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# Solid-Phase Reagents on the Modified Silica Gel Base for the Rapid Determination of Nickel(II) Ions

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**Abstract.** The new solid-phase reagent is synthesized by previous modifying the surface of silica gel with 1-(4-sulfophenyl)-3-methyl-5-(benzoxazol-2-yl)formazan for Ni(II) ions determination in water. The high effective colour change ( $\Delta\lambda = 200$  nm) is observed due to complex-formation reaction reagent with nickel (II) ions. Increasing toxicant concentration is accompanied by growth the colour intensity of the solid-phase sorbent, which form the basis of colour scale development for the Ni(II) ions determination in the range 10-100 mkg/ml. Ni(II) ions determination can't be disturbed by the presence of Co(II) ions, alkali or alkali-earth metal ions. Proposed technique is high rapid (its max duration is 2-3 min), ordinary (it is required neither expensive equipment nor qualified staff to use the technique) and ecological friendly.

## 1. Introduction

During last decades a new tendency has appeared in chemical analysis and monitoring quality of water and wastewater when the chemical analysis is conducted where the water sample is placed rather than in the chemical laboratory [1]. Sophisticated and expensive equipment to conduct multicomponent analysis can be used the only in a laboratory whereas using simple and reliable test-systems allows conducting in situ online analysis to assess ecological risk faster so that to take measures for improving the situation.

Nickel compounds are known to be harmful for humans. Increased nickel concentrations in water can cause endemic diseases or bronchial cancer. It's the main reason why the effective monitoring nickel (II) ions presence level in industrial wastewater and natural water ecosystems is so important environmental and analytical issue.

Complex-forming chemically modified silica gels with organic ligands immobilized onto their surface are often used as a matrix (supporter) for solid phase determination of metal ion concentrations. The ligands were chosen because they form coloured complexes [2] while contacting with metal ions in solution. It is reported [3], silica gel doesn't absorb light in visible spectrum and, being a mineral matrix, it ensures high rate of mass transfer (due to arrangement of functional groups at the surface), moreover silica gel is chemically stable, abrasion resistant and high adsorption capacity.

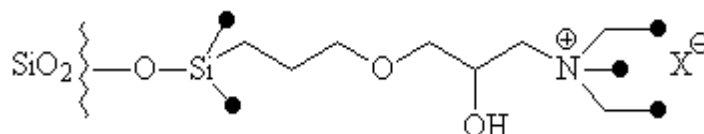
Heterocyclic compounds (class formazans) have been proven to work well as chromogenic reagents due to their extremely high tautomeric and conformational lability [4,5]. It was the promotion for high effective complex-formation reactions with metal ions that was used to develop techniques for metal ion analysis.



This report is devoted to investigating the silica-based testing technique for visual and spectral Ni(II) ion determination in aqua solution.

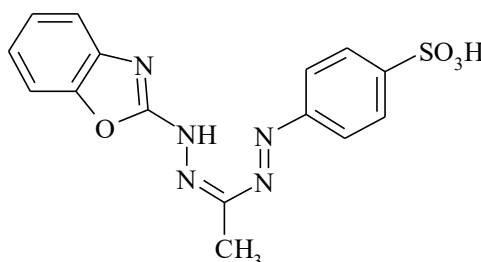
## 2. Results and discussion

Silica gel DIASORB-100-TA (Figure 1, average diameter of pores 100 Å) with trimethylammonium groups on the mineral matrix surface as a result of the covalent bonding was used for the investigation (number of fixed groups - 0,32 mMol/g).



**Figure 1.** Structure of Silica gel DIASORB-100-TA

Just these groups take part in immobilization 1-(4-sulfophenyl)-3-methyl-5-(benzoxazol-2-yl)formazan (Figure 2) onto silica gel surface due to electrostatic interaction (ionic mechanism).

