

DETERMINATION OF POTASSIUM IODIDE IN TABLE SALT

M.B. Rajković¹

Abstract: The samples of table salt bought in Belgrade supermarkets are analysed in this paper. The method of indirect iodometry was used in the process of the analysis, and received results were converted into the content of KI in mg/kg of salt. Beside the content of KI, the content of NaCl was also determined, counted to dry meter and water content, and received results were compared with the requests determined by Regulations of Table Salt Quality Meant for Human Diet and by manufacturers' declaration.

Received results show that the volumetric method of indirect iodometry, applied in this analysis, is very reliable for determination of potassium iodine in table salt, because of its high precision and reproducibility of the analysis results.

All received results show that the samples of table salt which can be bought in supermarkets are according to the demands given by Regulations. Only one sample (evaporated salt) has significantly less mass of KI than it is determined by Regulations, but also by manufacturer's declaration.

Measured humidity in the samples of table salt received from sea salt (sample 1) is higher than humidity in the samples received from rock salt as the result of magnesium presence in sea salt, which is hygroscopic material. Although samples 5 and 6 also originate from sea salt, their smaller humidity is the result of additional heating and salt processing.

Key words: table salt, iodine, method of iodimetry, thiosulphate solutions.

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Introduction

Sodium chloride (*table salt, sea salt, salt rock, evaporated salt*) or simply salt (*greek, ἅλς, sea salt*) is the product which is used as additional substance in food production, for direct human consumption and as the carrier for additives or nutrients (Official Gazette of FRY, 2005). Table salt is not a spice in a classical sense (herbal), but a mineral used for flavouring a dish. The basic substance for salt production is sodium chloride crystal.

According to the way of derivation sodium chloride can be (Lačnjevac and Rajković, 2005): 1) evaporated sodium chloride crystal - received by evaporation of salt water; 2) sea sodium chloride crystal - received by evaporation of sea water; 3) rock sodium chloride crystal - derived by processing rock salt.

Salt is the crystal shape product which must fulfil the following requests:

1) that the content of sodium chloride is at least 97 % counted the dry meter without additives;

2) that the content of iodine is from 12-18 mg/kg of salt or 16-24 mg of potassium iodine per kg of salt;

3) that the content of water is 3 % upmost except with *fine* salt where the content of water can't be higher than 0.5 %;

4) that it is white, but it can have slightly noticeable shade of other colour, and without odour;

5) that it doesn't include ingredients which are not allowed by Regulations;

6) that it doesn't include more than 0.05 % of mineral ingredients insoluble in chlorohydric acid;

7) that granulation of *fine* salt is such that 90 % of salt can pass through sieve with square holes dimension of 0.5 mm, of small grained salt - 1.25 mm and large grained salt - 3 mm.

The manufacturer is obliged to make manufacturer's specification before the start of salt production. Manufacturer's specification must contain a short description of technological process of production, as well as the report of completed analysis of salt quality index (sanitary status, sensor, physical and chemical characteristics). Salt can be sold only in original packaging made of materials which preserve the quality of the product till the moment of opening in the expiration date.

Iodine is a part of thyroid gland hormones (thyroxine and triiodothyronine), that influence to normal growing and developing, regulate the speed of basic metabolism, create the energy and regulate the body temperature, influence on synthesis of proteins, changing β -carotene into vitamin A, synthesis of cholesterol, functioning of nerve and muscular tissue etc. (Grujić, 2000).

If there is not enough iodine in food, it comes to the significant decreasing of the amount of synthesised thyroxine which influence to the metabolism intensity. The deficiency of iodine in organism cause several functional and developing abnormalities known under the common name as "iodine deficiency disorders" or IDD, some of which are the heaviest types of brain damage and mental retardation (cretinism) (Dealnge, 1994). The deficiency of iodine in nourishment is especially dangerous in the period of fetus development, which can cause physical and mental disturbances.



According to World Health Organisation (WHO) data and International Council for Control of Iodine Deficiency Disorders (ICCIDD) more than 200 million of people suffer from goiter, most of them in Africa, and the cause of illness for over 4 % of all cases is iodine deficiency in food.

