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IONIZING RADIATION AS OPTIMIZATION METHOD FOR ALUMINUM DETECTION FROM DRINKING WATER SAMPLES

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ABSTRACT

The presence of organic compounds in water samples is often responsible for metal complexation; depending on the analytic method, the organic fraction may dissemble the evaluation of the real values of metal concentration. Pre-treatment of the samples is advised when organic compounds are interfering agents, and thus sample mineralization may be accomplished by several chemical and/or physical methods. Here, the ionizing radiation was used as an advanced oxidation process (AOP), for sample pre-treatment before the analytic determination of total and dissolved aluminum by ICP-OES in drinking water samples from wells and spring source located at Billings dam region. Before irradiation, the spring source and wells' samples showed aluminum levels of 0.020 mg/l and 0.2 mg/l respectively; after irradiation, both samples showed a 8-fold increase of aluminum concentration. These results are discussed considering other physical and chemical parameters and peculiarities of sample sources.

Keywords: drinking water, ionizing radiation, aluminum
.

1. INTRODUCTION

The high demand for water by agricultural and industrial production increased the damage to the environment [1]. Mining, smelting operations and tanning are the industrial activities that cause the highest level of heavy metal contamination in freshwater sources. This contamination represents a threat to both the human and the environment health, even in low concentrations [2].

Aluminum is not classified as a heavy metal; however, it also is toxic to humans and the environment. In the natural environment, aluminum is an abounding metallic element found on soils and rocks; it is typically present on concentrations from 7% to 10% on highly insoluble compounds, which cannot be leached with percolating water. However, under highly acidic conditions many natural aluminum compounds are dissolved, releasing the cations Al^{3+} and $Al(OH)^{2+}$. In the same way, highly alkaline conditions release anions $Al(OH)^{-}$. These ions are water soluble and have high toxicity to the natural life [3].

On humans, aluminum contamination occurs mostly through oral route and its sources are drinking water, cooking utensils, packaging, anti-acid and antiperspirant formulations [4]. Seborrhea, hair loss, cerebral sclerosis (Alzheimer), irritability, displacement of calcium and magnesium from bones (osteoporosis) are human diseases related to aluminum contamination [5,6].

Environmental regulatory authorities established maximum limits for the presence of metals in drinking water. It is expressed as the *maximum contaminant levels* (MCL) and, according to European and Brazilian regulations, the maximum concentration of aluminum in drinking water is 200 ppb; for US-EPA regulations these limits are between 50~200 ppb.

Aluminum ions can be determined by several techniques and methods [7,8]; sample pre-treatment and precision limits must be considered before analytic quantification.

As sample pre-treatment, mineralization may be accomplished by ionizing radiation, a kind of AOP. The irradiation process promotes degradation of organic matter and it is done without additives. This is an advantage, because it does not introduce any kind of contaminant. Also, the irradiation process may be attractive if the water sampling occurs close to the bottom of the well – the organic matter turbidity may be decreased by irradiation process and all metals released for quantification; it may also eliminate a filtration step before quantitative analysis [9].

In this work, drinking water samples from wells and spring source located at Billings dam region (São Paulo city – Brazil) were pre-treated by irradiation process for organic matter degradation. The aluminum specie was determined by ICP-OES in irradiated and non-irradiated samples. Other physical and chemical parameters and peculiarities of sample sources were measured and these all results were discussed.

2. EXPERIMENTAL METHODOLOGY

2.1. Sampling site localization

Bororé “island” is a tiny continental strip of land on the Billings dam. It is located south of São Paulo city and its main access is by ferry boat (Fig.1). Three domestic wells were chosen (points P1, P8 and P9) for sampling drinking water. These are low depth wells (less than 20 m) and thus they are classified as shallow or pond wells. Another characteristic of these sampling points is their closeness to the septic tanks and to the dam shore (about 6-10 meters). These samples did not undergo any antibacterial treatment. Other water samples were collected from the Billings dam and from a spring source in the studied region. The samples were collected between August 2011 and July 2012.