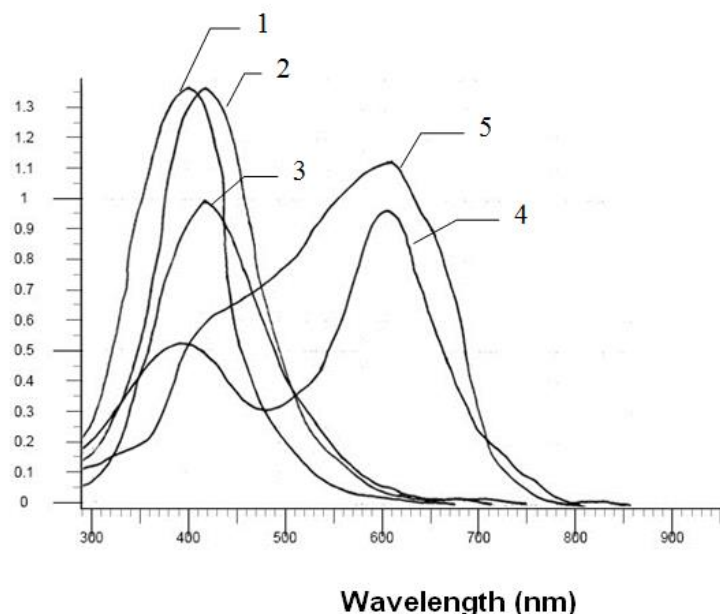


**Figure 2.** Structure of 1-(4-sulfophenyl)-3-methyl-5-(benzoxazol-2-yl)formazan.

The silica gel modified with a formazan is characterized by the absorption band around 420 nm which corresponds to a maximum absorption of partially polar formazan in the solution (Figure 3).

As optimal parameters to realize analytical reaction on the modified with benzoxazolylformazan silica gel surface, the following conditions were provided: metal solution pH  $5.5 \pm 0.5$ , concentration of benzoxazolylformazan analytical groups fixed onto silica gel (0.1 g)  $3 \cdot 10^{-4}$  M, duration when the indicator powder interacts with Ni(II) solution - 2-3 min.

Sorbent had been kept in Ni(II) nitrate solvent with mass concentration of the metal in range 30-300 mkg/ml. After that spectra of coloured matrix diffuse reflection were registered relatively those of the modified with benzoxazolylformazan silica gel. It has been established the maximum of absorption band of Ni(II) formazanate on the DIASORB-100-TA surface was observed when 610 nm and corresponds to the maximum of absorption spectrum of complex compound with composition 1Ni:1L in the solution (Figure 3).



**Figure 3.** Electronic spectra of: 1 – absorption spectrum of formazan in the solution; 2 – absorption spectrum of partially polar formazan ; 3 - absorption spectrum of formazan in solid phase; 4 - absorption spectrum of complex compound Ni(II) in the solution; 5 - absorption spectrum of complex compound Ni(II) in solid phase.

While interacting with Ni(II) nitrate, colour of an indicator powder had been changing from original red-orange to blue and its intensity depends on ion metal concentrations 30-45-65-80-100 mkg/ml. Empirically obtained colour scale was taken as a base to develop high rapid visual technique to determine Ni(II) ion concentrations in the solution (Figure 4).

<i>Colour of sorbent</i>						
<i>Ni(II) ion concentration in the solution, mkg/ml</i>	0	30	45	65	80	100

**Figure 4.** Colour scale for semi-quantitative determination of Ni(II) ion concentration.

The method was applied to model solutions analysis. Accuracy of determination had been checking with a technique «added–found». Minimal concentration of Ni(II) ions can be determined is 15 mkg/ml, standard deviation 0.15. Ni(II) ions determination can't be disturbed by the presence of Co(II) ions (when presence 1:5), alkali or alkali-earth metal ions. Ni(II) ions concentrations determined with the previously prepared colour scale were corrected by solid-phase spectroscopic technique.

### 3. Conclusion

Thus, proposed technique is high rapid (its max duration is 2-3 min), ordinary (it is required neither expensive equipment nor qualified staff to use the technique) and ecological friendly. The benefit of the developed solid-based reagent is its stability to light and air oxygen. The sorbent colour has not been changing during 3 years so it is available to use solid-phase reagent as ready-to-use analytical reagent 1-(4-sulphophenyl)-3-methyl-5-(benzoxazol-2-yl)formazan.

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