Quality of an Underground Hydro Resource in Order to Classify it as Natural Mineral Water for Thermalism: The Case of the New Resource "Termas de São Tiago"

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Abstract. A Medical Spa in Portugal to work legally needs to be supplied by a groundwater that is classified as natural mineral water. After the referred classification, that water is considered as a geological resource and of the public domain, that is, protected by the Portuguese State. Among several studies, mainly hydrogeological, the quality of that natural resource and its physicochemical and microbiological stability, over a hydrological year, is absolutely crucial to obtain the official classification of the resource. Thus, the main objective of this paper is to present the fundamental elements of that domain, in the sense of presenting a case study, which serves as an example to other situations. The results of physicochemical and microbiological analysis, performed monthly, for 12 consecutive months, as well as, the results of a very complete analysis with trace chemical elements, gases, radiological parameters, mineral oils and polycyclic aromatic hydrocarbons, are presented, analysed and discussed. Finally, the main hydrogeochemical classifications of that natural resource are presented, which led to the new mineral water called as "Termas de São Tiago", and which was responsible for the fact that Portugal can currently count on a new medical spa in operation.

1. Introduction
This work is part of the research line of author who has taken in partnership between the University of Beira Interior and several regional entities, such as the Municipality of Penamacor and the Thermal Spa Development Group of Portugal (GDTP), in order to study the quality of underground water masses, elaborate scenarios or hydrogeological models, in order to lead to new abstractions that serve as an anchor for new groundwater exploration projects, namely for thermalism, geothermal applications and bottling.

The study area is located immediately south of the urban area of the border town of Penamacor, about 47 km to NE from its district capital, Castelo Branco, interior region central Portugal (Figure 1).
In geomorphological and geological terms, the region of interest to the present study is associated with the granitoids of Penamacor-Monsanto plutonite. The area of greatest interest to the aquifer system that was selected to be explored, is integrated in a small sub-basin of a very faint waterline and only of grade 1 in the Strahler classification [2]. That sub-basin is generally included within a rectangle of only 500m by 900m, with the largest side according to the maximum development of the water line, with the highest terrain elevation in the order of 483m asl and the lowest in the groundwater abstraction site in the order of 461m asl. That area is surrounded laterally and especially to the West and East by other contiguous sub-basins, with development for upstream, up to the highest urban area of Penamacor at terrain elevation of the order of 605m asl. The distance from the groundwater abstraction site to the most upstream area is in straight line about 1200m.

Geomorphological, geological, hydrogeological and other elements, with some detail were presented in an unprecedented report [3] presented to the Portuguese state to propose superiorly the legalization of the new natural mineral water. The main aspects on those subjects, namely the conceptual hydrogeological model, the characteristics of the groundwater abstraction, among others, were presented in Ferreira Gomes [1]. It should be noted that the resource to be studied comes from confined to semi-confined aquifer system, of granite rocks, of the fissural type, and is captured in a hole, with 324m deep, with an allowable flow rate of 0.7 L/s. After the analytical control of the quality of the resource for 12 consecutive months, and studies of the vulnerability of aquifer systems, definition of the Protection Perimeter, establishment of the Exploration Plan, study on the economic viability, among others, it was possible the Portuguese state to grant a new concession [4], and lead to the current reality: the São Tiago Medical Spa.

2. Methodology

After making available groundwater abstraction, Well P1, there was a need to check whether its resource would have quality and stability of its parameters, in order to be worthy of complying with the rules for to be classified as natural mineral water.

A sampling plan was made, in accordance with Portuguese law [5], in order to perform monthly physicochemical and microbiological analysis for 12 consecutive months, in addition to other unique
analysis, punctually (once only during the 12 months - complete physicochemical analysis), such as research of trace elements, radioactive elements, gases, hydrocarbons, and others.

The various samples were carried out, after being pumped 24 hours in a row at the allowable flow rate of exploration. The physical and chemical analysis were performed by LAIST - Laboratory of Analysis of the Instituto Superior Técnico (of Lisbon). It should be noted that its analysis methodologies are accredited by IPAC according to NP EN ISO/IEC 17025 [6]; The microbiological analysis were performed by the ULS Laboratory, and it should be noted that it is a Public Health Laboratory, where the requirements of Decree-Law 156/98 [7] and Decree 1220/2000 [8] were followed. All the norms and procedures used in the search for the various parameters are recorded together with the presentation of the results (Table 1 to 4).

