Electronic supplementary materials *Mendeleev Commun.*, 2024, **34**, 896–898

The preparation method of cells with boron nanoparticles to determine boron by direct current arc atomic emission spectrometry

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In vitro experiment design

Composition 2 and 3 (V = 0.5 ml) were added to U87 cells, and, the ratio of BNPs to nutrient medium is 1 to 10. Cells were incubated at 37°C in an atmosphere with 5% CO_2 for 24 h. Than the nutrient medium was removed. Cells attached to the substrate were washed with the addition of versene (Biolot, Russia). After removing versene, tripsin-versen (1:1, Biolot, Russia) was added to the cells and incubated at 37°C for 5 min. Tripsin-versene detached the cells from the substrate. Nutrient medium was then added to the cells floating in the trepsin-versene. This is stops tripsin to prevent cell damage. Cells were counted on a Countess automatic cell counter (Invitrogen, Waltham, MA, USA). Cells and nutrient medium were separated by centrifugation for 5 min at 1000 rpm. The supernatant was removed and the cells were placed in tubes of $1 \cdot 10^6$ cells.

BNPs size

Small-angle X-ray scattering method (SAXS) on the device WAXS/SAXS X-ray System (Xenocs, USA) was also used. Data SAXS: Rg = 23.8 ± 3.4 nm [1.74 < qR < 2.48], d = 2 Rg sqtr(5/3) = 61 ± 9 nm. Top right shows the Guinier approximations.

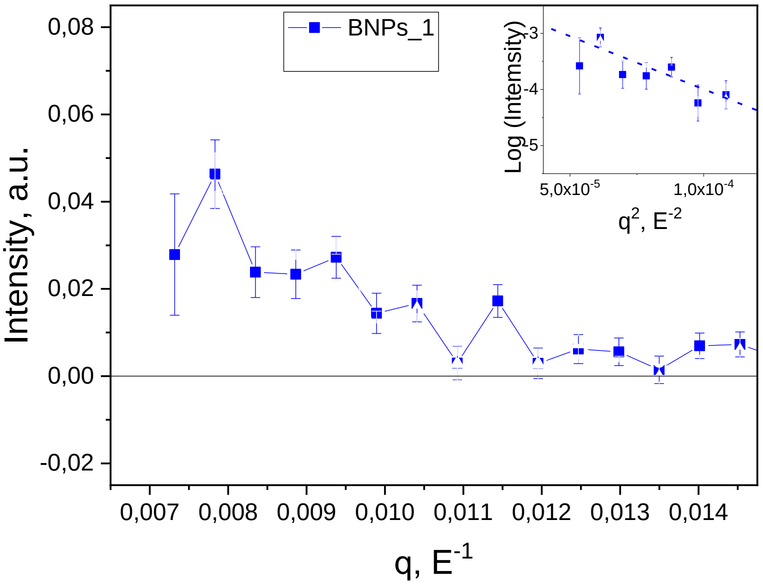

Figure S1. Small-angle scattering curve for BNPs nanoparticles.

BNPs/HEC composition

Highly dispersed BNPs were stabilized in an aqueous solution of hydroxyethylcellulose 3 g·l^{-1} (HEC). The BNPs concentration was 100, 210 and 500 mg l^{-1} (BNPs/HEC composition 1, 2 and 3, respectively). The aggregation stability of BNPs was assessed using the dynamic light scattering method, and the size and ζ-potential were assessed over a period of 90 days (Figure S2).

