

Erratum

In Article “Chemical quality of bottled mineral waters from markets of Curitiba-PR-Brazil”, with the number of DOI: <http://dx.doi.org/10.1590/1678-4324-2016160111>, published in journal Brazilian Archives of Biology and Technology, vol. 59, the 01 page.

That read:

“Éder José dos Santos^{1*}, Dasio Roberto de Oliveira Junior¹, Amanda Beatriz Hermann¹, Ralph Edwatd Sturgeon².”

¹ Instituto de Tecnologia do Paraná – TECPAR – Centro de Tecnologia em Saúde e Meio Ambiente, Curitiba, Paraná, Brasil; ²Measurement Science and Standards, National Research Council Canada, Ottawa, Canada.”

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“In Brazil, in accordance with RDC Resolution No. 274/2005 of the Ministry of Health, mineral water is “water obtained directly from natural or ground water extraction sources”. Concern over the quality of mineral water on the market is highlighted in several articles devoted to this subject. Ikem et al. investigated the chemical quality of twenty-five bottled waters from cities in the state of Alabama, USA, through a one-way parametric analysis of variance (ANOVA) and showed that several samples available on the market had physical-chemical parameters (pH and conductivity) as well as essential and non-essential elements exceeding the EU and US EPA drinking water standards (Ikem et al., 2002).”

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“In these studies, the concentrations of inorganic ions as well as parameters such as pH, electrolytic conductivity and inorganic and total organic carbon were determined. For this purpose, the main analytical techniques employed were ion chromatography (IC), inductively coupled plasma optical emission spectrometry (ICP OES) and inductively coupled plasma mass spectrometry (ICP-MS). In Brazil, studies have also highlighted concern over the quality and composition of bottled mineral waters available on the market. Godoy et al. investigated the natural radionuclides ^{228}Ra , ^{226}Ra and ^{210}Pb in Brazilian bottled mineral waters (Godoy et al., 2001).”

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Table 1. ICP OES, CVG-ICP OES and ICP-MS instrumental parameters.

	ICP OES	CVG-ICP OES	ICP-MS
Radiofrequency	40 MHz	40 MHz	27.12 MHz
Forward power	1.25 kW	1.2 kW	1.4 kW
Plasma gas flow rate	15.0 L min ⁻¹	15.0 L min ⁻¹	18.0 L min ⁻¹
Auxiliary gas flow rate	1.5 L min ⁻¹	1.5 L min ⁻¹	1.8 L min ⁻¹
Sheath gas flow	-----	-----	0.21 L min ⁻¹
Carrier gas flow rate	-----	120 mL min ⁻¹	-----
Nebulizer pressure	180 kPa	-----	-----
Nebulizer type	Concentric glass K	-----	-----
Spray chamber	Standard Axial Varian	Standard Axial Varian	-----
Replicate read time	15 s with 4 replicates	15 s with 4 replicates	20 s with 8 replicates
Torch type	1.8 mm I.D. alumina injector tube	Quartz for axial view	Quartz
NaBH ₄ concentration	-----	0.3 % (m/v) in 0.4%, m/v, NaOH	-----
HCl concentration	-----	5 mol L ⁻¹	-----
Analytical lines / isotopes (ICP-MS)	Al: 396.152 nm, Ba: 455.403 nm, Cd: 214.439 nm, Cr: 267.716 nm, Cu: 327.395 nm; Fe: 238.204 nm, Li: 610.365 nm, Mn: 257.610 nm, Ni: 231.604 nm, Sr: 421.552 nm, V: 292.401 nm, Zn: 213.857 nm	Hg: 252.652 nm	^{75}As , ^{121}Sb , $^{206,207,208}\text{Pb}$ and ^{78}Se
Collisional gas CRI	-----	-----	H ₂ (^{78}Se)
Internal standard	-----	-----	^{89}Y , ^{115}In