The amount of iodine in organism is about 25 mg, small part of it is in muscles, while 20% (1 000 times more than in muscles) of iodine is placed in thyroid gland. The rest of iodine is in skin and bones and very small amount (under 1.0 %) in blood.



Iodine is given in food to prevent the consequences of iodine deficiency. It is proved that table salt is, consumed in relatively constant amounts (about 10 g/day), the great carrier of iodine, so it proved to be the most reliable way to take in iodine (WHO/NUT/94.6). World Health Organisation (WHO) and ICCIDD recommend that the amount of added iodine should be 20-40 mg/kg of salt (Diosady et al., 1998).

The percentage of countries in the world where salt iodination was done successfully and used in nourishment is shown in Figure 1.

For salt imported as the salt for human diet and which doesn't fulfil demands of iodine content, the iodination is obligatory after the import, that is, before selling. Iodination of sodium chloride crystal can be done with potassium iodide, sodium iodide and potassium iodate. The content of iodine in salt must always be under the constant supervision of Republic

Commission for Prevention of Iodine Deficiency Disorders, for stopping illnesses caused by iodine deficiency.

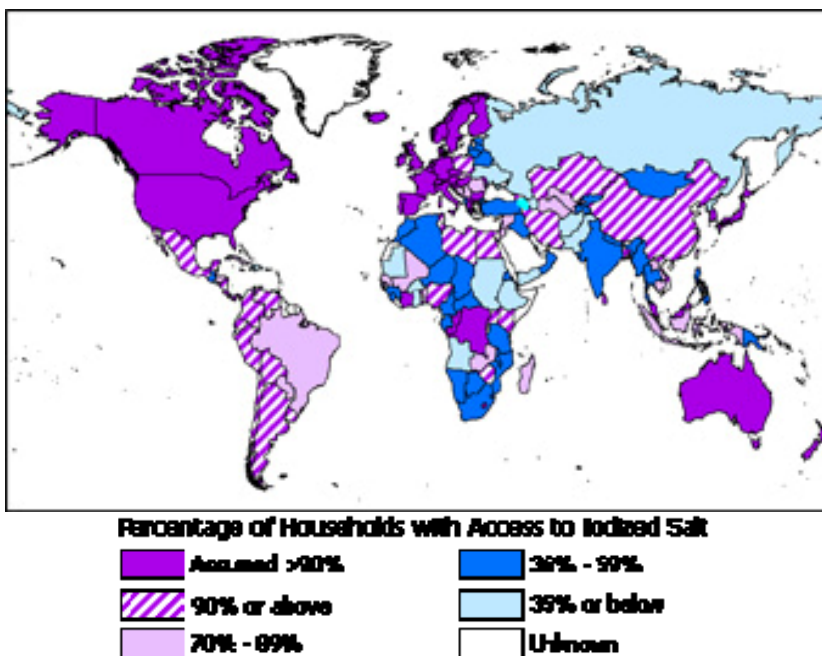


Fig. 1. - The percentage of countries in the world where salt iodination is used

Salt in Serbia is mostly imported, which is done by about 200 distributors, what makes control more difficult. There is no systematic control of salt distribution, because it is common opinion that salt is not perishable goods. However, as it was said in the analysis of Belgrade Consumer's Organisation, taken during 2005, the salt quality on domestic market is bad, for less or higher iodination, presence of particles or bed declaration, and more than a half of the tested samples failed on the quality test.

That was the reason for the analysis of potassium iodide content in table salt of different origin that could be found in Belgrade market. The potassium iodide content and humidity of samples were analysed in this paper, and received results were compared with values determined by Regulations (Official Gazette of FRY, 2005).

Material and Methods

All analysed samples of table salt have manufacturer's specification that is determined by Law (Official Gazette of FRY, 2005), and which is given in Table 1.

Manufacturer keeps records that must contain the following data (manufacturer's specification): 1) evidence number of specification; 2) date of manufacturer's specification; 3) name and seat of the company; 4) product's name and its brand name 5) date of the beginning of production according to the manufacturer's specification; 6) date of completed analysis of product quality indicators; 7) functional group of used additives, their name and number; 8) type and amount of nutrients used to increase the product's biological value; 9) the expiration date and mark "best before"; 10) storage conditions.

