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QUANTITATIVE DETERMINATION OF THE MASS FRACTION OF TOTAL IODINE IN MARINE HYDROBIONTS BY THE SPECTROPHOTOMETRIC METHOD

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The technique of quantitative analysis of total iodine is given: the mineralization of biomass in the presence of KOH followed by bromine oxidation of inorganic iodine compounds to iodates with removing the excess bromine with formate. Iodates in acidic medium are reduced with iodide to elemental iodine, which is extracted with chloroform and detected by light absorption in the range of 502–510 nm. The lower detection limit is 10^{-6} g. To boost sensitivity, a 10% alcohol solution of potassium iodide is added to the chloroform fraction. After potassium triiodide is formed, iodine is detected by light absorption in the range of 225 or 325 nm. The lower detection limit is 10^{-9} g.

Keywords: marine organisms, organoiodine compounds

Essential elements play a vital role in marine ecosystems. The analysis of metabolic pathways, the determination of their quantitative content in hydrobionts, and many other applied aspects deserve close attention of researchers. Among these elements, iodine stands out both in theoretical and practical terms. Despite its importance in the diet, including for humans, relatively few publications are focused on studies of iodine metabolism in marine hydrobionts. This is partly due to the fact that many methods for determining iodine are quite labor-intensive and expensive, or have low sensitivity. When choosing the proper technique, it is reasonable to consider its analytical capabilities, the availability of technical measuring instruments, and also iodine content in the object of study. In laboratory practice, the following analysis methods are used: photometric [Bryanskaya, Leskova, 2006; GOST 26185-84, 2004; Laboratornyi praktikum, 1991; Mázor, 1986; Pochinok, 1976; R 4.1.1672-03, 2004], titrimetric [Bryanskaya, Leskova, 2006; Ermolenko et al., 2010; GOST 25832-89, 1996; GOST 31505-2012, 2013; MUK 4.1.1106-02, 2002; Mázor, 1986; R 4.1.1672-03, 2004], kinetic [Bozina, Ruvinsky, 2003; Ermolenko et al., 2010; Farkhutdinov et al., 2013; GOST 28458-90, 2006], chromatographic [Bozina, Ruvinsky, 2003; Mishra et al., 2000; Nitschke, Stengel, 2015; Yeh et al., 2014], radiochemical [Bozina, Ruvinsky, 2003], and electrochemical ones [GOST 31505-2012, 2013; GOST 31660-2012, 2013; MUK 4.1.1481-03, 2003], as well as methods of atomic emission spectrometry [Milinovic et al., 2021] and mass spectrometry [GOST EN 15111-2015, 2017; Mello et al., 2013].

The aim of the study is to develop a theoretical basis for working out a simple spectrophotometric method for determining the mass fraction of total iodine in the biomass of hydrobionts.

RESULTS AND DISCUSSION

In the biomass of hydrobionts, iodine occurs mainly in organic form. During sample preparation (biomass mineralization), organic and inorganic iodine compounds are oxidized, which may result in iodine deficiency due to the sublimation of its vapors. To prevent its loss, biomass mineralization is carried out in the presence of alkali:

$$3\,I_2 + 6\,KOH \longrightarrow KIO_3 + 5\,KI + 3\,H_2O\,.$$

Mineralization must be carried out in the presence of KOH at a temperature of +500…+550 °C, since potassium iodate decomposes releasing oxygen at +560…+650 °C, while sodium iodate has a decomposition temperature significantly below +500 °C.

The resulting iodide compounds are oxidized by bromine in an alkaline medium to iodates during mineralization (IO_3^-):

$$I^- + 3\,Br_2 + 6\,OH^- \longrightarrow IO_3^- + 3\,H_2O + 6\,Br^-\,.$$

For example, in the presence of KOH:

$$KI + 3\,Br_2 + 6\,KOH \longrightarrow KIO_3 + 3\,H_2O + 6\,KBr\,.$$

Excess elemental bromine is removed using formate after its formation in an alkaline medium:

$$HCOOH + KOH \longrightarrow H_2O + KCOOH\,;$$
$$KCOOH + Br_2 \longrightarrow H_2CO_3 + 2\,KBr\,.$$

Then, it is acidified with sulfuric acid.

Iodates in an acidic medium are reduced with iodide to elemental iodine:

$$IO_3^- + 5\,I^- + 6\,H^+ \longrightarrow 3\,I_2 + 3\,H_2O\,.$$

For example, with potassium iodide in the presence of formic acid:

$$KIO_3 + 5\,KI + 6\,HCOOH \longrightarrow 3\,I_2 + 3\,H_2O + 6\,KCOOH\,.$$

Iodine is concentrated from an aqueous solution by extraction with chloroform and detected by light absorption in the wavelength range of 502–510 nm. The lower detection limit is 10^{-6} g. To boost sensitivity, a 10% alcohol solution of potassium iodide is added to the chloroform fraction resulting in the formation of potassium triiodide:

$$KI + I_2 \longrightarrow KI_3\,.$$

Iodine is determined in the ultraviolet range at a wavelength of 225 or 325 nm. Thus, the sensitivity can be boosted 20-fold. The lower detection limit is 10^{-9} g.

The proposed simple method is suitable for routine measuring. It is time effective and does not require expensive reagents and equipment.

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КОЛИЧЕСТВЕННОЕ ОПРЕДЕЛЕНИЕ МАССОВОЙ ДОЛИ СУММАРНОГО ЙОДА В МОРСКИХ ГИДРОБИОНТАХ СПЕКТРОФОТОМЕТРИЧЕСКИМ МЕТОДОМ

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Приведена методика количественного определения суммарного йода в гидробионтах, заключающаяся в минерализации биомассы в присутствии KOH и окислении неорганических соединений йода бромом до йодатов с удалением излишков брома формиатом. Йодаты в кислой среде восстанавливают йодидом до элементарного йода, который экстрагируют хлороформом и определяют по поглощению света в диапазоне 502–510 нм; нижняя граница определяемого содержания — 10^{-6} г. Для увеличения чувствительности в хлороформную фракцию добавляют 10%-ный спиртовой раствор йодида калия. После образования трииодида калия определяют йод по поглощению света в области 225 или 325 нм; нижняя граница определяемого содержания — 10^{-9} г.

Ключевые слова: морские организмы, йодорганические соединения