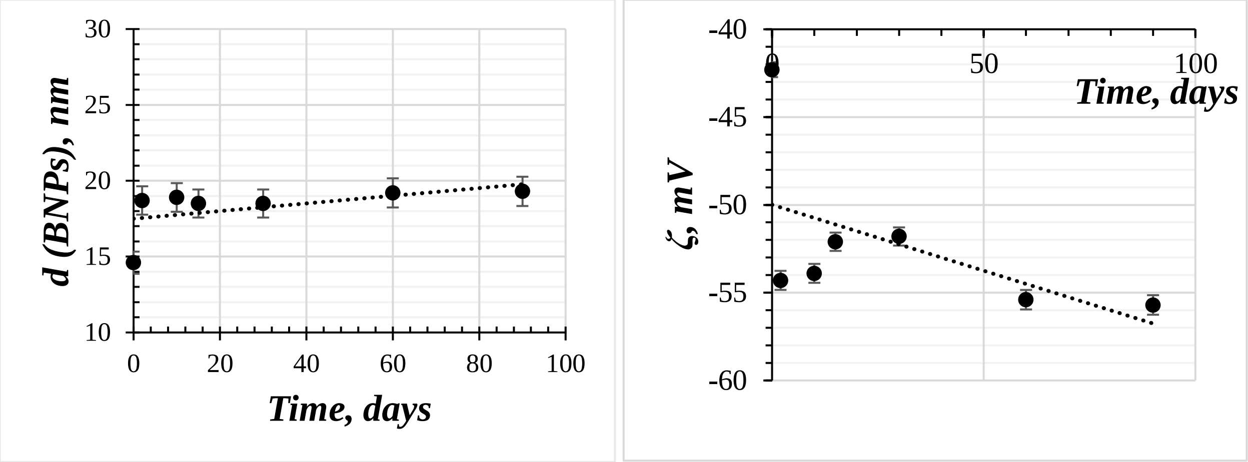

Figure S2. Characteristics of BNPs in an aqueous solution of HEC: size, ζ-potential.

DCA AES analysis

A modernized spectrograph PGS-2 (Carl Zeiss Jena, Germany) and a Fireball FB-25 electric arc generator (VMK-Optoelectronics, Russia) were used for DCA AES analysis. A spectrograph is equipped with an 8-crystal photodiode array for registration analytical signals. The photodiode array is named a multichannel analyzer of emission spectra MAES (VMK-Optoelectronics, Russia). The spectra were registered at an arc current of 13 A for 22 s. Graphite electrodes for spectral analysis (ø 6 mm, Grafi, Russia) was used. Truncated cones as anodes, and electrodes with craters (depth and diameter 4 mm) as cathodes were used.

Design of spike experiment

Boron from the standard solution was added to the HEC solution (3 g·l^{-1}). Then 0.04 ml of spiked HEC was pipetted into 100 mg of spectral buffer and mixed after drying under an IR lamp. The resulting mixture is placed in the craters of graphite electrodes. The boron concentration introduced into the spectral buffer was 5 mg·l^{-1}.

Table S1. The spike experiments results (n = 5, P = 0.95) of boron determination by DCA AES. Measured concentration of boron presented as $\bar{X} \pm \Delta$, where $\Delta = {S \cdot t}/{\sqrt{n}}$.

Line, nm	Spiked concentration, mg·l^{-1}	Measured concentration, mg·l^{-1}	Recovery, %
249.677	5	4.9±0.9	98
249.772	5	5.4±1.7	107

ICP AES analysis

A high-resolution spectrometer iCap-6500 Duo (Thermo Fisher Scientific, USA) was used for ICP AES analysis. The sample solution was supplied into the plasma using a pneumatic spray system. A SeaSpray (Glass Expansion, Australia) type nebulizer was used, the peristaltic pump rate was 0.7 ml·min^{-1}. Quantitative measurements were carried out at instrumental parameters recommended by the manufacturer: cooling argon flow = 12 l·min^{-1}, secondary = 0.5 l·min^{-1}, registration time = 5 s, power supplied to an ICP inductor = 1150 W. Registration was performed with an axial view of the plasma to ensure high analytical signals.

The cells were decomposed by heating at 95 ± 1 °C in a digital block heater (Dry Block Heater, Ika, Germany) for 30–40 min with nitric acid (1.0 ml). The obtained solutions were diluted with ultra-pure water after cooling to room temperature. For the control nutrient medium was prepared in the same way.

Dissolution of BNPs/HEC composition

The dissolution of BNPs/HEC composition in several solvents was studied. Nitric acid, a mixture of nitric and hydrochloric acids, a mixture of nitric acid and hydrogen peroxide or a mixture of nitric, perchloric and hydrofluoric acids were added to aliquots of BNPs/HEC composition 3 (500 mg·l^{-1}). Then the samples with solvents were heated at a temperature not exceeding 95°C. The boron concentration in the resulting solutions was determined by the ICP AES method. Analysis results shown in Table S2. The found boron concentration is presented as a percentage of the theoretical concentration (recovery). As can be seen from Table S2, the recovery is no more than 35%. The use of an internal standard (IS) after dissolving samples in a mixture of nitric and hydrochloric acids does not increase recovery. It can be concluded that BNPs do not dissolve completely.

Table S2. Results of ICP AES analysis of BNPs/HEC composition 3 after using various solvents

Theoretical concentration, mg·l^{-1}	Solvents	Recovery, %
$5.0 \cdot 10^2$	HNO_3	24
	HNO_3 +HCl	27
	$HNO_3 + H_2O_2$	30
	HNO_3 + HCl, IS	33
	$HNO_3 + HClO_4$ + HF	35