Calcium iron (II) cyanide (*potassium ferrous cyanide*) (declare as $\text{Fe}(\text{CN})_6$), or E536 (YU mark for additives 7.5) was added as the anti caking agents. According to the decision of Codex Alimentarius (CA) (group A(1)), the origin – synthetic (S) (Modić, 2001; Official Gazette of FRY, 2003).

Potassium and sodium iron (II) cyanide can be used in the amount up to 20 mg/kg in the process of salt rock derivation.

Tab. 1. – Analysed commercial samples of table salt

Sample	Name of product	Manufacturer	Special suggestion	Content of NaCl (in %)	Content of KI (mg/kg)	Content of H ₂ O (in %)	Additive
sample 1	Kuhinjska sitna MORSKA SO Paška	SOLANA PAG d.d., Pag, Croatia	iodated	98.80	16-24	0.180	E-536 2-10 mg/kg
sample 1-a	Kuhinjska sitna MORSKA SO Paška	SOLANA PAG d.d., Pag, Croatia	iodated	98.800	20-30	0.180	E-536 2-10 mg/kg
sample 2	Kuhinjska varena SO	DP „SoProdukt” Srbija i Crna Gora	iodated	98-99.80	16-24	–	E-536
sample 3	Morska kuhinjska-jodirana SO	DP „SoProdukt” Serbia and Montenegro	iodated	min. 99.0	16-24	–	E-536
sample 4	Tuzlanska so kuhinjska varena so	„SOLANA” d.d. Tuzla, Bosnia and Hercegovina	iodated	99-99.5	–	–	max. E-536 10 mg/kg
sample 5	Rafinisana morska SO	„Kristal so” d.o.o., Beograd SCG poreklo Izrael	Refined 100% natural	99-99.90	16-24	–	max. E-536 10 mg/kg
sample 6	Morska, prirodna, jodirana, kuhinjska so, ultra PEA	PERLA GREEK SALT LTD, Greek	natural	97.00	16-24	1,0	max. E-536 10 mg/kg

Method of potassium iodide determination in table salt

Principle of the method

The method of potassium-iodine determination in table salt is based on Γ ion oxidation (from KI added to table salt) in iodate ion (IO_3^-), and then on its reduction to elementary iodine (I_2) which is determined by titration of standard solution of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$).

Reagents:

- Samples of table salt;
- Indicator **methyl red**: 0.2000 g of methyl red indicator dissolves in mixture of 60.00 cm³ of 95 % ethanol and 40.00 cm³ of distilled water;
- Solution of **hydrochloric acid**, of 0.1000 mol/dm³ concentration;
- Solution of **sodium thiosulphate**, of 0.01 mol/dm³ concentration. Sodium thiosulphate crystallises with five molecules of water ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). However, crystal salt is not stable in air, it easily loses water. Because of that crystal salt doesn't have absolutely defined content, that is, doesn't have attributes of primary standard. Sodium thiosulphate dissolves in a solution under the influences of oxygen from air and bacteria, and light also has catalytic influence to the dissolving process. That's why the solution of thiosulphate must be kept in dark well closed bottles. Fresh prepared solution visibly changes during the first few days, and when it becomes stable and is kept well, it can be steady for 2-3 months. Stability of thiosulphate solution is highest in the pH value interval from 7.00 to 9.00. In order to regulate pH values of the solution it is recommended to add small mass of sodium carbonate. The stability of the thiosulphate solution in this range of pH is explained by the fact that in this interval the influence of bacteria is the smallest.

For preparation of 1 dm³ solution of sodium thiosulphate of 0.01 mol/dm³ concentration, 2.4800 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ was measured on a technical scale. This was dissolved in 300 cm³ of boiled and cooled distilled water, and then 0.05-0.1000 g of Na_2CO_3 was added and filled by distilled water till 1 dm³. Such prepared solution was left in a dark bottle for 7 days and then it was standardised (Rajković, 2007).