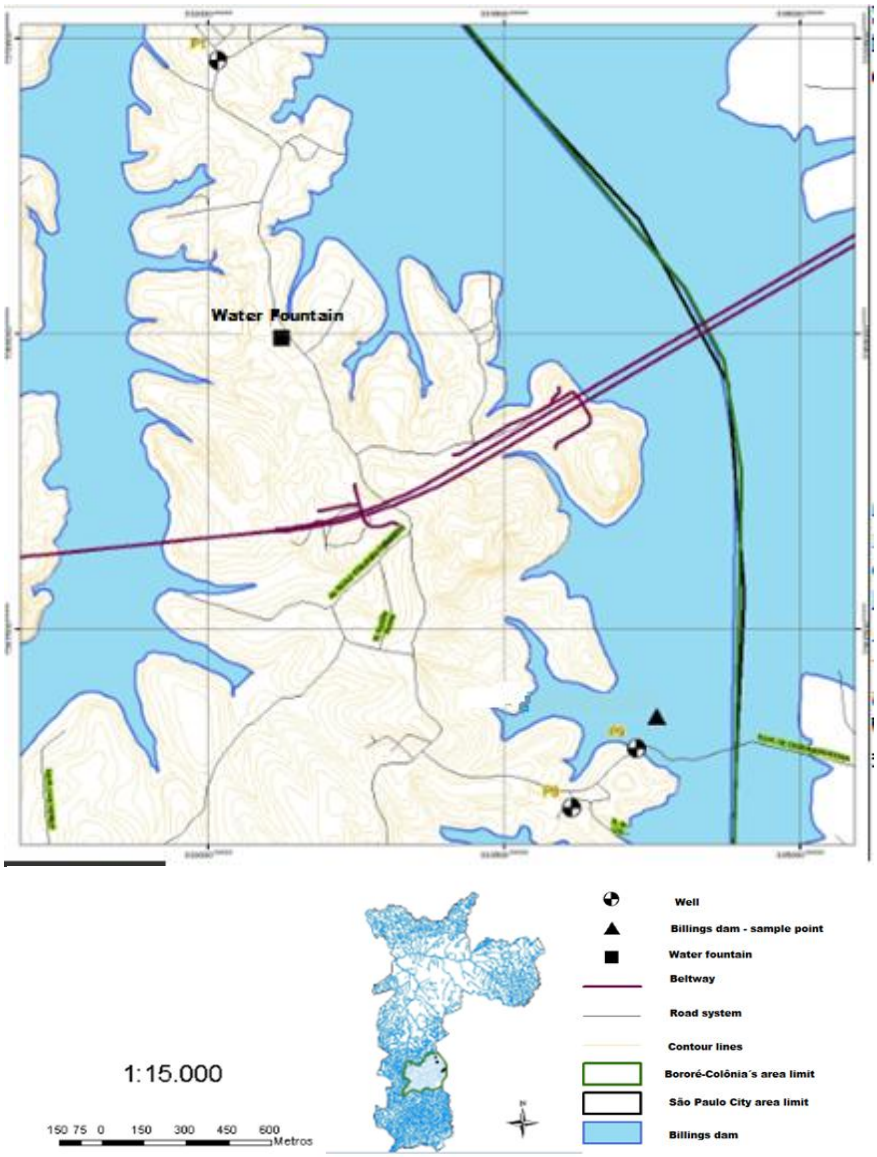

Figure 1. Map of sampling points

Two types of samples were collected: the first one was filtered on the field with manual filtration equipment equipped with a 0.45µm membrane (Millipore ®) as active filtering element; the other sample did not undergo any kind of treatment after being collected. For sampling ground water from wells it was used an appropriate sample device - a disposable bailer sampler. The sampling operation was based on ASTM D6699 – 01(2010) standard, which describes the procedure for sampling stratified or un-stratified water from wells.

2.2. Physical and chemical parameters

The following physical-chemical analyses were based in APHA (American Public Health Association – 1999) and were also measured in the field by a multi-parameter probe model 9828 Hanna: Total Dissolved Solids (TDS–mg^{-1}), electrical conductivity (µS/cm), turbidity (NTU), temperature of water (^{o}C) and hydrogen potential (pH). The methodology for these measurements was based in APHA (1999).