3. Results and discussions
The results of the physicochemical analysis carried out monthly over a year, of the groundwater of Well P1, at the time of its legalization, are presented in Table 1. The graphical evolution of the main physical-chemical parameters is shown in Figure 2. In order to better clarify the results, Piper, Schoeller-Berkaloff and Stiff diagrams are presented in figures 3 and 4.

By the elements presented, generally the physicochemical stability of the various parameters is very good, because the global parameters and main ions almost globally have a relative standard deviation (DP%) of less than 10%, except for Iron with 13%.

On the other hand, a detailed observation of the graphical evolution of the results over time (Figure 2), shows that there is no any evolution over the year, with the values always being very convergent as is evidenced by figures 3 and 4.

It is also emphasized that some elements, where it was not possible to make statistics of them, but when analyzing them, results were always lower than the limit of quantification, as it was the case of the case: NH\textsubscript{4}+, CO\textsubscript{3}-, NO\textsubscript{3}-, NO\textsubscript{2}-, SH\textsuperscript{-} e H\textsubscript{2}S\textsuperscript{2-}. These situations also reinforce the meaning of chemical stability of the water under study.

The results of the vestigiary species are presented in Table 2. It should be noted the very significant occurrence of Mn with 540 \(\mu\)g/L and then Sr and As with 39 and 35 \(\mu\)g/L, respectively, and still with traces (<\(\mu\)g/L) other elements, which according to their content, from highest to lowest, are the following: Mn> Sr> As> Rb> I> Al> Cs> Nb> U>Nb >U.

In relation to some special elements obtained during the complete physicochemical analysis, the synthesis of the results of gases, radiological parameters and hydrocarbons and pesticides is shown in Tables 3 and 4. From those results, it is emphasized the particularity of not being detected any value for hydrocarbons, pesticides and cyanide, which, if they occur, would make the legalization of water as a natural mineral unfeasible.

About the chemical classification of the water under study was presented in the report of the complete physicochemical analysis, which according to the traditional classification of the "Institute of Hydrology of Lisbon" [9] as a weakly mineralized water, with acid to soft reaction, and silicated. It should be noted that this water presents 24% of silica in non-ionized form, in relation to total mineralization.

Of the main ions, the only anion with significance is Bicarbonate, and in relation to cations, there is the occurrence with some significance of Sodium, Calcium and Magnesium. Thus, in a simple way in ionic terms, water can be called Sodium-Calcium-Magnesium Bicarbonate.
Considering the classifications for total mineralization and hardness [10], the present water is classified as weakly mineralized and slightly hard, respectively.

Regarding resource temperature, considering the water temperature at the head of the Well P1 (18°C), the present groundwater in terms of classification [11] is called hypothermal (< 25°C).

From the microbiological surveys performed monthly throughout the year, the results presented in Table 5 were verified. It should be noted that of all the analysis there are none that are considered inappropriate, with only two samples presenting Cultivable microorganisms above the recommended value.

Table 1. Results of the monthly physicochemical quality control, during one hydrological year, of the groundwater from Well P1, at the time of its legalization.