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Collisional gas CRI	-----	-----	H ₂ (⁷⁸ Se)
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“**Reagents and samples:** All chemicals were of analytical grade, unless otherwise specified. High-purity water (18.2 MΩ cm resistivity) was de-ionized in a Milli-Q system (Bedford, MA, USA). The following Merck reagents (Darmstadt, Germany) were used: NaBH₄ to prepare a 0.3 % m/v solution in NaOH; 65% v/v HNO₃suprapur® ; 37% v/v HCl from which a 5 mol L⁻¹ solution was prepared. Buffer solutions for pH 4.00 and pH 7.00 were supplied by Êxodo (Hortolândia, Brasil). Monoelemental standard solutions containing 1000 µg mL⁻¹ of Al, As, Hg, In, K⁺, Mn, Na⁺, Pb, Se, Sb, Sr, V, Y, Zn, Cl⁻, NO₂⁻ and NH₄⁺ were obtained from AccuStandard (New Haven, USA); those for Ba, Cd, Cr, Ca²⁺, Cu, Fe, Li, Mg²⁺ and Ni from UltraScientific (North Kingstown, USA), while solutions of F⁻, NO₃⁻ and SO₄²⁻ were supplied by FlukaAnalytical (St. Louis, USA). A conductivity standard of 84 µS cm⁻¹ was obtained from Mettler Toledo. Quality control Certified Reference Materials comprising acidified aqueous solutions containing 100 µg mL⁻¹ As, Sb, Ca, Cd, Cr, Cu, Fe, Li, Mg, Mn, Ni, Pb, Se, Sr, V and Zn (QCS-01-5), 100 µg mL⁻¹ of Al, Ba, K and Na (QCS-02-R1-5) and 20 µg mL⁻¹ of Hg (TCLP-02-1) were obtained from AccuStandard. These served as quality control standards.”

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Table 4. Figures of merit and results for analysis of quality control materials and standards (n=7).

Analyte	Linear correlation coefficients	Limit of detection ($\mu\text{g L}^{-1}$)	Limit of quantification ($\mu\text{g L}^{-1}$)	of expected concentration ($\mu\text{g L}^{-1}$)	recovery	RSD, %
Al	0.9998	2	10	10.0	10.0 ± 0.2	± 2.0
As	0.9997	0.1	1.0	10.0	10.3 ± 0.2	± 1.9
Ba	0.9999	1	5	10.0	11.0 ± 0.1	± 0.9
Ca ²⁺	0.9999	10	100	100	106 ± 4	4
Cd	0.9999	0.2	1.0	10.0	9.6 ± 0.1	1.0
Cu	0.9996	1	10	10.0	10.1 ± 0.2	± 2.0
Cr	0.9999	0.2	1.0	10.0	9.8 ± 0.1	1.0
Cl ⁻	0.9999	30	100	100	101 ± 10	10
F ⁻	0.9993	1	20	10.0	9.1 ± 0.3	3.3
Fe	0.9999	2	10	10.0	10.5 ± 0.1	± 1.0
Hg	0.9980	0.04	0.2	1.0	0.9 ± 0.03	± 3.3
K ⁺	0.9999	10	100	100	102 ± 3	3
Li	0.9996	2	10	10.0	11.2 ± 0.2	± 1.8
Mn	0.9999	0.2	1.0	10.0	10.2 ± 0.2	± 1.8
Mg ²⁺	0.9999	5	100	100	98 ± 2	2
NO ₃ ⁻	0.9999	5	100	100	89 ± 1	1
NO ₂ ⁻	0.9999	1	10	10.0	10.2 ± 0.3	± 2.9
NH ₄ ⁺	0.9999	10	30	30.0	33.0 ± 2.1	± 6.4
Na ⁺	0.9999	10	100	100	100 ± 4	4
Ni	0.9999	2	10	10.0	9.5 ± 0.3	3.2
Pb	0.9995	0.1	1.0	10.0	10.2 ± 0.2	± 2.0
Sb	0.9996	0.2	1.0	10.0	11.1 ± 0.3	± 2.7
Sr	0.9997	1	5	10.0	10.0 ± 0.1	± 1.0
Se	0.9987	0.1	1.0	10.0	10.1 ± 0.1	± 1.0