2.3. Irradiation Process

The mineralization process of the water samples was performed by gamma radiation from a ^{60}Co source of a Gammacell-220. The maximum dose rate was 1.33 kGy/h and the absorbed doses applied were: 10 kGy, 20 kGy, 40 kGy and 60 kGy. A process blank (distilled water) was also used to check for possible interfering reagents or inadequate decontamination of containers.

2.4. Analysis of Aluminum by ICP-OES

The aluminum specie was determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), on an iCAP 600 Thermo Scientific spectrophotometer (ASL laboratory accredited by INMETRO). The methodology was based on SW 846, US EPA 6010c.

For each calibration curve, six standards were used from dilution of three stock standards (aluminum calibration curves developed in three ranges). The ASTM® Type I water (from a Millipore® filtration system) acidified with supra-pure nitric acid (Merck® - Germany) was used as calibration blank and for all dilutions. After obtaining the calibration curves - which showed correlation coefficients of 0.9999 - two quality control (QC) samples (one being a CCB, i.e., continuing calibration blank) were run to monitoring the instrument performance and to evaluate the long-term stability. NIST® certified drinking-water reference material CRM 1643e and high purity standards certified reference material for drinking water (CRM-TMDW-B) were used for validating the developed method.

2.5 TOC analysis

TOC (Total Organic Carbon) analysis was recorded by a TOC analyser Shimadzu 5000A. No sample pretreatment was necessary. All results were compared to a calibration curve obtained by potassium biphthalate solutions in several concentrations.

3. RESULTS AND DISCUSSION

3.1. Physical and chemical parameters

Table 1 shows the results of physical-chemical parameters measured every two months.

3.1.1. Turbidity

Until 1984 the Brazilian governmental regulations recommended the turbidity of water should be kept lower than 25 NTU, since higher values could interfere in the potability of drinking water. In 1984, the guidelines of WHO (World Health Organization) recommended turbidity should be lower than 5 NTU; in disinfected waters, values lower than 1 NTU were

recommended. At this time, drinking water with turbidity lower than 1 NTU was considered safe if it was disinfected by chlorine with a Cl_2 free residual of 0.5 mg/l maintained for 30 minutes at pH lower than 8; this dose was considered to be sufficient to obtain safe drinking water free of bacterial and viral pathogens [10].

The turbidity results in Table 1 shows an interesting behavior: all studied wells (P1, P8 and P9) have seasonal influences – turbidity is high during both the dry season (July/August) and the wet season (December); during spring and autumn, the turbidity is close to 1 NTU. Of the three studied wells, only P9 shown higher NTU values in December (about 6.7 NTU) than in July/August (about 2.3 NTU). The water concentration in the dry season and the dragging of soil elements by rainfall and storms during the wet season may be responsible by these high values; the turbidity in P9 may be more influenced by storms.

The turbidity of dam water may be influenced both by the weather as and human activities: large values (between 12 and 18 NTU) are observed in wet, spring and autumn seasons. In July and May (dry season and autumn) the turbidity has values of about 5.5 NTU. The large value in August (32.2 NTU) may be related to the dryness of season, wind action aggravated by human activities.

The water of spring source presented values close to 1 NTU during the entire sampling period, which suggests this water source is not influenced by seasonality.

3.1.2. Temperature

Changes in temperature also affect the pH. For example, when the temperature of pure water is raised far above 25^{o}C, its pH decreases by 0.45. In ground water the thermal amplitude is low – that is, the temperature of wells is not affected by the temperature of the atmosphere except for shallow wells. Salve and Hiware [11] observed that during summer, water temperature in shallow wells is high due the low water level.

For spring source and dam water, the temperature changes (ΔT) were minimal for the entire period of study, with ΔT=3.5 ^{o}C and ΔT=4 ^{o}C. This behavior is expected once the source's temperature values keep in equilibrium with the atmosphere temperature. By the other hand, the temperature values of wells water presented high changes: differences of 10 ^{o}C, 7 ^{o}C and 11 ^{o}C were observed respectively for P1, P8 and P9 in the studied period. This behavior is characteristic for shallow wells, originated typically of non-confined aquifers.

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Table 1: Results physical-chemical parameters.