| Parameter       | Method          | Month (*) | Statistic |
|-----------------|-----------------|-----------|-----------|
| pH              | SMEWW 4500 H.P.| 1 2 3 4 5 6 7 8 9 10 11 12 m a M SD(%) |          |
| C. (µS.cm⁻¹)    | NP EN 27888-C. | 196 193 199 196 196 187 185 187 185 184 188 196 185 192 199 | 2.5      |
| A₇ – (CaCO₃)   | SMEWW 2320 B-V. | 86.6 84.9 89.5 93.1 87.4 87.9 89.2 88.5 85.3 90.0 83.2 88.7 82.2 87.3 93.1 | 3.4      |
| H₂S – (mg/L)   | MM (COL)-Col. | <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 <0.5 | -0.5     |
| Silica (mg/L)  | SMEWW 4500 Si-C | 50 52 52 53 52 53 53 50 50 52 52 50 50 52 53 | 2.3      |
| Ha - (CaCO₃)   | SMEWW 2340-calc. | 69 69 65 67 67 64 63 63 62 67 61 61 65 69 | 4.1      |
| Rₒ at 180°C    | SMEWW 1030-G. | 161 162 162 165 162 165 158 159 158 165 156 158 156 161 161 | 1.7      |
| M₇ (mg/L)      | MM 2.1.11 calc. | 214 214 220 222 215 217 209 213 210 220 207 212 207 214 224 | 2.1      |
| Na⁺            | EPA 300.7-C.I  | 18.6 18.1 17.3 17.0 17.3 16.8 16.3 16.0 16.7 17.3 17.1 16.1 16.0 17.1 18.6 | 4.3      |
| Ca²⁺           | EPA 300.7-C.I  | 14.3 14.5 13.2 13.6 13.9 13.3 12.8 12.9 12.9 13.8 13.6 14.1 12.8 13.6 14.5 | 4.0      |
| K⁺             | EPA 300.7-C.I  | 1.4 1.4 1.4 1.2 1.3 1.3 1.2 1.2 1.3 1.4 1.2 1.3 1.4 1.4 5.8    |
| Ca²⁺           | EPA 300.7-C.I  | 8.1 7.9 7.7 8.0 7.8 7.6 7.6 7.4 7.3 7.8 6.7 8.1 6.7 7.7 8.1 | 4.9      |
| NH₄⁺           | MM 4.1 EAM     | <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 | -0.05     |
| Li⁺            | EPA 300.7-C.I  | <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 | -0.1     |
| Fe²⁺           | EPA 300.7-C.I  | 2.4 2.4 2.7 2.3 2.3 1.9 2.8 2.7 3.1 2.8 2.9 2.8 | 2.6 3.1 11.8 |
| HCO₃⁻          | SMEWW 2320 B-V/P. | 105 103 108 113 106 106 99 107 103 109 101 107 | 99 106 106 113 3.4   |
| CO₃⁻           | SMEWW 2320 B-V/P | <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 | -2 -2     |
| Cl⁻            | SMEWW 4110-C.I | 8.8 8.6 8.6 8.4 8.9 8.9 8.6 8.5 8.7 8.8 8.5 8.2 8.2 8.6 8.9 | 2.3      |
| Anions SO₂⁻²   | SMEWW 4110-C.I | 5.6 5.9 5.5 5.0 5.6 6.0 6.0 6.7 6.4 6.4 6.0 | 5.0 5.9 6.7 7.4 |
| F⁻             | SMEWW 4110-C.I | 1.2 1.1 1.1 1.1 1.0 1.0 1.0 1.0 1.0 | 0.9 1.0 1.2 7.2    |
| NO₃⁻           | SMEWW 4110-C.I | <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 <0.3 | -2 6 -7    |
| NO₂⁻           | SMEWW 4500-(EAM) | <0.01 <0.01 <0.01 <0.01 | <0.01 <0.01 <0.01 | <0.01 | -0.01 |
| SH²            | calc.          | <0.5 <0.5 <0.5 <0.5 <0.5 <0.3 <0.5 | <0.5 | -0.5 |

(*) the first analysis was performed in May 2014.
Figure 2. Graphical evolution of the main physicochemical parameters over the course of a year, from water analysis of the Well P1, at the time of its legalization.

Table 2. Results of the search for vestigial elements from water analyzes of the Well P1, at the time of its legalization.

| Element | Ag | Al | As | B | Ba | Be | Bi | Br | Cd | Cr | Cu | Hg | I | Mn |
|---------|----|----|----|---|----|----|----|----|----|----|----|----|---|---|
| Technique | MM | MM | MM | ICP | ISO | 11885 ICP | MM | 11885 ICP | SME | ICP | MM | MM | MM | W- |
| Value | < 1 | 6 | 35 | <30 | <30 | 0.6 | <50 | <100 | <1 | <2 | <1 | <2 | <2 | <0.2 | 7 | 540 |
| µg/L | | | | | | | | | | | | | | |

| Element | Mo | Nb | Ni | Pb | Rb | Sb | Se | Sn | Sr | U | V | W | Y | Zn | Zr |
|---------|----|----|----|----|----|----|----|----|----|---|---|---|---|----|----|
| Technique | MM | MET | MSF | MM | MM | MET | MM | 5.4 | AA- | Iso | Iso | Iso | Iso | 11885 ICP | 11885 ICP |
| Value | <5.0 | 2.6 | <5.0 | <3.0 | 7.1 | <1.0 | 0.4 | <5.0 | 39 | 1.8 | <10 | <1.0 | <0.5 | <50 | <50 |
| µg/L | | | | | | | | | | | | | | | |