SO ₄ ²⁻	0.9999	10	100	100	98 ± 2	2.0
V	0.9999	2	10	10.0	9.0 ± 0.1	1.1
Zn	0.9999	2	10	10.0	9.6 ± 0.3	3.1

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“Tables 5 and 6 summarize results obtained characterizing the twenty-seven commercial samples purchased for this study. The presence of NH₄⁺ and NO₂⁻ are indicative of microbiological contamination. The concentration of NH₄⁺ was below the limit of quantification but for NO₂⁻ samples 14, 19 and 21 (0.01 mg L⁻¹) concentrations near the maximum limit set by Brazilian legislation were evident whereas that for sample 16 (0.04 mg L⁻¹) exceeded the maximum limit set by RDC N° 274, 2005, i.e., 0.02 mg/L. ”

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“Iron is only detected in two imported samples from Italy: $10 \text{ } \mu\text{g L}^{-1}$ (sample 26) and $21 \text{ } \mu\text{g L}^{-1}$ (sample 27), but its concentration is not declared on the sample labels. Selenium is present in the range $1.2 - 6.4 \text{ } \mu\text{g L}^{-1}$ (four samples) and V in the range $10 -150 \text{ } \mu\text{g L}^{-1}$ (ten samples). Sample 24, imported from France, and sample 17, from Paraná State, are identified as containing the highest concentrations of Se: $6.4 \pm 0.3 \text{ } \mu\text{g L}^{-1}$ and V: $150 \pm 5 \text{ } \mu\text{g L}^{-1}$, respectively. According to Brazilian legislation, these samples should also be classified as mineral waters “seleniada”, i.e., that contains selenium at minimum $6 \text{ } \mu\text{g L}^{-1}$ and maximum of $10 \text{ } \mu\text{g L}^{-1}$, and “vanadica”, i.e., that contain vanadium at minimum $30 \text{ } \mu\text{g L}^{-1}$ (Portaria DNPM 540, 2014 and RDC N° 274, 2005). However, no statement on the label can be found. With respect to trace element content and potential contaminants, all samples should have more information concerning their concentrations.”

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“Although the physico-chemical parameter pH showed values different from those declared on the labels, as summarized in Table 3, these differences were not significant and in these cases the producers indicate that the values correspond to those characterizing the source of the water. These differences are acceptable since chemical transformations may occur over time that could slightly affect the pH. The pH range varies from mildly acidic (5.53 ± 0.01 , sample 23) to slightly alkaline (8.89 ± 0.10 , sample 24). Alkalinity due to the presence of CO_3^{2-} was detected in four samples where the reported pH was 8.49 ± 0.01 (sample 14), 8.79 ± 0.03 (sample 17), 8.44 ± 0.05 (sample 19) and 8.89 ± 0.01 (sample 24). Figure 1 shows the correlation between conductivity and total dissolved solids (TDS) parameters which indicates that the higher the sample conductivity, the greater the amount of TDS present. These two parameters are strongly correlated, especially with imported samples 24 (France), 25 (Portugal) and 26 (Italy). Conductivity is in close agreement with declared values except for sample 01 for which a value of $3.7 \pm 0.1 \text{ } \mu\text{S cm}^{-1}$ was determined whereas $153 \text{ } \mu\text{S cm}^{-1}$ declared, and the producer will require notification.”