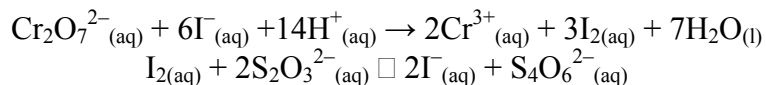
Standardization of sodium thiosulphate solution

Standardization of sodium thiosulphate was done by the solution of potassium dichromate, of 0.0033 mol/dm³ concentration. Solution was

prepared in the way to measure 0.0970 g of $K_2Cr_2O_7$ on an analytical scale and put into a 100 cm^3 measurement dish and filled by distilled water till the measurement line. 10.00 cm^3 of solution was put by pipette into the measurement dish and filled by distilled water till the measurement line. Then 10.00 cm^3 of potassium dichromate solution was measured by pipette in Erlenmeyer flask till 250 cm^3 , and 2.50 cm^3 of 5 % solution of potassium iodide, 5.00 cm^3 of 2 mol/dm^3 hydrochloric acid solution and distilled water till 100 cm^3 were added. Such prepared solution for titration was left for 5 minutes on room temperature, and was titrated by the solution of sodium thiosulphate till light yellow colour showed. Then 2-3 drops of starch solution were added and it was continued with titration until it lost blue colour of indicator (Rajković and Novaković, 2005).

The average value of measurement was: $V_s = 1.96\text{ cm}^3$, as the result of four measurements.

The reactions which appeared during standardisation can be represented by the following equations (Rajković, 2007):



Stoichiometric ratio of pro-reacted amounts of potassium dichromate and sodium thiosulphate, considered through iodine amount is:

1 mol of $K_2Cr_2O_7$ frees 3 mol of I_2 which reacts with 6 mol of $Na_2S_2O_3$

so the concentration of sodium thiosulphate solution can be calculated through equation:

$$c(Na_2S_2O_3) = 6 \cdot \frac{c(K_2Cr_2O_7) \cdot V(K_2Cr_2O_7)}{V_s(Na_2S_2O_3)}$$

where the received value is: $c(Na_2S_2O_3) = 0.0101\text{ mol/dm}^3$.

– **Bromine water:** 1.00 cm^3 of bromine was placed into a 100 cm^3 measurement dish and filled by distilled water till the measurement line.

All measurements were done on a semiautomatic technical scale with two decimals ET-111 and on an analytical scale, TEHTNICA, Zelezniki, Slovenia, with accuracy on four decimals, and measurement of samples' humidity were done on drier SUTJESKA, factory of medical devices Sutjeska.

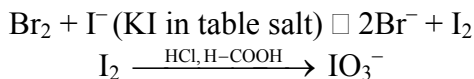
Determination of potassium-iodine in table salt

There was measured 50 g of table salt on technical scale and put into 500 cm³ Erlenmeyer flask. Salt was dissolved in 200 cm³ of distilled water. Solution, in which 3 drops of indicator methyl red were added, was titrated with hydrochloric acid solution of 0.1000 mol/dm³ concentration till the changing of colour from pale yellow to pale pink (first steady colour which is steady). Immediately after the changing of colour, 1.50 cm³ of bromine water was added to the solution and the solution was heated till boiling and cooked for 5 minutes. After that the solution was taken from a heater, slightly cooled (under pipe water stream), and both 1.00 cm³ of formic acid (H-COOH) solution, and 1.00 cm³ of phosphoric acid were added and then it was cooled till room temperature.

In the solution both 1.00 cm³ of concentrated phosphoric acid, and 0.1000 g of potassium iodide were added, solution was stirred and left for 5 minutes in dark. After that, the solution was titrated with sodium thiosulphate solution, which was previously standardised, till changing of colour from yellow to light yellow (lemon yellow). In that moment 1.00 cm³ of starch solution was added and solution changed colour into blue, and solution was titrated until it lost blue colour of indicator.

Results and Discussion

Determination of added potassium-iodine in table salt was done by the method of indirect iodometry (*iodometry*), by titration of free iodine with sodium thiosulphate solution. Reaction is based on previous oxidation of iodide ion (I⁻) (from KI which was added into common salt) with hydrochloric acid, bromine water and formic acid (H-COOH) solution in iodate-ion (IO₃⁻):



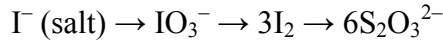
By adding KI into the solution, it came to separation of iodine in elementary condition, according to the following equation:



Free iodine was then titrated by sodium- thiosulphate solution:



Scheme of method of determination can be presented on the following way:



where it can be concluded that the ratio of amounts of I^- (KI) and $\text{S}_2\text{O}_3^{2-}$ ($\text{Na}_2\text{S}_2\text{O}_3$), with reaction "through iodine" is: $n(\text{I}^-) : n(\text{Na}_2\text{S}_2\text{O}_3) = 1 : 6$, that is:

$$6 \cdot n(\text{I}^-) = n(\text{Na}_2\text{S}_2\text{O}_3)$$

As: $c = \frac{n}{V}$ and $n = \frac{m}{M}$, it can be written:

$$\frac{m(\text{KI})}{M(\text{KI})} = \frac{1}{6} \cdot c(\text{Na}_2\text{S}_2\text{O}_3) \cdot V(\text{Na}_2\text{S}_2\text{O}_3) \text{ odn. } m(\text{KI}) =$$

$$\frac{M(\text{KI})}{6} \cdot c(\text{Na}_2\text{S}_2\text{O}_3) \cdot V(\text{Na}_2\text{S}_2\text{O}_3)$$

As $M(\text{KI}) = 166 \text{ g/mol}$, and calculated concentration of sodium thiosulphate – $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.0101 \text{ mol/dm}^3$, there is:

$$m(\text{KI}) = 27,67 \cdot 0,0101 \cdot V(\text{Na}_2\text{S}_2\text{O}_3) \text{ g}$$

and when it is changed to 1 kg of salt (and 50 g were taken, so it means that received result should be multiplied with coefficient 20) and having in mind that spent cubage (V) is in cm^3 and concentration in mol/dm^3 we get the final expression for calculation of KI in analysed table salt in mg/kg :

$$m(\text{KI}) = 0,2795 \cdot 20 \cdot V(\text{Na}_2\text{S}_2\text{O}_3) \text{ mg KI/kg}$$

which was used for calculation.

Received results of the analysis of KI content (in mg/kg of salt) in analysed samples of table salt are given in Table 2. Spent cubage of $\text{Na}_2\text{S}_2\text{O}_3$ represents the average value received after five titrations.

Tab. 2. – Results of KI determination in table salt samples

Samples	Consumption of $\text{Na}_2\text{S}_2\text{O}_3$ (in cm^3)	Content of KI (in mg/kg)	Manufacturer's declaration (in mg/kg)
sample 1	5.40	30.19	16-24
sample 1-a	3.85	21.52	20-30
sample 2	2.15	12.02	16-24
sample 3	3.10	17.33	16-24
sample 4	3.50	19.56	-
sample 5	3.20	17.89	16-24
sample 6	3.05	17.05	16-24

Statistic data processing of the results of KI determination (in mg/kg of salt) in analysed samples of table salt are given in Table 3.

Tab. 3. – Statistic data processing of the results of KI determination in analysed samples of table salt

Samples	Content of KI (in mg/kg)	Standard deviation (in %)	Average deviation (in %)	Relative mean Deviations (in %)
sample 1	30.19	0.035	0.016	0.81
sample 1-a	21.52	0.050	0.022	1.61
sample 2	12.02	0.051	0.023	2.94
sample 3	17.33	0.047	0.021	1.88
sample 4	19.56	0.074	0.033	2.62
sample 5	17.89	0.043	0.019	1.67
sample 6	17.05	0.072	0.032	2.94

Received results showed that sample 1 withdraws from manufacturer's declaration, because of too high value received for KI. As the second bought sample showed significantly less value, and on declaration was written other (higher) value, we concluded that the wrong label was put on the product. All other samples showed the KI content was at lower limit (about 17 mg/kg), and sample 2 (the only evaporated salt) didn't contain recommended (and regulated by Law) content of potassium iodide. As the process of obtaining evaporated (or cooked) salt is based on evaporation in evaporation devices, we think that part of iodine was lost in that way.

Samples of sea salt (samples 5 and 6) during the dissolving process contained small crystals (size of salt crystal) that were insoluble in water, which can be explained by the origin of common salt in sea water (Fig. 2).



Fig. 2. – Crystals of sea salt

Content of sodium chloride, counted to dry mater, and humidity measured in analysed samples of salt is shown in table 4.