Note: the values indicated with "<" correspond to the quantification limits of the respective parameters.
Figure 3. Chemical diagrams for all water samples from Well P1, at the time of its legalization.
Figure 4. Stiff diagrams for all water samples from Well P1, at the time of its legalization.

Table 3. Results of the survey of gases and radiological parameters of the water samples from Well P1, at the time of its legalization.

| Parameter                  | Technique                | Result |
|----------------------------|--------------------------|--------|
| Gases                      |                          |        |
| Free CO₂ – (mg/L)          | MM 2.2.7 – Calculation   | 42     |
| Radon – Rn (Bq/L)          | W-RN222LSC               | 105    |
| Sulphidic acid (H₂S)       | MM – Colorimetry         | < 0.50 |
| Dissolved oxygen (O₂)      | SMEWW 4500 O B and C - Volumetry | < 1.0 |
| Radiological parameters    |                          |        |
| 𝛼total (Bq/L)              | W-GAA-SCI                | 0.118  |
| 𝑝total (Bq/L)              | W-GBA-PRO                | 0.220  |

Note: the values indicated with “<“ correspond to the quantification limits of the respective parameters.
Table 4. Results of the search for special parameters of the water samples from Well P1, at the time of its legalization.

| Parameter                          | Technique   | Result  |
|------------------------------------|-------------|---------|
| Hydrocarbons - Mineral oils (µg/L) | MM 8.12 FTIR| < 10    |
| Polycyclic Aromatic Hydrocarbons  | Fluoranthene| MM 6.5.1 HPLC| < 0.005 |
| (PAH's) (µg/l)                     | Benzo(b) fluoranthene| MM 6.5.1 HPLC| < 0.005 |
|                                   | Benzo (k) fluoranthene| M M 6.5.1 HPLC| < 0.005 |
|                                   | Benzo(a) pyrene| M M 6.5.1 HPLC| < 0.005 |
|                                   | Benzo(g, h, i) perylene| M M 6.5.1 HPLC| < 0.005 |
|                                   | Indeno (1,2,3-cd) pyrene| M M 6.5.1 HPLC| < 0.005 |
| Pesticides (µg/L)                 | Linuron     | M M 6.5.1 HPLC| < 0.05  |
| Cyanide (µg/L)                     | -           | EAM     | < 10    |

Note: the values indicated with “<” correspond to the quantification limits of the respective parameters.

Table 5. Results of the monthly microbiological control, during one hydrological year, of the groundwater from Well P1, at the time of its legalization.

| Survey | VR(1) | VL 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|--------|-------|------|---|---|---|---|---|---|---|---|----|----|----|
| 1      | 5     | 1    | 0 | 2 | 0 | 7 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 2      | 20    | -    | 0 | 100| 0 | 0 | 16| 0 | 3 | 2 | 1  | 0  | 0  |
| 3      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 4      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 5      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 6      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 7      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |
| 8      | -     | 0    | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0  | 0  | 0  |

(1) Surveys and Techniques:
1. Quantification of Cultivable microorganisms at 37º C (UFC/1 mL) ISO 6222 :1999
2. Quantification of Cultivable microorganisms at 22º C (UFC/1 mL) ISO 6222:1999
3. Research and Quantification of coliforms (UFC/250 mL) LSPG PE 02 MB (2013-10-16)
4. Research and Quantification of Faecal coliform (UFC/250mL) LSPG PE 50 MB (2014-01-22)
5. Research and Quantification of Escherichia coli (UFC/250 mL) LSPG PE 02 MB (2013-10-16)
6. Research and Quantification of Enterococcus faecalis (UFC/250 mL) ISO 7899-2:2000
7. Research and Quantification of Pseudomonas aeruginosa (UFC/250 mL) LSPG PE 05 MB (2012-09-14)
8. Research and Quantification of spores of Anaerobic sulfite-reducing bacteria (UFC/50 mL) EN 26 461-2:1994

(2) the values presented in this column are those recommended for ingestion and contact with the respiratory mucous membranes, however for baths and showers, the values are 20 and 100, respectively. (3) (* 3) the first month of surveys was in May.