Samples*	TDS ppm					Turbidity (NTU)					pH					Electrical conductivity (µS/cm)					Temperature of water (^{o}C)				
	P1	P8	P9	Billings dam	Spring source	P1	P8	P9	Billings dam	Spring source	P1	P8	P9	Billings dam	Spring source	P1	P8	P9	Billings dam	Spring source	P1	P8	P9	Billings dam	Spring source
Months																									
August	93.0	73.0	145	84.0	23.0	3.02	14.6	2.29	5.41	0.95	4.62	4.94	5.01	9.00	6.50	186	146	289	164	33.0	24.00	19.00	20.00	19.00	18.00
October	285	69.0	168	90.0	22.0	0.85	0.86	0.89	17.8	0.56	4.67	4.40	4.73	6.70	4.70	569	137	336	166	45.0	18.00	20.00	21.00	21.00	18.20
December	86.0	82.0	99.6	80.0	20.0	1.38	4.52	6.69	11.9	0.80	4.74	5.69	4.90	7.20	4.40	173	163	199	171	121	21.00	20.00	21.00	20.00	19.60
March	25.0	44.0	230	81.0	22.0	0.83	0.89	0.53	14.2	0.88	4.67	5.21	5.00	8.89	6.50	49.4	88.3	350	157	20.0	14.00	14.00	12.00	23.00	21.00
May	60.0	50.0	89.0	85.0	20.0	0.96	1.15	0.95	5.60	1.10	4.57	4.99	4.90	7.40	4.40	120	100	178	170	15.0	21.00	21.00	25.00	23.00	19.00
July	56.0	58.0	133	84.0	14.0	3.02	14.6	2.29	32.2	0.91	4.46	4.71	5.04	7.49	5.00	112	116	267	165	22.0	20.00	20.00	19.00	19.70	17.50
MCL 2914/11	N.A.					5					6 to 9					N.A.					N.A.				

*The samples are not filtered and disinfected by chlorine. MCL: Maximum Concentration Limited (not applicant to sample Billings dam); N.A.: not applied

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3.1.3 Total Dissolved Solids (TDS) and electrical conductivity

The Total Dissolved Solids (TDS) is the term used to describe the inorganic salts and traces of organic matter present in solution. The principal constituents are usually calcium, magnesium, sodium and potassium cations and carbonates, hydrogen carbonate, chloride, sulfate and nitrate anions. The presence of dissolved solids in water may affect its taste [1]. According to WHO [12] the TDS levels can be classified as: excellent, less than 300 mg/L: good, 300 at 600 mg/L; fair, between 600 and 900 mg/L; poor, between 900 and 1200 mg/L; and unacceptable, greater than 1200 mg/L. Water with extremely low concentrations of TDS may also be unacceptable because of its flat, insipid taste.

There is no recent data on health effects associated to the ingestion of high TDS drinking water. However, the relationship between diseases and hardness rather than TDS concentration has been investigated in many studies [12]. In early studies, inverse relationships were reported between TDS concentration in drinking water and the incidence of cancer, coronary heart disease, atherosclerosis heart disease, and cardiovascular disease. The results of a limited epidemiological study, over a 5-year period increased with the mean level of dry residue in the groundwater in the former Soviet Union indicated the average number of cases of inflammation of the gallbladder and gallstones.

TDS results for spring source and dam water are both in the same order of magnitude for the entire period of study: about 19 mg/L and 85 mg/L respectively. This parameter is not influenced by seasonality in these both cases.

However, for the studied wells, TDS values change according to the seasons: P1 and P9 presented high TDS values during spring (285 mg/L) and autumn (230 mg/L). The low water volume in dry season and the the dragging of soil elements by rainfall and storms in wet season may have influenced these values in these specific periods.

P1 showed increased TDS values from March (25 mg/L) to October (285 mg/L) but P9 did not present any periodic tendency. Also, P8 showed increased TDS values from March to December (respectively 44 mg/L and 82 mg/L). Although these results represent the characteristic behavior for shallow wells and it is high for P1 and P8, the case of P9 is specific – this shallow well may receive other influences besides seasonality.

The electrical conductivity results are related to the media dispersed ions. Higher ions concentration, higher is this parameter. The electrical conductivity is done by cations and anions from inorganic salts, frequently calcium, magnesium, sodium, potassium, hydrogencarbonate, chloride, sulphate and nitrate.