Tab. 4. – Content of sodium chloride, and water in analysed samples of table salt

Samples	Content of NaCl (in %)	Manufacturer's Declaration (in %)	Content of water (in %)	Manufacturer's Declaration (in %)
sample 1	99.78	98.80	0.215	0.18
sample 1-a	99.78	98.80	0.215	0.18
sample 2	99.97	98-99.80	0.025	-
sample 3	99.93	99.00	0.070	-
sample 4	99.925	99-99.50	0.075	-
sample 5	99.94	99-99.90	0.065	-
sample 6	99.98	97.00	0.020	1.00

From received results it can be seen that the content of sodium chloride is mostly according to manufacturer's declaration which is given with the product, but that also fulfills the demands of Regulations (Official Gazette of FRY, 2005), which says that table salt must contain at least 97 % of sodium chloride, counted to dry mater.

Content of water is also according to the demands of Regulations (Official Gazette of FRY, 2005), where it is said that content of salt is upmost 3 % and with fine salt it can't be higher than 0.50 %.

Conclusion

Received results show that volumetric method of indirect audiometric, applied in this analysis, is very reliable for determination of potassium iodide in table salt, because of its high precision and reproducibility of the analysis results.

All received results show that samples of table salt that can be bought in supermarkets are according to the demands of Regulations, except one sample (evaporated salt) which contains significantly less mass of KI than it is determined by Regulation, but also by manufacturer's declaration.

Measured humidity in samples of table salt received from sea salt (sample 1) is higher than humidity in samples received from rock salt as the result of magnesium presence in sea salt, which is hygroscopic material. Although samples 5 and 6 also originate from sea salt, their smaller humidity is the result of additional heating and salt processing.

Salt, which is used in human diet, should be kept in closed dishes because of iodine degradation under the influence of light and air.

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ADDITION:

Regulations of quality and other demands for salt in human diet, which preceded to the present Regulation (Official Gazette of FRY, 2005):

1. PRAVILNIK O KVALITETU I DRUGIM ZAHTEVIMA ZA KUHINJSKU SO I SO ZA PREHRAMBENU INDUSTRIJU (Službeni list SRJ, br. 39 od 20.jula 2001. god. i Službeni list SCG, br. 56/2003 - dr. pravilnik, 4/2004 - dr. pravilnik i 47/2004).
2. PRAVILNIK O KVALITETU I DRUGIM ZAHTEVIMA ZA KUHINJSKU SO I SO ZA PREHRAMBENU INDUSTRIJU (Službeni list SRJ, br. 39/2001 i Službeni list SCG, br. 56/2003 - dr. pravilnik, 4/2004 - dr. pravilnik i 47/2004).

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ODREĐIVANJE KALIJUM-JODIDA U KUHINJSKOJ SOLI

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Rezime

U radu su ispitivani uzorci kuhinjske soli koji se mogu nabaviti u beogradskim prodavnicama. U radu je primenjena metoda indirektna jodimetrija, a dobijeni rezultati su prečunavani na sadržaj KI u mg/kg soli. Osim sadržaja KI, određivan je i sadržaj NaCl, računato na suhu materiju i sadržaj vode, a dobijeni rezultati upoređeni su sa zahtevima koji su predviđeni Pravilnikom o kvalitetu kuhinjske soli predviđenim za ljudsku ishranu i proizvođačkom deklaracijom.

Dobijeni rezultati su pokazali da je volumetrijska metoda indirektna jodimetrija, primenjena u ovom radu, veoma pouzdana za određivanje kalijum-jodida u kuhinjskoj soli, usled velike preciznosti i reproduktivnosti rezultata ispitivanja.

Svi dobijeni rezultati pokazali su da uzorci kuhinjske soli koji se mogu nabaviti u trgovini odgovaraju zahtevima Pravilnika, osim jednog uzorka (varena so) koji je sadržao znatno manju masu KI nego što je predviđeno Pravilnikom, ali i proizvođačkom deklaracijom.

Izmerena vlažnost u uzorcima kuhinjske soli dobijene iz morske vode (uzorak 1.) veća je od vlažnosti u uzorcima dobijenim iz kamene soli kao posledica prisustva magnezijuma u morskoj vodi, koji je higroskopni materijal. Iako su i uzorci 5. i 6. poreklom iz morske vode, njihova mala vlažnost posledica je dodatnog zagrevanja i dorade soli.

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