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“Likewise, the hardness present in the sample can be correlated mainly to the presence of Ca and Mg. Samples 24-26 are again highlighted for their significant values present. The main constituent elements reported on the sample labels include: HCO_3^- , F⁻, Cl⁻, NO_3^- , SO_4^{2-} , Na⁺, K⁺, Mg²⁺, Ca²⁺, Ba, Li and Sr. Principal component analysis (PCA) for the concentrations of these analytes is presented in Figure 2, where PC1 is the first principal component, PC2 is the second and PC3 is the third (Dos Santos et al., 2008, Dos Santos et al., 2012 and Miller, J. N. and Miller, J. C., 2000). PC1 accounts for 47% of the variance of the data and discriminates samples through the influence of parameters related to HCO_3^- , F⁻, Cl⁻, Ca²⁺, and Li. PC2 accounts for 17% of the variance of the data and discriminates samples on the basis of SO_4^{2-} , while PC3 accounts for 12% of the variance of the data and is mainly correlated with the presence of Ba and Sr. Together, 76% of the variability of the original data is thus accounted for. As evident from Figure 2, the samples are clustered in a main group but five are located in different regions of the graph. Samples 23 to 26 are imported: 23 and 24 from France, 25 from Portugal and 26 from Italy. Only sample 22 is from Minas Gerais State, Brazil. It is evident that these samples have different physicochemical compositions, being characterized by relatively high concentrations of salts, mainly HCO_3^- and Sr. Legislation does not specify a value for Sr, and the HCO_3^- ion is an essential component of mineral waters (Decreto-Lei 7841, 1945). These five samples are also rich in F⁻, providing a classification as “fluoridated mineral water”, as defined by Brazilian legislation (Portaria DNPM 540, 2014). The concentration of Li is high in samples 22, 24, 25 and 26, also a characteristic of mineral water in accordance with Brazilian legislation (Portaria DNPM 540, 2014). The concentration of Ba in sample 22 is the highest: $386 \pm 5 \mu\text{g L}^{-1}$, but below the maximum specified by Brazilian legislation, i.e., $700 \mu\text{g L}^{-1}$ (RDC N° 274, 2005). The main cluster comprises the majority of the samples that are bottled in the State of Paraná, with the exception of sample 11 from São Paulo State and sample 27 from Italy. This cluster is characteristic of the presence of NO_3^- . Nitrate may have either a mineral or microbiological origin. Its determinable concentration ranges from 0.10 mg L^{-1} - 63.05 mg L^{-1} while samples 01, 15 and 25 are below the LOQ. Only sample 10 (63.05 mg L^{-1}) is above the maximum specified by Brazilian legislation, i.e., 50 mg/L (RDC N° 274, 2005), **but the label states a value of 3.47 mg L^{-1} (Table 3)**. The elevated concentration may be indicative of microbiological contamination which may be harmful to human health (Astel et al., 2014). Samples 01, 10 and 21 are classified as “fluoridated mineral water”, according to their labels; however, in this study, F⁻ was not detected above 0.02 mg L^{-1} , the minimum, according to Brazilian legislation

(Portaria DNPM 540, 2014), needed for water to be classified as such. Since the declared values are close to the minimum, analyte sorption by the packaging materials may have occurred. It is evident that samples 3 and 4 have the same mineral composition. In reality, these samples are from the same region (Apucarana city, State of Paraná), but on the market they have a different identification and no indication that they are from the same producer. In general, it is clear that information declared on the label is different from that arising from analysis of the samples in this work, but it must also be recognized that as the samples are rich in salts, several factors may lead to changes in the composition, however small, including decomposition of HCO_3^- , sorption of analytes onto the walls of the container material, the time on the market shelf, and proper care during transport and the ambient storage temperature. To make a more qualified analysis of the national samples, a scatter plot based on only national Brazilian samples is presented in Figure 3. Here the PCs account for 73% of the variability of the original data. As in Figure 2, variability in the compositions of samples are evident wherein the difference of mineral content of sample 22 in relation to others is evident. The sample 10 possibly is contaminated and the samples 15 and 17 show F^- content and Na^+ higher concentrations than others. Clearly, the PCA statistical tool can be very useful for identification and comparison of compositions of bottled mineral waters on the market.”

