ significantly of the modelled and estimated ages, being difficult to establish an appropriate evaluation of the $^{14}$C data at this stage.

However, when the corrected U content and AR values of the analyzed groundwaters are taken into account, it is not possible to apply the U-isotopes modelling, because, instead of the expected redox variations, other effects are achieved, for instance, an enhancement of $^{234}$U in solution in accordance with an increase of the dissolution of calcium from the rock matrices (the AR changed from 1.3 in sample 1 up to 2.3 in sample 2, while the Ca$^{2+}$ content varied from 0.1 up to 0.9 mg/L). Such process is not the rule, because if the profile BB is considered, then, is possible to see that the AR does not increase accompanying the increase of the Ca$^{2+}$ content.

Other elements commonly reported to be redox sensible like Fe, Mn, and Cr didn’t exhibit significant variations in their concentrations like observed for the Eh redox potential, and, as already referred to, the main modifications in the groundwater chemistry are due to anthropogenic inputs (municipal and industrial landfills, industrial lagoons, septic sewage systems, livestock feedlots, agricultural chemicals, waste spills) or natural water-rock/soil interactions. Under this aspect, the ease with which the fine particulates may be freed from the rock surface and subsequently dissolved, the inhomogeneous distribution of elements in the multi-grain aggregates, surface roughness of the aquifer rocks, unequal exposed areas to etching/leaching, or irregular distribution of micaceous minerals in the rock surface are parameters that can affect the mobilization of species to the liquid phase and that are difficult/impossible to be properly evaluated. Thus, the obtained data in the studied part of the aquifer confirm that is not simple to perform the dating of groundwater masses, even when many variables are taken
into account, because of the complexity of the systems and premisses necessary to obtain reliable data and to apply the models for estimating the ages.

References