4. Conclusions
The Well P1, vertical, with 324m depth, with characteristics of definitive abstraction, presents a resource (groundwater) which after a quality control, according to Portuguese legislation, proved to be an adequate water to be used in a medical spa, because it presents stability in its quality, with a stable chemical composition, without evolutionary trends over a hydrological year, does not present chemical elements or organic and inorganic compounds of anthropogenic origin that are harmful to human health, and in microbiological terms is appropriate for applications in the activity for a medical spa. So, the water under study was classified as natural mineral water.

According to the classical classifications, in global terms it is a weakly mineralized water (total mineralization ≈ 214 mg/L), slightly hard (Hardness - CaCO₃ ≈ 65 mg/L), with acid to soft reaction (pH ≈ 6.72), silicated (silica ≈ 52mg/L), and hypothermal (T ≈18°C). In ionic terms it is called sodium-calcium-magnesium bicarbonate water.
That water, after classification as natural mineral water, was subject to a medical-hydrological study under the responsibility of a doctor, having proved its suitability for rheumatic, musculoskeletal, and respiratory diseases [12], and can therefore definitely be used in the activity of a medical spa.

Finally, it is emphasized that Portugal currently offers a new medical spa, in association with a Hotel [13], and that it will have a lasting future, if among many others, above all preserve the stability of the quality of the resource, and for this it is absolutely essential to implement monitoring systems, namely for water flow rate control to be extracted at groundwater abstraction (Well P1), consequent piezometric levels, quality of the resource, in addition to the imposition of appropriate restrictions regarding activities and occupation of the territory, within the protection zones of the aquifer system (Immediate Protection Zone, Intermediate Protection Zone, and Extended Protection Zone).

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Abbreviations
a = average
AA-CG = Atomic Absorption Spectrophotometry with Graphite Chamber
AA-GH = Atomic Absorption Spectrophotometry with Hydride Generator
A_T = Total Alkalinity
calc. = calculation
C. = Conductivity
C. = Condutimetry
C.I. = Ionic Chromatography
Col. = Colorimetry
EA-VF = Atomic Absorption Spectroscopy with cold vapour generator
EAM = Molecular Absorption Spectrophotometry
EN = European Norm
EPA = Environmental Protection Agency
FTIR = Fourier Transform Infrared Spectroscopy
G. = Gravimetry
Ha = Hardness
HPA = Health Protection Agency
ICP = Optical Emission Spectrometry with Coupled Inductive Plasma
IEC = International Electrotechnical Commission
IPAC = Portuguese Accreditation Institute
ISO = International Standard Organisation
LQ = Quantification Limit
LSPG PE xx MB = Microbiological Internal Laboratory Procedure
m = minimum
M = maximum
MM = Internal Method
M_T = Total mineralization
n.d. = undetected
NE = Northeast
NP = Portuguese Norm
NP EN = Portuguese version of the European Norm
P. = Potentiometry
R_D = Dry residue
SDR = Relative Standard Deviation
SMEWW = Standard Methods for the Examination of Water and Wastewater
UFC = Colony Forming Units
ULS = Local Unit of Health
VL = Legal Limit Value
VR = Recommended Value
V. = Volumetry
V./P. = Volumetry /Potentiometry
W-METMSFX3 (ICP-MS) = Mass Spectrometry with coupled inductive plasma
W-GAA-SCI = Determination of gross alpha activity by measuring of evaporated residue in a mixture with ZnS (Ag) scintillator.
W-GBA-PRO = Determination of gross beta activity by measuring of evaporated residue by means of proportional detector and determination of gross beta activity corrected for potassium 40 by calculation from measured values
W-RN222 LSC = Determination of Rn-222 by liquid scintillation counting method

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