The results for electrical conductivity are complementary of TDS; the same considerations are appointed for electrical conductivity in all period for water wells and superficial waters (Billings dam and spring source).

3.1.4. pH

Although the pH of water usually has no direct impact on human health, it is one of the most important operational parameter regarding quality of water. Contamination of drinking water and adverse effects on its taste, odor, and appearance can be related to the changes of pH values. Careful attention to the pH control is necessary in all stages of water treatment to ensure satisfactory water clarification and disinfection. The optimum pH in drinking-water is often in the range 6.5 – 9.5 [15], but these values may be different according to the composition of the water and the nature of the construction materials used in the distribution system. Also, this parameter is important for the corrosion control on water pipelines [12,13].

High concentration of carbon dioxide decrease the pH in water; on the other hand, the presence in high concentration of bicarbonate, carbonate and hidroxyl ions will increase the water pH values. The pH of water is a measure of the acid-base equilibrium and in many natural waters is controlled by the carbon dioxide-bicarbonate-carbonate equilibrium system. Photosynthetic activity prevents the assimilation of carbon dioxide and bicarbonate ion which are responsible for increase in pH [14].

The pH value for spring source is around 6.5 in dry season (August) and in early autumn (March); in the other studied periods the pH values are between 4.4 and 5.0. These results suggest an acidic characteristic for this water source; however, it has a tendency to became less acid when the flux of water is low (winter season in August) or when the evaporation is high (late summer season in March); in both situations the high concentration of salts in soil's surface layer may have contributed to these values.

The behavior of pH values for dam water is similar to those of the spring source: in August's dry season and in early autumn (March) the pH values are around 9 and in the other period, the average pH is around 7.2. These results suggest the pH of Billings dam water is slightly alkaline, but both in dry winter season and in early autumn season a decreasing in water volume occurs due to winter's low humidity and/or summer's high evaporation; thus the concentration of alkaline salts concentration may increase and/or the concentration of carbon dioxide may decrease due the high photosynthetic activities of algae proliferation, which is typical of dry season [14].

The pH did not change in all period for all studied wells. For P1 pH values are between 4.5 and 4.7; P8 presented pH values between 4.4 and 5.7 and for P9, pH values are between 4.7 and 5.0. This parameter indicates an acidity for well waters and it does not depend on seasonality; this behavior is typical for confined waters.

3.2. Aluminum analysis

Table 2 shows the total and dissolved aluminum measured by ICP-OES for non-irradiated and irradiated water samples.

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Table 2: Total and dissolved aluminum in irradiated and non-irradiated in water samples (December/2011) by ICP-OES.

Water Samples	Total Aluminum – mg.l^{-1}**					Dissolved Aluminum – mg.l^{-1}				
	Non-irradiated	Gamma Irradiation (kGy)				Non-irradiated	Gamma Irradiation (kGy)			
		10	20	40	60		10	20	40	60
P1	**0.643**	3.000	5.157	2.010	2.900	**0.589**	0.697	0.782	2.760	2.130
P8	**0.628**	3.060	0.589	1.090	1.500	**0.519**	0.920	0.109	0.510	0.600
P9	**1.546**	2.182	1.280	4.510	5.490	**1.389**	1.590	1.170	2.190	3.900
Spring source	**0.019**	0.444	NA	0.350	0.520	**<LQ**	<LQ	NA*	0.350	0.190
Billings dam***	**0.507**	0.320	0.309	1.320	0.890	**0,098**	0,093	<LQ	0.770	0.510
Blank	**<LQ**	<LQ	<LQ	<LQ	<LQ	**<LQ**	<LQ	<LQ	<LQ	<LQ

NA = not applied; ** Maximum Concentration Limit (MCL) of the Ministry of Health 2914/11 (old law 518/05) - MCL = 0.2 mg.l^{-1};
*** Applies the MCL of Resolution 430/11 (and modifying the 357/05) - MCL = 0.1 mg.l^{-1}; LQ: 0.05 mg.l^{-1} (Limit of Quantification)

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For water samples from the water wells, filtered samples show 80% to 90% of the total aluminum concentration detected on non-filtered samples. For water samples from Billings dam, this concentration is much lower: filtered water samples show 20% of the total aluminum concentration detected on non-filtered samples. All samples show levels of aluminum concentration above the MCL. For water samples from spring source, both total and dissolved aluminum concentrations are below the MCL for the non-irradiated samples.