Read:

“Likewise, the hardness present in the sample can be correlated mainly to the presence of Ca and Mg. Samples 24-26 are again highlighted for their significant values present. The main constituent elements reported on the sample labels include: HCO_3^- , F^- , Cl^- , NO_3^- , SO_4^{2-} , Na^+ , K^+ , Mg^{2+} , Ca^{2+} , Ba, Li and Sr. Principal component analysis (PCA) for the concentrations of these analytes is presented in Figure 2, where PC1 is the first principal component, PC2 is the second and PC3 is the third (Dos Santos et al., 2008, Dos Santos et al., 2012 and Miller, J. N. and Miller, J. C., 2000). PC1 accounts for 47% of the variance of the data and discriminates samples through the influence of parameters related to HCO_3^- , F^- , Cl^- , Ca^{2+} , and Li. PC2 accounts for 17% of the variance of the data and discriminates samples on the basis of SO_4^{2-} , while PC3 accounts for 12% of the variance of the data and is mainly correlated with the presence of Ba and Sr. Together, 76% of the variability of the original data is thus accounted for. As evident from Figure 2, the samples are clustered in a main group but five are located in different regions of the graph. Samples 23 to 26 are imported: 23 and 24 from France, 25 from Portugal and 26 from Italy. Only sample 22 is from Minas Gerais State, Brazil. It is evident that these samples have different physicochemical compositions, being characterized by relatively high concentrations of salts, mainly HCO_3^- and Sr. Legislation does not specify a value for Sr, and the HCO_3^- ion is an essential component of mineral waters (Decreto-Lei 7841, 1945). These five samples are also rich in F^- , providing a classification as “fluoridated mineral water”, as defined by Brazilian legislation (Portaria DNPM 540, 2014). The concentration of Li is high in samples 22, 24, 25 and 26, also a characteristic of mineral water in accordance with Brazilian legislation (Portaria DNPM 540, 2014). The concentration of Ba in sample 22 is the highest: $386 \pm 5 \mu\text{g L}^{-1}$, but below the maximum specified by Brazilian legislation, i.e., $700 \mu\text{g L}^{-1}$ (RDC N° 274, 2005). The main cluster comprises the majority of the samples that are bottled in the State of Paraná, with the exception of sample 11 from São Paulo State and sample 27 from Italy. This cluster is characteristic of the presence of NO_3^- . Nitrate may have either a mineral or microbiological origin. Its determinable concentration ranges from 0.10 mg L^{-1} - 63.05 mg L^{-1} while samples 01, 15 and 25 are below the LOQ. Only sample 10 (63.05 mg L^{-1}) is above the maximum specified by Brazilian legislation, i.e., 50 mg/L (RDC N° 274, 2005), but the label states a value of 3.47 mg L^{-1} (Table 3). The elevated concentration may be indicative of microbiological contamination which may be harmful to human health (Astel et al., 2014). Samples 01, 10 and 21 are classified as “fluoridated mineral water”, according to their labels; however, in this study, F^- was not detected above 0.02 mg L^{-1} , the minimum, according to Brazilian legislation (Portaria DNPM 540, 2014), needed for water to be classified as such. Since the declared values are close to the

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The present study reveals that, in general, the majority of parameters determined in commercial bottled mineral waters from markets in Curitiba-PR-Brazil are below limits specified by Brazilian legislation. Only one sample was detected as having a high concentration of NO_3^- , indicating microbiological contamination; some results for NO_2^- may also reflect the same contamination. Improvements in monitoring throughout the process of filling and points of sale of the samples, with the aim of securing the microbiological quality of bottled mineral waters sold in the region under study may be needed. The comparison of labelled composition to analytical results highlights a difference for most elements, but it should be noted that intrinsic instability as well as other factors such as sorption of components onto container surfaces as well as the length of time spent on the shelf, care during transport, and the ambient storage temperature may alter the composition; perhaps a “best before date” could eliminate such discrepancies. In such cases, a further study of the stability and interaction with the packaging should be performed. Although a perfect division of samples from

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