According to a report by Indiana Department of Environmental Management, filtering is necessary (dissolved aluminum); otherwise, this will impart the sampling process and will lead to inappropriately constructed sites, which increase turbidity levels of the samples [16].

The high concentration of aluminum in these water samples is related to the geological formation of the region. The soil nearby the Billings dam is characterized by three distinct layers: (1) clayish surface layer, with a low fraction of sand and silt; (2) intermediate layer, which is even more clayish, sandy and silt and (3) the deepest layer, where sand and silt prevail over clay [17]. Borges [18] described the composition of this clay, which is composed by aluminum silicate, iron and other alkaline earths.

The aluminum concentration from irradiated water samples show interesting results. As a general behavior, the aluminum release does not increase monotonically with the absorbed dose; there is a saturation dose for which aluminum release is maximum.

Results for non-filtered water from the wells follow:

1. P1 water sample presented high aluminum concentration (8 times higher) after low dose absorption (20 kGy);
2. P8 water sample showed about 5 times the aluminum concentration after 10 kGy of absorbed dose, compared to the non-irradiated sample of the same origin;
3. P9 water sample demanded the highest absorbed dose (60 kGy) to achieve about 4 times the aluminum concentration, compared with non-irradiated samples of the same origin.

For superficial water samples from Billings dam and spring source, high absorption doses (40 kGy and 60 kGy respectively) were necessary to achieve the maximum aluminum concentrations of 3 and 25 times, respectively, compared to non-irradiated samples of the same origins. For filtered samples, the behavior and absorbed dose for maximum aluminum release are the same of corresponding non-filtered samples, except for P1 and spring source water samples.

These results showed the necessity for sample pre-treatment to degrade the organic matter of these natural samples. Organic matter combined with aluminum is critical in these samples because of human consumption. The measurement of physical-chemical parameters (TDS and turbidity) are preliminary indicatives of contamination by organic matter; finally, the

irradiation process was the fundamental process for aluminum decomplexation from organic matter.

Classic wet methods may also be used to destroy the organic matter in water samples, such as heating, addition of oxidizing agents or a combination of both. However, the sample contamination may be a disadvantage, especially in the case of trace metal levels. Moreover, the process presents high consumption of reagents is high and features a long mineralization time. On the other hand, this work presents an alternative, clean process for achieving the same goal [19, 20].

The release of Al^{3+} from organic matter degradation may be verified by the decrease of Total Organic Carbon (TOC) as shown in Table 3; because of the decomposition of organic matter, their organic molecules break forming carbon dioxide (CO_2) and water (H_2O) allowing for full mineralization [20, 21].

Table 3: Total Organic Carbon (TOC) of non-irradiated e irradiated samples.

Samples		Concentration (ppm)		
		TC	IC	TOC
Billings dam	Gamma irradiation	**1.360**	**0.832**	**0.528**
	non-irradiated	1.880	1.000	0.880
		1.490	0.651	0.839
		0.873	0.245	0.628
		17.100	11.100	6.000
P8	Gamma irradiation	**1.060**	**0.762**	**0.298**
	non-irradiated	12.200	5.670	6.530
P9	Gamma irradiation	**2.750**	**2.440**	**0.310**
	non-irradiated	3.280	0.743	2.537
Spring source	Gamma irradiation	**2.490**	**2.150**	**0.340**
	non-irradiated	1.680	1.290	0.390
		1.650	1.230	0.420

TC: Total Carbon; IC: Inorganic Carbon

4. CONCLUSIONS

Gamma irradiation process applied as a pre-treatment of natural waters represented an alternative AOP to degrade the organic matter. After ionizing irradiation process the well samples released aluminum up to 8 times the quantity detected in non-irradiated samples. Organic matter in these samples is critical since the wells chosen for this study were shallow wells (non-confined water), providing water that is consumed by local community. Since the water of these wells is non-confined, the organic matter found on them may be originating from other sites just as the water of the said wells most certainly is.

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